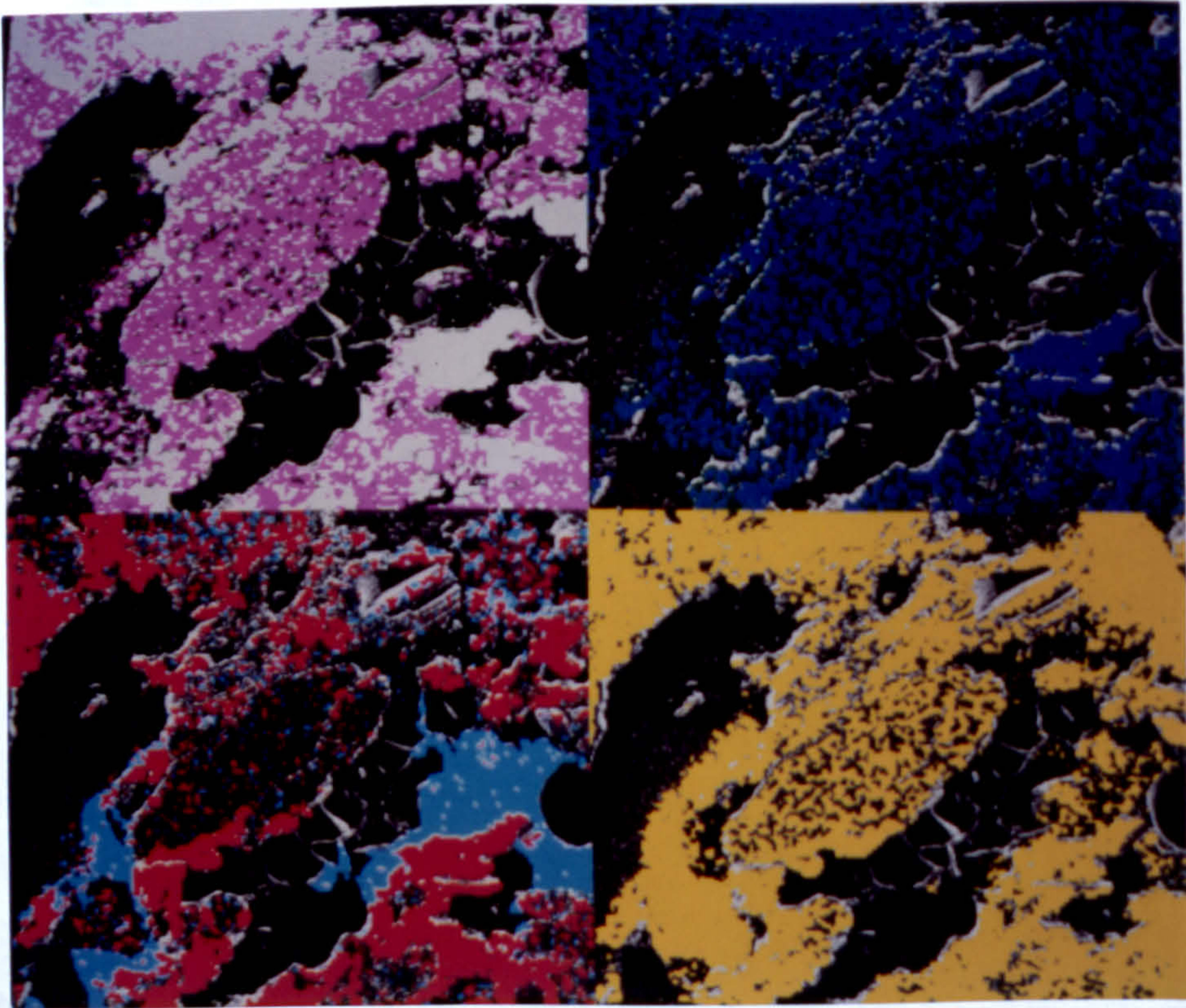




FRONTISPIECE

False colour images of sample GGRAN1
showing silicon, calcium, aluminium
and iron rich areas.

ELECTRICAL CONDUCTIVITY OF ROCK SAMPLES

SUBJECTED TO

HIGH TEMPERATURES AND PRESSURES

VOLUME 1

THEORY, EXPERIMENTAL DESIGN AND PROCEDURE

by

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A thesis submitted to the University of
East Anglia in fulfilment of the degree
of Doctor of Philosophy

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Norwich .



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To Julie

All people dream: but not equally.

Those who dream by night in the dusty recesses of their minds
wake in the day to find that it was vanity:
but the dreamers of the day are the dangerous ones,
for they may act their dream with open eyes,
to make it possible.

T.E. LAWRENCE

ABSTRACT

The field determinations of crustal electrical conductivity/depth profiles show anomalously high conductivities in the lower crust. This has yet to be explained by a consistent theory and interpretation of field results is difficult due to the lack of laboratory conductivity measurements of saturated rocks at lower crustal temperatures.

A cell was designed to measure the electrical conductivity of saturated rocks up to the lower crustal conditions of; confining pressures of 1 GPa, pore-fluid pressures of 1 GPa and temperatures of 900°C. This complex exercise required the use of a metal sleeve and the use of guard-ring techniques to remove leakage currents induced by the sleeve. The development of the cell involved several years work and is a breakthrough in measurement techniques as it has enabled the measurement of saturated rock conductivities at lower crustal temperatures and high pressures for the first time.

The conductivity of 14 samples of acidic and metabasic rocks was measured at a variety of confining pressures (<0.2 GPa), pore-fluid pressures (<0.2 GPa), temperatures (<900°C) and saturation fluids.

The pressure variations showed that the basic rocks had a conductivity too high to be explained by conduction through saturating electrolyte alone. The temperature variation showed a dramatic difference between acidic and basic rocks. The acidic rocks showed large decreases in conductivity above 350°C after initial rapid increases in conductivity. The basic rocks showed no such reduction in conductivity indicating that a conduction mechanism in addition to pore-fluid conduction was present.

Results indicate that saturated rocks at high temperatures have conductivities sufficient to explain the high conductivity layers in the upper lower crust whereas acidic rocks do not. The results also demonstrate that graphite may be an important additional conduction mechanism possibly accounting for the moderately high conductivities in the lower lower crust even in the absence of electrolyte conduction.

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CREDITS

In the course of this work I have read many theses but not until now have I spent any time reading the acknowledgements they contain. This is a profound oversight for if it were not for the effort and enthusiasm of many of our predecessors, colleagues and teachers for their science there would be no foundation on which I could hope to build upon. My thanks are stretched out to these and my hopes are that this research will stand the test of being built upon in a similar way.

On a more personal level there are many people who have helped during the progress of this research:

Firstly my special thanks to Professor F.J. Vine for his friendly help, suggestions and guidance. Instigator of this project, advisor, ally and supervisor, his support has been invaluable.

Equally I am indebted to Dr. Russell Ross. His encouragement, support and uncompromising standards have made him the best teacher of Physics that I have ever had the fortune to work with and the pleasure to know.

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DEBITS

I thought long and hard before including this short section. It is my belief that a thesis such as this should include nothing but the research report for which it was intended. I do think, however, that, having gratefully acknowledged all those who have helped me during the course of this research, it only correct to point out that the sources of dissatisfaction, disillusionment and difficulty implicit in doing a Ph.D. have been increased many fold by financial cutbacks imposed by the government on university departments and to student grants.

I make no political claims that another government would not cause the same lack of resources, nor do I pretend that the higher educational establishment did not need to be put on a more up-to-date footing, just this: the funding currently experienced by British Universities is desperately insufficient for effective research and teaching in a technologically based modern world. Likewise the continuing drop in the value of the student grant has reached a point at which no one can study without additional financial support. This will mean that universities will increasingly lose good students who do not consider it viable to live at university, they will become less universal and standards will drop.

Before I started on this work it was pointed out to me that to do a Ph.D. I would need to be a romantic or a fool. I didn't believe it! Luckily I was both. How many more are there out there I wonder?

In an increasingly technologically based modern world that uses knowledge of the natural world to improve the lot of its inhabitants, I would just like to remind the government that;

NOTHING COMES FROM NOTHING

Lucretius (55 BC)

NOTATION

SYMBOLS

SYMBOL	DESCRIPTION	UNITS
α	Absorbed fluid content	-
α	Thickness aspect ratio	-
α	σ/T variation factor	-
a	Archie constant	-
A	Area	m^2, cm^2, mm^2
A_o	Radiogenic heat production of surface rocks per unit vol	$\mu W m^{-3}$
b	Archie exponent	-
B	Clay counterion mobility	$S g m^{-4}$
c	Additional conduction term	$S m^{-1}$
d	Depth	km
d	Clay cation charge density	$C m^{-2}$
D	Heat flow characteristic depth	km
ϵ	Crack density parameter	$3\phi/8E(k)\alpha$
e	Charge on charge carriers	C
E	Electric field strength	$V m^{-1}$
E	Youngs modulus	$N m^{-2}$
$E(k), K(k)$	Complete elliptical integrals	-
E_o	Activation energy	eV
ϕ	Porosity	-
γ	Planform aspect ratio	-
$g, g(x)$	Acceleration due to gravity	$m s^{-1}$
G	Conductance	S
I	Electric current	A
i	$(-1)^{1/2}$	-
J	Electrical current density	$A m^{-2}$
k	Hoenig k parameter	$(1-\gamma^2)^2$
k	Boltzmann's constant	$8.6171 \cdot 10^{-5} eV K^{-1}$
k_d	disassociation constant	-
L	Length	mm, cm, m, km
L	Crustal thermal conductivity	$g m^{-1} k^{-1}$
L	Displacement, Length	mm, cm, m
μ	Charge carrier mobility	$m^2 V^{-1} s^{-1}$
n	ionic number density	mm^{-3}, cm^{-3}, m^{-3}
n	Effective pressure law factor	-
N	Amount of a substance	M
P	Pressure	GPa
q_o	Reduced heat flow	$\mu W m^{-2}$
Q	Surface heat flow	$\mu W m^{-2}$
Q_v	Cation exchange capacity	meq/100g, $m^3 g^{-1}$
ρ	Resistivity	$\Omega m, ohm m$
r	pore geometry 'constant'	-

NOTATION CONTINUED;

SYMBOL	DESCRIPTION	UNITS
R	Resistance	Ω
$\rho, \rho(x)$	Mass density	$\text{g cm}^{-3}, \text{kg m}^{-3}$
σ	Conductivity	S m^{-1}
T	Temperature	$^{\circ}\text{C}, \text{K}$
v	RMS charge carrier velocity	m s^{-1}
V	Volume	$\text{mm}^3, \text{cm}^3, \text{m}^3$
V	Potential difference	V
Ω	Saturation factor	-
w	Angular frequency	rads s^{-1}
W	Weight	g, kg
W_1	Initial weight	g
W_2	Dry weight	g
W_3	Saturated weight	g
x	Depth variable	km

NOTATION CONTINUED;

SUPERSCRIPTS AND SUBSCRIPTS

All sub- and superscripts are given relating to a variable X which is defined in the section on symbols.

SUBSCRIPT	DESCRIPTION
X_p	Relating to pore fluid or pore pressure
X_c	Relating to confining pressure
X_∞	At infinite temperature
X_T	At temperature T
X_r	Relating to the whole rock
X_m	Relating to the rock matrix
X_0	At 0°C
X_c^r	Critical value
$(X)_d$	Evaluated for depth d
X_i	i th term in summation
X_1	1st value etc.
X_h	Hydrostatic value
X_e	Effective value
X_{cT}	Relating to clay conduction at temperature T
$X_{c\infty}$	Relating to clay conductivity at infinite T
X_{EGR}	Measurement between or across the emitter and the guard-ring electrodes of the measurement cell
X_{CGR}	Measurement between or across the collector and the guard-ring electrodes of the measurement cell
X_{CE}	Measurement between or across the collector and the emitter electrodes of the measurement cell
X_{EC}	Measurement across or between the emitter and the collector electrodes of the measurement cell
X_{GR}	The value at the guard-ring electrode
X_C	The value at the collector electrode
X_E	The value at the emitter electrode

SUPERSCRIPT	DESCRIPTION
X^*	Measured value
X'	Real part of complex variable
X''	Imaginary part of real variable

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CHAPTER 1 : INTRODUCTION

1.1. THE LOWER CRUSTAL HIGH CONDUCTIVITY LAYER

During the last fifteen years there has been a steadily growing body of data concerned with the electrical conductivity of the earth's lower continental crust. The data has broadly fallen into two groups depending on its source:

1. Magneto-telluric and controlled source geomagnetic deep sounding measurements.
2. Laboratory measurements on rock samples subjected to lower crustal conditions.

The results of these studies have not been particularly well combined by any theory of lower crustal electrical properties of a global relevance.

Magneto-telluric and geomagnetic deep sounding measurements have been done in many parts of the world (Ingham and Hutton, 1982a; Jones, 1982; Beamish, 1986). These commonly reveal a zone of high electrical conductivity (around $0.01 - 0.1 \text{ S m}^{-1}$) within the earth's crust. This value should be compared with the average conductivity of the more resistive upper crust (around 10^{-5} S m^{-1}). The high conductivity zone appears at depths of around 10 - 20 km (Jones, 1981) and seems to be a world-wide phenomenon. Constable (1984) refers to 23 locations where measurements have indicated a high conductivity zone of regional scale.

Many theories have been advanced to explain such observations.

All theories require there to be an interconnected film or network of highly conducting medium. One medium that has been suggested is that of a partial melt (Waff, 1973). Others include metallic ores and non-metallic graphite (Stesky and Brace, 1973; Alabi et al, 1975). One of the most enduring theories suggests the presence of an aqueous electrolyte (Gough, 1986; Jones, 1987; and many others). Although the presence of water as a sufficiently conducting medium was initially suggested many years ago, it recently has seen many refinements. These enable it to overcome some of the criticisms that have been used to discredit it. These criticisms are briefly that:

1. Rocks become non-porous due to cracks closing under high pressures and temperatures thus excluding a fully interconnected pore fluid network (Brace, 1971).
2. The presence of water would instigate retrograde metamorphism in the anhydrous granulite grade metamorphic rocks that are thought to be dominant in the lower crust (Yardley, 1986).
3. A source for the water needs to be found.

Some solutions to these problems will be dealt with in greater detail in Chapter 3 it being sufficient to say in introduction that the hypothesis of an aqueous medium is still the most credible one available to explain the field data.

1.2. LABORATORY EXPERIMENTS UNDER SIMULATED CRUSTAL CONDITIONS

Ultimately the resolution of the apparent problem of lower crustal conductivity depends upon greater understanding of the electrical properties of the rocks and minerals in those areas. It is obviously impossible to make such measurements in situ. Measurements must be made upon samples thought to be representative of that part of the crust of interest. These measurements have to be made in an environment carefully created to mimic that thought to exist at these levels in the crust. Such measurements are potentially of very great value as they not only accurately characterise each individual sample's electrical behaviour and set bounds on the overall behaviour of the lower crust, but conditions can be accurately controlled. In fact in such an experiment the conditions of temperature, pore pressure, confining pressure and saturation state must all be carefully controlled to enable a lower crustal environment to be as close to reality as possible. The method is open to uncertainties due to unrepresentative sampling arising from the uncertainty as to the composition and mineralogy of the rocks existing in the lower crust, and the problems in obtaining such types of rock considering that the only possible access to such samples has been in uplifted lower crustal exposures and xenoliths (Brodie and Rutter, 1987; Fountain and Salisbury, 1981; Kay and Kay, 1981). Nevertheless, simulated high pressure and temperature measurements have produced a large body of electrical property data on many types of rocks and minerals under greatly varying conditions. However, none of the laboratory studies attempted

so far have succeeded in controlling all the conditions necessary to enable direct measurements of electrical conductivity in lower crustal conditions. Some laboratory experiments have been done to extreme pressures or temperatures on dry and wet rocks but never has a simulation of lower crustal confining pressure, pore pressure, temperature and saturation state been attempted. Some form of extrapolation of data has always been necessary to obtain speculative electrical conductivity measurements in true lower crustal conditions (Brace, 1971). The lack of unextrapolated measurements taken in a carefully controlled lower crustal environment has been due to the extreme difficulty in designing an experiment that recreates lower crustal conditions in the comparatively vacuous and cold conditions of a surface laboratory. Experiments that have been done have been directed towards the interpretation of the field data without attempting to control so many parameters that the experiment became technologically complex. These studies have been on low porosity crystalline rocks which are dry or have been saturated with an electrolyte of known molarity. Most of the experiments controlled the confining pressure and sometimes the temperature but rarely has the pore pressure been controlled and then only up to upper crustal conditions (Lee et al, 1983; Evans, 1980). Some studies have also concentrated on the specific mechanisms that may go some way towards explaining the lower crustal high conductivity zone (Jackson, 1984).

1.3. CRUSTAL ROCK CONDUCTIVITY - AN OVERVIEW

It was realised at an early stage that most commonly occurring rocks are very highly resistive even at high temperatures and pressures. The addition of a pore fluid makes even rocks of quite a low porosity relatively highly conducting. Thus the action of a changing pressure and temperature environment upon the properties of the fluid filled cracks controls the bulk rock conductivity (Brace et al, 1965).

Consequently the magnitude of the electrical conductivity of a rock sample has been modelled in the following simple way. The rock matrix is assumed to be an electrical insulator. All conduction occurs due to ion movement in an electrolyte that fills the pores and cracks of the rock matrix. In this model the bulk rock conductivity is a function of electrolyte conductivity and rock porosity (Archie, 1942).

This model works very well for some of the more unweathered and unmetamorphosed acid rocks (Evans, 1980). More basic rocks tend to diverge from this simple model by being more conductive than the model predicts (Jackson, 1984; Lee et al, 1983). This extra contribution to conduction, that is unaccounted for by the simple model, suggests that there is an additional conduction mechanism operating in the suite of more basic rocks.

Increasing the confining pressure on any saturated rock reduces its electrical conductivity. This is because increasing confining pressure cuts off conduction pathways due to pore and

crack closure. This decrease in conductivity is far less marked in basic rocks than in acid rocks. The acid rocks undergo a decrease consistent with closure of their high aspect ratio cracks and the model outlined above. The basic rocks tend to have more low aspect ratio cracks and would be expected to undergo a greater decrease in conductivity as the confining pressure is increased. As this is not so an additional mechanism for conduction must be inferred (Jackson, 1984).

Increasing the temperature of a saturated rock held at high pressure produces an increase in the electrical conductivity of the rock due to the increase in the conductivity of the electrolyte (Drury, 1979). The conductivity of basic rocks rises much faster than would be expected from the rise in conductivity of the electrolyte alone (Lee, 1977). This is also suggestive of another conduction mechanism.

Further unmodelled increases in the conductivity of the more basic rock types as temperatures and pressures equal those found in the lower crust may well explain the observed lower crustal high conductivity zone. More data is needed at these higher temperatures and pressures to enable models of the enhanced conduction apparent in basic rocks to be formed.

The awareness of the different electrical responses of acidic and basic rocks to changes in pressure and temperature has resulted in a twofold classification defined by Evans (1980) and used by Jackson (1984) and throughout this thesis.

The enhanced conduction has been attributed to mineral conduction (Evans, 1980; Parkhomenko, 1982), crack structure (Lee, 1977), hydrous minerals and clay alteration products (Jackson, 1984) and magnetite in veins (Stesky and Brace, 1973).

1.4. OBJECTIVES

Much of the data collected so far is extrapolable to the lower crust. Such extrapolations are, however, no replacement for measurements made in true lower crustal conditions. These measurements involve the accurate control of confining pressure and pore pressure up to 1 GPa, temperatures up to 800 °C and the saturation state of the sample. The control of these parameters involves building an electrical conductivity measuring cell that will withstand such conditions. This requires the solution of many problems in Physics, Metallurgy and Materials Science.

The data from such studies may be combined with what is already known about the electrical properties of crustal rocks and crustal models to produce a theory that effectively explains the mid-crustal high conductivity layer. It is hoped that the extension of confining pressure, pore pressure and temperature conditions will enable more sense to be made of the high conducting areas of the continental crust.

Bearing this in mind, the main objectives of this work were to:

1. Design, test and build an electrical conductivity measuring cell to work up to the following specifications;
 - Electrolyte saturation
 - Confining pressure, P_c 1 GPa
 - Pore pressure, P_p 1 GPa
 - Temperature, T 800 °C
2. Redesign drying and saturating apparatus to enable better control of the saturation state of the samples.
3. Arrange an experiment to measure the electrical conductivity of a limited number of rock samples including some collected from the Ivrea Zone, N. Italy by Dr. K. Brodie and Dr. E. Rutter using the measurement cell and controlling;
 - Confining pressure, P_c
 - Pore pressure, P_p
 - Temperature, T
 - Saturation state
 - Time
4. Interpret the measurements of conductivity in relation to the physical processes occurring at differing values of pressure, temperature etc.
5. Attempt to correlate the measured electrical conductivities to their composition



using;

- Scanning Electron Microscopy (SEM)
- Energy Dispersive X-Ray Analysis (EDS)
- X-Ray Fluorescence (XRF)
- X-Ray Diffraction (XRD)
- Optical Mineralogy

The experimental cell was designed to the specifications stated above. It was hoped to extend the pressure from 0.4 GPa (Lee, 1977; Jackson, 1984; Evans, 1980) to 1 GPa and the temperature from 250 °C to around 800 °C. In effect the inability to obtain a sufficiently large gas pressure vessel has restricted the use of the cell. Although the cell has been tested up to 0.45 GPa and 250 °C in an oil pressure vessel, it has not been used above 0.2 GPa in measurements of electrical conductivity at high temperatures (800 °C). This is not as large a disadvantage as it first may seem because the sensitivity of rock conductivity to temperature is considerably greater than that for pressure and hence much more important to understand in any study relating to lower crustal high conductivity layers. Accurate extrapolation for increased pressure has been possible using measurements taken at high temperatures (800 °C) during variation of confining pressure. These measurements show considerably less change in conductivity takes place as confining pressure is varied at progressively higher temperatures. Confining pressure runs of saturated rock samples at such high temperatures have never been done before and have provided data that should enable more accurate extrapolations for confining pressure to be done in

future. It is important that the rock is under some confining and pore pressures to ensure that the pore fluid remains in the fluid state at very high temperatures, this has been possible using pressures up to 0.2 GPa. High temperature studies have previously been restricted to measuring dry rocks due to this requirement for an applied pressure. The cell should be able to be used to its full design specification when a large gas pressure vessel becomes available. The values of electrical conductivity obtained using this equipment under the high temperature, moderate pressure and full saturation will be closest to the values existing at depth so far measured once the simple extrapolation for pressure is done.

This introductory chapter is followed by a chapter on the basic theory of rock conduction and related research and a chapter reviewing the current state of thought on the lower crust. Chapter 4 describes sample preparation and analysis and is followed by three chapters concerned with the experiment design, cell design and experimental procedure. These are followed by the results, their discussion and a final chapter in conclusion. A full list of references can be found in the rear of this volume and a table of symbols can be found at its beginning. S.I. units are used throughout.

CHAPTER 2 : BACKGROUND THEORY AND RELATED WORK

2.1. INTRODUCTION

This chapter is split into three sections. Firstly there is a brief description of electrical conductivity and different types of conduction mechanism. Secondly there is a more detailed description of the conductivity of rocks in simulated crustal conditions. Finally there is an outline of how the electrical conductivity of rocks is affected by changes of pore pressure, overburden pressure and temperature and the various hypotheses that may account for such behaviour.

2.2. ELECTRICAL CONDUCTIVITY - A THEORETICAL BACKGROUND

The nature of electrical conductivity and its mechanisms must be discussed before there can be any detailed analysis of its functional relationship to pressure, temperature and other factors.

The electrical properties of a material depends on the response of that material to a change in the electromagnetic environment in the space around or within it. The electromagnetic environment is defined by either a distribution of static electric charges or by a distribution of dynamic electric charges. The arrangement of static charges in a space gives rise to an electric field within that space. The distribution of dynamic charge flows (current flows) through a space defines a current density in that space (Lorraine and Corson, 1970). The electric field strength and the current density are related by

Maxwell's equations.

All materials contain charged particles. In some materials these charged particles are free to move about in a random fashion. This movement is a result of the thermal energy of each particle and increases as more energy is introduced into the system. If a steady electrical field, E , is present there is a biasing in the direction in which the charged particles move. This movement of charged particles forms an electric current. The electric current density, J , describes the effect of this flux of charged particles. For one type of charge carrier carrying a charge, e , the electric current density, J , is given by;

$$J = nev \quad \text{E2.1.}$$

where n = number of charged particles / unit volume
 e = charge on each particle
 v = particle drift velocity

Obviously each particle will have a different drift velocity. The velocity distribution of the particles is usually described by a root mean square velocity which defines the average behaviour of the particles. Rigorous consideration of Boltzmann distributions of charge carrier velocities and energies is beyond the scope of this thesis. Subsequently it will be assumed that particle drift velocity means root mean square velocity with all that implies.

For k types of charge carrying particle each of charge, e_1 , and velocity, v_1 ;

$$J = \sum_{i=1}^{i=k} n_i e_i v_i \quad \text{E2.2.}$$

Subsequently it will be assumed that the only charge carrier responsible for charge conduction is the electron with charge, e , although the following analysis is just as valid for any positive or negative ion of charge, e .

The drift velocity of charge carriers is usually proportional to the magnitude of the applied electric field. The constant of proportionality is called the charge carrier mobility, μ .

$$v = \mu E \quad \text{E2.3.}$$

From E2.1. and E2.3.

$$J = ne \mu E \quad \text{E2.4.}$$

In circumstances other than in the presence of high field strengths the charge carrier number density, n , and the charge carrier mobility, μ , are independent of the applied electric field strength. The charge, e , is constant. Thus it is possible to write;

$$J = \sigma E \quad \text{E2.5.}$$

where

$$\sigma = ne\mu \quad \text{E2.6.}$$

and the constant of proportionality, σ , is known as the electrical conductivity and is measured in siemens per metre, $S\ m^{-1}$. E2.5. is known as Ohm's Law and can be rewritten;

$$\sigma = \frac{J}{E} \quad E2.7.$$

E2.5. has been derived assuming the application of a steady electric field. If the steady field is replaced by one which varies with time the current flow may not be in phase with the field. If this is the case then the current can be represented by two components, one component in phase with the alternating applied field. The other part in quadrature to it. The representation of this situation mathematically requires the introduction of complex numbers into the solutions of Maxwell's equations and requires the electrical conductivity to be a complex number.

$$\sigma^* = \sigma' + i \sigma'' \quad E2.8.$$

where i is the square root of minus one. σ' represents the in-phase conductivity that gives rise to the conduction component $\sigma'E$. The term $i \sigma''$ is the quadrature, displacement (dielectric), current component of conductivity that gives rise to the displacement current, $i\omega\epsilon$, where ω is the angular frequency and ϵ is the dielectric permittivity.

Only the real part of the complex conductivity has been considered and measured in the following work. Subsequently whenever the term 'conductivity' is used it is to be considered

synonymous with the real part of the conductivity, σ' , in E2.8.

In normal measurement technique it is more usual to measure the electric current, I , passing through a sample than the current density, J . If a current, I passes through a space of area, A , then the current density is simply the current per unit area normal to the current flow. This can be written as;

$$I = J \cdot A \quad \text{E2.9.}$$

where $\cdot$ indicates the cross-product of two vectors.

The potential difference across a space, V , is related to the electric field and the displacement between the two points, L , across which the potential difference is measured. This can be written as;

$$V = E \cdot L \quad \text{E2.10.}$$

The ratio of electric current to the applied potential difference is called the conductance, G . This is measured in siemens, S .

$$I = GV \quad \text{E2.11.}$$

Conductivity and conductance are related by the geometry of the sample through which the current measured is passing. The relationship between measured conductance and conductivity of a sample of regular dimensions is given by;

$$\sigma = \frac{G L}{A} \quad \text{E2.12.}$$

where σ is the conductivity of the sample

G is the conductance of the sample

L is the sample length

A is the normal cross-sectional area of the sample.

2.3. THE MODES OF ELECTRIC CONDUCTION

This section is concerned with the main mechanisms of conduction and the classification of materials according to the extent to which each mechanism operates.

The electrical properties of a material depend on the number and type of charge carriers as well as their mobility. It is this last parameter that is the most important as a material may contain an abundance of electrons, protons and ions but if they all have a relatively low mobility it will require a high electric field to form a current. Solid materials may be classified into three main groups each of which has a different response to an applied electric field.

- A. Metallic conductors
- B. Insulators
- C. Semiconductors

These three groups cover a conductivity range of $10^{10} - 10^{-10} \text{ S m}^{-1}$.

Each mode of conduction will be dealt with in turn and described using 'Band Theory'.

A. METALLIC CONDUCTORS undergo metallic bonding in which the valence electrons have such a high energy that they no longer remain within the sphere of influence of the parent atom. They are free to roam around the space lattice of the metal in a diffuse electron cloud. This can be described by band theory in the following way: In a solid crystal lattice the energies of electrons can be envisaged as forming bands in which each electron is distributed according to its energy. The valence band covers the range of energies that an electron has when occupying the bonding energy level. These electrons take part in bonding but are not usually able to move freely away from their parent atom to take part in conduction. To take part in conduction they need to enter the higher energy conduction band. In the case of metals the conduction and valence bands overlap, see figure F2.1. (Ashcroft and Mermin, 1976). Some of the electrons in the valence band already have sufficient energy to be involved in conduction as they are already in the conduction band. Other electrons can gain energy by being excited by an applied electric or thermal field. These electrons can freely move into the conduction band. As there is no gap between the two overlapping bands promotion of electrons is easy and there is no threshold energy that the electrons need to acquire (Kittel, 1971).

The conductivity of metals at room temperature and pressure is typically of the order of 10^7 S m^{-1} . A few naturally occurring minerals such as graphite and pyrites are good conductors but these are usually so dispersed within rocks that they make negligible difference to their overall conductivity. They will be discussed with other more commonly occurring minerals in section 2.8.

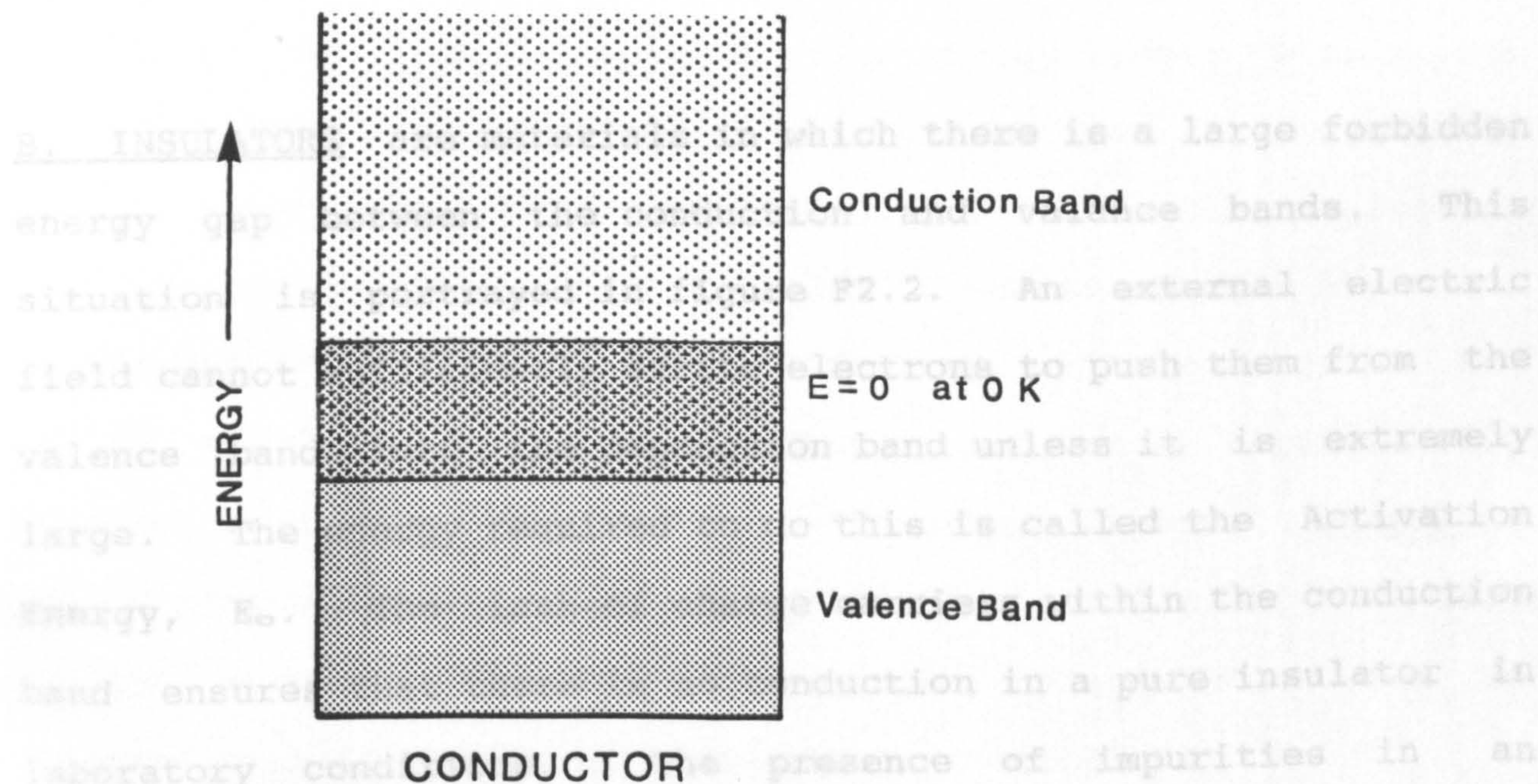


FIGURE F2.1. Band theory diagram of a metallic conductor.

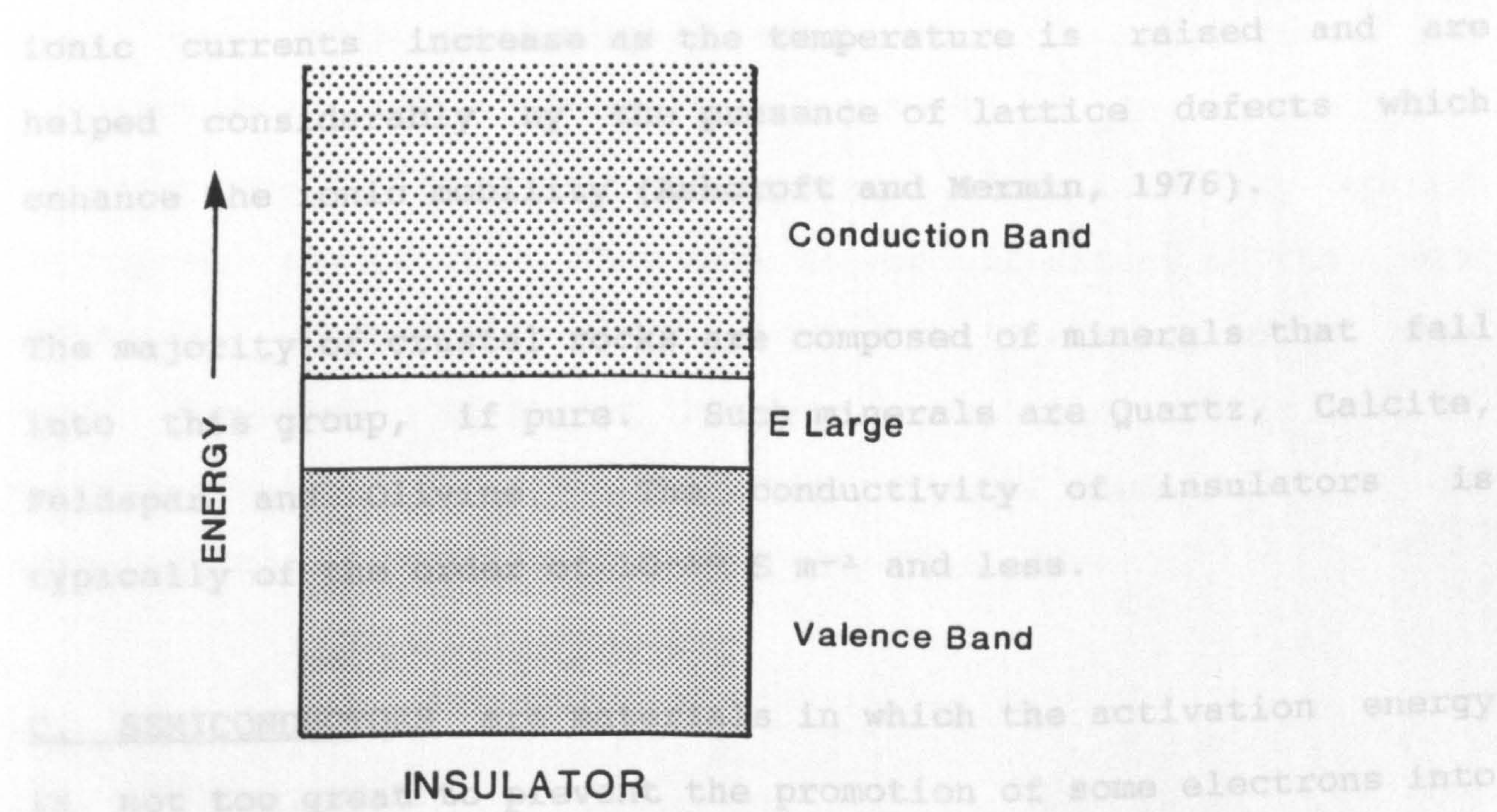


FIGURE F2.2. Band theory diagram of an insulator.

The conductivity of metals at room temperature and pressure is typically of the order of 10^7 S m^{-1} . A few naturally occurring minerals such as graphite and pyrites are good conductors but these are usually so dispersed within rocks that they make negligible difference to their overall conductivity. They will be discussed with other more commonly occurring minerals in section 2.8.

B. INSULATORS are materials in which there is a large forbidden energy gap between the conduction and valence bands. This situation is portrayed in figure F2.2. An external electric field cannot sufficiently excite electrons to push them from the valence band into the conduction band unless it is extremely large. The energy required to do this is called the Activation Energy, E_a . The lack of charge carriers within the conduction band ensures that there is no conduction in a pure insulator in laboratory conditions. The presence of impurities in an insulator can give rise to small ionic currents. These small ionic currents increase as the temperature is raised and are helped considerably by the presence of lattice defects which enhance the ionic mobility (Ashcroft and Mermin, 1976).

The majority of crustal rocks are composed of minerals that fall into this group, if pure. Such minerals are Quartz, Calcite, Feldspar and Olivine. The conductivity of insulators is typically of the order of $10^{-10} \text{ S m}^{-1}$ and less.

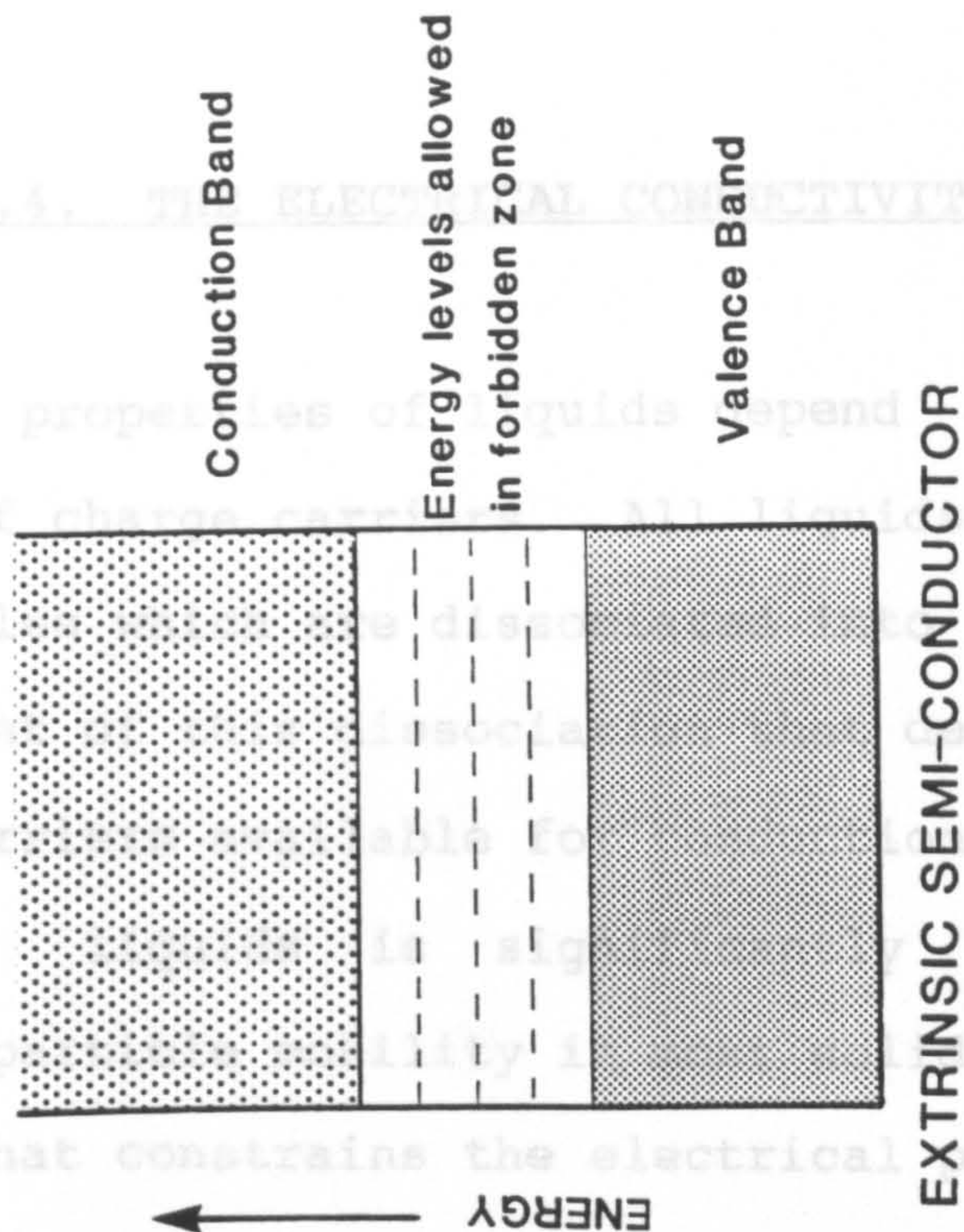
C. SEMICONDUCTORS are materials in which the activation energy is not too great to prevent the promotion of some electrons into

the conduction band. Thermal excitation of electrons enables them to overcome the band gap and be available in the conduction band for conduction provided that an electric field is present. These materials are known as intrinsic semiconductors. See figure F2.3.

If an insulator or semiconductor contains a significant amount of impurities or lattice defects then energy levels within the forbidden band become allowed. This can be seen in figure F2.4. Some of these levels may be sufficiently close to the conduction band to enable small electric fields to excite enough electrons to the conduction band to cause conduction. These materials are called extrinsic n-type semiconductors and can be artificially produced by doping semiconductors with other elements. Extrinsic p-type semiconductors cause conduction in a similar way but rely on the movement of positively charged 'holes' which can be understood as the space which an electron occupied.

Most ores such as galena, magnetite and sphalerite are semiconductors but although they may be found in economically workable quantities the location of such sites is widely spread throughout the crust. They have no overall effect on the bulk electrical conductivity of the crust even if they produce some local electrical anomalies that may be exploited by the use of electromagnetic prospecting methods. Typical conductivities of semiconductors range from 10^5 S m^{-1} to 10^{-7} S m^{-1} .

FIGURE F2.4. Band theory diagram of an extrinsic semi-conductor.



Conduction Band

E Small

Valence Band

INTRINSIC SEMI-CONDUCTOR

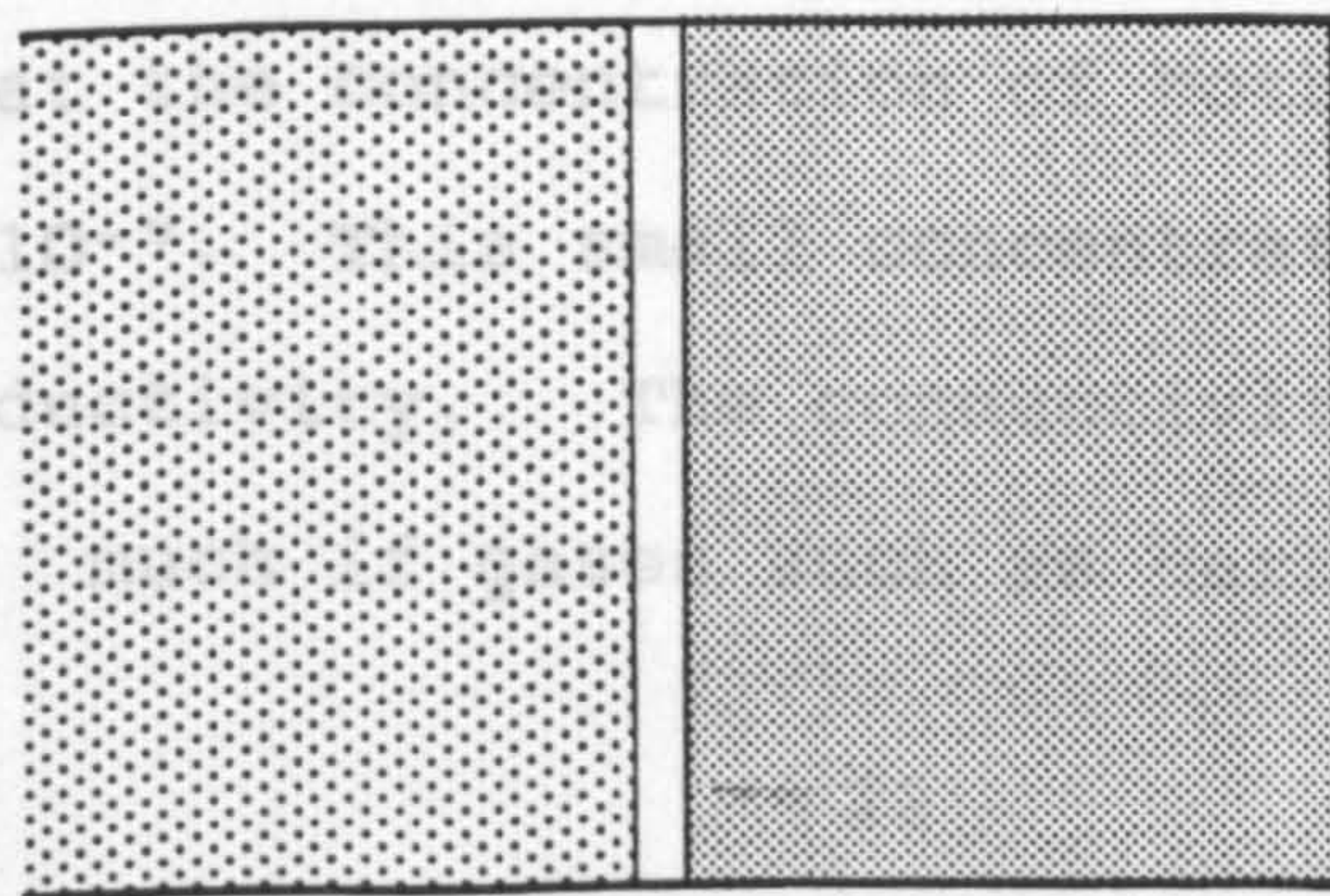


FIGURE F2.3. Band theory diagram of an intrinsic semi-conductor.

2.4. THE ELECTRICAL CONDUCTIVITY OF SOLUTIONS

The electrical properties of liquids depend on the type, number and mobility of charge carriers. All liquids consist of randomly moving molecules which are dissociated into ions to some extent. It is the extent of this dissociation that determines the number of charge carriers available for conduction. The mobility of particles in liquids is significantly greater than the corresponding particle mobility in most solids. It is not only the mobility that constrains the electrical properties of liquids but type and amount of charge carriers as well.

Pure covalent liquids consist of covalently bonded molecules with no nett charge. Individual atoms within the molecule may appear as slightly positive or negative due to bonding electron bias towards different atoms. This is usually particularly the case for hydrogen and some particularly electrophilic atoms. These charges give rise to weak inter-molecular forces which are often called hydrogen bonds. These covalent liquids undergo a small amount of dissociation to ions even at 25 °C. Pure water has a dissociation constant of about 10^{-14} at 25 °C. It dissociates into hydrogen and hydroxyl ions.



This implies that the concentration of both hydrogen and hydroxyl ions is about 10^{-7} . This small concentration of ions gives rise to a small conductivity. The conductivity may increase up to five times as much if gases such as carbon dioxide from the

atmosphere dissolve in it. Carbon dioxide will dissolve in water to give HCO_3^- and hydrogen ions.



Even when pure the conductivity of water is sensitive to increasing temperature. The addition of heat to the liquids increases the dissociation of molecules and increases the concentrations of both hydrogen and hydroxyl ions.

The conductivity of a covalent liquid may be further enhanced by introducing more ions into the system. An ionically bonded solid material such as sodium chloride is electrically neutral on a macroscopic scale due to the gross summation of an equal number of positive and negative ions arranged in a three dimensional lattice. It dissociates into its oppositely charged ions when it is dissolved in a covalent liquid such as water. Sodium chloride, NaCl , dissociates into sodium ions, Na^+ , and chloride ions, Cl^- .



These ions can move around in the relatively free environment of the liquid in which they are dissolved. The conductivity of such a solution is given by E2.6.

$$\sigma = ne\mu \quad \text{E2.6.}$$

where n is the total number density of ions

e is the charge on each ion

μ is the ionic mobility

The chemical equation E2.15. represents how the salt dissolves in a solvent. The extent to which a salt dissolves in a solvent and hence the availability of ions is dependant upon pressure and temperature. The relative amounts of ions in solution can be described by a dissociation constant, k_d .

The mobilities of ions commonly found in groundwater are all approximately the same (Parkhomenkho, 1967). The dominant salt in groundwater is sodium chloride. The content of different salts in groundwater is conveniently expressed in terms of the equivalent content of sodium chloride alone (Dunlap and Hawthorne, 1951).

For a constant temperature and pressure the mobility, μ , of all ions is approximately constant. The charge carried by each type of ion is always constant. It can be seen from E2.6. that the conductivity is directly proportional to the number density of ions in the liquid.

$$\sigma \propto n \quad \text{E2.16.}$$

And for k types of ions each of charge, e_i and number density, n_i

$$\sigma = \mu \sum_{i=1}^{i=k} n_i e_i \quad \text{E2.17.}$$

E2.17. does not hold for very high concentrations. This is due to the mobility of the ions being reduced by ionic interactions (Horne, 1969). This can be seen in table T2.1.

T2.1. NaCl Aqueous Solution (After Lee, 1977)	
Molarity /moles	Conductivity /X10 ⁻¹ S m ⁻¹
0.01	1.21
0.10	12.00
1.00	85.00

The electrical properties of aqueous solutions are affected by pressure and temperature changes by several mechanisms. The effect of temperature upon dissociation constants has already been discussed. Once ions are in the solution it is the variation of mobility with pressure and temperature that causes conductivity variations. A good overview of aqueous solution transport properties and the effect on them by varying pressure and temperature is given by Horne (1969). Little data is available on the pressure dependence as this effect is small and there has consequently been little work done on it. Much greater amounts of work have been done on the temperature dependence of electrolytes but much of this is quite old and does not usually involve measurements above 100 °C. Quist and Marshall (1968, 1969) have studied the properties of electrolytes to pressures of 0.4 GPa and temperatures of 600 °C. Figure F2.5. shows the

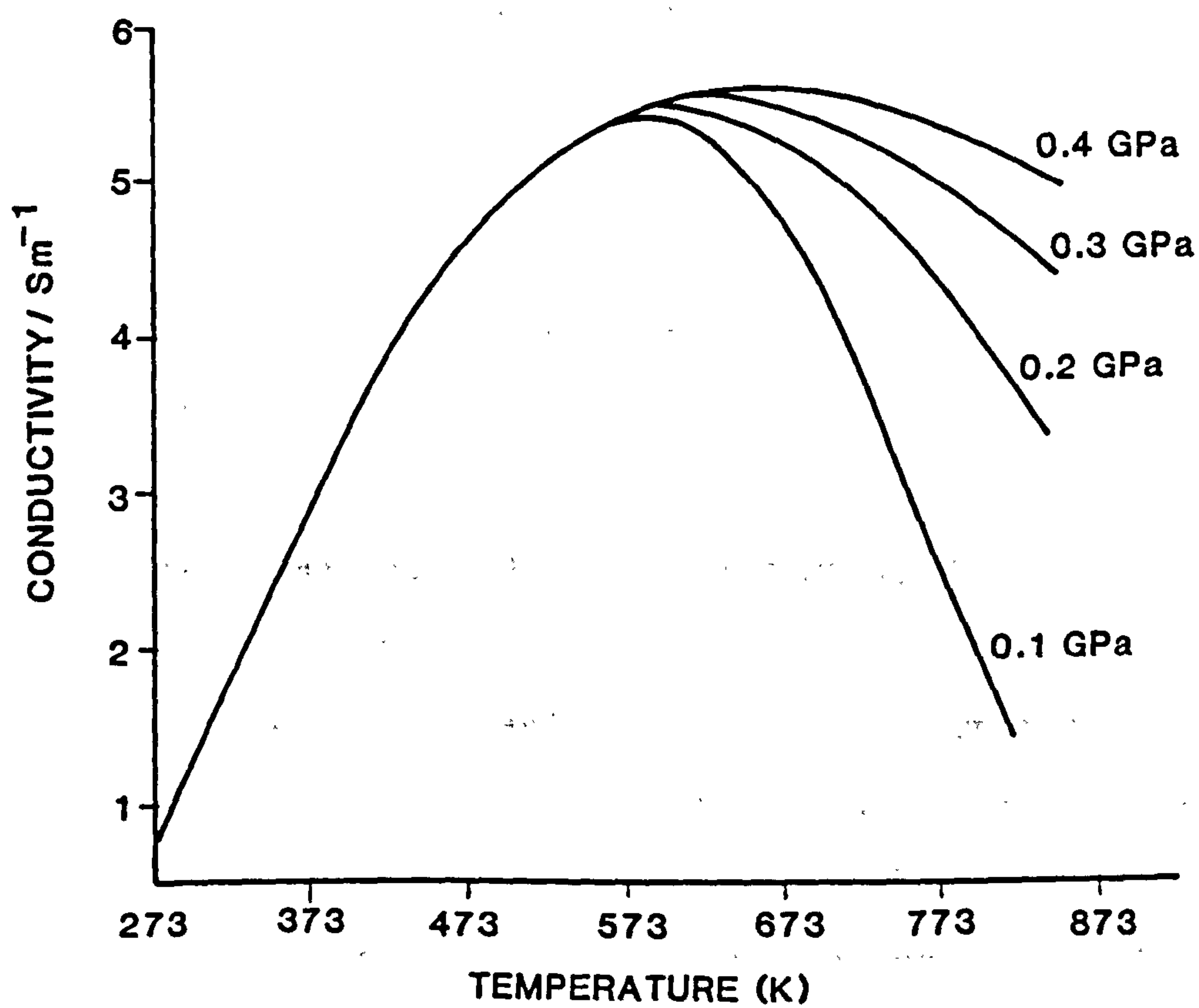


FIGURE F2.5. The variation of the conductivity of a 1 M NaCl solution with temperature and pressure (After Quist and Marshall, 1967).

pressure and temperature variations in conductivity of a 0.1 M solution of NaCl. The behaviour shown in F2.5 has been described by Quist, Marshall and others in the following way. Initially the electrolyte viscosity decreases rapidly as the temperature increases (Bird et al, 1963). The mobility of ions increases. The electrical conductivity increases in step with the ion mobility. This increase is linear at the lower temperatures and can be described by the following equation.

$$\sigma_T = \sigma_0 (1 + \alpha T) \quad \text{E2.18}$$

where σ_T is the conductivity at T °C
 σ_0 is the conductivity at 0 °C
T is the temperature
 α is a constant

According to Parkhomenkho (1967), $\alpha = 0.0022/^\circ\text{C}$ for a 1 M NaCl solution. At temperatures of 350 °C and greater the rate of viscosity decrease with increasing temperature diminishes. This ensures that the ionic mobilities increase at a much lower rate than at lower temperatures. Isobaric density has been decreasing steadily as the temperature has been increasing. This has reduced the ionic number density. The higher temperatures has also increased the probability that interaction and association between oppositely charged ions will occur. The previous three points collude to ensure that a conductivity maximum occurs between 300 and 400 °C. The temperature at which the conductivity maximum occurs represents the temperature at which the increase in ionic mobility is offset by a decrease in the

ionic number density due to inter-ionic association and diminishing isobaric density. Note that F2.5. shows no pressure dependence below 250 °C.

A graph of $\log_{10} \sigma$ against the reciprocal of temperature, measured in kelvin, gives a straight line if the conductivity can be represented by an expression of the form of equation E2.34. Section 2.7. discusses the representation of conductivity data in this way and how the straight line gradients can give an indication of the activation energy of a particular conduction mechanism at a particular temperature. Figure F2.12 is obtained if this is done for a solution of 0.5 M NaCl. There are two values of effective activation energy depending on the temperature range (Jackson, 1984; Waxman and Smits, 1968);

0.16 - 0.20 eV	for	0 - 80 °C
0.14	for	80 - 190 °C

It can be seen from Table T2.1. that even dilute solutions of NaCl have a much greater conductivity than most dry crustal rocks. A 0.1 M NaCl solution has a conductivity of $12 \times 10^{-1} \text{ S m}^{-1}$ whereas dry crustal rocks are of the order of 10^{-5} to 10^{-8} S m^{-1} . The presence of even small quantities of groundwater in dry crustal rocks will significantly modify their conductivities.

2.5. POROSITY, CRACKS AND MICROCRACKS

The following section will deal with the type and amount of voidspace within dry crustal rocks and its effect upon electrical conductivity particularly when filled with pore fluids.

In section 2.4 a comparison was made between the conductivities of 0.1 M aqueous solution of NaCl and that of dry crustal rocks. It was recognised that the presence of electrolytes in voidspaces within the rock will have a significant effect on the bulk conductivity of the rock. The bulk conductivity of the rock depends on;

1. Conductivity of the rock matrix
2. Conductivity of the pore network
3. Surface conduction along associated grain boundaries.

The way in which these parameters can be used to describe the bulk conductivity of the rock in a quantitative way will be dealt with in section 2.6.

The volume and arrangement of airspace within a dry rock has an effect upon the amount of electrolyte that the rock may take up when saturated and hence upon the bulk conductivity of the rock. The following pore parameters have an effect upon the bulk rock conductivity.

1. Overall porosity
2. Size distribution of pores
3. Shape distribution of pores
4. Percentage saturation of pores
5. Connectivity of the pores

There is a characteristic and large pressure dependence in the physical properties of crystalline crustal rocks at relatively low pressures (up to 0.1 GPa). This has been attributed to the closure of many small 'microcracks' within the rock. These microcracks have been classified according to their shape.

The shape of a microcrack is considered to be that of an ellipsoid with orthogonal semi-axes a , b and c . The size of these axes defines the overall size of the microcrack. Their relative sizes define the shape of the microcrack. Two ratios are defined (Evans et al. 1982);

$$\alpha = c/b \quad \text{-Thickness Aspect Ratio} \quad \text{E2.19a}$$

$$\gamma = b/a \quad \text{-Planform Aspect Ratio} \quad \text{E2.19b}$$

A spherical microcrack has $a=b=c$ and hence $\alpha = 1$ and $\gamma = 1$, a coin-shaped microcrack has $a=b \gg c$ so $\alpha \ll 1$ and $\gamma = 1$ and a cigar-shaped microcrack has $a \gg b > c$ and therefore $\alpha < 1$ and $\gamma \ll 1$. A microcrack which exhibits a high degree of sphericity has α and γ close to unity and is called a high aspect ratio microcrack (HARC). A flattened or elongated microcrack has either α or γ or both of these ratios significantly less than unity and is called a low aspect ratio microcrack (LARC). The crack structure of a rock

can be described by a crack spectrum in which the amounts of porosity in each of a range of aspect ratio are given.

It was first suggested by Walsh (1965b) that the closure of microcracks under pressure was related to the aspect ratio and the inherent strength of the rock matrix. He suggested that the closure of a particular microcrack might occur at a critical pressure, P_{cr} which is related to the aspect ratio and the rock matrix strength in the following way.

$$P_{cr} \propto E \quad E2.20.$$

where E is the Youngs modulus of the rock.

Initially, for rock matrices of the same Youngs modulus, only the low aspect ratio cracks close as pressure increases. Walsh argued that it was the closure of these low aspect ratio microcracks at pressures below 0.1 GPa that was responsible for the large change in the physical properties of the rocks. The closure of the microcracks effectively repairs a weakness in the rock. This implies that as the confining pressure is increased even further the measured physical properties of the rocks approach those of a microcrackless crystalline crustal rock.

The cause of microcracks is considered to be the presence of high local stresses within the rock. Microcracks occur in certain small volumes within the rock where the local stresses exceeds the local strength of the rock (Simmons and Richter, 1974). The existence of localised high points within the stress field is due

to anisotropy of crystal properties. Localised high stress areas occurring when there is a change in the pressure and temperature environment cause anisotropic expansion or contraction.

Theoretical studies of crack closure and its effect upon conductivity and seismic velocities have been done by only a few workers. Most of the studies have concentrated upon modelling the crack distribution by inversion of seismic velocity data. O'Connell and Budiansky (1974) and Budiansky and O'Connell (1976) have shown that a crack density parameter and crack saturation percentage can be derived from the values of compressional and shear seismic wave velocities as a function of pressure. Their 'self-consistency theory' assumes a low concentration of randomly distributed coin-shaped cracks. The crack density, essentially a function of the surface area of the cracks, controls the seismic velocities. Although some good agreements between predictions made by this theory and observed conductivities have been found (Evans, 1980) difficulties arise as the theory only indirectly relates porosity to pressure. The theory is also limited in its usefulness as it does not give the relative concentrations of microcracks of different aspect ratios.

Another theory was postulated by Kuster and Toksoz (1974). They developed the work of Walsh (1965a; 1965b) to produce a 'non-interactive theory' which predicts the effect of a range of concentrations of different aspect ratio oblate spheroid cracks on seismic velocities. The 'non-interactive theory' assumes no interaction between neighbouring coin-shaped cracks and so is only applicable to rocks with sufficiently low porosities.

Further work enabled the 'non-interactive theory' to be used to invert seismic velocity/pressure data to provide the pore aspect ratio spectrum of a rock (Cheng and Toksoz, 1976; 1979). This inversion technique has been successfully used to provide rock pore aspect ratio spectra of a range of rocks (Jackson, 1984).

The effect of crack distribution and high pressure crack closure upon the conductivity of a saturated rock has also been modelled but not as extensively as in the case of seismic velocities (Greenberg and Brace, 1969; Shankland and Waff, 1974). The only detailed analysis so far has been the Hoenig Theory (Hoenig, 1979) which is a development of the 'self-consistent theory' of O'Connell and Budiansky (1974). The theory of Hoenig is described in greater detail in section 2.9.

The effectiveness of these theories has been tested by using them to produce predicted rock conductivity/pressure variations which were compared with those observed (Evans et al, 1982). This work involved the simultaneous measurement of compressional and shear wave velocities and electrical conductivity. The seismic velocities were used with the 'non-interactive theory' to calculate the crack spectrum for each rock studied. These rock crack aspect ratio spectra were combined with the theory of Hoenig and with Archie's Law to predict the electrical conductivity of the rock as a function of pressure. The predicted electrical conductivities were found to be in good agreement to those observed. It was, however, necessary to assume the cracks to be in the form of a cigar-shape for use with Hoenig's theory as this theory seems to produce unreasonable

results when used with a coin-shaped crack despite the theory being a development of the 'self-consistent (coin-shaped crack assuming) theory'. More work needs to be done in this area to develop further the quantitative theory of pressure on a cracked solid.

2.6. THE THEORY OF SATURATED ROCK CONDUCTIVITY

The conductance of a rock sample is made up of two contributions, one from conduction through the rock matrix and the other from electrolytic conduction through the pore fluid. It can be seen by comparing table T2.1. and the conductivity range of dry crystalline crustal rocks (table T2.4.) that the major conductivity contribution in saturated crystalline crustal rocks will be from pore fluid conduction.

The measured conductance, G^* , of a saturated rock can be expressed as;

$$G^* = G_M + G_P \quad \text{E2.21.}$$

where G^* is the measured conductance
 G_M is the rock matrix conductance
 G_P is the pore fluid network conductance

All individual pore paths through a sample can be expressed as a single, regular pore of cross-sectional area, A_P and length, L , through a sample of normal cross-sectional area, A_M and length,

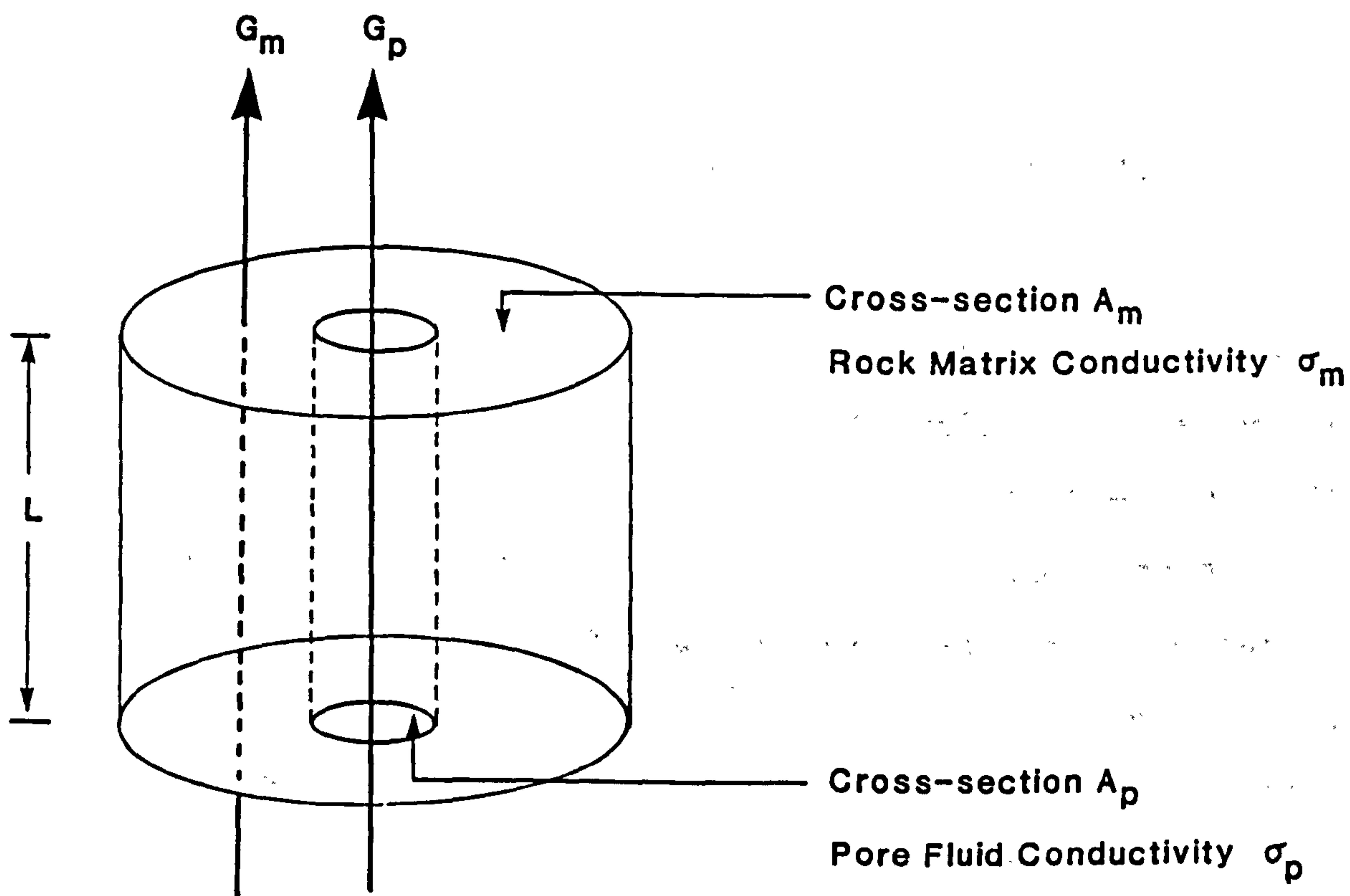


FIGURE P2.6. Schematic diagram of the idealised pore fluid/rock matrix conductor model.

L, as shown in figure F2.6. (Lee, 1977).

From E2.12.
$$G_M = \frac{\sigma_M A_M}{L} \quad \text{E2.22.}$$

And
$$G_P = \frac{\sigma_P A_P}{L} \quad \text{E2.23.}$$

From E2.22. and E2.23.

$$\frac{G_M}{G_P} = \frac{\sigma_M A_M}{\sigma_P A_P} \quad \text{E2.24.}$$

Reference to section 2.4. shows that an average value for σ_M/σ_P is about 10^{-5} . Bearing this in mind E2.24 implies that a value of A_P/A_M of only 10^{-5} is required to ensure that the the bulk conductivity is dominated by the pore fluid conduction contribution. For values of A_P/A_M greater than 10^{-5} domination by the pore fluid term in the conductance equation, E2.21, effectively makes the rock matrix term of negligible significance except in the case of some rocks where an anomalously high conducting mineral content is present.

The values of G_P and G_M for a particular rock can be obtained by measuring rock samples in both their dry and saturated conditions. This usually shows that $G_P \gg G_M$. Bearing this in mind and from E2.21. and E2.23.;

$$G^* \approx G_P = \frac{\sigma_P A_P}{L} \quad \text{E2.25.}$$

$$\text{And as} \qquad A_M \gg A_P \qquad E2.26.$$

$$G^* \approx \frac{\sigma^* A_M}{L} \qquad E2.27.$$

where σ^* is the measured conductivity of the saturated sample.

From E2.25. and E2.27.

$$\sigma^* \approx \frac{\sigma_P A_P}{A_M} \qquad E2.28.$$

The porosity of the sample, ϕ , is given by the ratio of pore volume to the total volume and as $V_{TOTAL} = A_{TOTAL}L \approx V_M = A_M L$;

$$\phi = \frac{A_P L}{A_M L} = \frac{A_P}{A_M} \qquad E2.29.$$

and therefore;

$$\sigma^* = \sigma_P \phi \qquad E2.30.$$

The previous treatment is valid only for the simplified case of a single regular pore. The three dimensional networks of pores of irregular cross-section found in real rocks cannot be treated using equation E2.30. The most common way of relating the measured conductivity of a rock to the pore fluid conductivity is by the relationship;

$$\sigma^* = a \sigma_p \phi^b$$

E2.31.

where a and b are empirically derived constants for a particular rock type. The pore parameter, b, depends in a complex way upon the pore parameters mentioned in section 2.5. For compacted crystalline rocks the constant a can be taken as unity and the constant b as 2 (Brace et al, 1965). E2.31. is known as Archie's Law (Greenberg and Brace, 1969). It was originally derived for use in the oil industry to describe the conductivity behaviour of highly porous sandstones and limestones. Brace and others discovered that it was also valid for many rocks with very low porosities (Brace et al, 1965; Brace and Orange, 1968). The validity of Archie's Law for all rock types, especially those that are severely altered or carry a significant ore content, has been questioned recently (Patinode and Wyllie, 1950; Duba et al, 1978; Barker, 1979). It has been suggested that in some cases mineral conduction is a significant contributor to the bulk conductivity and should be included in a modified form of Archie's Law (Patinode and Wyllie, 1950).

$$\sigma^* = a \sigma_p \phi^b + c$$

E2.32.

where c is a contribution from mineral conduction.

2.7. THE CONDUCTIVITY OF DRY ROCKS AND MINERALS

The electrical conductivity of a rock depends on the individual electrical conductivities and quantities of the various mineral components present in the rock. The conductivity also depends to a lesser extent upon the arrangement of different minerals within the rock. Anisotropic conductivity can be related to the rock texture. In any research into mineral aggregates it is important that the bulk physical and electrical properties of the individual minerals are understood as fully as possible.

Table T2.2. shows the range of conductivities which rocks and minerals possess. It can be seen that rock conductivities cover a wide range of orders of magnitude. Common rock forming minerals are also spread over many orders of magnitude but on the whole tend to be more highly resistive than the rocks which they make up. This is perhaps only to be expected as small amounts of conducting impurities in rocks will make them slightly more conductive than expected purely from their majority composition. If the rocks are wet they have conductivities that are considerably higher than the sum of their mineral constituents would indicate. Clearly, and as we have already said in section 2.4., the most important factor affecting the overall rock conductivity in wet rocks is the presence of conduction through the pore fluids.

The effect of small amounts of water on the electrical conductivity of otherwise dry rocks has been studied by Olhoeft (1975; 1976; 1982). He found an increase in conductivity of over

T2.2. THE RANGE OF CONDUCTIVITIES OF SOME COMMON ROCKS AND MINERALS. (After Jackson, 1984 and Parkhomenko, 1967.).

CONDUCTIVITY/S m ⁻¹	ROCKS	MINERALS	OTHER
10 ⁸			METALS
10 ⁶			
10 ⁴		GRAPHITE GALENA, MAGNETITE	
10 ²			TELLURIUM
10 ⁰		SULPHIDES	SEA WATER GERMANIUM
10 ⁻²	WET BASALT	CARBONATES ZINC ORES	SILICON
10 ⁻⁴	PERIDOTITE		
10 ⁻⁶	QTZ DIORITE LIMESTONE	METAL OXIDES IRON ORES, ALBITE	ICE
10 ⁻⁸	GNEISS SANDSTONES	GARNET	
10 ⁻¹⁰	DRY BASALT		IODINE
10 ⁻¹²	PYROPHYLLITE	SILICATE MINERALS OLIVINE	POLYTHENE
10 ⁻¹⁴		QUARTZ	PARAFFIN PTFE
10 ⁻¹⁶		DIAMOND	

two magnitudes upon the addition of a very small amount of water to the dry basalt in dry air, figure F2.7. Pore fluid conduction is the dominant conduction mechanism. The variations of rock matrix conductivity with pressure and temperature are now of very limited importance compared with the variations of conductivity due to the effect of pressure and temperature upon the pore fluid. A review of the effect of pressure and temperature upon aqueous pore fluids can be seen in section 2.4.

The published data concerning rock electrical conductivities exhibits a large degree of scatter. In many cases the value of

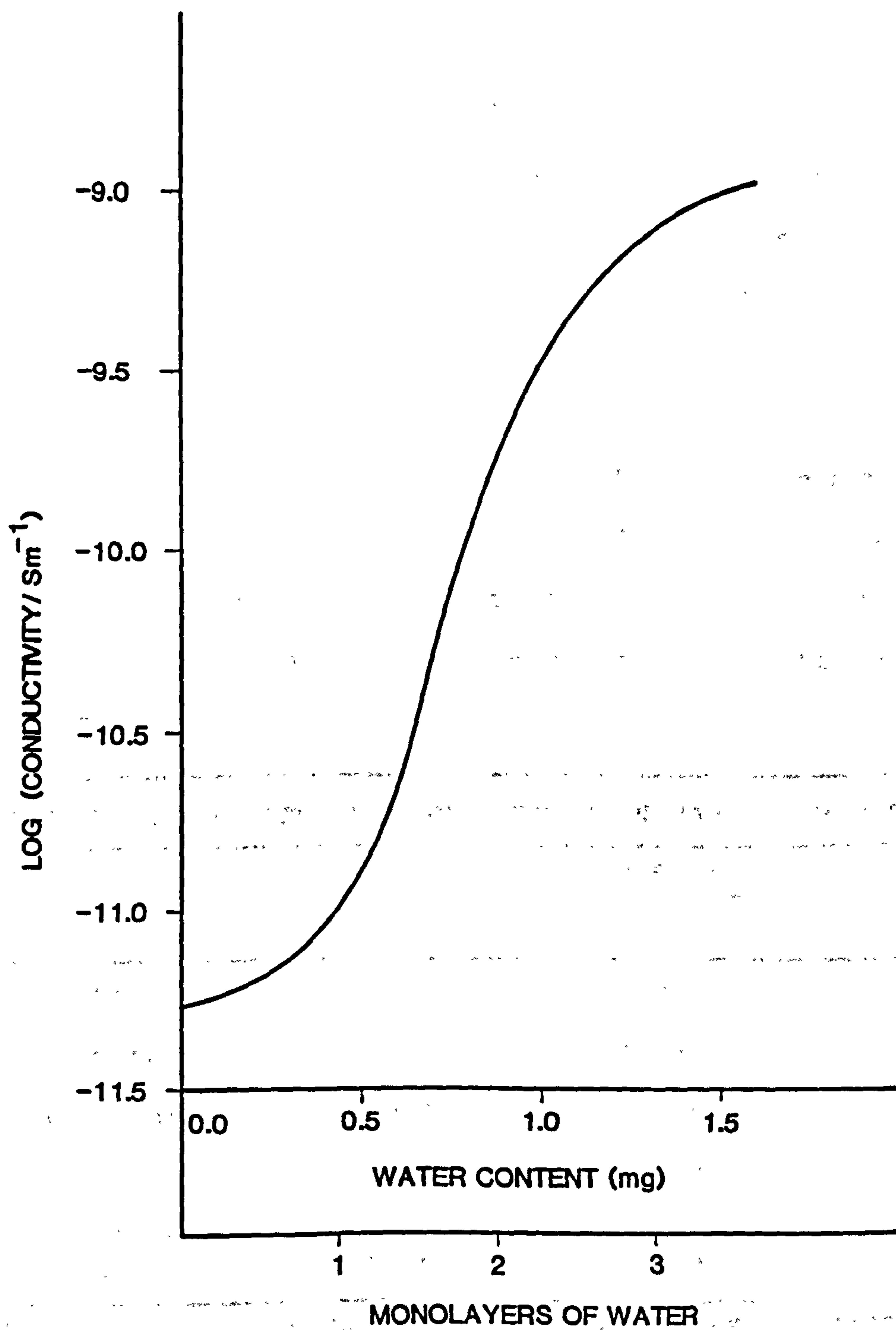


FIGURE F2.7. The variation of rock conductivity as a function of small amounts of added water (After Jackson, 1984)

conductivity measurements on reportedly identical mineral aggregates produces values that vary by several orders of magnitude. Some of such cases are documented by Parkhomenko (1967). One possible reason for such a spread of measured values is anisotropy. In most published data there has been no determination of the direction of measurement relative to the texture of the rock. The geometry of rock texture relative to the measurement direction varies from measurement to measurement producing a scatter in the conductivity value obtained. Conductivity anisotropy in minerals is due to charge carrier funnelling along crack planes as has recently been shown in core flooding experiments carried out at B.P. research laboratories in Sunbury. Rock conductivity anisotropy is similarly due to greater charge carrier mobility along bedding or cleavage planes or along foliations. Some examples of anisotropy are given in Table T2.3.

TABLE T2.3. CONDUCTIVITY ANISOTROPY IN VARIOUS ROCKS AND MINERALS			
MATERIAL	CONDUCTIVITY/S m ⁻¹		REFERENCE
	PARALLEL TO LAYERING	NORMAL TO LAYERING	
Biotite mica	1X10 ⁻⁹	2X10 ⁻¹¹	1
Enstatite	5X10 ⁻⁷	4X10 ⁻⁸	2
Shaly Slate	2X10 ⁻³	1X10 ⁻⁵	3
Galena	1X10 ³	3X10 ⁻³	3
Crystalline Slate	2X10 ⁻⁶	3X10 ⁻⁸	3
REFERENCES: 1 - TOLLAND (1973); 2 - DVORAK and SCHLOESSIN (1973); 3 - PARKHOMENKO (1967).			

Another source of discrepancy between different measurements of apparently identical dry rocks is the degree of their dessiccation. It has been shown that samples measured in a vacuum are much less conductive than samples measured in the relatively humid air of the laboratory. This can produce a very significant error in rocks of very low conductivities (St Amant and Strangway, 1970).

Even in the absence of the two effects hitherto mentioned it is hardly surprising that there is some scatter in the electrical conductivities of seemingly identical rocks as small variations of grain size and amounts of impurities will have some effect.

In a small number of rocks conducting minerals may play a significant role in conduction. The relatively highly conducting mineral graphite may have a large effect on the conductivity of the bulk rock even if it is present only in small amounts. In this case the distribution of the highly conducting mineral within the fabric of the rock may cause a certain conductivity anisotropy. Other relatively conducting minerals such as metallic oxides and clay minerals in general do not have a significant effect upon the bulk saturated rock conductivity unless they are present in such a critical concentration that continuous or near continuous conducting pathways are formed (Tolland and Strens, 1972). At concentrations near the critical concentration small changes in the percentage volume of conducting mineral has considerable effect on the bulk conductivity, figure F2.8 (Madden, 1976).

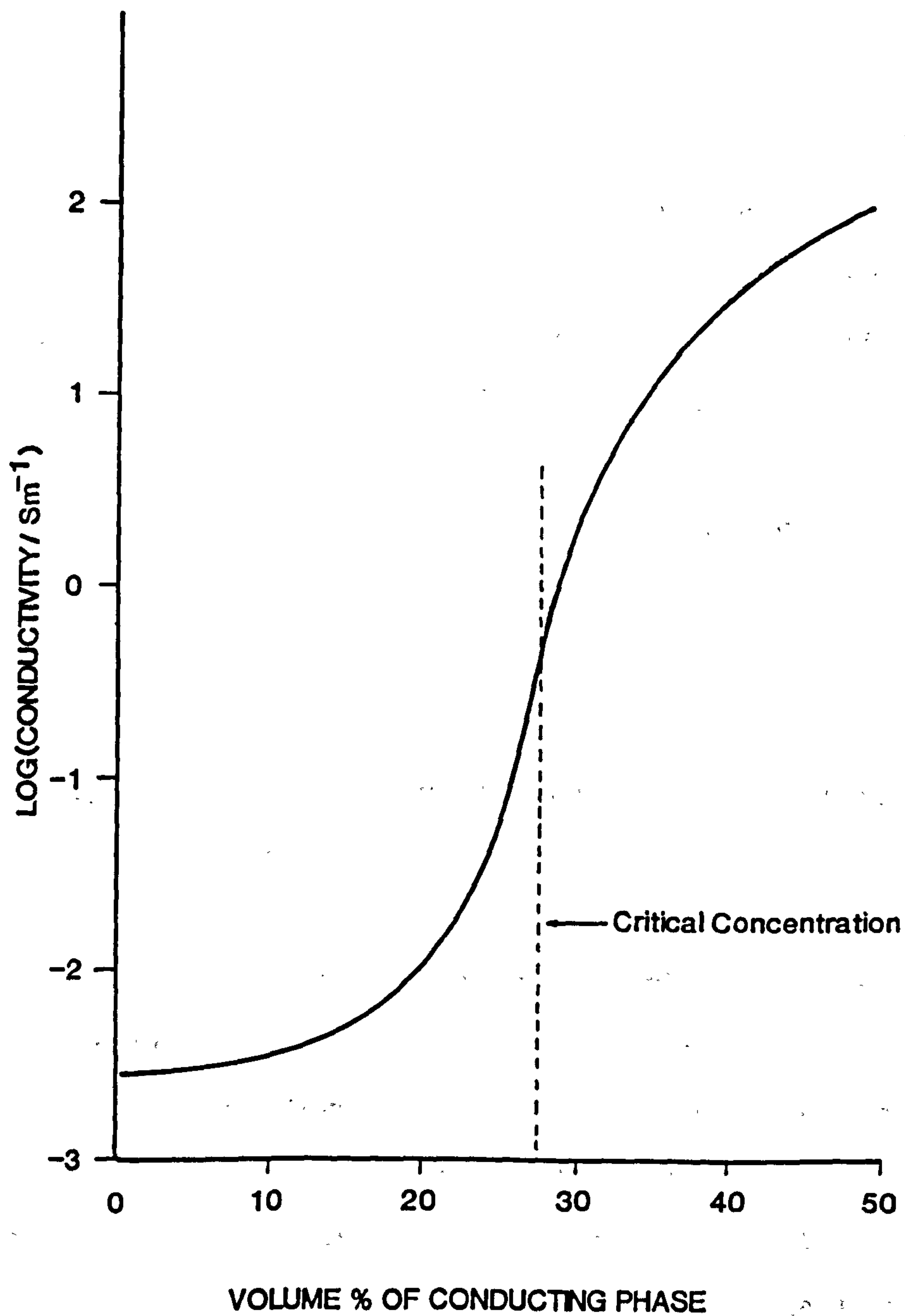


FIGURE F2.8.

The variation of rock conductivity as a function of small percentage increases of a conducting mineral (After Lee, 1977).

A recent study of rock conductivities has indicated that there is a rise in conductivity with rising content of the metallic oxides FeO, Fe₂O₃, TiO₂ and/or with increasing content of Ni, Cr, Co (Lastovickova and Kropacek, 1980; Lastovickova, 1984). Cermak and Lastovickova (1987) have applied percolation theory to a set of alkaline and calc-alkaline rocks of a wide range of TiO₂ content (0.1 - 6.0 %wt) and FeO + Fe₂O₃ content (2.5 - 17.0 %wt). They report the success of percolation theory up to 500°C where it breaks down due to electronic conduction through the heated mineral, mentioned above, becoming significant (Lastovickova and Kropacek, 1983). The electrical conductivity of Fe-Mg-SiO₂ minerals, the main rock forming minerals of basaltic rocks, has been investigated by Kropacek and Lastovickova (1980). They found that as a general rule the electrical conductivity decreased with increasing oxidation.

Table T2.4. shows the room temperature conductivity of some dry rocks, ores and minerals.

The effect of confining pressure upon the electrical conductivity of dry rocks is very limited and is related to the rock incompressibility (Dvorak, 1973). The effect of changes of temperature upon dry rock conductivities is much greater. Raising the temperature of a metal decreases its conductivity due to scattering of charge carriers by metal lattice vibrations. Raising the temperature of insulators and semiconductors, however, increases their electrical conductivity by increasing the number of charge carriers. The electrical conductivity of insulators increases exponentially with temperature (Ashcroft and

**TABLE T2.4. THE CONDUCTIVITY OF SOME COMMON ROCKS AND MINERALS.
(After Parkhomenko, 1967 and Keller, 1966)**

MATERIAL	CONDUCTIVITY/S m ⁻¹
GRAPHITE	1.0X10 ⁵
GALENA	4.0X10 ⁴ - 3.0X10 ⁰
BORNITE	4.0X10 ⁴ - 1.2X10 ⁻²
PYRITES	3.0X10 ⁴ - 6.0X10 ⁻¹
MAGNETITE	1.2X10 ⁴ - 1.0X10 ¹
IRON ORES	2.0X10 ⁰ - 3.0X10 ⁻⁷
ZINC ORES	1.4X10 ⁰ - 7.0X10 ⁻³
PERIDOTITE	1.5X10 ⁻⁴
QUARTZ DIORITE	5.0X10 ⁻⁶
DIABASE	3.0X10 ⁻⁶ - 6.0X10 ⁻¹¹
GNEISS	3.0X10 ⁻⁷
GARNET	3.0X10 ⁻⁷
ALBITE	2.5X10 ⁻⁷
BASALT	8.0X10 ⁻⁸
PYROPHYLLITE	1.0X10 ⁻¹²
MUSCOVITE	1.0X10 ⁻¹² - 1.0X10 ⁻¹⁴
QUARTZ	5.0X10 ⁻¹⁵

Mermin, 1976; Kittel, 1971). Most common rock forming minerals can be considered to be insulators and to undergo this exponential rise in electrical conductivity with temperature. The conductivity at a temperature, T, σ_T is given by;

$$\sigma_T = \sigma_{\infty} e^{(-E_0/kT)} \quad E2.33.$$

where σ_{∞} is the conductivity at infinite temperature and is

assumed to be temperature independent, k is Boltzmann's constant, E_0 is the activation energy and T is the temperature in kelvin.

A graph of $\log_e \sigma_T$ against $1/T$ gives a straight line whose gradient is $-E_0/k$ (Nakhromenko and Mkrtchyan, 1974). The value of $-E_0/k$ is a measure of the movement of charge carriers, figure F2.9. Different conduction mechanisms give rise to different values of $-E_0/k$. A change in the slope of the $\log_e \sigma_T$ against $1/T$ graph at a certain temperature indicates that a change in conduction mechanism has occurred near that temperature. If a graph such as that shown in figure F2.9. has more than one slope E2.33 has to be modified and becomes;

$$\sigma_T = \sigma_1 e^{-E_0'/kT} + \sigma_2 e^{-E_0''/kT} \quad \text{E2.34.}$$

The first term relates to the low temperature conduction mechanism and the second to the mechanism that becomes dominant at higher temperatures. In some cases more terms are required each corresponding to a single conduction mechanism. It can be seen that it is therefore very important to quote the temperature regime whenever an activation energy is reported.

Figure F2.10. shows $\log_e \sigma_T / 1/T$ graphs for various rock types (Cermak and Lastovickova, 1987).

Comparison of the log conductivity against $1/T$ graphs for rocks of differing origins and composition shows that there is an overlap of the conductivity/ $1/T$ regimes of granites and granodiorites, figure F2.11. (Volarovich and Parkhomenko, 1976).

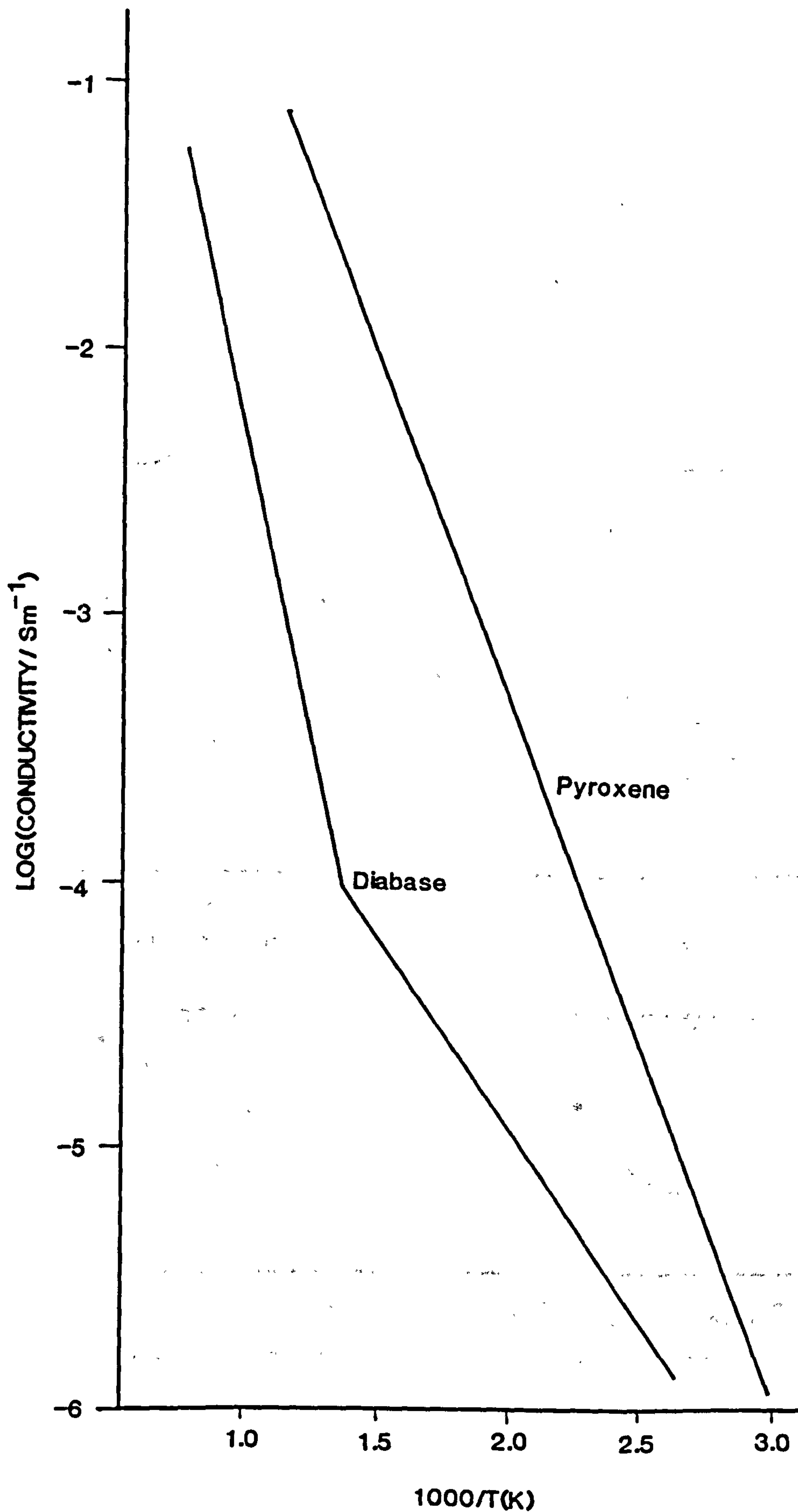


FIGURE F2.9. The variation of dry rock conductivity as a function of $1000/T(K)$ for different samples (After Nakhromenko and Mkrtchyan, 1974; Parkhomenko, 1967).

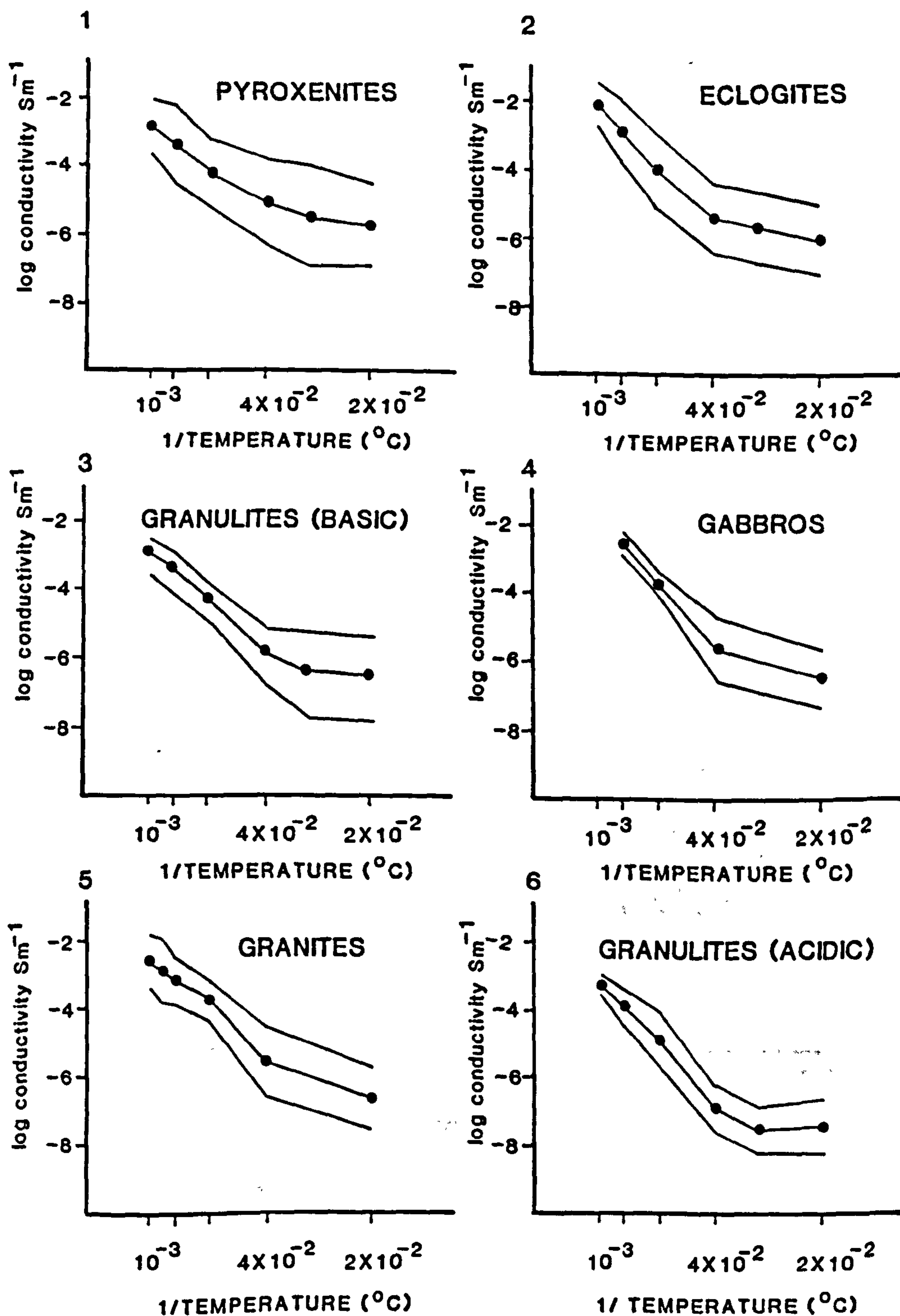


FIGURE F2.10

The variation of the electrical conductivity of some dry rock types as a function of $1/T(^{\circ}\text{C})$. Central line - the average behaviour, envelope - the variation away from the mean behaviour. (After Cermak and Lastovickova, 1987).

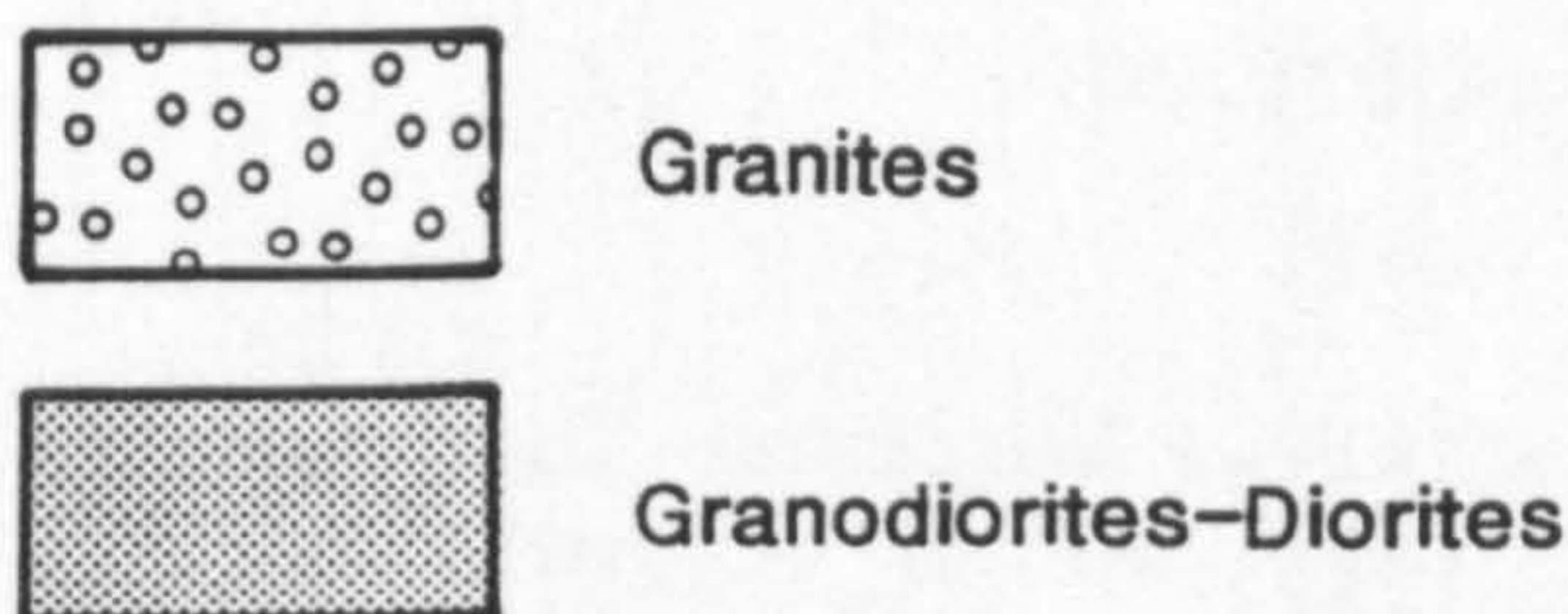
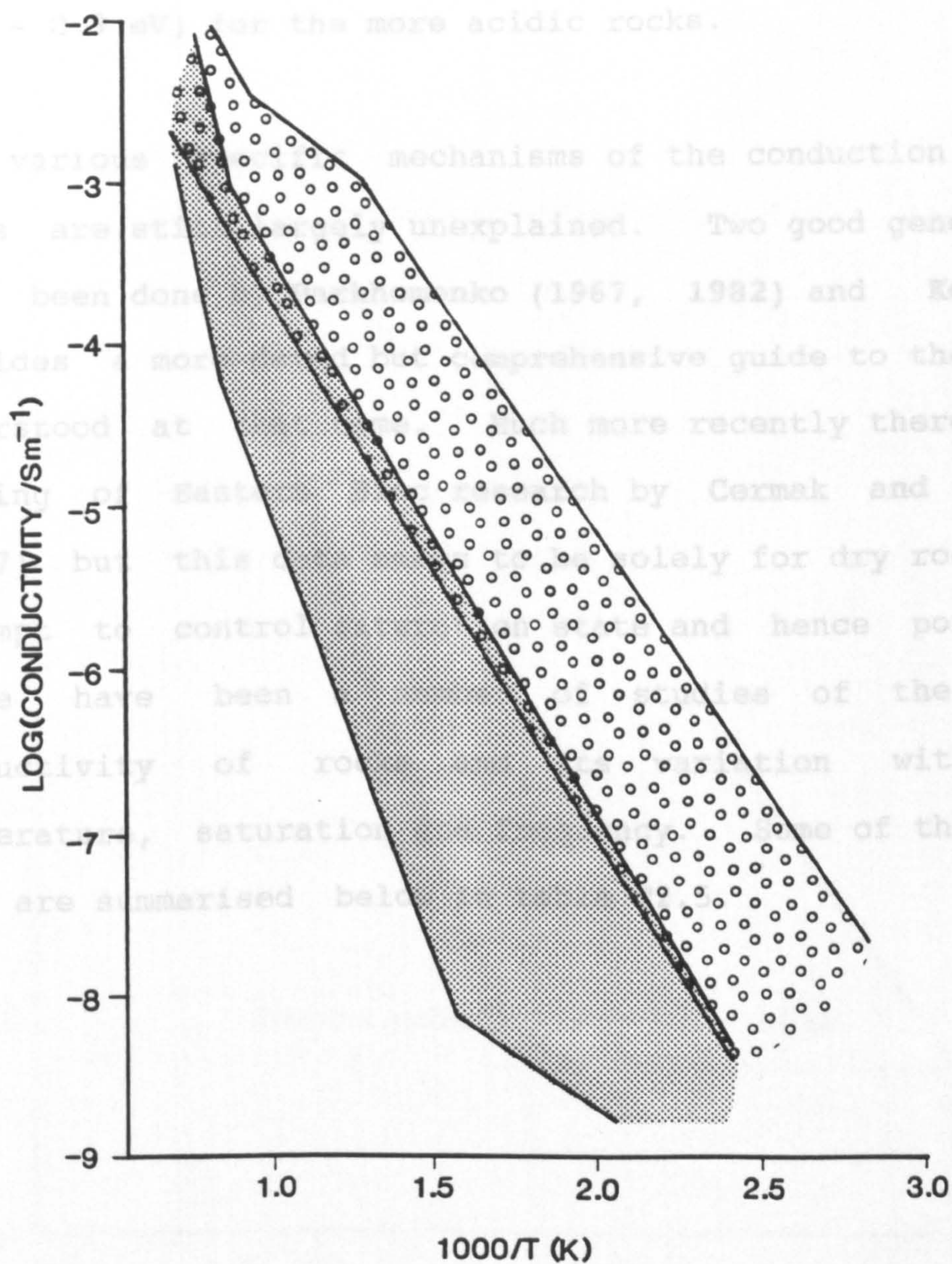


FIGURE F2.11. The variation of rock conductivity as a function of $1000/T(K)$ for a range of granites and granodiorites/diorites (After Volarovich and Parkhomenko, 1976).

The range of conductivities for both types of rock at low temperatures is quite great but decreases considerably as the temperature is increased. As the temperature increases the amount of overlap tends to increase as the activation energies of the more mafic rocks (0.6 - 0.8 eV) tends to be less than that (0.7 - 0.9 eV) for the more acidic rocks.

The various specific mechanisms of the conduction process in rocks are still largely unexplained. Two good general reviews have been done by Parkhomenko (1967, 1982) and Keller (1966) provides a more dated but comprehensive guide to the subject as understood at that time. Much more recently there has been a pooling of Eastern Bloc research by Cermak and Lastovickova (1987) but this data seems to be solely for dry rocks with no attempt to control saturation state and hence pore pressure. There have been a number of studies of the electrical conductivity of rocks and its variation with pressure, temperature, saturation and frequency. Some of the more useful ones are summarised below in table T2.5.

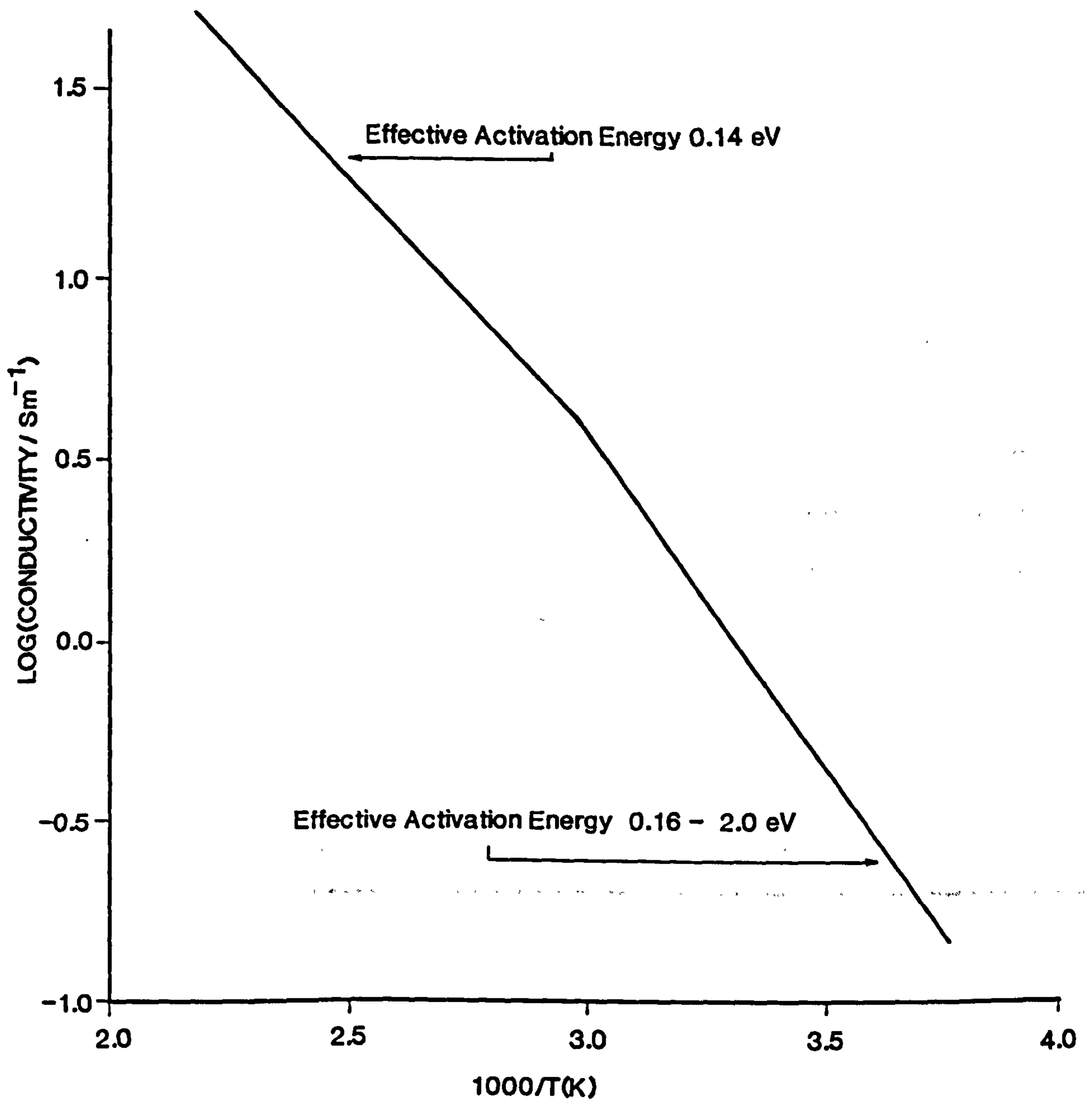


FIGURE F2.12. The estimated variation of rock conductivity as a function of $1000/T(K)$ for a 0.5 M NaCl solution (After Jackson, 1984).

TABLE T2.5. SOME MAJOR REFERENCES AND THEIR COVERAGE.

AUTHORS	DATE	NOTES
Dvorak	1973	Mafic and ultramafic rocks High P and T
Dvorak and Schloessin	1973	High P and T
Parkhomenko	1967	Mafic and ultramafic rocks High P and T
Parkhomenko	1973	High P and T
Parkhomenko and Dvorak	1971	Frequency dependency High P and T
Duba	1972	High P and T
Duba et al	1973	Pyroxene and olivines
Duba et al	1974	Pyroxene and olivines
Duba et al	1978	Quartz
Bradley et al	1964	Pyroxene and olivines
Stesky and Brace	1973	Serpentinities Surface T
Tolland	1973	High T, Surface P
Tolland and Strens	1972	High T, Surface P
Nakhromenko and Mkrtchyan	1974	High T and P
Bondarenko et al	1976	High T and P
Katsube and Collett	1973a	Frequency dependency Surface T and P
Katsube and Collett	1973b	Frequency dependency Surface T and P
Olhoeft et al	1973	Frequency dependency High T, Surface P
Bondarenko	1972	Frequency dependency High T, Surface P
Budzinski et al	1985	High T
Kropacek et al	1985	
Lastovickova	1984	
Jelenska et al	1978	
Lastovickova and Kropacek	1983	
Lastovickova et al	1985	
Cermak and Lastovickova	1987	High T

2.8. THE EFFECT OF TEMPERATURE UPON THE ELECTRICAL CONDUCTIVITY OF SATURATED ROCKS

Increasing the temperature of any insulator produces a corresponding increase in its electrical conductivity as has been discussed in section 2.7. This also applies to rocks whatever the state of their saturation.

Little data is available concerning the effect of temperature upon the electrical conductivity of rocks held at high pressures. Lee (1977) and Evans (1980) have measured the electrical conductivity of rocks as a function of temperature up to 250 °C at a confining pressure of 0.4 GPa. Jackson (1984) has extended this work by making measurements up to 240 °C at 0.35 GPa and up to 900 °C at atmospheric pressure on dry rocks. All this research has found increases in the electrical conductivity as temperature is increased. Maintenance of high pressures and temperatures produced a transient decrease of the electrical conductivity. Once the transient decrease has occurred subsequent temperature cycling produces repeatable conductivity values. The effect of the transient conductivity decrease is noticeable in both saturated and unsaturated rock samples, although it is smaller in unsaturated samples. Lee (1977) and Evans (1980) attributed the decrease to a transient creep process. It is conceivable that the presence of an aqueous solution may be able to lubricate such a process and hence make it more extensive. This could account for the decrease seeming to be more marked in saturated rock samples. A generalised view of the electrical conductivity of a rock sample as a function of

temperature is shown in figure F2.13.

The range of activation energies obtained by Lee (1977) was from 0.1 to 0.4 eV. Pyrophyllite gave a value around 0.13 eV whereas amphibolites of various types gave values of around 0.4 eV. Lee found that the average value for the activation energy of the rocks he measured was around 0.2 eV. This value can be compared with the values of the activation energy quoted for 'brine' of 0.2 eV (Parkhomenko, 1982) and 0.5 M NaCl of 0.14 eV (Waxman and Smits, 1968). The activation energies of dry igneous rocks have been studied some while ago by Parkhomenko and Bondarenko (1962). They found that the activation energies of dry basic and ultrabasic rocks (diabase, basalt and peridotite) measured between 100 °C and 800 °C varied between 0.6 and 0.8 eV. The similarity between the activation energies of saturated rocks and an aqueous solution of 0.5 M NaCl in comparison with the activation energies of dry rocks suggests that the presence of the conducting fluid is the major factor in influencing the electrical behaviour of the saturated rock.

Lee (1977) has found that some rocks exhibit a more sensitive variation of conductivity as a function of temperature than saturation with a salt solution might suggest. This effect has also been observed by other authors (Evans, 1980; Drury, 1976; 1979; Stesky and Brace, 1973 and Parkhomenko, 1972). The activation energies of these basic amphibolites and basic gneisses are around 0.3 to 0.4 eV. It is possible that this effect is caused by additional conduction through rock minerals as well as through the pore fluid. There have been some

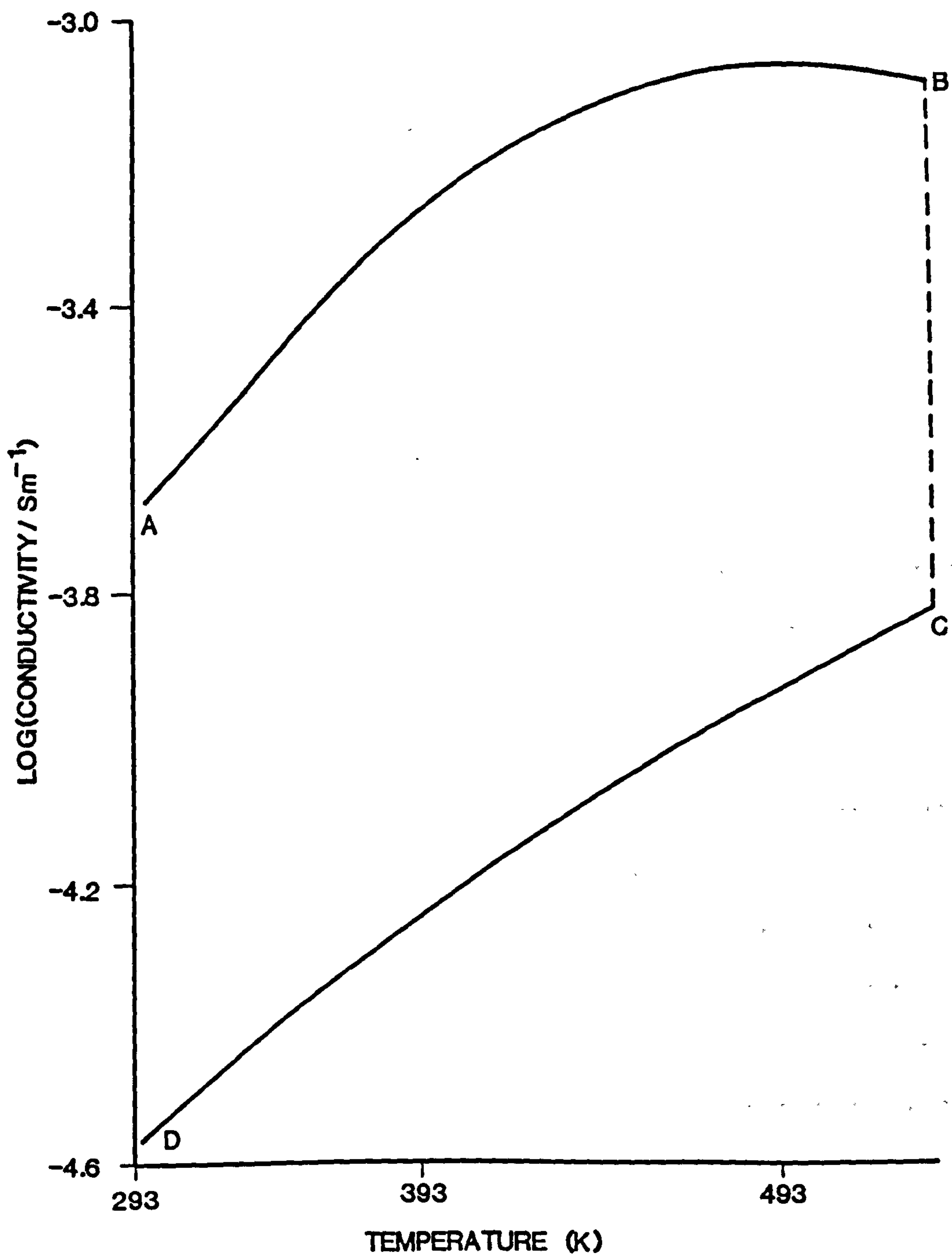


FIGURE P2.13. The variation of the electrical conductivity of a saturated rock as a function of temperature and at a constant high pressure (After Lee, 1977).

suggestions that the presence of enhanced conduction is as a result of conduction through clay minerals or through deposits of magnetite along grain boundaries. The partitioning of the saturated rock conductivity into two groups is shown in figure F2.14. (After Lee (1977)) and diagrammatically in figure F2.15.

Interestingly it is the rocks that exhibit a conductivity similar to that of a molar salt solution that have room temperature conductivities predictable by Archie's Law (Evans, 1980).

2.9. THE EFFECT OF CONFINING PRESSURE UPON THE ELECTRICAL CONDUCTIVITY OF SATURATED ROCKS

The conductivity of a saturated rock is a sensitive function of additional electrolyte especially at low concentrations of added electrolyte. As we have said in section 2.5. the electrical conductivity is dependent upon pore parameters. It can be summed up by stating that the electrical conductivity is largely controlled by the percentage volume of the saturated part of the pore cross-section perpendicular to the current flow as well as the pore connectivity.

In considering a rock matrix/pore fluid system with no conduction mechanism apart from electrolyte conduction it may be said that providing each element does not interact with the other that its conductivity should lie between the low conductivity of the rock matrix and the conductivity of the pore fluid. This is not particularly useful except to define the absolute limits in which

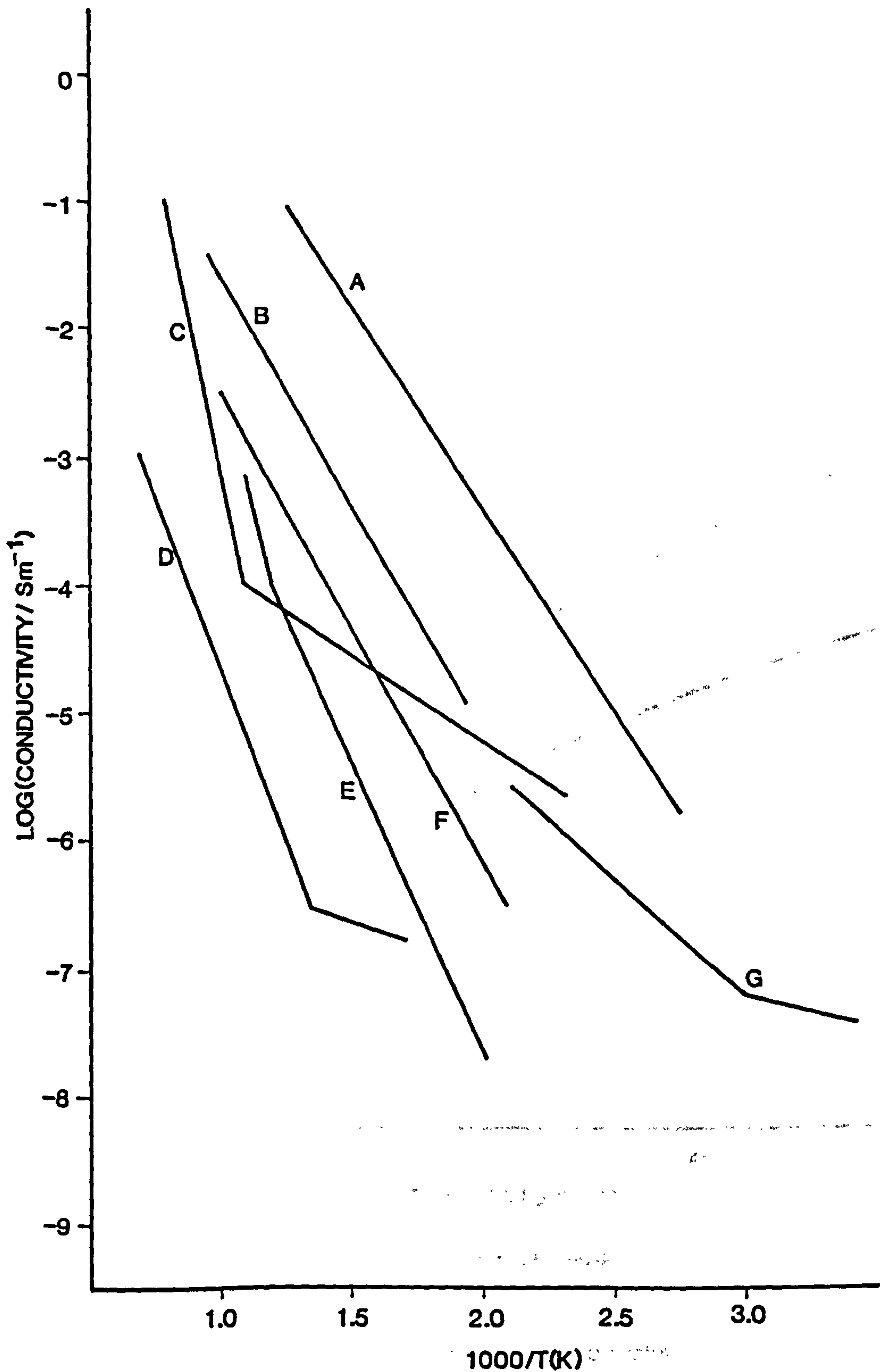


FIGURE F2.14. The variation of the electrical conductivity of a range of rocks and minerals as a function of $1000/T(K)$ (After Lee, 1977).

A - Diabase	Parkhomenko, 1967
B - Basalt	Parkhomenko, 1967
C - Pyroxene	Nakhromenko and Mkrtchyan, 1974
D - Granite	Parkhomenko, 1967
E - Diorite	Parkhomenko, 1967
F - Eclogite	Parkhomenko, 1967
G - Enstatite	Dvorak and Schloessin, 1973.

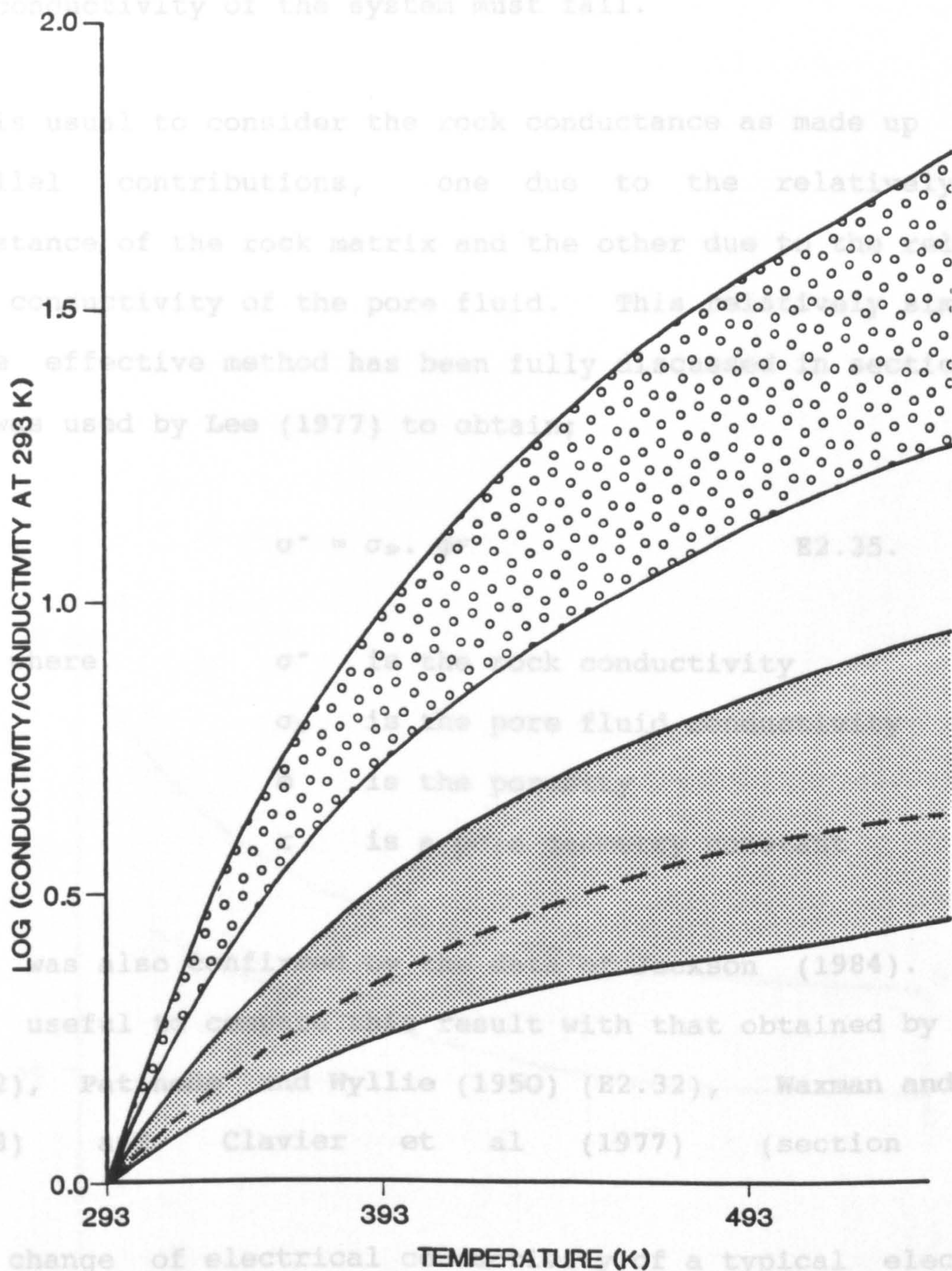


FIGURE F2.15. A comparison of the increase of electrical conductivity of saturated rock samples as a function of temperature for rocks held at high pressure. (After Lee, 1977; Drury, 1979; Evans, 1980).

the conductivity of the system must fall.

It is usual to consider the rock conductance as made up of two parallel contributions, one due to the relatively high resistance of the rock matrix and the other due to the relatively high conductivity of the pore fluid. This relatively simple but quite effective method has been fully discussed in section 2.6. and was used by Lee (1977) to obtain;

$$\sigma^* = \sigma_P \cdot \phi^r \quad \text{E2.35.}$$

where

σ^*	is the rock conductivity
σ_P	is the pore fluid conductivity
ϕ	is the porosity
r	is a pore geometry constant

This was also confirmed by the data of Jackson (1984). It is also useful to compare this result with that obtained by Archie (1942), Patinode and Wyllie (1950) (E2.32), Waxman and Smits (1968) and Clavier et al (1977) (section 2.11).

The change of electrical conductivity of a typical electrolyte saturated rock as a function of confining pressure is shown as figure F2.16. As pressure initially increases (A-B, F2.16.) the electrical conductivity decreases swiftly. The rate of decrease lessens as the confining pressure gets higher. This variation is caused by the reduction of porosity due to the increasing closure of pore fluid pathways through the rock matrix.

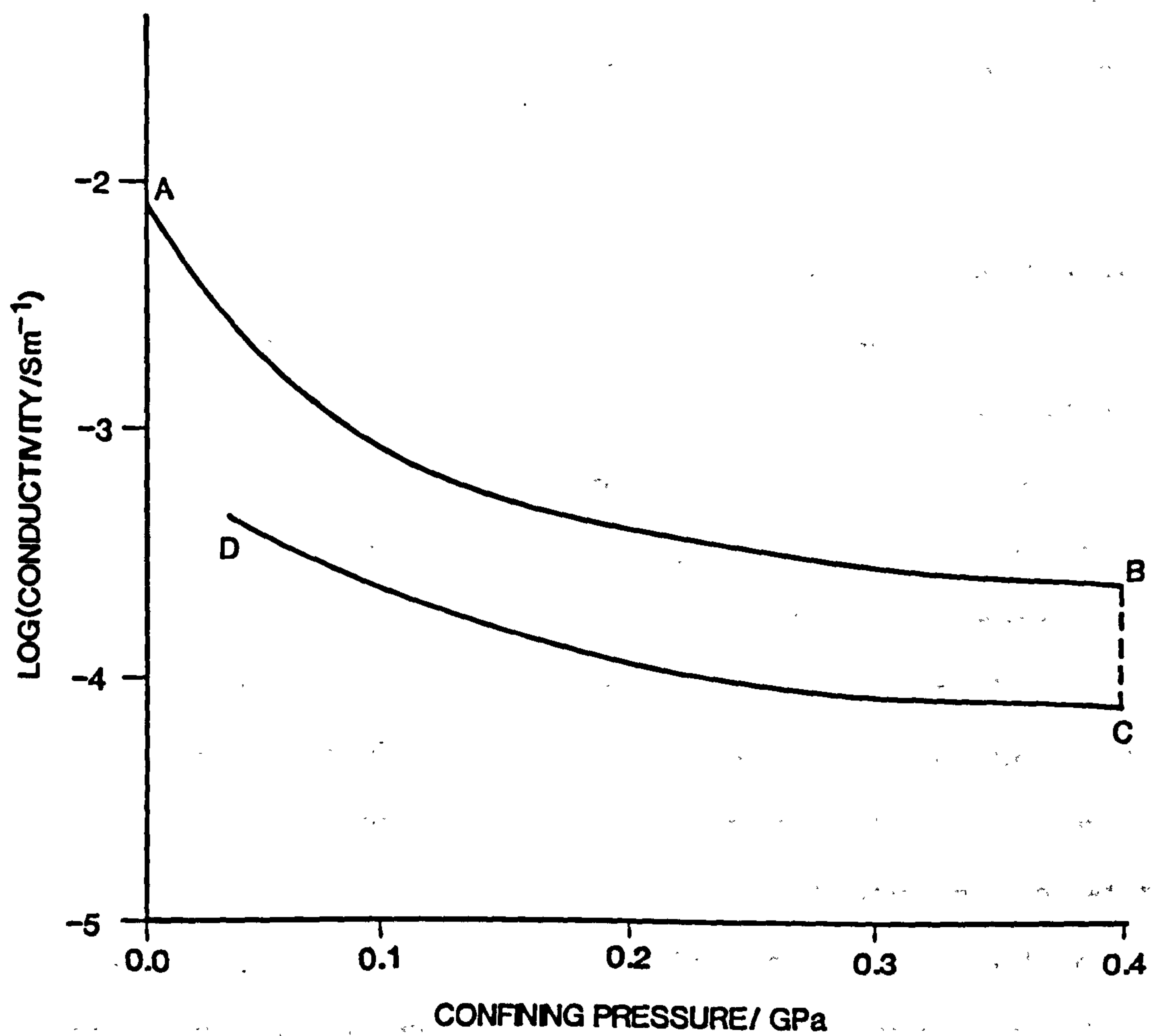


FIGURE F2.16. The variation of the electrical conductivity as a function of confining pressure (After Lee, 1977).

At higher pressures (>0.3 GPa) the conductivity of low porosity crystalline rocks depends on the square of the porosity (Brace et al, 1965). This is the pressure regime where all the long thin cracks have closed and only high aspect ratio cracks remain decreasing the overall porosity (Walsh, 1965). Lee (1977) and Evans (1980) have found that this form of Archie's Law ($r = 2$) is effective in describing electrical conductivity of acidic crystalline rocks at high pressures. They found that the correlation was especially good for those rocks which were quartz rich. There is a discrepancy when this model is applied to basic and ultrabasic rocks.

There is also a difference in the behaviour of acidic and basic rocks as confining pressure increases. Figure F2.17. shows the relative variations of the electrical conductivity of basic and acidic rocks with confining pressure. The basic rocks seem to have a linear dependence of $\log_e(\text{conductivity})$ with pressure compared with the non-linear behaviour of acidic rocks. This has been attributed to differences in crack distribution between acidic and basic rocks (Lee, 1977). Acidic rocks were assumed to have a greater proportion of low aspect ratio cracks compared with basic rocks. Work by Jackson (1984) has indicated that the converse situation exists. Jackson has used data from conductivity and seismic velocity measurements to produce crack porosity distributions. He used inversions derived from Hoenig's equation (Hoenig, 1979) and the non-interactive theory of Cheng and Toksoz (1979). Jackson found that a high proportion of the total porosity of quartz rich rocks is in the form of high aspect ratio cracks compared with more basic rocks. Jackson attributes

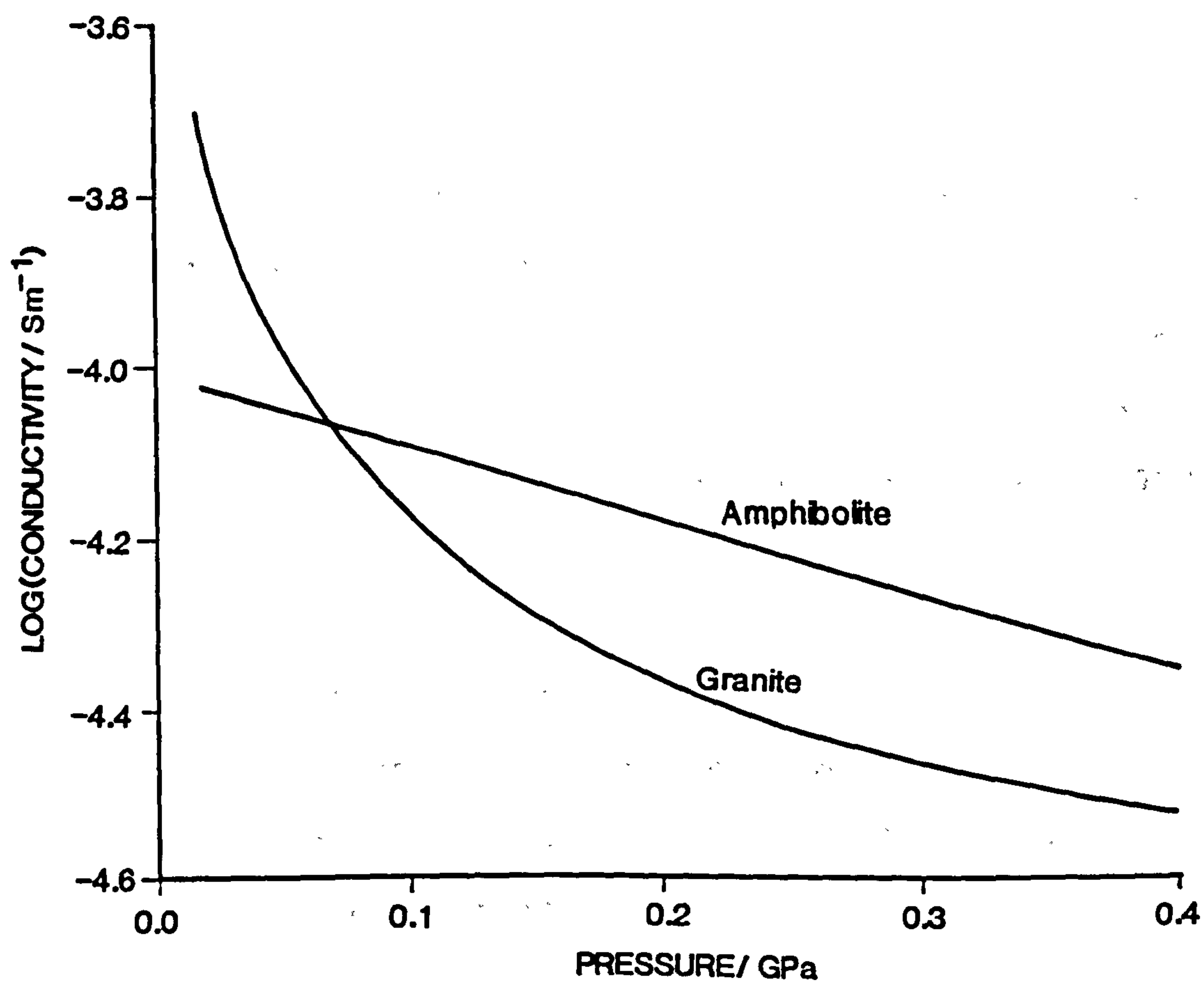


FIGURE F2.17. Schematic variation of the electrical conductivity of electrolyte saturated granitic and amphibolitic rock samples as a function of confining pressure (After Evans, 1980).

the discrepancy between the conductivity/pressure variations of acidic and basic rocks to the presence of an enhanced conduction mechanism in the basic rocks which is less sensitive to pressure than the closure of pore fluid conducting cracks. In the case of basic rocks an enhanced conduction mechanism should make the overall conductivity proportionably less reduced compared with acidic rocks where no such mechanism is important. This can produce the required conductivity/pressure characteristics.

It has been demonstrated that a value for the Archie's Law pore geometry exponent of unity is associated with low aspect ratio cracks (Shankland, 1975). A value of this parameter closer to 2 has been shown to be more applicable to higher aspect ratio cracks such as those that are still open at higher pressures (>0.4 GPa) (Brace et al, 1965; Lee, 1977 and Evans, 1980).

There have been only a few theoretical studies of the electrical conductivity of saturated rocks which match the detail attained in the theoretical modelling of seismic velocities. Such theories as there are (e.g. Hoenig (1979) and Walsh (1975)) are based upon the self-consistency theory of O'Connell and Budiansky (1974).

The usual approach involves idealising a crack shape. Walsh (1975) used thin cracks, tubes and spheres to produce a theory which was difficult to apply. Hoenig's work was based upon a spectrum of ellipsoidal cracks such as that shown in figure F2.18. in which a , b and c are the ellipsoid's principal semi-axes. Hoenig used coin-shaped ellipsoids, where $a = b \gg c$,

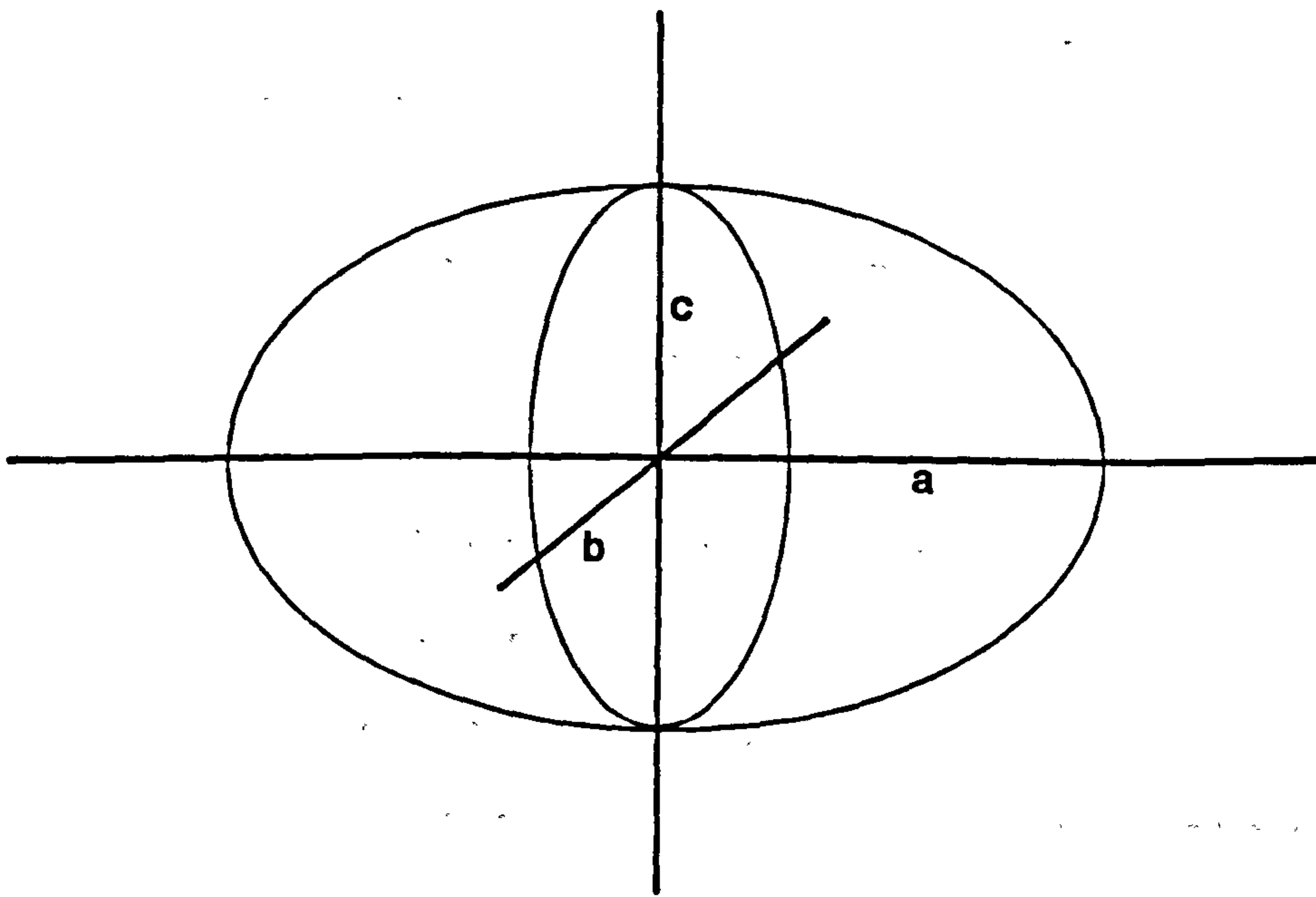


FIGURE F2.18. The 'penny-shaped' cracks with semi-axes a , b and c . Variation of the relative and absolute sizes of a , b and c defines a spectrum of different sized and shaped cracks and micro-cracks (After Jackson, 1984).

embedded in an elastic matrix. Two aspect ratios were derived from a, b and c and these were able to be varied to give a spectrum of different shaped cracks.

$$\text{Thickness aspect ratio} \quad \alpha = c / b \quad \text{E2.19a}$$

$$\text{Planform aspect ratio} \quad \gamma = b / a \quad \text{E2.19b}$$

Hoenig kept $\gamma = 1$ ($a = b$) and varied α .

The size of the cracks was altered by changing the overall volume of pore space with respect to matrix. Hoenig obtained;

$$\frac{\sigma_M}{\sigma^*} = 1 - \frac{8E(k)\epsilon}{9} \left[\frac{1}{(\sigma^*/\sigma_M \Omega + \gamma^2 \{k^2(K(k) - E(k))\})} \right] + \left[\frac{-1}{(\sigma^*/\sigma_M \Omega + 1/\{k^2(E(k) - \gamma^2 K(k))\})} \right] \quad \text{E2.36a}$$

where; σ^* - effective conductivity

σ_M - matrix conductivity

σ_P - pore fluid conductivity

ϵ - crack density parameter

ϕ - porosity

Ω - saturation factor

$K(k)$, $E(k)$ - complete elliptical integrals (see O'Connell and Budiansky (1974))

γ - planform aspect ratio

α - thickness aspect ratio

$$\text{and;} \quad - k^2 = (1 - \gamma^2)^{1/2} \quad \text{E2.36b}$$

$$- \epsilon = 3\phi / 8E(k) \quad \text{E2.36c}$$

$$- \Omega = \sigma_M / \alpha \sigma_P \quad \text{E2.36d}$$

Evans et al (1982) showed that a modified form of this equation specific to cigar-shaped cracks predicted the observed

conductivity at around 0.4 GPa better than Archie's Law. Jackson (1984) has discussed the theory modification in detail.

Reference to figure F2.16. shows the subsequent variation of conductivity with pressure after the initial variation (F2.16, A - B). If the saturated rock is kept at high pressures for a number of hours then there is a drop in the electrical conductivity (F2.16, B - C). Lee (1977) attributes this drop to a compaction process resulting from motion along grain boundaries and better alignment of crack edges. Jackson (1984) has subsequently shown this motion to be partially reversible but on a longer timescale than the original compaction. Lee (1977) and Jackson (1984) showed that subsequent cycling of confining pressure produced repeatable electrical conductivity curves of a slightly different geometry to the original (F2.16, C - D). This is considered to be the post-transient elastic response state of the rock.

2.10. THE EFFECT OF PORE PRESSURE UPON THE ELECTRICAL CONDUCTIVITY OF SATURATED ROCKS

The variation of pore pressure has a large effect upon the electrical conductivity of all saturated rocks. An increase in the pore pressure produces an increase in the electrical conductivity whatever the value of confining pressure. This variation can be seen in figure F2.19. The knowledge that pore pressure and confining pressure act in opposition to each other has long been recognised. Increasing the confining pressure

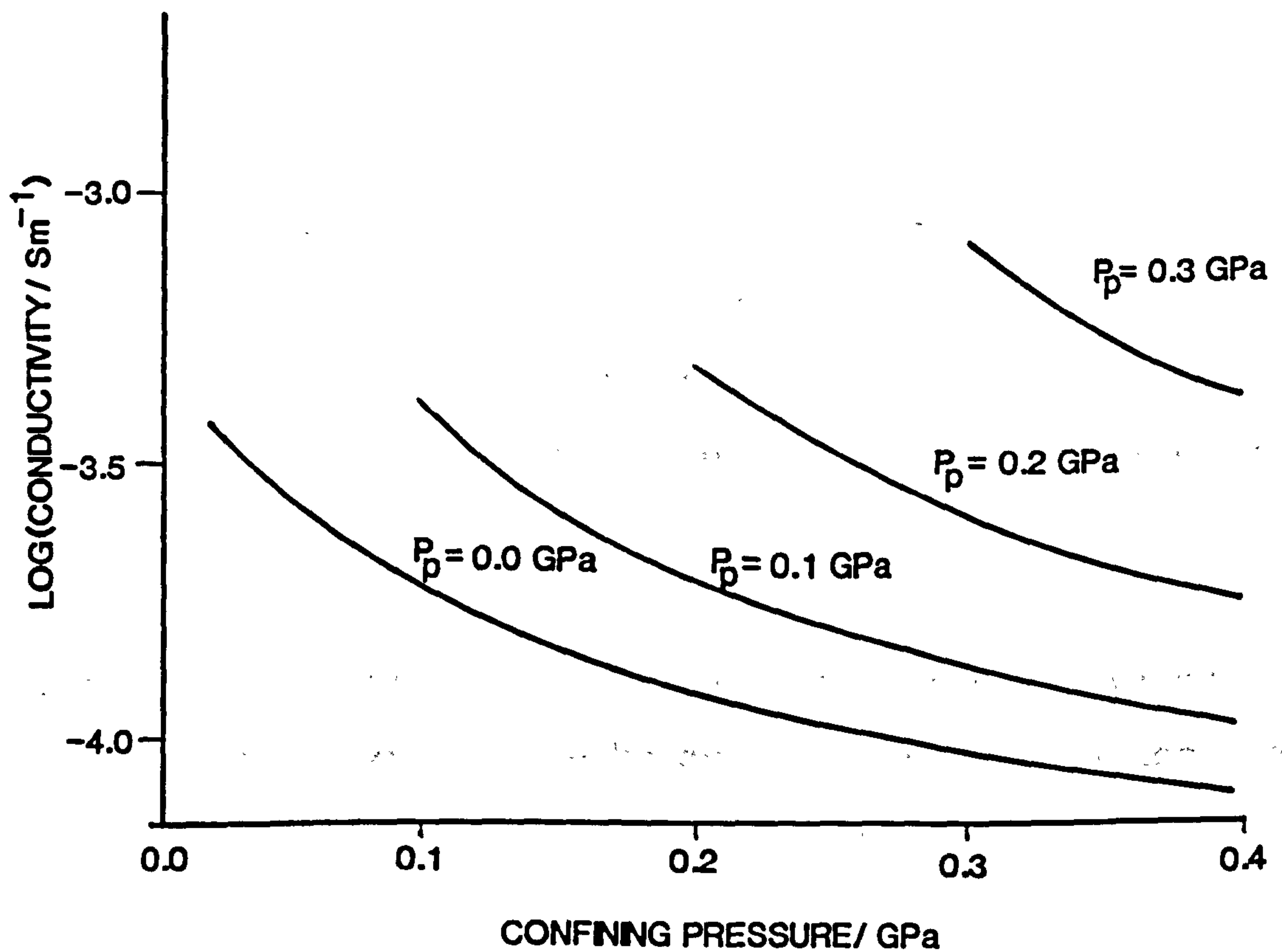


FIGURE F2.19. The variation of the electrical conductivity of a saturated rock as a function of confining pressure for differing pore fluid pressures (After Jackson, 1984).

tends to reduce the conducting pathways available for conduction whereas an increase in the pore pressure tends to increase the number of conducting pathways available by opening up healed cracks. If the rock system acts in an elastic manner to changes in both pore pressure and confining pressure then the opposition may be stated quantitatively as an effective pressure law (Robin, 1973);

$$P_e = P_c - n P_p \quad \text{E2.37.}$$

where P_e is the effective pressure
 P_c is the confining pressure
 P_p is the pore pressure

The constant, n , is accepted to be close to unity for electrical conductivity.

Similar effective pressure laws but with different values of n have been used to explain the behaviour of seismic velocities and other rock parameters (Brace, 1977; Spencer and Nur, 1976).

Lee (1977) found that the effective pressure law was approximately obeyed for electrical conduction by plotting effective pressure against conductivity as shown in figure F2.20. Nevertheless he found that a few rocks did not obey the law. An example of such is given as figure F2.21.

The effective pressure law assumes that the elastic response of the rock matrix to an increase of pore pressure is the same magnitude but in the opposite sense to the elastic response of

FIGURE F2.21. The variation of electrical conductivity of a saturated rock as a function of the effective pressure for a sample disobeying the effective pressure law (After Jackson, 1984).

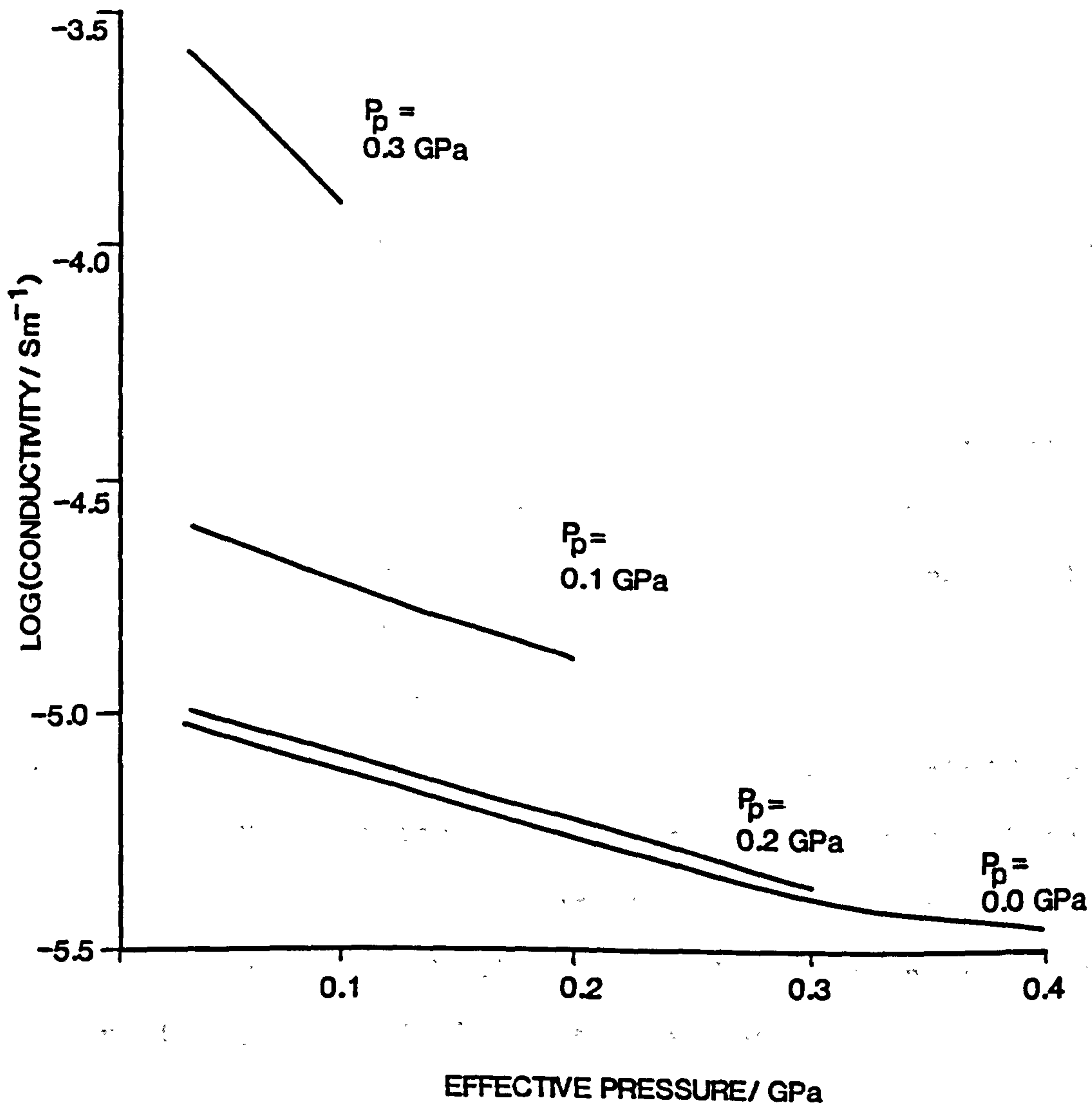
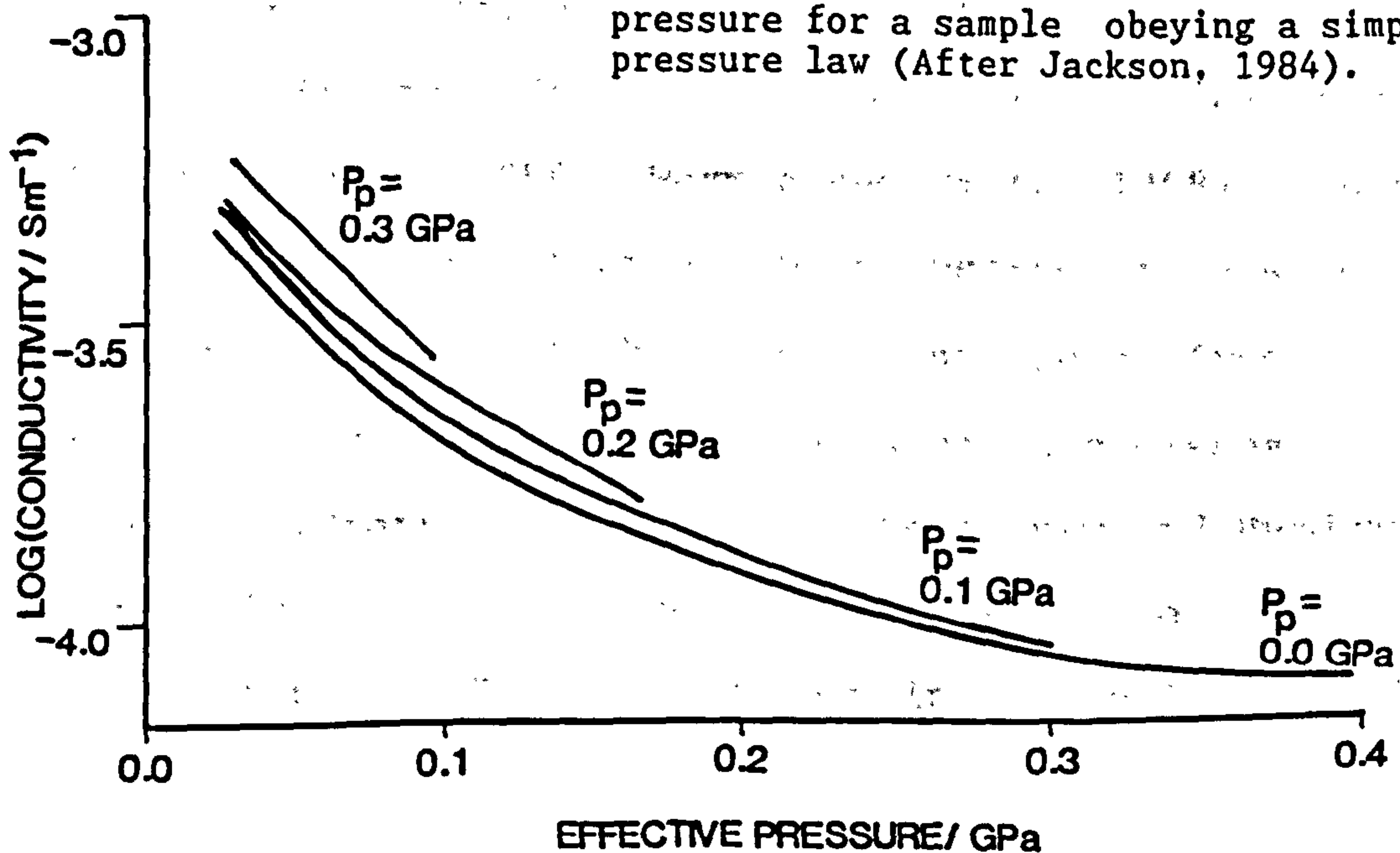


FIGURE F2.20. The variation of the electrical conductivity of a saturated rock as a function of effective pressure for a sample obeying a simple effective pressure law (After Jackson, 1984).



the rock to the same magnitude of applied confining pressure.

2.11. NON PORE FLUID CONDUCTION

So far it has been assumed that conduction by any mechanism involving the rock matrix is negligible compared with that due to electrolytic conduction through the pore fluid. Although it has been noted in section 2.6. that non pore fluid conduction may be significant in some rocks, the formulae so far discussed (Archie's Law and Hoenig's Equation) assume that volume rock conduction occurs only via the saturating fluid. As this is not always the case some quantitative and qualitative understanding needs to be drawn which models the non-pore fluid conduction mechanism. Many authors have suggested that Archie's Law be modified by the addition of some term that accounts for this non-pore fluid conduction (Hill et al, 1979; Waxman and Smits, 1968; Clavier et al, 1977; Patinode and Wyllie, 1950).

Surface conduction and cation exchange within pores in sands with some clay mineralogy have been shown to be mechanisms that can have a significant effect upon the overall rock conductivity (Hill and Milburn, 1956; Waxman and Smits, 1968). Subsequently non-pore fluid conduction has been observed in a wide range of sedimentary rocks. It has also, but less frequently, been observed in crystalline rocks such as basic gneisses (Lee, 1977), amphibolites (Evans, 1980) and basalts (Drury and Hyndman, 1979). Non-pore fluid conduction seems to be associated with the more basic rock types. This can be seen in figure F2.15. which shows

that the increase in conductivity of basic rocks with temperature exceeds that of acidic rocks indicating an additional conduction mechanism other than through the pore fluid that is also a sensitive function of temperature. The work of Jackson (1984) collated much of the work of the previously named authors and, with additional data, produced a model for rock conductivity depending upon pore parameters and non-pore fluid mechanisms. This will be discussed later in this section. First I will briefly describe the way wet clay mineralogy is thought to affect the rock conductivity and a couple of attempts to quantify this effect.

Clay minerals can enhance conductivity in the following way: Clay minerals are electrically neutral and insulating in their bulk dry state. In this state excess charge that arises due to substitution of Al^{3+} for Si^{4+} in the clay mineral lattice is compensated by changes in the structure (Bailey, 1980). Addition of an electrolyte generates a negative charge within the clay surface. This charge attracts positively charged hydrated cations from the electrolyte. An electrically heterogeneous double layer is formed on the clay surface. Van Olphen (1977) showed that conduction can take place along this layer by cation migration. The extent to which a mineral can generate a sub-surface negative charge in a wet environment depends on its structure and controls the extent to which cations are exchanged with the electrolyte. Thus the surface density of cations available for conduction, and hence conductivity, depends upon intrinsic properties of the clay mineral. The capacity of a mineral to exchange cations is measured by the value of the

cation exchange per unit volume, Q_v , which can be measured empirically. While the value of this parameter is high in clays it can be quite low in some hydrated minerals such as chlorite and biotite (Jackson, 1984).

The aforementioned additional term to Archie's Law to account for such behaviour (c in E2.32) has been the subject of study of several authors. Waxman and Smits (1968) suggest that;

$$c = \phi^2 B Q_v \quad \text{E2.38.}$$

where B is the mobility of the cations in the electrical double layer and Q_v is the cation exchange per unit volume. Clavier et al (1977) propose;

$$c = \phi^2 d A \quad \text{E2.39.}$$

where d is the charge density and A is the available surface area.

Both of these relationships were derived for sandstones which failed to obey Archie's Law and are quite good at predicting their conductivities. The applicability of these equations to other rock types is less clear. Jackson (1984) has analysed the crack spectra of hard crystalline rocks with obvious conductivity enhancement and found that they contain higher proportions of low aspect ratio cracks such are often associated with grain boundaries. This type of crack has a large surface area and closes at relatively low pressures. If there were an hydrous or

altered clayey layer existing at the grain boundaries, as can often be the case due to alteration of plagioclase crystals for instance, then although the crack may close physically it may not do so electrically. The decrease of conductivity on increase of pressure would occur at a much more gradual rate than the physical closure of a pore conduction path would predict. This seems to explain the variation that is seen in the pressure dependence of basic rocks compared with more acidic rocks (figure F2.16) as well as giving a mechanism for enabling the overall basic rock conductivity to be higher.

Increasing the temperature of a system undergoing solely this type of conduction may be expected to produce an increase in the conductivity of the system due to greater cation mobility. This has not been experimentally checked but is expected to obey;

$$\sigma_{CT} = \sigma_{\infty} e^{(-E_{C\infty} / kT)} \quad \text{E2.40.}$$

in a similar manner to equation E2.33. where the subscript, c, denotes value for ionic double layer conduction (Horne, 1969; Madden and Marshall, 1959).

It must also be noted that if altered clay products fill all or a considerable proportion of the initial pore space then a drop in conductivity may be encountered due to the decrease in porosity and the closing of paths for pore fluid conduction (Madden and Marshall, 1959).

It has been suggested that additional hydroxyl ions can affect the

electrical conductivity of rocks (Parkhomenko, 1982).

There have been claims that the electrical conductivity of dry rocks may depend upon the chemical composition of their mineral assemblages (Parkhomenko, 1982; Lebedev and Shepel, 1981). Although any effect may be wholly overwhelmed in a saturated rock regime there seems to be some evidence (Jackson, 1984) to support the idea that minerals that contain the larger cations (Fe^{3+} , Fe^{2+} , K^+ and Na^+) are more likely to be more conducting than those that contain a greater proportion of the smaller cations (Si^{4+} , Ca^{2+} , Mg^{2+} and Al^{3+}). This may be explained by assuming the smaller cations to be more tightly bound than those with greater ionic radii.

2.12. VARIATIONS OF PORE PRESSURE, CONFINING PRESSURE AND TEMPERATURE IN THE CRUST

There are four major environmental factors that affect the measured electrical conductivity of rocks. These are;

- A. Confining pressure
- B. Pore pressure
- C. Temperature
- D. Saturation state

Any attempt to measure the electrical conductivity of rocks under simulated crustal conditions must control these parameters. It is also important that the variation of these parameters with depth is understood as much as possible. This section will deal with each parameter in turn describing what is known about the

variation of each parameter with depth and the various sources of error that may affect each judgement.

A. CONFINING PRESSURE, P_c is the pressure exerted on an amount of material at a depth, d , due to the action of gravity on a column of material of density, ρ above it. The confining pressure is given by the equation;

$$P_c = \rho g d \quad \text{E2.41.}$$

where g is the acceleration due to gravity (about 9.81 m s^{-1}). The pressure can be considered to be lithostatic on the scale of the samples used in this study (diameter 20 - 26 mm). The acceleration due to gravity and the density are, however, functions of depth. E2.41. now becomes;

$$(P_c)_d = \int_{x=0}^{x=d} (\rho(x)g(x)) \cdot dx \quad \text{E2.42.}$$

where x is the depth measured into the crust.

For estimations of the value of $(P_c)_d$ in the crust it is usual to take the acceleration due to gravity to be a constant and equal to that at the surface ($g(x) = g = 9.81 \text{ m s}^{-1}$). Average density of overburden strata, $\bar{\rho}_x$ is calculated and assumed to be constant throughout the crust ($\rho(x) = \bar{\rho}_x$). These approximations give a value for $(P_c)_d$ without the need to consider the complex way in which the rock densities and the acceleration due to gravity vary with depth. Equation E2.41 can be used to approximately predict the confining pressure at depth due to the

rocks.

Typical values for $(\partial P_c / \partial x)_d$ are around 0.03 GPa km^{-1} although this may be reduced to around $0.025 \text{ GPa km}^{-1}$ in a sialic terrain such as a granite batholith.

The uncertainty inherent in a calculation with such sweeping assumptions may be very great and will increase as the depth of burial increases. It has been estimated that the error in estimating the depth of burial simulated at 0.4 GPa may be as much as 10 percent (Evans, 1980). Extrapolation of this figure suggests that at around 1 GPa it may be as much as 25 percent.

B. THE PORE PRESSURE, P_p is usually considered to lie somewhere between the hydrostatic pressure, P_h , (the case where all pores can be envisaged as being connected to the surface) and the confining pressure, P_c , (the case where all the pores are isolated in the matrix). If this is the situation then;

$$P_h \leq P_p \leq P_c = \bar{\rho}_r g d \quad \text{E2.43.}$$

Now $P_h = \rho_p g d \quad \text{E2.44.}$

where ρ_p is the density of the pore fluid.

So $\rho_p g d \leq P_p \leq \bar{\rho}_r g d \quad \text{E2.43.}$

And as $\bar{\rho}_r \approx 3\rho_p \quad \text{E2.46.}$

Then it follows that;

$$P_c/3 \leq P_p \leq P_c \quad \text{E2.47.}$$

This equation seems to be applicable to stable areas of continental crust. There is some evidence to suggest that pore

pressure can exceed the confining pressure in tectonically active areas undergoing metamorphic processes producing water (Wyllie, 1971).

C. TEMPERATURE, T. Geothermal gradients are based upon down-hole determinations. Such measurements indicate large regional variations in geothermal gradients which can be correlated to the type of tectonic province in which the measurements were taken (Roy et al, 1972).

The geothermal heat-flow through a crustal layer depends on the trans-basal heat-flow and the intra-layer heat production. This is summed up in an empirical relationship between surface heat-flow, Q , and the radiogenic heat production of the surface crustal rocks, A_0 ;

$$Q = q_0 + D A_0 \quad \text{E2.48.}$$

where q_0 is the so called reduced heat-flow below a certain layer in which local variations of the concentrations of radio-isotopes produce variations in the surface heat-flow. Reduced heat-flow is presumed to be constant in large regions which have undergone similar tectonic evolution. Such regions have been called tectonic provinces (Roy et al, 1972). The parameter D has dimensions of length. The term $D A_0$ characterises the vertical distribution of the heat sources. Two models for the variation of the $D A_0$ term are usually quoted. It is beyond the remit of this work to examine them here but they can be found in Roy et al (1968) and Lachenbruch (1968).

Roy et al (1972) categorised tectonic provinces on the basis of heat-flow measurements. The examination of such provinces in the U.S.A. shows three broad categories of continental crust. These are shown in table T2.6, each province being represented by a typical area.

TABLE T2.6 THE RELATIONSHIP OF GEOTHERMAL GRADIENT TO TECTONIC PROVINCE (after Roy et al (1972)).		
TECTONIC PROVINCE	GEOTHERMAL GRADIENT (°C km ⁻¹)	
Sierra Nevada	10.5	LOW
Eastern United States	15.3	MEDIUM
Basin and Range	25.2	HIGH

Figure F2.22. and table T2.6. show geothermal gradients for different European tectonic units (Cermak, 1982a; Cermak and Lastovickova, 1987). In this study the Lachenbruch model (Lachenbruch, 1968) has been used with values of $D = 10\text{ km}$ and $A_0 = 2\text{ }\mu\text{W m}^{-3}$. The value of D is the mean of values of D for all tectonic provinces the world over. D varies between 4 km and 16 km (Morgan and Sass, 1984).

Errors arise in the determination of geothermal gradients from the limited penetration of the boreholes in which temperature measurements are made. The boreholes, usually less than 1 km

deep, suffer from long wavelength perturbations due to long period surface temperature changes. These perturbations must be filtered out. Errors can also arise from other sources such as from borehole convection. There has been a categorisation of the reliability of surface heat-flow in the U.K. (Rollin, 1982; 1983) a full description of which can be found in a report by Glover (1984). A considerable error in the geothermal gradient can be produced when the data is extrapolated to depths of around 10 -

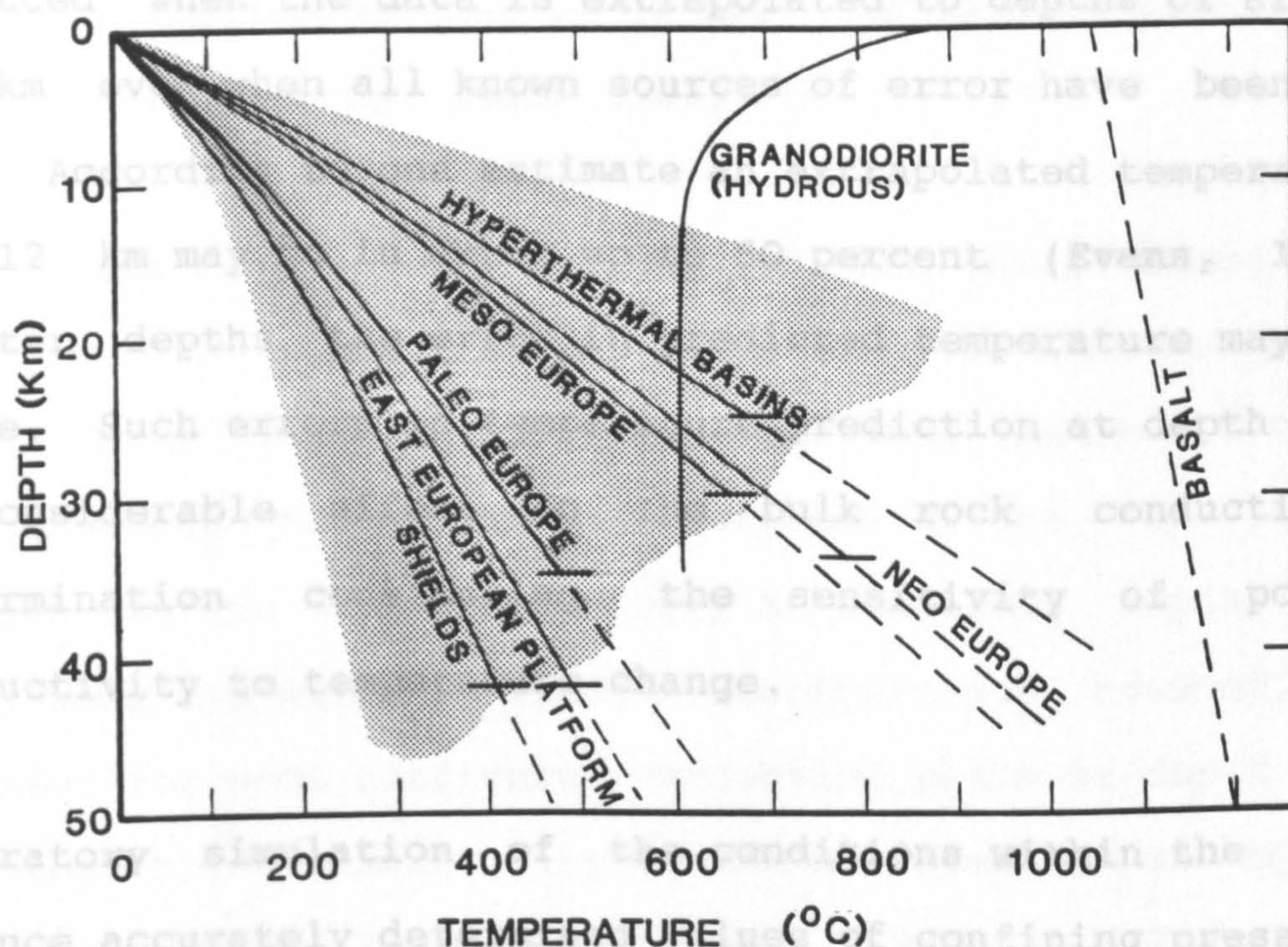


FIGURE F2.22. One dimensional steady state temperature profiles for specific European tectonic units (After Cermak and Lastovickova, 1987).

D. THE SATURATION STATE of crustal rocks is probably the least understood parameter of the four. There has been much debate as to whether rocks in the deep crust are associated with an aqueous phase (Gough, 1986; Jones, 1987; Yardley, 1986). Initially it

deep, suffer from long wavelength perturbations due to long period surface temperature changes. These perturbations must be filtered out. Errors can also arise from other sources such as from borehole convection. There has been a categorisation of the reliability of surface heat-flow in the U.K. (Rollin, 1982; 1983) a full description of which can be found in a report by Glover (1984). A considerable error in the geothermal gradient can be produced when the data is extrapolated to depths of around 10 - 20 km even when all known sources of error have been adjusted for. According to one estimate an extrapolated temperature value at 12 km may be in error up to 50 percent (Evans, 1980). At greater depths the error in predicted temperature may be very large. Such errors in temperature prediction at depth could have a considerable effect on the bulk rock conductivity/depth determination considering the sensitivity of pore fluid conductivity to temperature change.

Laboratory simulation of the conditions within the earth can produce accurately determined values of confining pressure, pore pressure and temperature. These values cannot at present be accurately translated into a depth of burial although some idea may be had of the rough position in the crust that particular values of confining pressure, pore pressure and temperature correspond to.

D. THE SATURATION STATE of crustal rocks is probably the least understood parameter of the four. There has been much debate as to whether rocks in the deep crust are associated with an aqueous phase (Gough, 1986; Jones, 1987; Yardley, 1986). Initially it

may be thought that at lower crustal depths the porosity of the rocks would be reduced so much as to make the presence of aqueous pore fluid unlikely (Dvorak, 1975; Keller, Anderson and Prichard, 1966). Recent works have shown the presence of large scale thrusting at depths which may afford a path for water to percolate into the lower crust (Beamish and Smythe, 1986). The world's deepest borehole on the Kola Peninsula reported free water circulation as deep as 12 km, its deepest point, before it collapsed (Kozlovsky, 1984; 1987).

Geochemists are of the opinion that water cannot exist at depth in association with the types of rocks that are thought to occur there due to rehydration reactions that would take place and result in retrograde metamorphism of the rock to a form thought to be more common only at shallower depths (Yardley, 1986; Wood and Walther, 1986). Geophysicists, however, point out the need for some continuous conducting phase at depth to account for the high conductivity values measured there by magnetotelluric methods (Ingham and Hutton, 1982a; 1982b; Jones, 1987; Gough, 1986, Beamish, 1986a; 1986b). This problem will be dealt with in more detail in chapter 3.

2.13. MEASUREMENT TECHNIQUES

The electrical properties of rocks and minerals have been studied under a wide range of physical conditions. High confining pressures, pore pressures, temperatures and saturation states have been used, but never have all these parameters been

extended to their limits together. This has been due to the constraints composed by the current measurement techniques and the properties of the materials from which the measurement cell needs to be constructed.

Pressures of up to 4.0 GPa have been obtained on cold, dry samples (Parkhomenko, 1967). Temperatures up to 1500 °C have been obtained by Gupta and Sharma (1978) and various states of saturation were used by Drury and Hyndman (1979). The use of pore pressure has only been employed by a few workers at the University of East Anglia (Lee, 1977; Jackson, 1984).

There are several basic measurement techniques that have been used which I will briefly review here.

The samples that have been used have been in the form of cylinders, disks and cubes, the former two being more common. Thickness of the sample varies between a few millimeters and a few tens of millimeters. This thickness is chosen to be sufficiently large to remove any errors due to grain size inhomogeneities.

Measurement has used direct current and low frequency (up to a few kilohertz) current techniques. Direct current measurements use both four and two electrode arrangements. The design of such a D.C. system is such that electrode/sample contact resistance is minimised. Other measurement problems include electrode polarisation and surface conduction. Polarisation is a particularly bad problem when dealing with D.C. measurements of

saturated rocks. In this case a four terminal electrode arrangement is usually used. The use of alternating current can remove much of the problems of electrode polarisation although surface conduction is still a problem (Ohloeft, 1982; Parkhomenko, 1967).

The largest source of error results from stray currents flowing over the surface of the sample. These can be reduced by the use of a guard-ring (Field, 1954; Cemic and Jansen, 1975; Will et al, 1979). Such a method has been used in this work due to the unusually conducting boundary conditions present in the measuring cell. Amey and Hamburger (1949) have studied the guard-ring arrangement and produced expressions that predict the efficiency of the guard-ring for various geometries. If the guard-ring is to be effective the geometry of the sample and electrodes needs to be carefully considered. Saturated samples also require jacketing which aggravates surface conduction. Good sample jacketing, electrode holder and seal design can overcome this problem at low temperatures. At high temperatures problems arise due to the non-availability of an electrically insulating, flexible, refractory jacketing material. This is one of the problems that this work has been concerned with.

Some comparisons have been made between direct current and low frequency alternating current measurements (Ohloeft, 1976; Lee, 1977). It seems that there is some correspondence between D.C. and 1 kHz A.C. measurements for high conductivity rocks. This may not be a particularly safe presumption on which to base a direct unconditional comparison of data. Table T2.7. shows a

comparison of some measurement methods (Parkhomenko, 1967).

TABLE T2.7. COMPARISON OF MEASUREMENT METHODS (after Parkhomenko (1967))			
ROCK TYPE	CONDUCTIVITY ($S\ m^{-1}$)		
	D.C. 4 Terminal	D.C. 2 Terminal	A.C. 1000 Hz
Dolomite	130	190	190
"	160	170	182
"	117	125	142
Sandstone	9	16	16
Arkose Sandstone	435	714	720

The practicalities of measurement cell design will be expanded upon on in Chapter 6 which is concerned with the design of a high temperature, high pressure cell for saturated rocks.

CHAPTER 3 : THE LOWER CONTINENTAL CRUST

3.1. INTRODUCTION

Discussion about the nature of the Earth's crust has been especially animated since the general acceptance of the theory of plate tectonics. A good understanding of the precise composition and structure of the crust and upper mantle was seen to be important in the confirmation and development of this theory. Although plate tectonic theory has now gained widespread acceptance, the understanding of the mid- and lower crustal environment has not developed in parallel with it. Observations and measurements on the upper crust are relatively easy whereas the important information about the structure and composition of the lower crust is not easily or directly attainable. This is because for the lower crust we lack the large scale natural sampling processes, such as the production of clastic sediments, which have helped simplify the analysis of the upper crust. This is unfortunate as the understanding of the lower crust is central to the understanding of the compositional and tectonic evolution of the continents.

There have been three main methods that the geophysicist and geologist have been using to extract more information from the crust:

1. Geophysical field measurements such as refraction seismology, deep reflection seismic and magneto-telluric measurements. These techniques can be used to obtain average values of the physical properties of the intra-crustal areas as well as

information about the location of transitions between these areas.

2. Study of the few tectonically exposed sections of lower crustal rocks. The composition and structural history of these zones can be of very great value in helping to define the mineralogy and deformational processes at depth.
3. Study of xenolith mineralogy. Mineralogical analysis of xenoliths found in kimberlite and nephelinite diatremes has produced a xenolithic stratigraphic column of the crust and part of the upper mantle.

This review chapter aims to report some of the data, information, interpretations and speculations concerning the lower continental crust. The review will be roughly structured around the three main areas of study as listed above.

3.2. CONTINENTAL CRUST MODELS

Unlike the upper continental crust the lower continental crust cannot be studied directly other than in a few occurrences where exposures of lower crustal material are believed to exist. This implies that the deep structure of the earth's continental crust must be deduced indirectly by using geophysical techniques and by making geochemical inferences.

The nature and composition of the whole crust must be established before any discussion of the lower crust can take place. It is a reasonable assumption that the continental crust must be derived in some way from mantle material by some long timescale geological process or processes. In the past this has been assumed to be the process of calc-alkaline volcanism typical of areas of orogenesis (Taylor, 1977). This source produces a sufficient volume of siliceous material and has the advantage of being presently observable. The idea that the continental crust is derived from andesitic volcanism in this way gave the theory its name; the andesite model. Many recent models of crustal evolution can be regarded as variations on the andesite theme (e.g. underplating by intrusion of tonalites (Weaver and Tarney, 1984)). The andesitic material produced by such volcanism has been used as a basis for a total crustal composition (Taylor, 1977). It seems, however, that at least three quarters of the continental crust was emplaced before the end of the Archean, and since volcanic activity in the Archean seems not to have produced much in the way of andesitic material another source of crustal material needs to be found. The assumption that the whole crust has been accreted by andesitic volcanism also generates values of Th/U and Ni/Cr which are not consistent with those observed (Gill, 1981). It seems that the andesite model may be the process by which crustal accretion has taken place since the Proterozoic.

The Archean crust is dominated by a bimodal basic-felsic suite of igneous rocks. These consist of basalts similar to MORBs (Mid

Ocean Ridge Basalts) and sodium rich felsic rocks (Taylor and McLennan, 1985). The ratio of basaltic to felsic rocks is around 2:1. It is possible that this is the sort of composition that makes up 75% of the continental crust. Such a distinction from the andesite model as this may have important implications for the lower crust. The basic bias of the composition of the lower crust from xenolith evidence (Arculus, 1984) seems to support this model of Archean crustal accretion.

There have been three main constraints imposed on the choice of the mechanism for crustal accretion. These are that;

- The derived crust has sufficient K, U, Th in the upper crust to produce the observed heat flow.
- The bulk crust composition must be capable of generating the upper crust by partial melting.
- Contributions to the crust by andesitic vulcanism must be restricted to the Post-Archean.

One such composition assumes 25% andesitic crust and 75% Archean crust and is given as figure F3.2. (Taylor and McLennan, 1985).

The internal structure of the continental crust has followed a simple thesis. The model which most people have accepted as being the best general description of the earth's continental crust is a simple one based upon deep seismic refraction studies. The model consists of a two layer crust overlying the mantle. The topmost layer is a 15-20 km thick 'Granitic' layer. This lies on top of a 15-20 km thick 'Basaltic' layer. The division between the two is called the Conrad Discontinuity. The base of

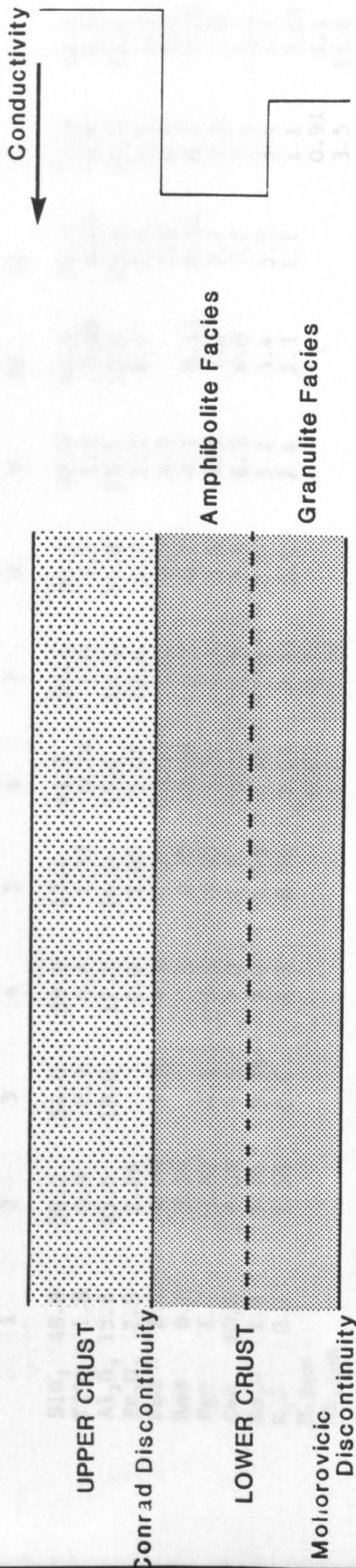


FIGURE F3.1.1. The general features of the two/three layered continental crust.

Fig. F3.2a. CRUSTAL COMPOSITIONS FROM SEVERAL AUTHORS

LOWER CRUST			LOWER CRUST PETROLOGICAL MODELS					WHOLE CRUST			UPPER CRUST	
IGNEOUS LAYER												
1	2	3	4	5	6	7	8	9	10	11	12	13
SiO ₂	48.8	58.2	59.0	57.4	61.2	54.4	57.1	60.0	62.5	52.4	57.3	62.5
TiO ₂	1.8	0.9	0.8	1.37	0.54	1.0	1.3	1.1	0.68	0.65	0.9	0.57
Al ₂ O ₃	15.6	15.5	17.1	16.0	15.6	16.1	15.2	15.1	15.6	15.8	15.9	15.8
Fe ₂ O ₃	2.8	2.86	2.0	2.05	3.07	10.6	2.3	2.3	6.1	2.6	9.1	1.2
FeO	8.2	4.78	4.1	7.3	2.57	0.2	5.7	4.9		5.6	0.1	3.4
MnO	0.2	0.19	0.1	0.15	0.08	0.2	0.2	0.1	0.13	0.14	0.1	0.09
MgO	8.7	3.85	3.4	5.65	3.36	6.3	5.6	4.5	3.2	7.7	5.3	2.2
CaO	10.8	6.05	7.0	7.1	5.57	8.5	7.5	6.3	6.0	7.7	7.4	3.3
Na ₂ O	2.3	3.10	3.9	1.95	4.42	2.8	3.0	3.2	3.4	3.5	3.1	3.7
K ₂ O	0.7	2.58	1.9	0.93	1.03	0.34	2.0	2.4	2.3	1.1	1.1	3.23
U, ppm					0.05	0.28					0.91	2.1
Th, ppm					1.0	1.06					3.5	13.0

The samples are as follows: 1. POLDERVAART, 1955; 2. RONOY AND YAROSHEVSKY, 1969; 3. TAYLOR, 1977; 4. SMITHSON, 1978; 5. MENHERT, 1975 (IVREA ZONE ROCKS); 6. SHERATON et al, 1973; 7. TAYLOR AND MCLENNAN, 1985; 8. PAKISSER AND ROBINSON, 1966; 9. HOLLAND AND LAMBERT, 1972; 10. McGETCHIN AND SILVER, 1972; 11. TAYLOR AND MCLENNAN, 1985; 13. EADE AND FAHRIG, 1968.

Fig. F3.2b. SEISMIC VELOCITIES IN VARIOUS ROCKS AND MINERALS (AFTER KAY AND KAY, 1981)

MEDIUM	VELOCITY km/s	DENSITY g/cm ³	VELOCITY km/s	VELOCITY(1kbar) ¹ km/s	VELOCITY(6kbar) ¹ km/s
<u>ANHYDROUS MINERALS</u>					
MICROCLINE	6.01	2.56			
QUARTZ	6.05	2.65			
PLAGIOCLASE					
An 9	6.07				
An29	6.35	2.61-2.76			
An55	6.62				
An70	6.75				
MAGNETITE	7.40	5.18			
DIOPSIDE(Cpx)	7.70	3.31			
BRONZITE(OpX)	7.84	3.34			
GARNET	8.53	4.16			
OLIVINE	8.43	3.38			
<u>HYDROUS MINERALS</u>					
SERPENTINE	5.10	2.6			
BIOTITE	5.26	3.05			
HORNBLende	7.04	3.15			
<u>ROCKS</u>					
PYROXENE HORNBLende GNEISS ²	6.49		6.49+2%		6.69+2%
PYROXENE GNEISS ³	6.81		6.82+2%		7.01+1%
GARNET PYROXENE GRANULITE ⁴	7.21		6.68+5%		7.40+3%
GABBRO(ECLOGITE) ⁵	(8.4)		6.9-7.1		6.9-7.1
DIORITE(GARNET GRANULITE) ⁶	(7.3)		6.6		6.6
GRANODIORITE(GARNET GRANULITE) ⁷	(7.2)		6.4		6.4
GRANITE ⁸	6.05				

¹ Calculated from mineral data at 1 and 6 kbar. The uncertainties are due to measured anisotropy in the rocks (data from Christensen and Fountain, 1975).

^{2,3,4,5} Christensen and Fountain, 1975. Value in parentheses from Ringwood, 1975.

^{6,7} Ringwood, 1975. Low pressure mineralogy values in parentheses.

⁸ Pakisser and Robinson, 1966.

the 'Basaltic' layer and the peridotite of the upper mantle are separated by a transition called the Mohorovicic Discontinuity (or MOHO).

The simple layer model suffers from considerable problems as a result of its elegant but unrealistic simplicity. The main problems can be summed up as four main points:

1. The mid-crustal Conrad discontinuity is not found in many seismic refraction surveys.
2. Seismic refraction surveys have discovered other intra-crustal discontinuities of a limited regional extent. An example of such discontinuities are the so-called F and G discontinuities found at a depth of about 10 km in certain locations in the USSR (Polshkov et al, 1973; Liebscher, 1964 a,b).
3. Lateral interruptions on a scale of 10-100 km exist in the Conrad and Mohorovicic discontinuities. Even where the Conrad discontinuity and Moho are well defined seismic reflection surveys show that lateral discontinuities on a scale of 10-100 km exist. This is the scale that characterises the seismic refraction method. The lateral discontinuity may imply gradational behaviour, lamination, quantisation (piecewise discontinuity) or extreme

folding in the transition zones of these discontinuities (Davydova, 1975; Smithson, 1978; Finlayson et al, 1979).

4. The layers from which the model is constructed are defined by seismic compressional wave velocities by most researchers. The mineralogy and types of rock found in these 'separate' layers is not unique. Therefore it should be borne in mind that although the layers are called 'Granitic' and 'Basaltic' the terms should not be seen as a description of the type of rock of which the layer is made but only as a label.

The geochemistry of the lower crust will be dealt with in section 3.3. It is sufficient to say here that whatever the bulk composition of the crust it should be such that an extraction of an upper crustal composition from it should leave a residue that is a possible lower crustal composition.

Despite its problems the simple two layer model is a useful general guide to the structure of the continental crust providing it is borne in mind that:

- A. It is probably only valid on a very large scale.
- B. It can be misleading as a guide to the detailed structure and composition of the crust.
- C. It is probably better to treat the upper/lower

crustal and the lower crust/mantle transitions as areas of smooth gradational change occurring over a set distance and cut through by oblique faults as has been reported by Cook et al (1979) and Smithson (1979).

Weaver and Tarney (1984) have suggested a three layer model consisting of:

1. 12 km of upper crust
2. 6 km of mid- crust of Lewisian type amphibolite grade rocks.
3. 18 km of lower crust of Lewisian type granulite facies rocks.

This model divides the lower crust into two subdivisions and is probably more applicable to the description of the lower crust in terms of its electrical conductivity than the two layer crust. It has two major problems. It overestimates the concentrations of K, U and Th and it generates a lower crust too rich in silica.

Figure F3.1. has been included to show the general features of the layered crust model.

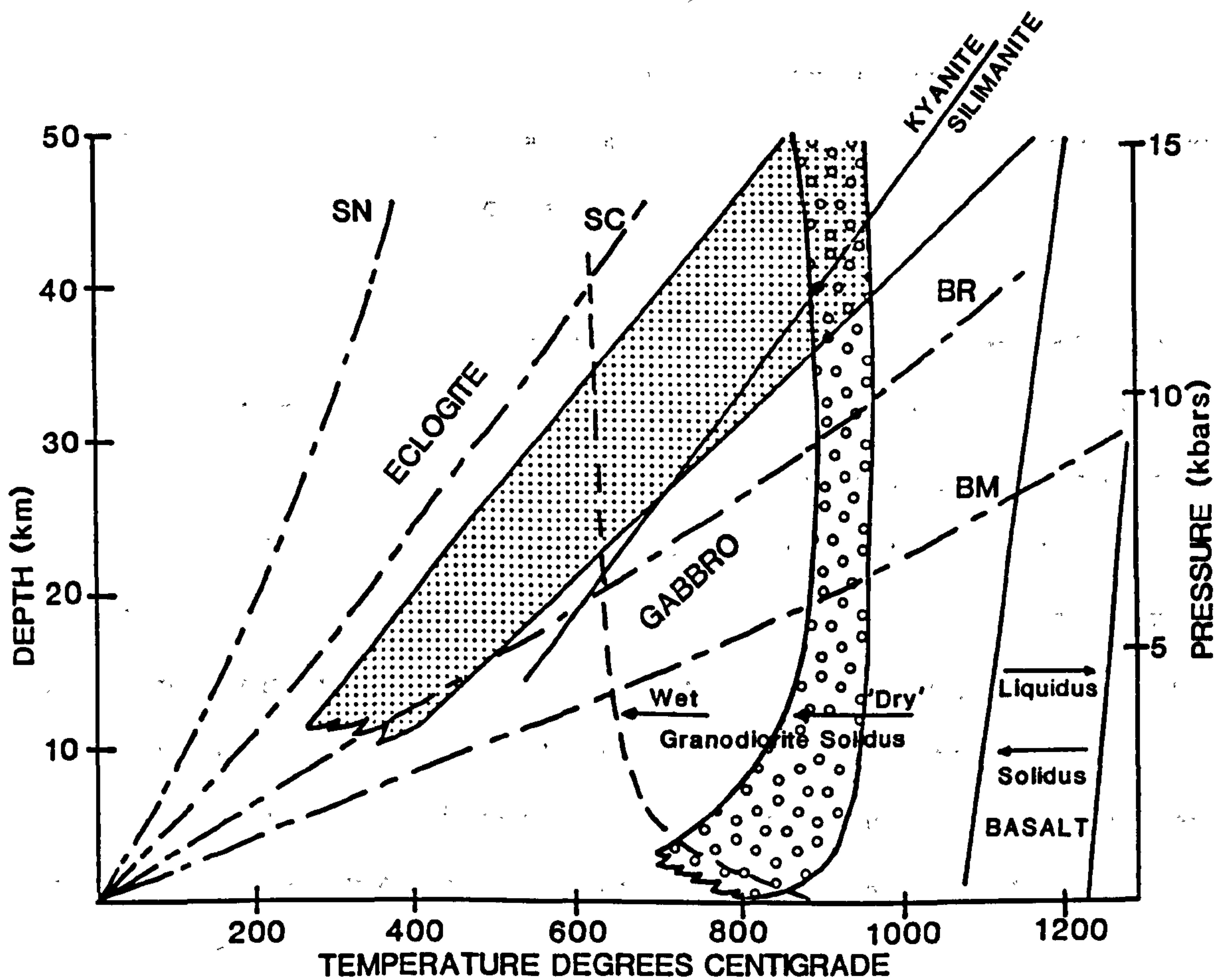
3.3. GEOCHEMISTRY AND THE TWO LAYER MODEL

The simple two layer model has influenced much of the geochemical theory of the lower continental crust. Ever since Poldervaart's petrological work (Poldervaart, 1955) the lower crust or 'Basaltic' layer has been believed to be composed of a rock of a gabbroic composition. Whatever the composition it must be attainable by considering it to be derived from the extraction of the upper crustal composition from the bulk crust composition discussed in section 3.2. The residue from such a process is thought to be of a tholeiitic composition (Taylor and McLennan, 1985).

The compressional P-wave seismic velocities of the bottom 10 km or so of the crust that are observed from seismic refraction studies agree well with laboratory measurements of P-wave velocities in basalt composition rocks of gabbroic mineralogy the laboratory measurements being carried out while the rock samples are subjected to lower crustal conditions of temperature and pressure. The values of P-wave velocities obtained have been in the range $6.9 - 7.1 \text{ km s}^{-1}$ (Christensen and Salisbury, 1975). P-wave velocities are shown in figure F3.2.

According to Ringwood (1975) there is probably an abundance of metamorphic rocks in the lower crust. Figure F3.3. shows the schematic stability ranges for rocks with tholeiitic basalt composition and reference geothermal gradients and figure F3.2. shows the P-wave velocities of different rocks and crust composition models from various studies. It can be seen

FIGURE F3.3. Schematic stability ranges for eclogite, garnet granulite and gabbro mineralogies of rocks with tholeiitic basalt composition shown in relation to the extrapolated geothermal gradients for regions of differing heat flow within the U.K. Other ranges and horizons are also shown. (After Kay and Kay, 1981).



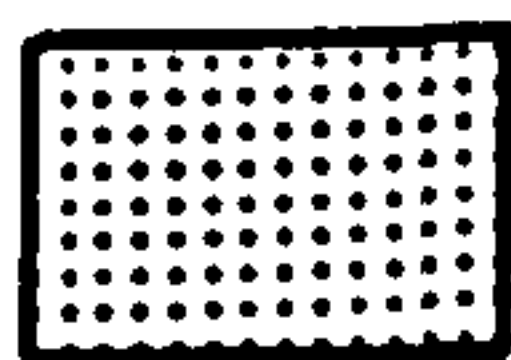
----- GEOTHERMAL GRADIENTS

SN Sierra Nevada (0.6 HFU)

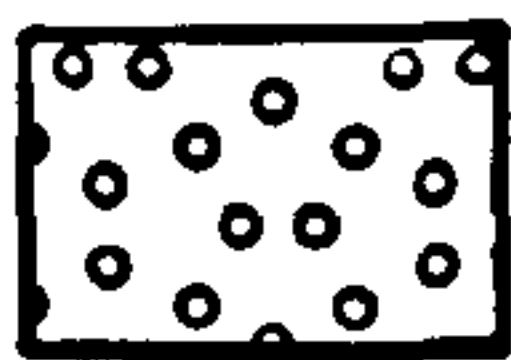
SC Stable Craton (1.3 HFU)

BR Basin and Range (2.1 HFU)

BM Battle Mountain High (3.0 HFU)



GARNET GRANULITE



AMPHIBOLE UPPER STABILITY RANGE (Schematic)

that the predicted mineral assemblages for lower crustal basaltic rocks will be garnet granulites and eclogites. These have higher P-wave velocities than gabbros. If lower crustal rocks do contain garnet granulites and eclogites then there must also be present a higher proportion of less dense rocks with lower P-wave velocities such as quartz and alkali feldspars to enable the overall P-wave profile of the crust to match that found by seismic field studies. Other possible accessory minerals apart from quartz and alkali feldspars might be plagioclase and hydrous mafic phases such as amphiboles and serpentines.

The biggest problem encountered when mineral composition is varied to obtain a seismic velocity profile match is the non-uniqueness of the solution. The set of compositional solutions of seismic velocity profile matching is infinitely large due to the number of possible minerals involved, each of which can be present in varying amounts. Extra information from some tectonically exposed sections of the lower crust and from mineralogical analysis of certain xenolith populations can help greatly to exclude abundances of certain minerals. This will be dealt with in greater detail in section 3.5.2.

Volcanic arc models of crustal evolution imply that the mean composition of the crust is andesitic (Taylor, 1977). By assuming the initial composition of the crust was andesitic and using the known composition of the upper crust Taylor produced a different lower crust composition. This can be seen in figure F3.2. It must be remembered however that the rate of formation and composition of new mantle derived crust has probably varied

over geological time and this may invalidate the Taylor method to a certain extent (Armstrong, 1981; Karig and Kay, 1981). The Taylor model is also totally dependent on the initial crustal composition which is debateable but does allow for regional variations which are needed to match the lower crustal composition to the regionally variable observed seismic P-wave crustal velocity profiles. More recent work, alluded to in section 3.2. (Taylor and McLennan, 1985), has derived a composition from a protocrust that is a mixture of andesitic and Archean. While this has all the advantages of the earlier Taylor composition it is more likely to take into account the changing type of vulcanism over geological time and hence be a more accurate composition of the lower crust.

It may be possible to get a direct estimate of the lower crustal composition from the proportion of rock types in exposed sections of possible lower crustal material. Areas of possible lower crustal exposure are well known but not necessarily representative of the lower crust as a whole. For this reason assumptions that are based upon data from these regions should be treated with circumspection. One area that is thought to be a complete cross-section of the lower continental crust is the Ivrea-Verbano Zone in northern Italy. The abundance of metasedimentary rocks, amphibolites and anorthosites in tectonically exposed sections through the lower crust and also in some xenolith populations (Padovani and Simmons, 1980; Fountain and Salisbury, 1981; Brodie and Rutter, 1987) should be treated as direct evidence for their presence in non-trivial amounts in the lower crust. This is important to bear in mind as estimates

of the lower crust composition from other methods favour eclogite and garnet granulite mineralogy. It is also important to remember that areas such as the Ivrea Zone have often had very complex histories that may devalue the data that they provide. In some rocks from lower crustal exposures there is a depletion of Th and U, some appear to have undergone granitic melt extraction while others show no obvious change in composition. Overall anhydrous mineralogy typifies the granulite facies rocks that are found in the 'lowermost' parts of most exposures. It seems that major compositional changes occur in the amphibolite facies before the onset of granulite grade metamorphism. Such reactions can include the formation and removal of granitic melts, dehydration reactions and the breakdown of micas and hornblende.

One constraint on the composition of the lower crust is the concentrations of U, Th, Rb and Cs. The upper crust must be enriched in these elements to account for the observed heat flow. Fyfe (1973) has pointed out the importance of the removal of water and granitic melts during granulite facies metamorphism. Extraction of water and granitic melts provides a mechanism for lower crustal depletion and upper crustal concentration of U, Th, Rb and Cs (Heier, 1973).

The dehydration that takes place during granulite facies metamorphism has also been invoked to explain the apparent mid-crustal high conductivity layer. Dehydration reactions start occurring at temperatures greater than 400 °C (Haak and Hutton, 1986; Spear and Selverstone, 1983; Walther and Orville, 1982;

Ringwood, 1975). If rocks are still sufficiently elastic at these temperatures to remain porous to any extent, as seems to be the case (Tullis and Yund, 1977; Brace, 1972; 1980), then the water released during burial or prograde metamorphism may be able to form a fully interconnected high conductivity pore fluid pathway. This is especially the case if the released water is prevented from rising too far through the crust by a zone of impermeable minerals (Etheridge et al, 1983). If such impermeable layers exist they would also prevent the redistribution of radioactive elements by the water as described by Heier (1973).

Estimates of lower crust composition based upon assuming certain minerals exist in equilibrium have been investigated by testing the stability of mineral mixtures under high pressure and temperature conditions. Initially a study by Kennedy into the gabbro-eclogite phase transformation showed that granulite-facies metamorphism characterised mineral assemblages which were stable up to the eclogite stability field and should be stable under lower crustal conditions (Kennedy, 1959). This has been confirmed by other studies (Yoder and Tilley, 1962; Cohen et al, 1967; Ito and Kennedy, 1970, 1971; and Kennedy and Ito, 1972). Work by Green and Ringwood has suggested that under anhydrous conditions eclogite and possibly garnet granulite are the stable phases of basaltic composition in the lower crust (Green and Ringwood, 1967a, 1967b; 1972).

Differences between observed crustal velocity profiles taken from different areas can be explained by a mainly basaltic lower

crust and a silicic upper crust by varying the composition of the crust from region to region as shown by Pakisser and Robinson (Pakisser and Robinson, 1976).

An example of varying the crust composition to match the observed physical properties of the lower crust is given by Jones (Jones, 1981). Jones has taken the varying crustal velocity and conductivity profiles that occur in Pre-Cambrian shield areas and classified them into three sets depending upon the observed dominant characteristics of the lower crust in those areas.

The Jones classification is;

A/ Type I - 'Normal'

V_P	6.55 - 6.85 km s ⁻¹
V_S	3.80 - 4.00 km s ⁻¹
Poisson's Ratio	0.241 - 0.252
Resistivity	10 ³ - 10 ⁴ ohm m
Surface Heat Flow	25 - 39 mW m ⁻²

B/ Type II - 'Intermediate'

V_P	6.70 - 7.20 km s ⁻¹
V_S	4.00 - 4.11 km s ⁻¹
Poisson's Ratio	0.25
Resistivity	100 - 300 ohm m

C/ Type III - 'Low'

V_P	6.8 km s ⁻¹
V_S	3.40 - 3.70 km s ⁻¹
Poisson's Ratio	> 3.00
Resistivity	10 - 50 ohm m

Where 'normal', 'intermediate' and 'low' refer to the resistivities of each area.

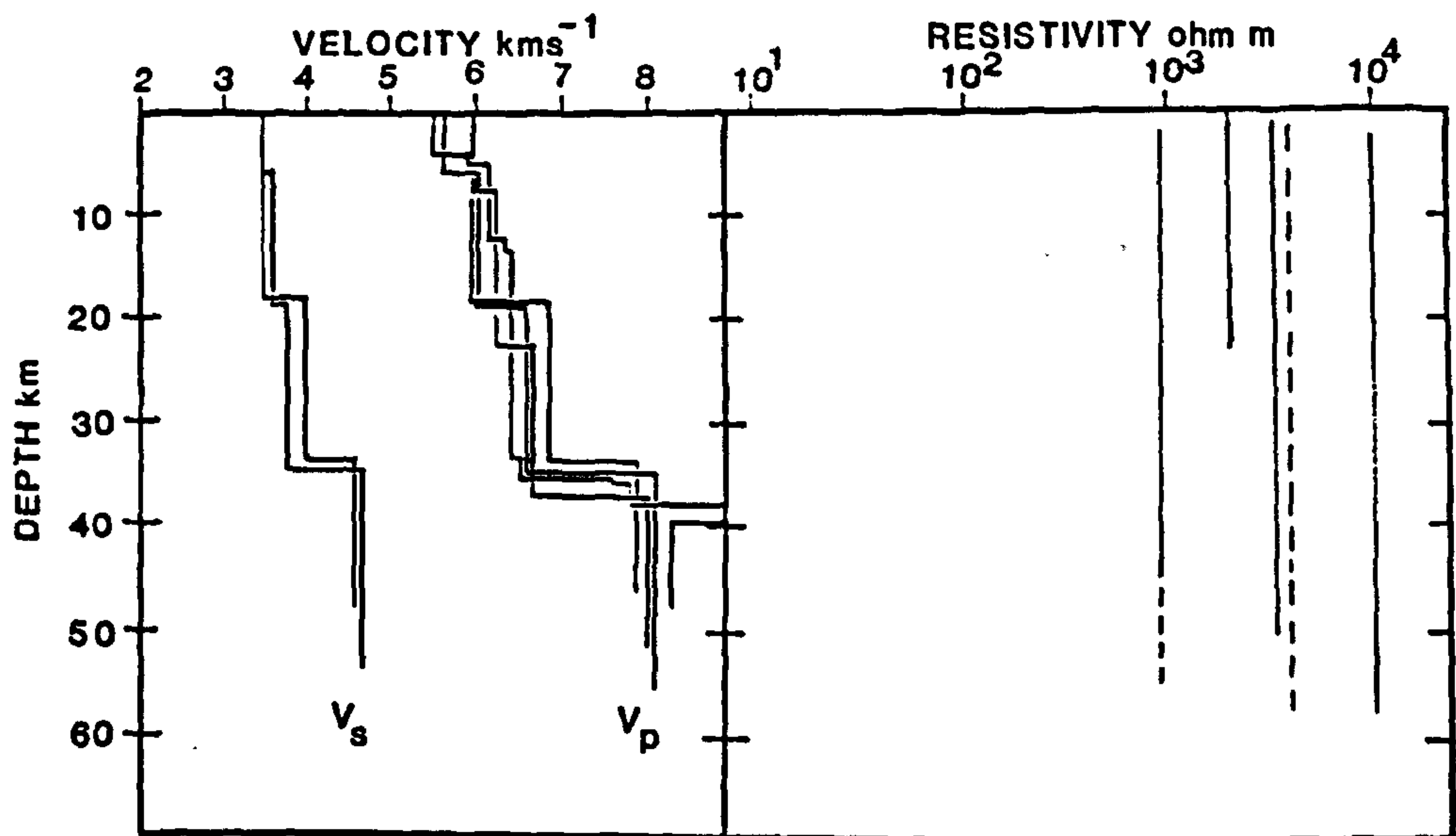
The variation of velocity and conductivity profiles between these

classes and within each class can be seen in figure F3.4. Jones has ascribed a possible rock type to each of these lower crustal layer classes indicating in each case the type of hydration characteristics which would produce the observed values of velocity and conductivity. These are;

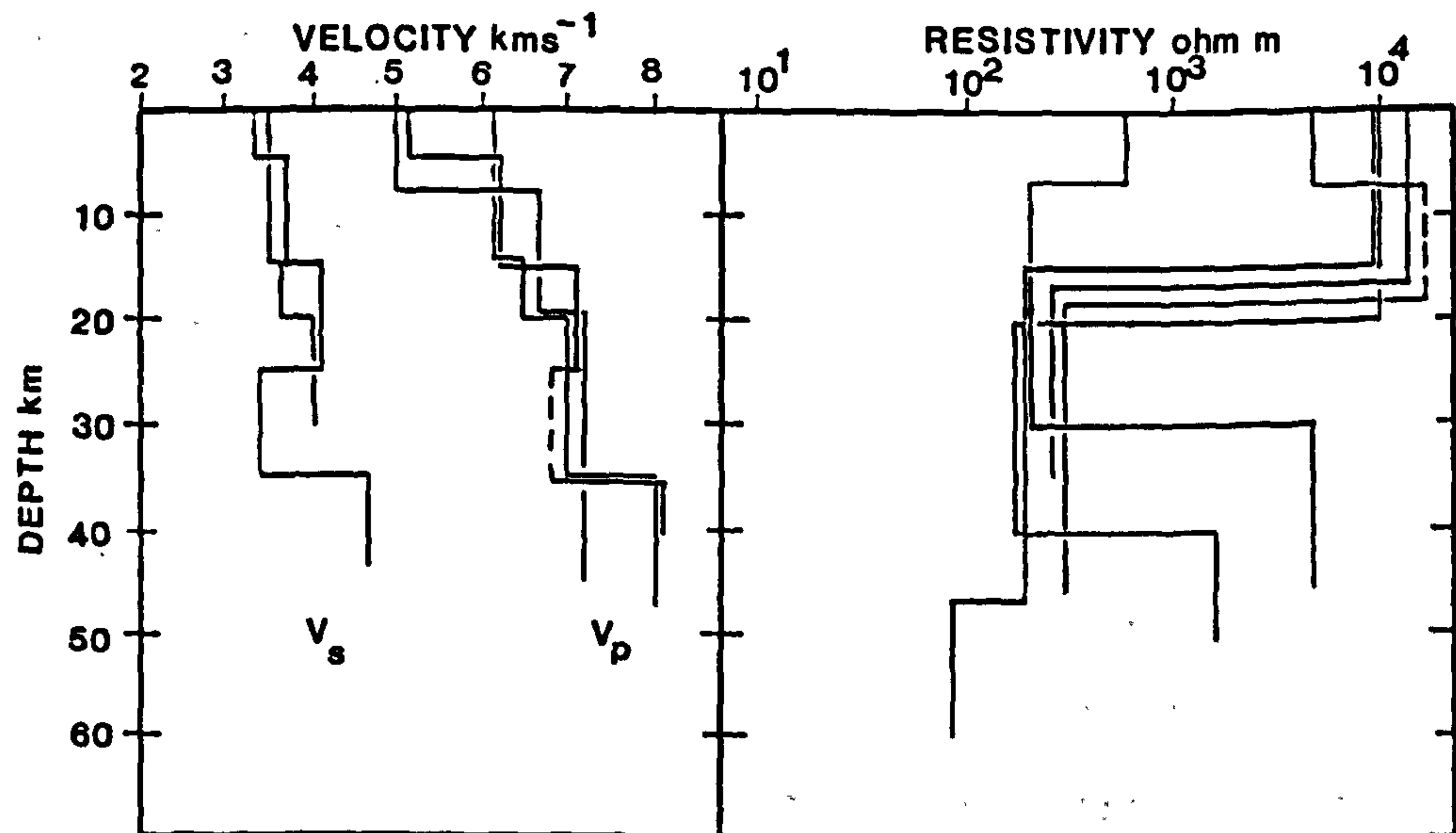
A/	TYPE I	Anhydrous	Quartz Dolerite
B/	TYPE II	Hydrous	Amphibolite
C/	TYPE III	Hydrous	Serpentinite

The lower crust is not a structural or compositional entity. The average composition of the crust may suggest a two layer model but on a more regional level the actual composition may vary greatly. In reality and in specific localities more complicated tectonomagmatic models may well best describe the true situation. The structure of each area must be judged on its own merits. An attempt at quantifying the relationships between different petrologies and the history of the crust in one particular area (Ivrea Zone) may be found in a paper by Brodie and Rutter (1987).

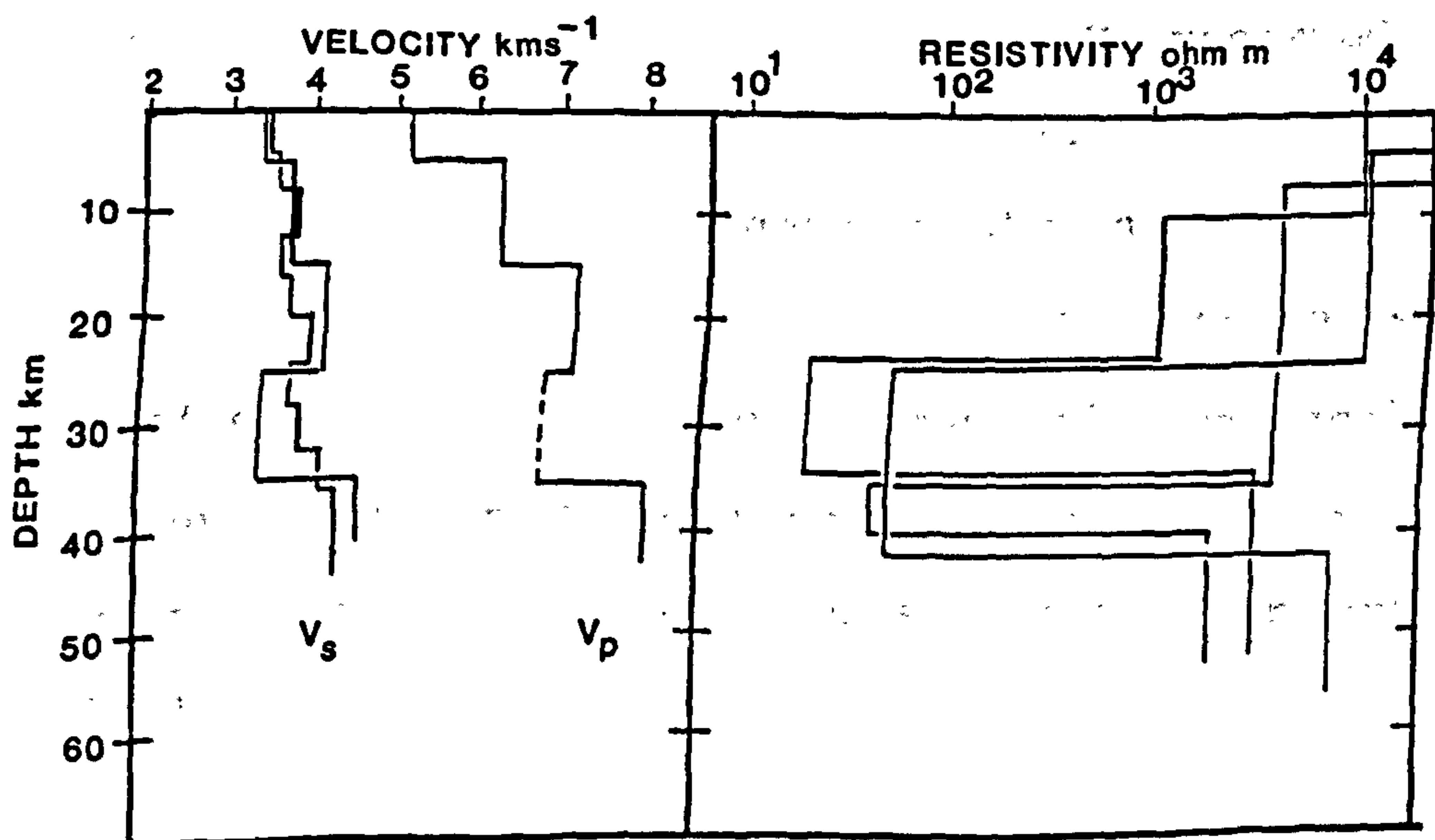
Knowledge about the mineralogy and composition is not complete. The general consensus amongst geochemists is that the lower parts of the crust are dominated by an anhydrous granulite facies mineralogy of tholeiitic composition and that this underlies an upper part of the lower crust consisting of an hydrous amphibolite facies rock of tholeiitic composition. Taylor and McLennan (1985) have discussed the problem in detail and arrive at a lower crustal composition by extracting a 25% granodiorite composition from a total crust composition based on the andesite and archean-bimodal crustal accretion models.



TYPE I LOWER CRUSTAL LAYERS



TYPE II LOWER CRUSTAL LAYERS



TYPE III LOWER CRUSTAL LAYERS

FIGURE F3.4. Seismic and resistivity profiles over areas classified into type I, II and III by Jones (1981). The dashed lines indicate minimum values. (After Jones, 1981)

3.4. GEOPHYSICAL EVIDENCE

3.4.1. SEISMIC EVIDENCE

The acoustic Moho is often recognised as the depth at which the compressional wave velocity, V_P , increases from its value of 6.8-7.0 km s⁻¹ in the lower crust to 8.0 - 8.2 km s⁻¹ in the upper mantle. Although there are many areas in which the presence of a seismically defined Moho is well recognised there are also areas where the seismic data may be better explained by a gradation of acoustic transmission properties between the mantle and the lower crust. Figure F3.5. shows the variation of V_P with depth.

Increased resolution in the field of seismic refraction experiments has shown coincidence of reflector horizons with boundaries of large refraction velocity contrasts (Mueller, 1977). Mueller admits that there are probably lateral discontinuities in his seismic sections but points out the overall consistent nature of the crust. He reported anomalous seismic velocity maxima and minima in most of his crustal cross sections which he ascribed to amphibolite and granitic layering by intrusion of granites in the lower crust. Another explanation for this effect would be the development of large scale layering due to deep level thrust faulting in a similar fashion to the mechanism that produces thrust faults at shallower levels (Cook et al, 1979).

models produced by Smithson, although emphasizing lateral changes in crustal composition, have overall depth zones of similar character in an attempt to preserve a general layer model while accounting for regional differences (Smithson, 1978, 1979). A study by Brown and others has departed from the single layer model even more by correlating zones of prominent and similar reflecting character to specific rock types and not constraining

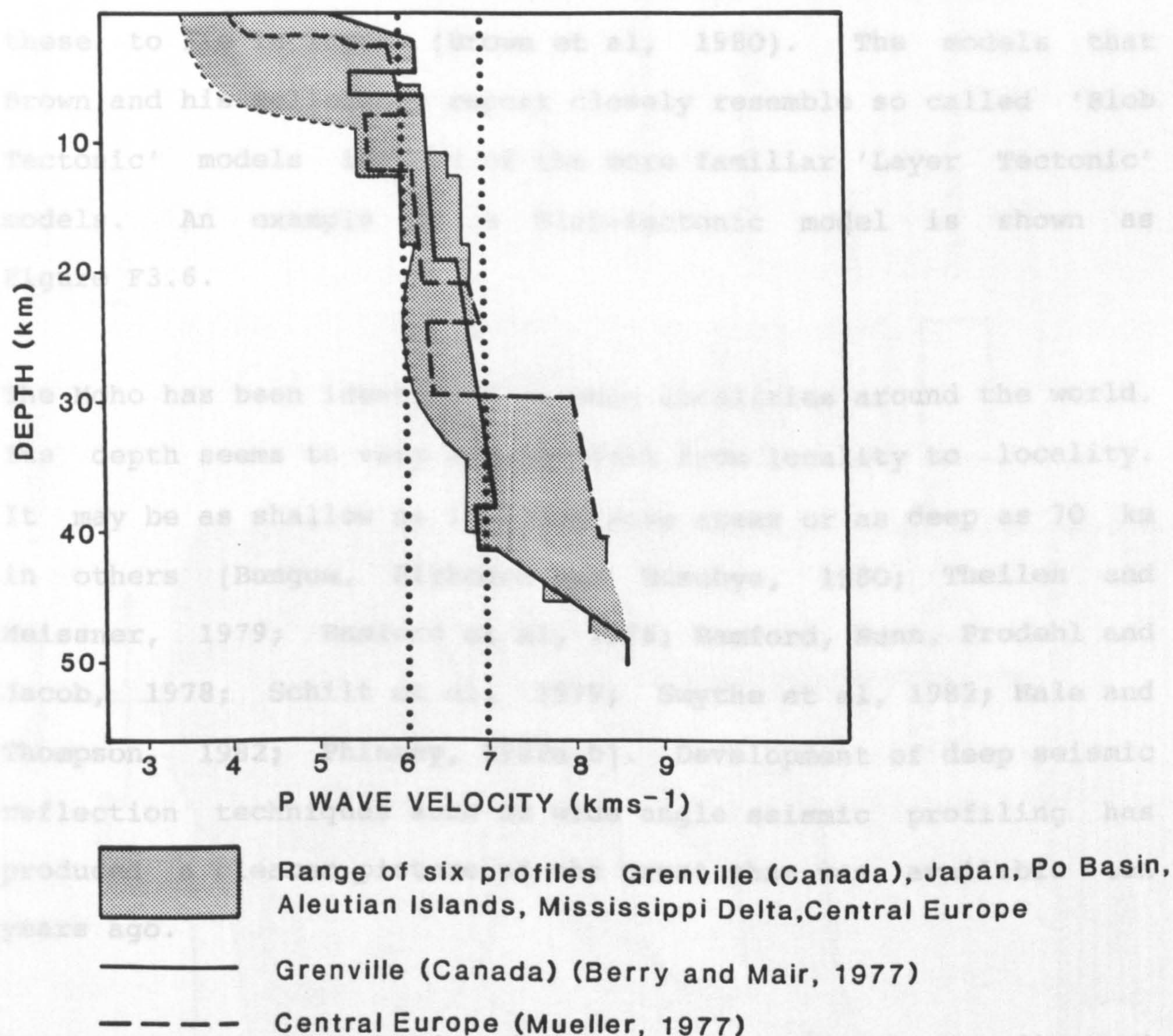


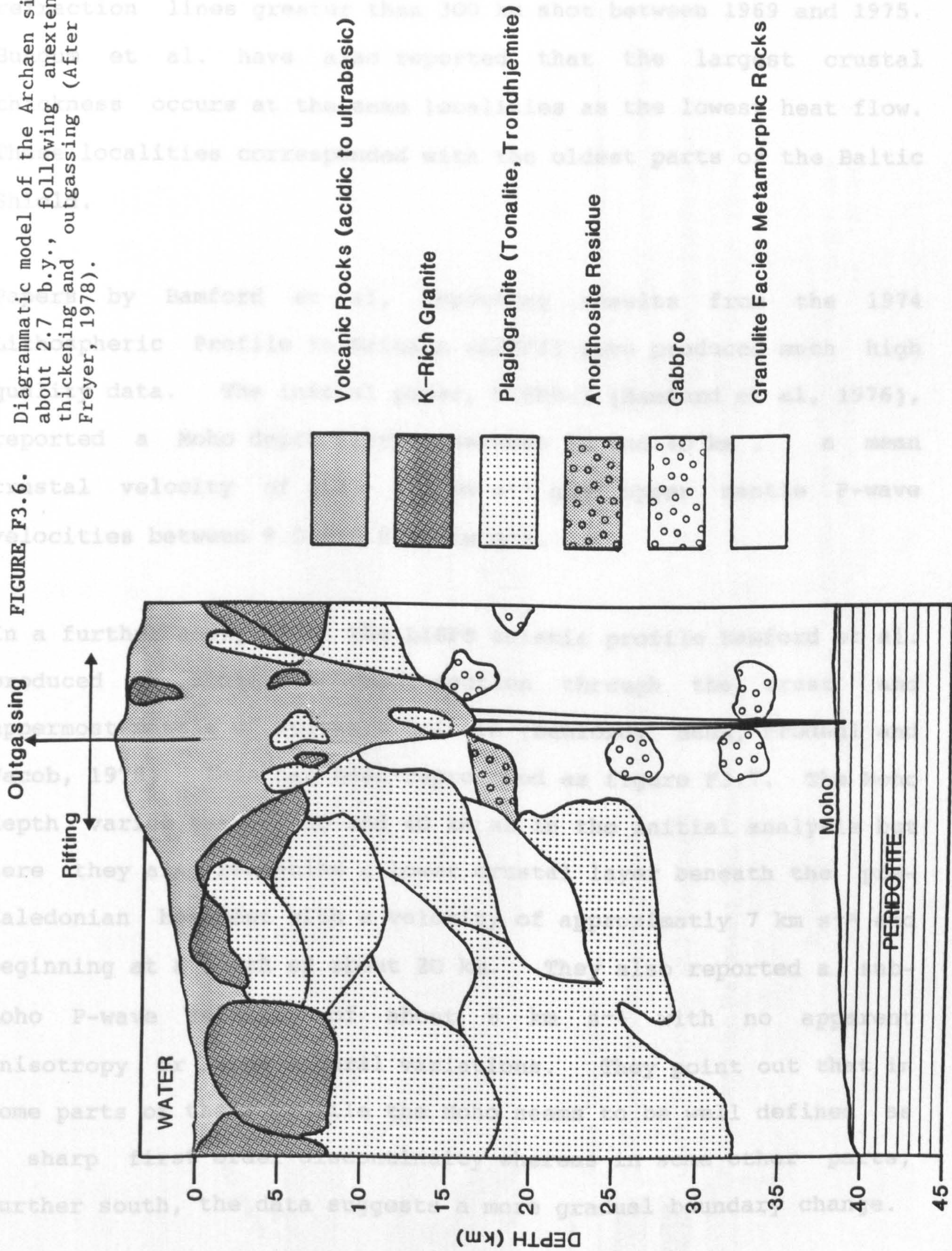
FIGURE F3.5. The depth variation of P-wave velocity on six seismic reflection profiles from various authors (After Kay and Kay, 1981).

Models produced by Smithson, although emphasising lateral changes in crustal composition, have overall depth zones of similar character in an attempt to preserve a general layer model while accounting for regional differences (Smithson, 1978, 1979). A study by Brown and others has departed from the simple layer model even more by correlating zones of prominent and similar reflecting character to specific rock types and not constraining these to lie in layers (Brown et al, 1980). The models that Brown and his colleagues report closely resemble so called 'Blob Tectonic' models instead of the more familiar 'Layer Tectonic' models. An example of a blob-tectonic model is shown as Figure F3.6.

The Moho has been identified in many localities around the world. Its depth seems to vary a great deal from locality to locality. It may be as shallow as 15 km in some areas or as deep as 70 km in others (Bungum, Pirhonen and Husebye, 1980; Theilen and Meissner, 1979; Bamford et al, 1976; Bamford, Nunn, Prodehl and Jacob, 1978; Schilt et al, 1979; Smythe et al, 1982; Hale and Thompson, 1982; Phinney, 1982a,b). Development of deep seismic reflection techniques such as wide angle seismic profiling has produced a clearer picture of the crust than was available ten years ago.

A considerable amount of work has been done in Fennoscandia and Northern England to trace the depth of the acoustic Moho. A paper by Bungum, Pirhonen and Husebye has reported a rather uniform crustal thickness of about 45 km in Fennoscandia, (Bungum, Pirhonen and Husebye, 1980). The value of Bungum et al.

FIGURE F3.6. Diagrammatic model of the Archaen sialic crust at about 2.7 b.y., following an extended period of thickening and outgassing (After Collerson and Freyer, 1978).



was based upon the spectral analysis of P-wave records from 11 seismographic stations in the area. The values obtained by this method agree well with Moho depths obtained from 11 seismic refraction lines greater than 300 km shot between 1969 and 1975. Bungum et al. have also reported that the largest crustal thickness occurs at the same localities as the lowest heat flow. These localities corresponded with the oldest parts of the Baltic Shield.

Papers by Bamford et al, reporting results from the 1974 Lithospheric Profile in Britain (LISPB) have produced much high quality data. The initial paper, LISPB-I (Bamford et al, 1976), reported a Moho depth varying between 30 and 40 km, a mean crustal velocity of $6.1 - 6.7 \text{ km s}^{-1}$ and upper mantle P-wave velocities between 8.0 and 8.25 km s^{-1} .

In a further analysis of the LISPB seismic profile Bamford et al. produced a schematic cross-section through the crust and uppermost mantle of northern Britain (Bamford, Nunn, Prodehl and Jacob, 1978). This has been reproduced as figure F3.7. The Moho depth varies between 30 and 40 km as in the initial analysis but here they also recognise a lower crustal layer beneath the pre-Caledonian basement with a velocity of approximately 7 km s^{-1} and beginning at a depth of about 20 km. They also reported a sub-Moho P-wave velocity of about 8 km s^{-1} with no apparent anisotropy or large lateral variations. They point out that in some parts of their profile the Moho seems to be well defined as a sharp first order discontinuity whereas in some other parts, further south, the data suggests a more gradual boundary change.

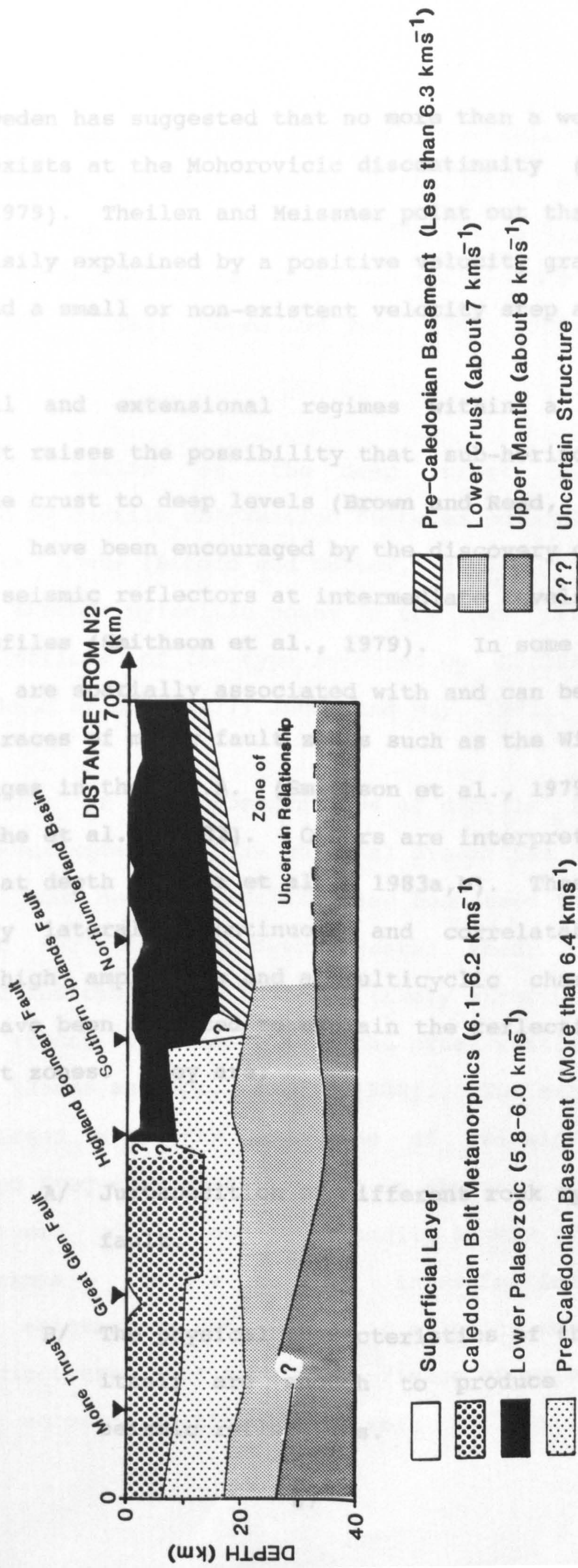


FIGURE F3.7. Schematic cross-section through the crust and uppermost mantle of northern Britain (After Bamford et al, 1978).

Work in Sweden has suggested that no more than a weak acoustic interface exists at the Mohorovicic discontinuity (Theilen and Meissner, 1979). Theilen and Meissner point out that their data could be easily explained by a positive velocity gradient within the crust and a small or non-existent velocity step at the Moho.

Compressional and extensional regimes within a tectonically active crust raises the possibility that sub-horizontal faults penetrate the crust to deep levels (Brown and Read, 1983). Such speculations have been encouraged by the discovery of prominent low angle seismic reflectors at intermediate levels in crustal seismic profiles (Smithson et al., 1979). In some cases these reflections are spatially associated with and can be correlated to surface traces of major fault zones such as the Wind River and Laramie ranges in the U.S.A. (Smithson et al., 1979) and Moine Thrust (Smythe et al., 1982). Others are interpreted as major fault zones at depth (Brewer et al., 1983a,b). These reflectors are usually laterally continuous and correlatable showing relatively high amplitudes and a multicyclic character. Two hypotheses have been advanced to explain the reflectivity of deep crustal fault zones. They are;

A/ Juxtaposition of different rock types across a fault.

B/ The physical characteristics of the fault zone itself are enough to produce the strong seismic reflections.

It is likely that deep fault zones in crystalline crust may usually juxtapose somewhat similar lithologies so the first hypothesis may not generally apply. The second hypothesis has been studied to determine whether it can explain the observations (Fountain et al., 1984; Jones and Nur, 1982; 1984; Fountain et al., 1988).

Sub-horizontal faults in the deep crust are probably characterised by ductile deformation features commonly associated with mylonite zones (Brodie and Rutter, 1987). The question arises as to whether mylonitic zones in the lower crust can cause seismic reflections of the type reported by COCORP and other groups (Smithson et al., 1977; Jones and Nur, 1982).

The combination of field observations of ductile fault zones, laboratory measurements of the physical properties of mylonites and true amplitude seismic modelling has been used to investigate the acoustic reflectors in deep crustal shear zones. These methods have indicated that the most likely structure that may produce the strong reflections from the crust/mantle transition is laminar (Jones and Nur, 1984; 1982). The structure they report is based upon the modelling of seismic reflection amplitudes and frequency spectra. It is thought to consist of alternate layers of high and low velocity layers of about 100 metres thickness. In a further investigation involving synthesising seismograms from models of mylonite zones Fountain et al have discovered that a zone comprising of mylonite laminae can produce coherent multicyclic seismic reflections with an

amplitude of response up to twice that of a single interface (Fountain et al, 1984). Jones and Nur also report that the P-wave velocity and density of the rocks are not greatly affected during deformation under mid-crustal conditions and they remain similar to those of the protolith. They ascribe the anomalously high reflections from some fault zones to the presence of a very high pore pressure within the fault zone but they admit that the maintenance of the pressure needed to account for this over a geologically significant timescale would imply rocks of an extremely low porosity. The major point is that the lower crust is structurally a lot more complex than at first thought. This has also been shown by Schilt et al. in their deep reflection surveys of the USA using Vibroseis techniques (Schilt et al., 1979). Schilt et al. stress the heterogeneity of the crust at a scale of a few kilometres or less and suggest that the Moho is not a single constant interface but more likely a complexly layered zone. They also report the common occurrence of mid-crustal discontinuities but not of a size indicative of a continental scale structure and as such they dismiss the idea that the Conrad discontinuity is a constant feature of continental crust.

In 1982 the MOIST seismic profile in the far north of Scotland was completed using marine seismic reflection techniques. Its aim was to map the top 40 km of the Earth in that area. The BIRPS MOIST team has reported an almost continuous Moho about 25 km deep and in good agreement with other values such as those from LISPB (Smythe et al, 1982). This profile was also able to map the major thrust planes of the area down to considerable depth.

They have reported a complexly structured crust as well. Other reports have also shown well reflecting horizontal or sub-horizontal structures at depths corresponding to the acoustic Moho and many of the authors also attribute them to laminations (Hale and Thompson, 1982; Phinney, 1982a). The deep crust has also been imaged by the BIRPS WINCH deep seismic profile. This has also revealed a complexly layered lower crust, the dipping horizon of the Iapetus suture, the Outer Isles Thrust and Flannan 'Thrust' with the latter seeming to penetrate down into the upper mantle, and a Moho depth of about 26 - 30 km (Beamish and Smythe, 1986; Brewer et al, 1983a). Phinney and Odom have reviewed the state of seismic profiling and point out that most workers in the field of research now accept the Earth to have a heterogeneous crust of a complex nature (Phinney and Odom, 1983) with a well reflecting Moho transitional zone.

The mineralogy of the lower crust necessary to give a value of seismic velocity comparable to the observed values is less well defined. There is data from experimental studies that indicates that granulite facies assemblages of acidic and intermediate compositions may be important stable phases in the lower crust, (Kennedy and Ito, 1972; Green and Ringwood, 1972; Green, 1970). To satisfy any valid petrological model of the lower crust the lower crustal rocks must have P-wave velocities comparable to those observed from seismic studies of the lower crust. The measurement of P and S-wave velocities of granulite samples under high pressure conditions, up to 1 GPa, has been done by Christensen and Fountain, (Christensen and Fountain, 1975). The range of velocities obtained was similar to the range of seismic

velocities reported for the lower crust from field studies. This strengthens the hypothesis that granulite rocks may be major constituents of the Earth's lower crust. There are two major problems with the report from Christensen and Fountain. Firstly, that the granulite results varied over quite a large range and secondly, that the values they obtained took no account of the effects of varying the temperature. This second point is important as it is very likely that the temperatures of the lower crust has a significant effect on the measured velocity of the samples as well as their stability. Christensen and Fountain were very careful to point out that their data does not rule out amphibolite facies from being a major constituent of the lower crust.

Ito and Kennedy (1970) have suggested that the relative densities of basalt, garnet granulite and eclogite should produce detectable seismic discontinuities. Moreover on the basis of mineralogical composition they estimated the P-wave velocity in garnet granulite to be about 7.5 kms^{-1} . Experimental evaluations of the seismic velocities in eclogites and granulites up to pressures of 1 GPa have shown that the elastic properties of garnet granulite are compatible with a $V_P = 6.8 - 7.7 \text{ kms}^{-1}$ lower crustal layer, (Manghnani, Ramanantoandro and Clark, 1974). It is therefore possible that garnet granulite is a major stable component of the Earth's lower crust.

Most authors seem to agree on the broadly layered structure of the Earth's crust, although some prefer a more disordered blob-tectonic model instead of the more traditional simplistic

layer model. There is evidence for both first order discontinuities as well as gradational behaviour within the lithosphere and in some cases there are reports of the occurrence of both but in different localities of the same survey. There is also evidence for gradational lateral behaviour in the zones of transition which produces a more complex architecture to the zones of mineralogical change. Disciples of the discrete layer model accept that seismic velocity maxima and minima that occur in seismic velocity profiles can be due to localised blobs of amphibolite or granulite while followers of the blob-tectonic models mention thrust faulting at depth as a possible mechanism for producing a more layered appearance to crustal cross sections and seismic profiles. Seismic reflection surveys (e.g. COCORP, BIRPS, ECORS) have revealed a more complex structure for parts of the lower continental crust (Schilt et al., 1979; Smythe et al., 1982; Jones and Nur, 1984) and lateral variations (Phinney and Odom, 1983). Some parts that appear strongly reflective could be areas of complex layering especially at the Moho.

Experiments to measure the seismic P-wave velocity through rocks of various mineralogies under simulated lower crustal conditions have confirmed that granulites including garnet granulite have values of P-wave velocity lying within the range of P-wave velocities in the lower crust and therefore do not rule them out as major constituents of the lower continental crust.

3.4.2. HEAT FLOW EVIDENCE

There is a linear relationship between surface heat flow and crustal heat production. This has been explained by assuming an exponentially decreasing heat production with depth with a scale depth of 7-10 km (Lachenbruch and Sass, 1977). Taking into consideration the structural and compositional complexities of the crust this simple exponential model is probably unrealistic (Smithson and Brown, 1977; Smithson et al, 1977). Many deep crustal rocks do show lower K, Th and U concentrations than their shallower counterparts. This can be accounted for by partial melting redistributing the radioactive elements depending on their relative concentrations between the solid and the melt phases (Lachenbruch and Sass, 1977). It also can be accounted for by the presence of a pervasive fluid phase in which the radioactive elements could become dissolved. Such a liquid phase may be able to transport the elements up to the upper crust by advective porous flow or along a diffusion gradient to leave the lower crust relatively depleted (Wells, 1979; Collerson and Freyer, 1978). The detailed mechanism of transfer of radioactive elements in the fluid phase is yet to be worked out but the work of Heier (1973) has gone some way towards explaining how water generated from dehydration reactions during granulite facies metamorphism can help in the redistribution of radioactive heat generating elements from an Andesitic or Archean protocrust (Taylor and McLennan, 1985).

The present geothermal gradient can be found from crustal heat flow and thermal conductivity values. Geothermal gradients

calculated in this way vary greatly from region to region. This is mainly due to the variability of the heat flow as can be seen in figure F3.8. (Lachenbruch and Sass, 1977).

Figure F3.9. shows the range of generalised steady state temperature profiles for various heat flow provinces in the United States. This demonstrates the range over which heat flows are distributed (Shankland and Ander, 1983).

The geothermal gradient is a primary controlling factor of the type of mineral assemblage formed for a given composition at a specific depth. This is due to the dependence of the stability of the mineral assemblage upon temperature. This can be seen clearly in figure F3.3. As the geothermal gradient varies from region to region it can be assumed that there are correlated variations in the mineral assemblages present from region to region. In particular metastable assemblages may be present at depths that would normally be considered unusual, such as gabbro in the eclogite field. Since the density differential between stable and metastable assemblages is quite large the effect on the P-wave velocity differential between them will also be quite considerable. It may be possible to gain some insight into the occurrence of the metastable assemblages from seismic field studies as these should be able to discriminate between stable and metastable assemblages using their relatively different P-wave velocities as a criterion (Kay and Kay, 1981).

It has been suggested that the depth of the high conductivity layer in the crust is correlated to the surface heat flow, the

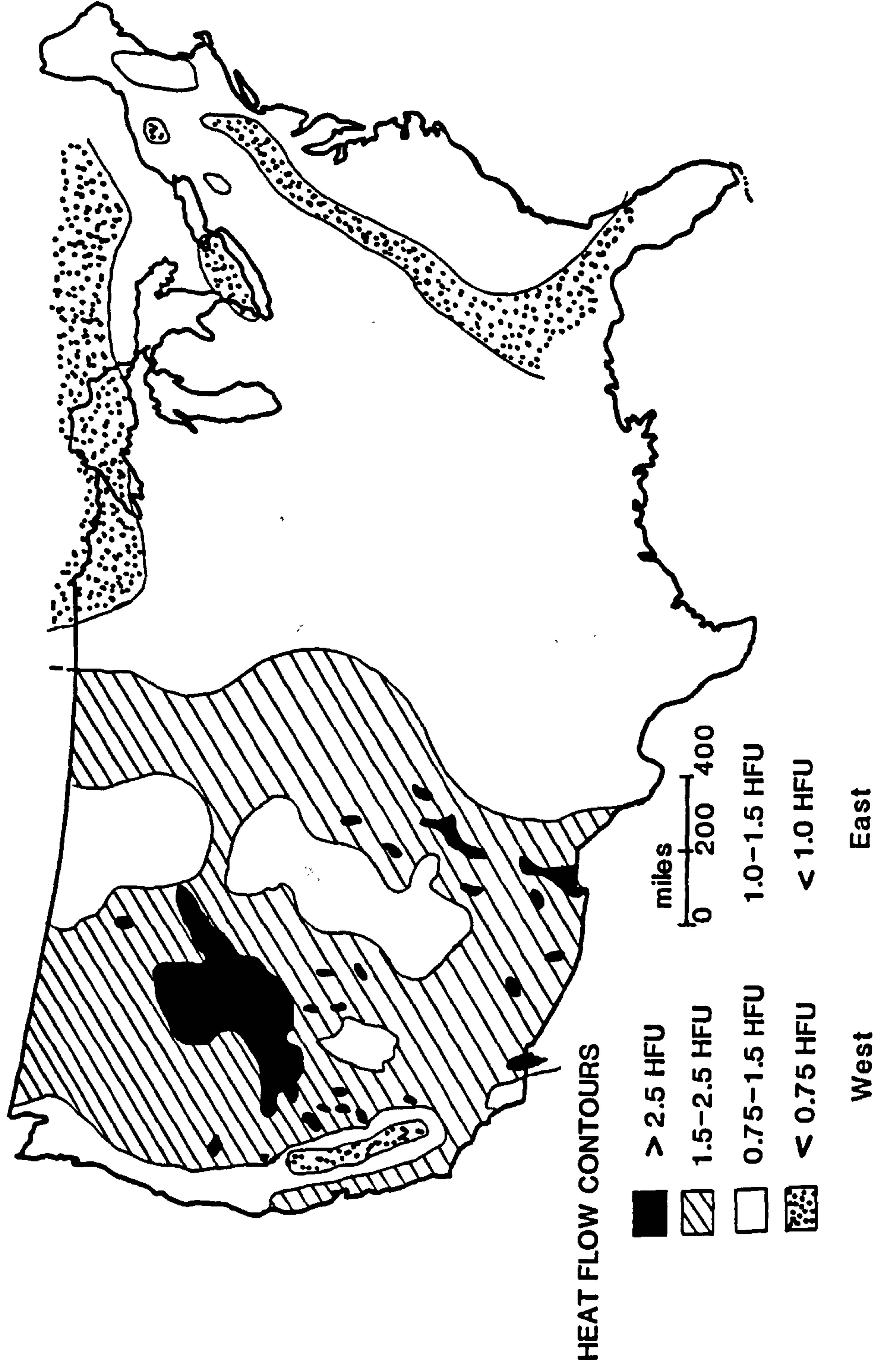


FIGURE F3.8. Generalised heat flow map of the U.S.A. (After Lachenbruch and Sass, 1977, figure 2).

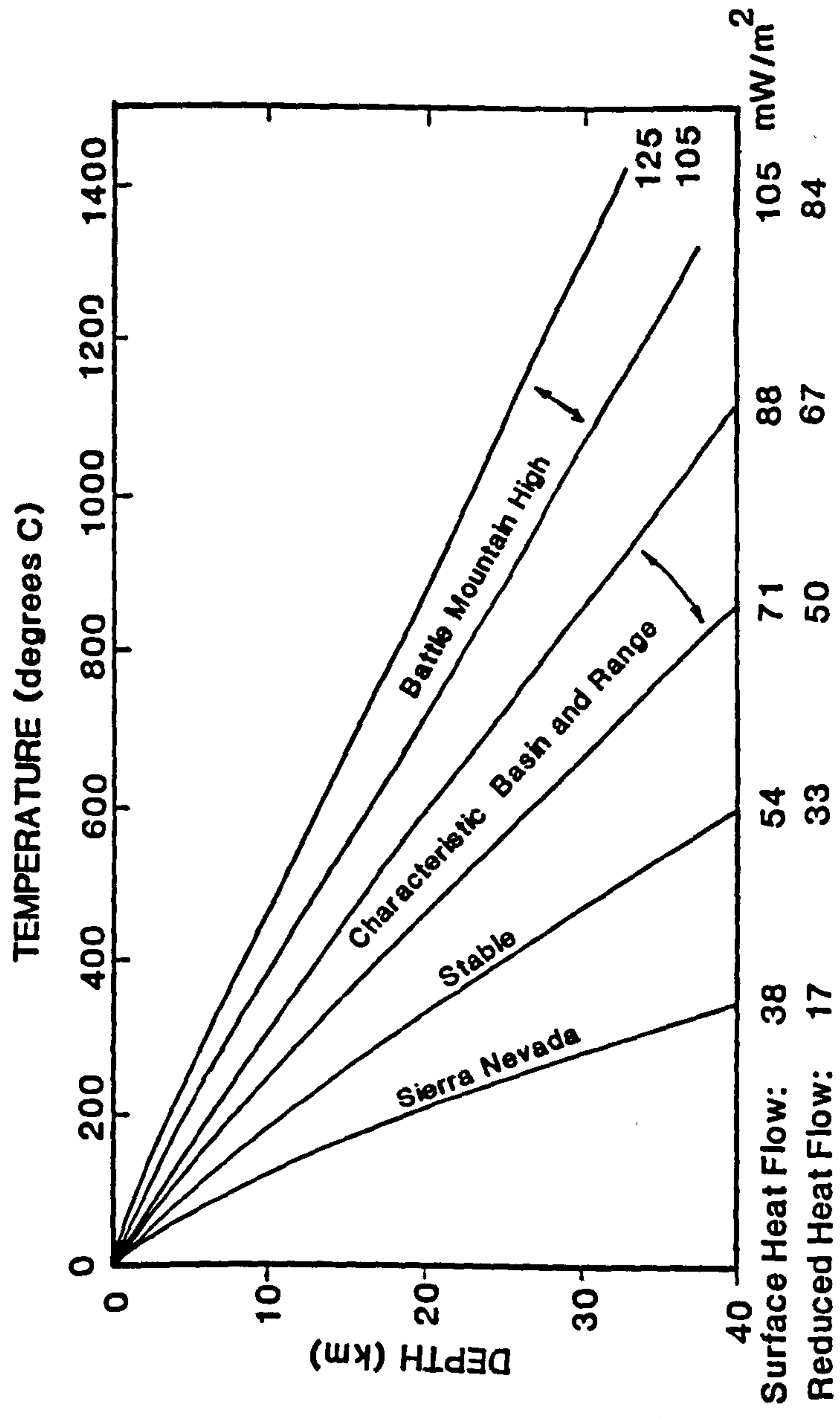


FIGURE F3.9. Generalised steady state temperature profiles for heat flow provinces in the U.S.A. Surface heat flow and reduced heat flow in mW m⁻¹ are indicated at the base of the figure. (After Shankland and Ander, 1983).

depth to the Curie point and regional tectonics (Ander et al, 1982; Kaufman and Keller, 1981). It has also been suggested that there is a possible relationship between continental heat-flow and the seismic reflectivity of the lower crust (Klemperer, 1987). In an investigation into any correlation between continental heat-flow and seismic reflectivity Klemperer has derived an isotherm map for the antral plane of the reflective lower crust. He reports temperatures varying between 300 and 400 °C and temperatures of about 390 °C in the western United States. Adam has also reported a correlation which he expressed as $Q=LT/h$ where the depth to conducting layers within the crust (h) is related to the surface heat-flow (Q) by the temperature (T) and thermal conductivity of the crust (L), (Adam, 1976). An analysis of Adam's correlation and data set by Klemperer has indicated that the temperature at crustal conducting and reflecting depths is around 470°C. The depths at which temperatures of 400 to 500 °C occur have been estimated at 25 - 30 km for a 'stable reference crust' according to Lachenbruch and Sass and 20 - 24 km from an extrapolation of the observed value of 180 °C at 10 - 12 km with a temperature gradient of 25 °Ckm⁻¹ measured in the Kola Peninsula borehole (Lachenbruch and Sass, 1977).

A further review of the data and correlations made by Czechoslovakian authors broadly agrees with these values (Cermak and Lastivickova, 1987).

Despite the linear relationship between surface heat flow and surface heat production the depth/heat production model has not

got an exponential form. The simple exponential model is a gross oversimplification when the complex nature of the continental crustal profile is taken into account. The one generalisation that is possible is that there is a greater concentration of radioactive heat producing elements at the surface than there is at depth for any given small volume. The abundance of radioactive heat producing elements at lower crustal depths is probably very small and would have a negligible heating effect upon the rock type present at that depth. The crustal heat flow of a particular locality and the local geothermal gradient are functionally interdependent. The geothermal gradient is important as it defines the temperature of the rocks within the Earth's crust. The value of the temperature is a primary rock and mineral assemblage stability determining factor. The heat flow varies between about 0.5 and 3.0 HFU from locality to locality and such variations may be partly indicative of lateral variations in the mineralogy of the mid- and lower crustal rocks. The heat flow variation may also be correlated in some way to the type of transition from the lower crustal to an upper mantle regime. The effect of geothermal gradient upon mineral stability in the lower crust is much more marked than that of mineral composition on the geothermal gradient due to small concentrations of radioactive elements.

3.4.3. MAGNETIC EVIDENCE

It has been suggested by several authors that long wavelength magnetic anomalies have their origins in the lower crust with the Moho acting as a magnetic boundary (Shuey et al., 1973; Wasilewsky et al., 1979). Wasilewsky pointed out that although peridotites are weakly magnetic the mantle will be non-magnetic or only very weakly magnetic as the temperatures at these depths approach the Curie Point of peridotite. If this is the case the Moho will act as a magnetic boundary.

Magnetic studies have found very large regional variations in magnetic field that are due to changes in the crust and upper mantle. These variations could represent regional changes in rock type or temperature. The magnetic intensities of these variations have long wavelengths which indicates that the depth of the causes of the variations is quite large.

It has been suggested that the Moho is a magnetic boundary as the Cr-Mg-Al spinels that are found in mantle peridotite are non magnetic at mantle temperatures but this is only the case if the lower crust can be shown to consist of rocks which are magnetic at the lower crustal temperatures. Haggerty maintains that magnetic anomalies that seem to be produced from the lower crust cannot be due to the natural magnetites that are present in granulites (Haggerty, 1978). More recent studies on magnetic minerals that may be present in the lower crust have indicated that lower crustal rocks probably do have Curie temperatures that exceed the average temperature in the lower crust by a sufficient

amount to allow the rock to have a significant magnetisation (Wasilewski and Padovani, 1980; Thomas et al., 1979). This work has centred around titanomagnetites and has produced several models which may describe the variation in magnetic properties of lower crustal rocks with depth. The work by Wasilewski and Padovani combines data from lower crustal xenoliths, mantle xenoliths, granulites from metamorphic terrains and a profile across the Ivrea Zone in North Italy. It also suggests that the Moho is a magnetic boundary and significantly reports the lack of any exotic minerals in their samples.

Large deep seated magnetic variations have been found all over the crust. These may be due to changes in mineralogy as a result of lateral changes in temperature or composition on a large scale. It is unlikely that the mantle is the location of the magnetic minerals that cause these variations due to the hyper-Curie temperatures found there. Indeed temperatures found in the lower crust in most geothermal regimes seems to exclude magnetisation of most magnetic minerals. Some reports have indicated that titanomagnetite spinels can remain magnetic at lower crustal temperatures. A fuller understanding of the precise magnetic minerals existing in the lower crust is needed before the possibilities of the broad magnetic variations can be understood.

3.4.4. ELECTRICAL CONDUCTIVITY EVIDENCE

The first and most important consideration when dealing with the electrical conductivity of the crust is the mechanism by which the electric charge is carried.

Electronic conduction through rocks can usually be discounted as a major mechanism below temperatures of about 700 °C as it is only at temperatures higher than this that the electron activation energy is exceeded (Brace, 1971).

Electrolytic conduction through the rock pore fluids is the major mechanism in the upper crust. It obeys the empirically derived Archie's Law (Archie, 1942). The critical controlling factor here is the rock porosity which decreases with depth as the overburden pressure increases and closes cracks, fractures and pores in the rock. Calculations by Dvorak and by Keller, Anderson and Prichard have suggested that conduction by pore fluids is likely to be quite small at depths of greater than 15 km due to the effect of overburden pressure (Dvorak, 1975; Keller, Anderson and Prichard, 1966).

Globally electrical profiles indicate that the average conductivity of the upper part of the Earth's crust is low, between 10^{-5} and 10^{-3} S m⁻¹ (Keller, Anderson and Prichard, 1966; Banks, 1969, 1971; Brace, 1971; Dvorak, 1975) whereas the middle part and to a certain extent the lower part of the Earth's crust has a relatively high conductivity of between 10^{-3} and 10^{-1} S m⁻¹ (Lienert and Bennett, 1977; Connerney et al., 1980; Van Zijl, 1977).

The variations of electrical conductivity at several locations in the continental crust from magneto-telluric and geomagnetic deep sounding measurements are shown in figure F3.10. It can be seen from the figure that the conductivity is several orders of magnitude higher than that predicted by Brace (Brace, 1971). Brace's estimates assume a systematic decrease of water saturated crack, fracture and pore volume with depth as the overburden pressure and temperature increases. He also assumes that the rock matrix consists of anhydrous minerals. Neither of these assumptions may hold true.

The apparent mid-crustal increase in electrical conductivity that is not found in Brace's estimates has been attributed to areas of more conductive partially molten zones (Lienert, 1979). Such zones are only possible in areas of high heat flow and Tertiary vulcanism such as the Basin and Range province of the USA. Mid-crustal high conductivity layers in regions of low heat flow cannot be explained by partial melting (Connerney et al., 1980; Van Zijl, 1977). It can be explained by the increase of temperature with depth combined with an increase of water filled pores or the presence of hydrous minerals (Connerney et al., 1980; Hyndman and Hyndman, 1968).

Resistivity/temperature curves compiled by Sternberg show that only rocks containing hydrous minerals have the required electrical resistivities of 50-5000 Ω m at depths of 20-30 km and temperatures of 300-400 °C (Sternberg, 1979). The Sternberg curves are shown as figure F3.11.

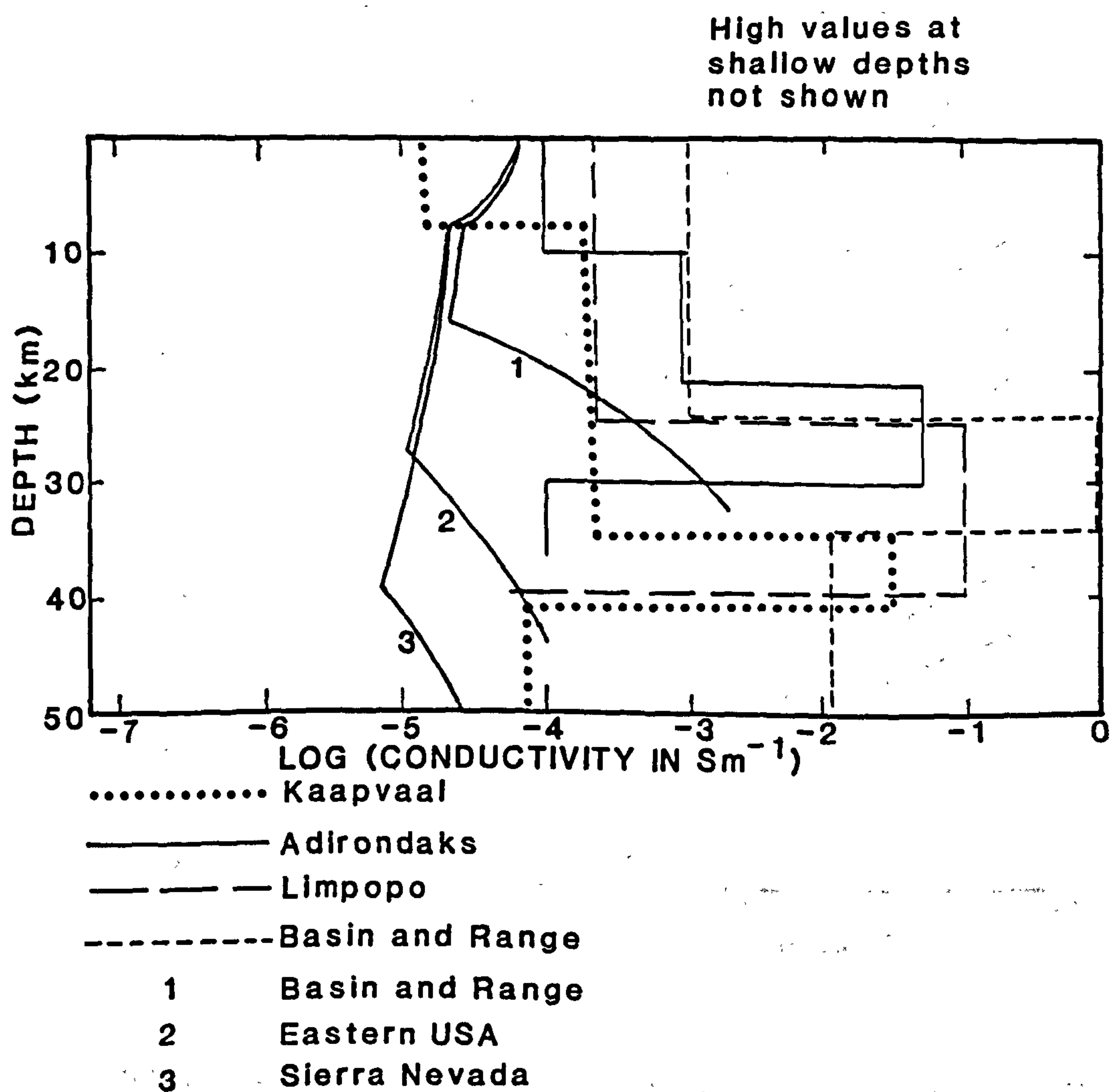


FIGURE F3.10. Conductivity/ depth profiles from various authors.

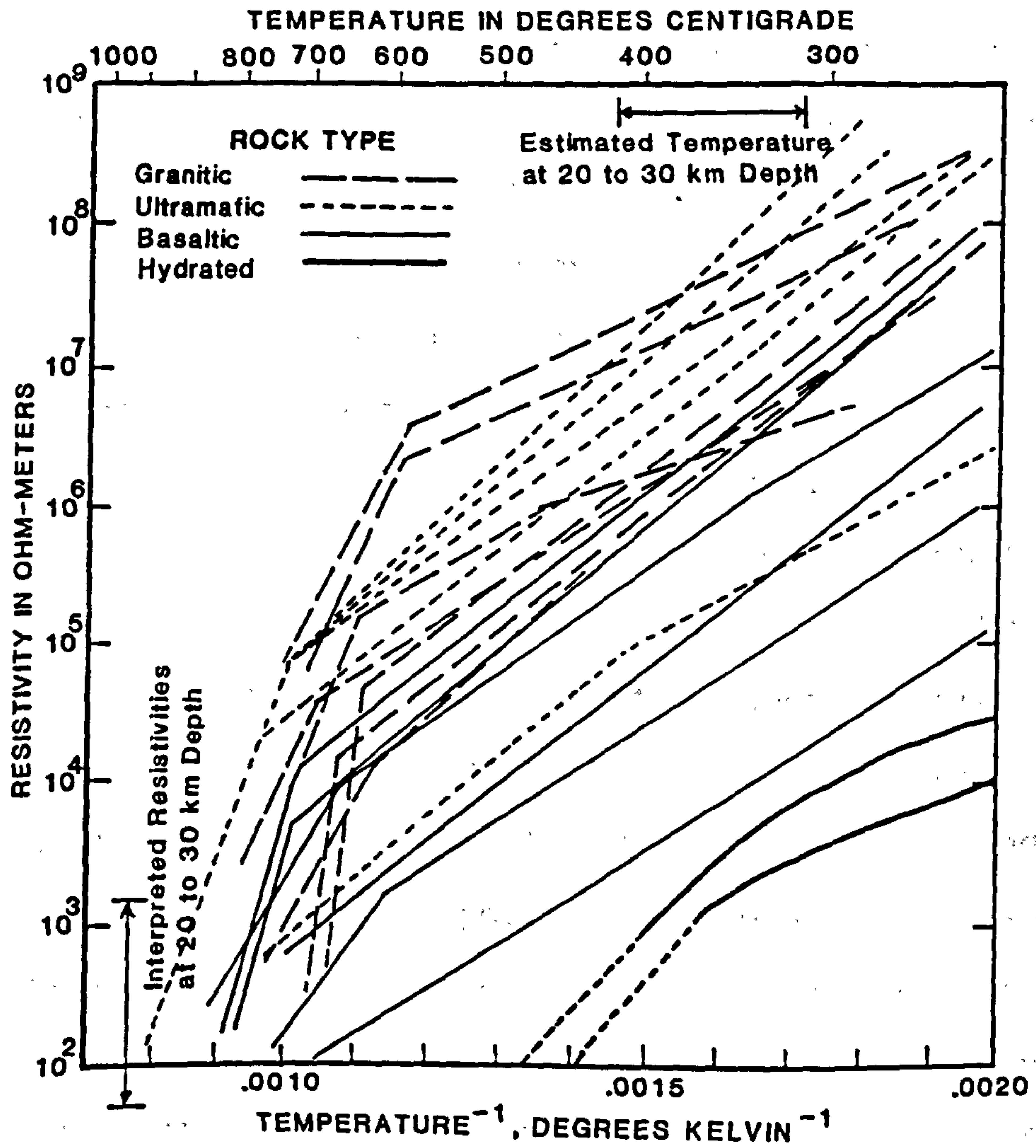


FIGURE F3.11. The electrical resistivity of various rocks as a function of inverse temperature (After Sternberg, 1979).

Although some hydrous mineral and magnetic oxide-bearing rocks, especially serpentinites, have high conductivities they are not necessarily characteristic of the type of rock that may be found in the lower crust. Conductivity data compiled from Soviet measurements of these types of rock has indicated that only a small number of the rocks with hydrous or magnetic mineral-bearing mineralogy have sufficiently high conductivities to be considered as possible sources of conductivity enhancement if they were present in the lower crust (Olhoeft, 1981). Ascribing high conductivity dominantly to the presence of conduction within structurally bound water requires the lower crust to be composed almost entirely of rocks consisting of hydrous minerals. It is extremely unlikely for this to be the case considering the prevailing pressures and temperatures (Shankland and Ander, 1983) although in some areas of special consideration a case may be made for hydrated rock mineralogies having a major effect in that particular locale (Taylor and Toksoz, 1982). It is useful to bear in mind that there may be sufficient rocks with structurally bound water in the upper part of the lower crust in amphibolite facies rocks. These may have the ability to significantly increase the conductivity of the upper part of the lower crust.

It is known that water is released from hydrous minerals during metamorphism. Berdichevsky et al. (1969) have estimated that the approximate 1-5% water that is contained in minerals by amphibolite facies rocks can be released by dehydration at the boundary with granulite facies. Such water if distributed intergranularly could produce electrical conductivities

comparable to those possible in the pure rock melt phase (Zadridze, 1967).

There is a rather narrow temperature range within which water becomes available for conduction from dehydration reactions and is then able to cause high conduction through interconnected pores (Jones, 1987). The lower limit on temperature is defined by the lowest temperature at which dehydration reactions occur (Haak and Hutton, 1986; Ringwood, 1975). The upper limit is defined by the temperature at which the rocks start undergoing plastic deformation as this decreases the interconnectivity of the pore fluids (Brace, 1972, 1980; Tullis and Yund, 1977). The temperature range is about 400 to 500°C. The depths at which temperatures of this order occur are 25 - 30 km according to the 'stable reference crust' of Lachenbruch and Sass (Lachenbruch and Sass, 1977) and 20 - 24 km according to extrapolation of measurements of 180 °C at 10 - 12 km and a temperature gradient of 25 °C km⁻¹ taken in the Kola Peninsula borehole.

Therefore although the decrease in porosity at depth tends to reduce pore volume it is possible that low resistivity may be obtained by the inter-granular distribution of water of crystallisation during the process of dehydration of hydrous minerals.

In 1968 a paper by Hyndman and Hyndman reported that geomagnetic and magneto-telluric evidence of high electrical conductivities at depths of 20-40 km in tectonically active continental areas could be due to water saturation of the rocks at these depths but

it was unclear how water might be transported to rocks at these depths or whether water was being provided by dehydration reactions (Hyndman and Hyndman, 1968). There have been suggestions that the correlation of certain dipping layers of high conductivity with tectonic zones of underthrusting may show that water can be carried down within such tectonically active layers to give the required relatively high conductivity at mid-crustal depths (Lienert, 1979). There may also be a possibility of this effect occurring at convergent plate boundaries (Fyfe, 1975). Recent work by Beamish and Smythe has concentrated on imaging the Iapetus Suture with deep seismic reflection data from the BIRPS WINCH project and magnetotelluric deep soundings. Their results seem to indicate a dipping highly conductive layer that correlates to the dipping suture zone imaged on the deep seismic cross-sections. This is thought to be an area of Benioff type subduction of the oceanic lithosphere when the Iapetus was closing. It may have a high conductivity due to the presence of electrolytes carried down during subduction (Beamish and Smythe, 1986). While suggesting the presence of a water saturated rock layer in tectonically active areas of the continental crust, Hyndman and Hyndman predicted the apparent absence of such a layer in some stable shield areas as a result of dehydration of once saturated rocks by the metamorphic process. Hyndman and Hyndman also discount the possibility that the mid-crustal electrical conductivity high is due to high temperature gradients or partial melting especially in areas of low heat flow as the required conditions would not be reached within the crust.

A relatively conductive material that may be found in the

lower crust is graphite. It is a common accessory mineral found in metasedimentary rocks primarily as a product of regional metamorphism of carbonaceous black shale. It can remain interconnected when present in amounts of only a few percent (Jackson, 1970). It has been invoked to explain the electrical conductivity in special areas (Alabi, 1975) and has been found in xenolith studies of lower crustal rocks (Padovani and Carter, 1977). Shankland and Ander dismiss the presence of graphite as a major influence on the lower crustal electrical conductivity. Although citing it as a possible source of enhanced conduction they do not think it can explain the observed conductivity/temperature curve for the crustal high conductivity layer (Shankland and Ander, 1983). Shankland and Ander also point out the need for a greater concentration of graphite to be present in tectonic rather than in shield regions if it were to be a major factor influencing the crustal conductivity. If the mechanisms of graphite formation have remained roughly constant over geological time one would expect the opposite of this to be the case with less graphite in more recently active zones. Shankland and Ander also dismiss sulphides as a medium for enhanced regional conduction in the crust with an argument that parallels that for graphite.

A recent report by Gough has tried to explain the broad physical differences between the upper and the lower crust by proposing a new model of water distribution within the crust (Gough, 1986). Gough's model tries to explain why the upper crust is relatively electrically resistive, seismically transparent, brittle, and, while behaving elastically to stress,

contains almost all the major intracontinental earthquake hypocentres, whereas the lower crust is relatively electrically conductive, ductile, aseismic and contains many seismic reflectors. His model proposes that water is present in the whole crust, in the upper crust it is isolated in cavities in compressively stressed rocks whereas in the lower crust it forms a continuous film around the crystals that make up the rock matrix and is not confined as the high pressures are hydrostatic. This explains the observations but is at variance with the geochemists view, as reviewed by Yardley (Yardley, 1986). The geochemists claim that water can only coexist with lower crustal granulite under conditions similar to those extremes at which the metamorphic grade formed, otherwise the water swiftly reacts with the granulite. The granulite undergoes a hydration reaction to become a lower grade hydrous facies. The high speed of the reaction (Wood and Walther, 1986) suggests that any water finding its way down into the lower crust will very quickly be combined into hydrous minerals.

It is interesting to note that the deepest borehole (Kola Peninsula, USSR) has recorded water percolation down to its greatest depth of 12 km (Kozlovsky, 1984; 1987). Another observation worth making at this point is that often one of the most striking features of some rocks of deep genesis is the presence of joints lined with hydrous minerals.

There is another new model to explain the mid-crust increase in conductivity in which the increase depends on the presence of water of dehydration being trapped by an impermeable layer

(Jones, 1987). It has been suggested that minerals in a layer in the crust that is at temperatures of between 400 and 500 °C and is undergoing dehydration reactions can precipitate out causing an impermeable layer that traps the water of dehydration beneath it (Etheridge et al, 1983, 1984). This gives rise to a three layer electrical conductivity structure for the 'cold' continental crust as shown in figure F3.12. The three layer model is briefly described below.

Layer 1 : Dry upper crust, no fluids, resistive (10^4 ohm m).

Layer 2 : The top part of the lower crust. Temperatures of 400 - 500 °C cause dehydration and water of dehydration is trapped under an impermeable layer of precipitated minerals giving a high conductivity zone (10 ohm m).

Layer 3 : Basal layer of lower crust. Contains free water but in isolated pockets not interconnected giving moderate conductivity (10^2 to 10^3 ohm m).

It may be correct to ascribe the position of the Conrad discontinuity and the often observed suite of sub-horizontal reflectors occurring typically between 6.0 and 6.5 TWT with the basal plane of layer 2 and therefore also with the impermeable zone at about 400 °C (Jodicke et al, 1983).

The most abundant fluid found in high temperature and pressure

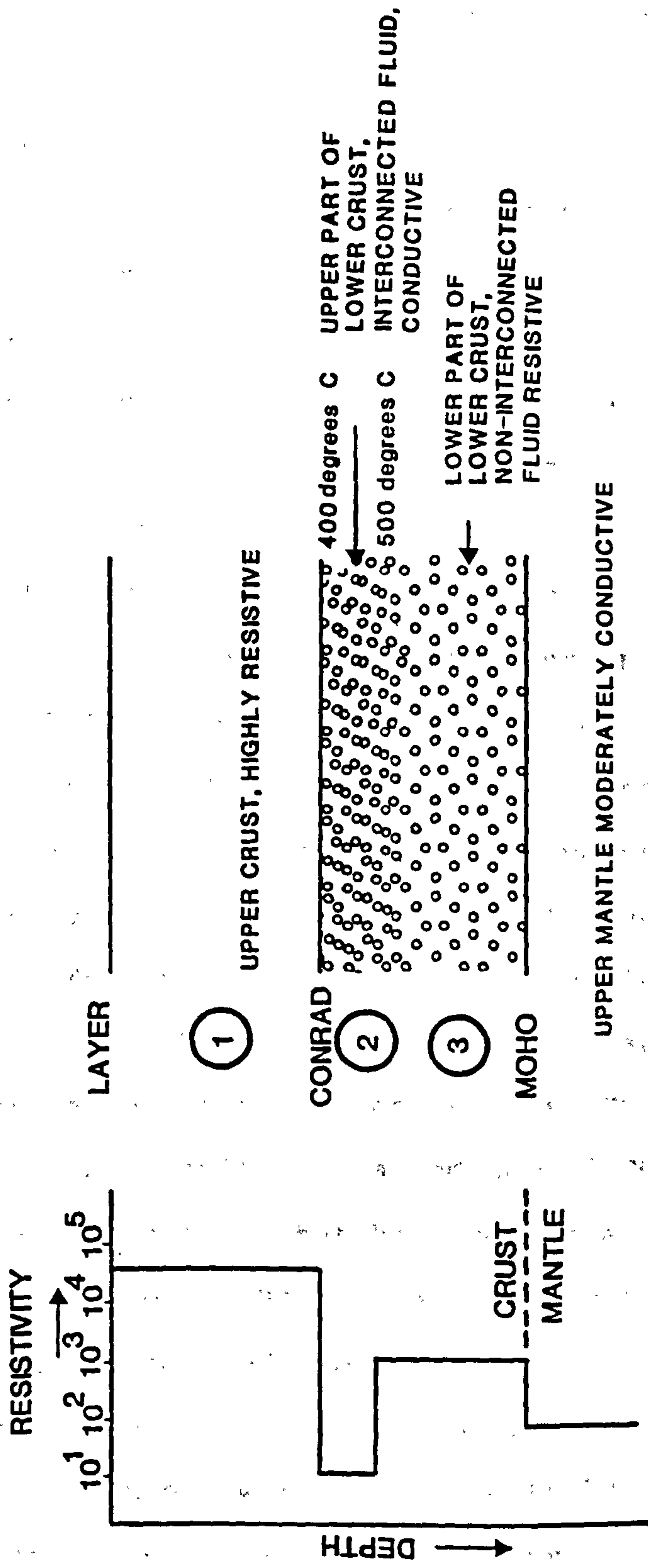


FIGURE F3.12. An electrical model of the continental crust (After Jones, 1987).

mineral inclusions of quartz, plagioclase, pyroxene etc in granulite facies rocks is CO_2 . It accounts for about 1% of the total volume of inclusion. It is also the most abundant fluid in high pressure and temperature xenoliths and exposed lower crustal rocks. It is a great deal more abundant than water (Luckscheiter and Morteani, 1980). The abundance of CO_2 rich inclusions has been interpreted as indicating that the CO_2 was the fluid in equilibrium with the granitic melt (Wendlandt, 1980) but to be sure of this it is important that we are sure that the fluid inclusions formed at depth. A mineral often reported in association with granulite facies rocks is the CO_2 and SO_2 -rich scapolite. This may also be a possible site for the storage of CO_2 of igneous origin in the lower crust (Goldsmith, 1976). Despite this abundance of CO_2 the mid-crustal conductivity rise is probably not due to an increased abundance of CO_2 in a supercritical fluid phase at temperatures of about 400 °C and pressures of around 0.8 GPa. If a 1% interconnected porosity is assumed together with an Archie's Law exponent of 2 then the measured conductivity of 10^{-2} to 10^{-1} S m^{-1} gives an estimate of 10^2 to 10^3 S m^{-1} for the conductivity of the fluid phase. These conductivities are high for a completely ionised electrolyte and impossible for a non-polar solvent such as CO_2 (Shankland and Ander, 1983). Thus, despite the abundance of CO_2 solvent and the significant solute content that has been detected in inclusions from the mantle (Green, 1974) it is unlikely that such solutions will have the required conductivities to explain the conductivities observed in the lower crust. Instead the conductivities in the lower crust have to be explained by the presence of intergranular water, hydrous silicates and other

materials.

Magneto-telluric measurements made along a linear traverse across southern Scotland by Ingham and Hutton have indicated a relatively high conductivity zone starting at about 20 km and extending as deep as 90 km (Ingham and Hutton, 1982a). They have produced one and two dimensional models from their data and found a considerable increase in conductivity at about 20 km in both. They found no detectable change in conductivity at depths suggested to be the Moho by the LISPB seismic profile. In a further paper Ingham and Hutton attempted an interpretation of the apparent geoelectrical structure of southern Scotland (Ingham and Hutton, 1982b). They ascribed the low resistivity at depths of 20-40 km to electrolytic conduction with water from dehydration of hydrous minerals at the amphibolite-granulite facies boundary with some electronic conduction in hydrated minerals still retaining their water at the shallower depths. The anomalously high electrical conductivity below about 40 km was ascribed to solid electronic conduction in basaltic and ultramafic rocks at temperatures greater than about 800 °C with the possibility of some partial melting.

The results of Ingham and Hutton tend to agree well with an interpretation made by A.G.Jones of Fennoscandian data in which he traced only a very small electrical discontinuity at the Moho (Jones, 1982).

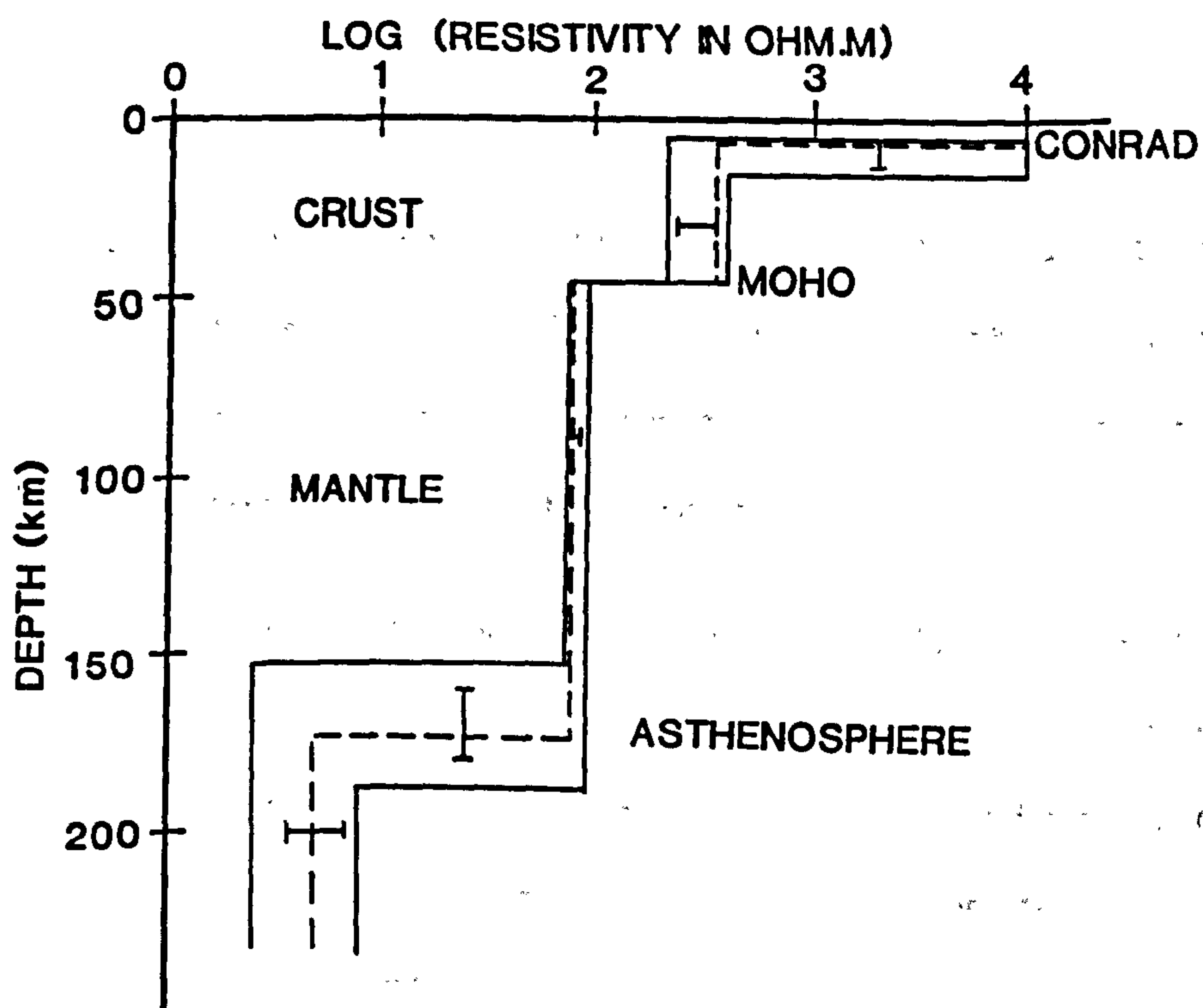
Geomagnetic interpretation of the Inductive Response Function observed in northern Sweden by Jones using a Monte-Carlo method

has shown a crust of 20-30 km thickness when the method assumed the crust was unpartitioned (Jones, 1982). In the same locality other workers derived a crustal thickness of 45-47 km from seismic evidence (Bungum, Pirhonen and Husebye, 1980). At one place Bungum et al. obtained an accurate determination of the Moho depth of 45.5 ± 0.6 km from eight seismic events. Jones repeated his procedure but allowed the method to assume a floating Conrad discontinuity partitioning a two layer crust so that he could compare his results with those of Bungum et al.. This interpretation gave a Moho depth of 46 km which is in good agreement with the values reported by Bungum et al.. The Jones partitioned crust interpretation is shown as figure F3.13.

The new Jones interpretation showed:

1. An intracrustal geoelectrical boundary at a depth of 6.5-16 km. This is comparable to recent estimates of the upper crustal thickness of 16 ± 4 km. This may be the geo-electrical equivalent of the seismic Conrad discontinuity.
2. A lower crustal layer of intermediate resistivity (215-400 ohm m) often correlateable to a seismic layer with a compressional P-wave velocity of about $6.8-7.2 \text{ km s}^{-1}$. Jones suggests that hydrated amphibolites may display the required characteristics for inclusion into this layer.
3. An electrical resistivity contrast across the Moho

FIGURE F3.13. Electrical resistivity as a function of depth from MT measurements (After Jones, 1981).



of only a small size (a factor of about 3.75) but corresponding very well with the seismic Moho.

The presence of a high conducting crustal layer is also reported by Beamish (Beamish, 1986a,b). He subjected several sets of magnetotelluric data to two inversion algorithms and obtained accurate determinations of the depth of the top of the high conductivity layer as it varied between localities. Beamish's models are shown as figure F3.14.

The study of the electrical parameters of the Earth's crust can be considered to fall into two areas:

- A. The laboratory measurement of rock samples and subsequent discussion and theory concerning the rock types, hydration and conduction mechanisms operating in the crust.
- B. The field measurements and interpretation of data. The results from the field are interpreted to give the electrical conductivity profiles that constrain the theoretical models to the real situation.

In summary, a simple model of the electrical situation in the Earth's crust has been discussed for almost 15 years. It is based upon electrolytic conduction through pore fluid and does not take into consideration the possibility of hydrous mineral conduction. It predicts a decrease in pore fluid conduction with

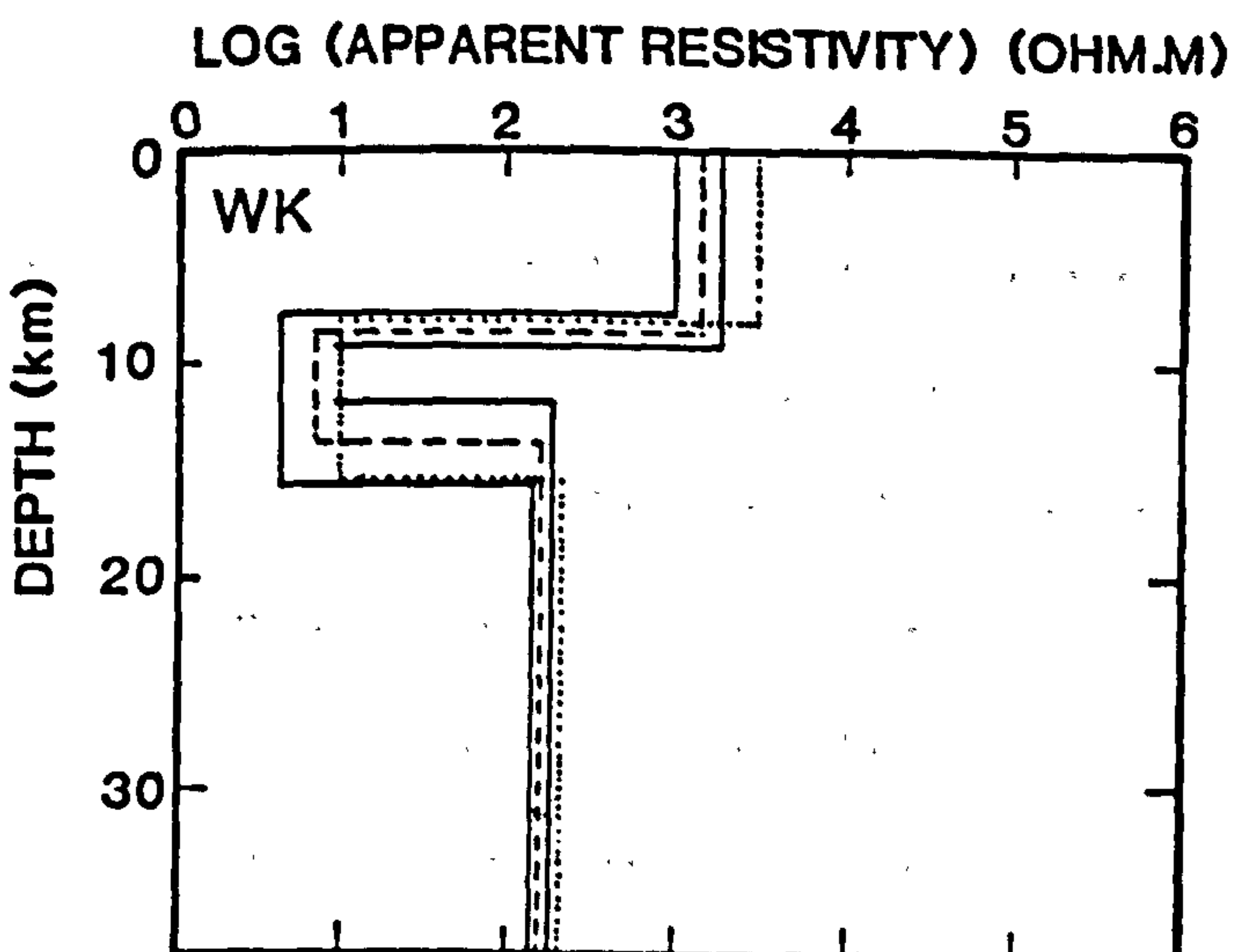
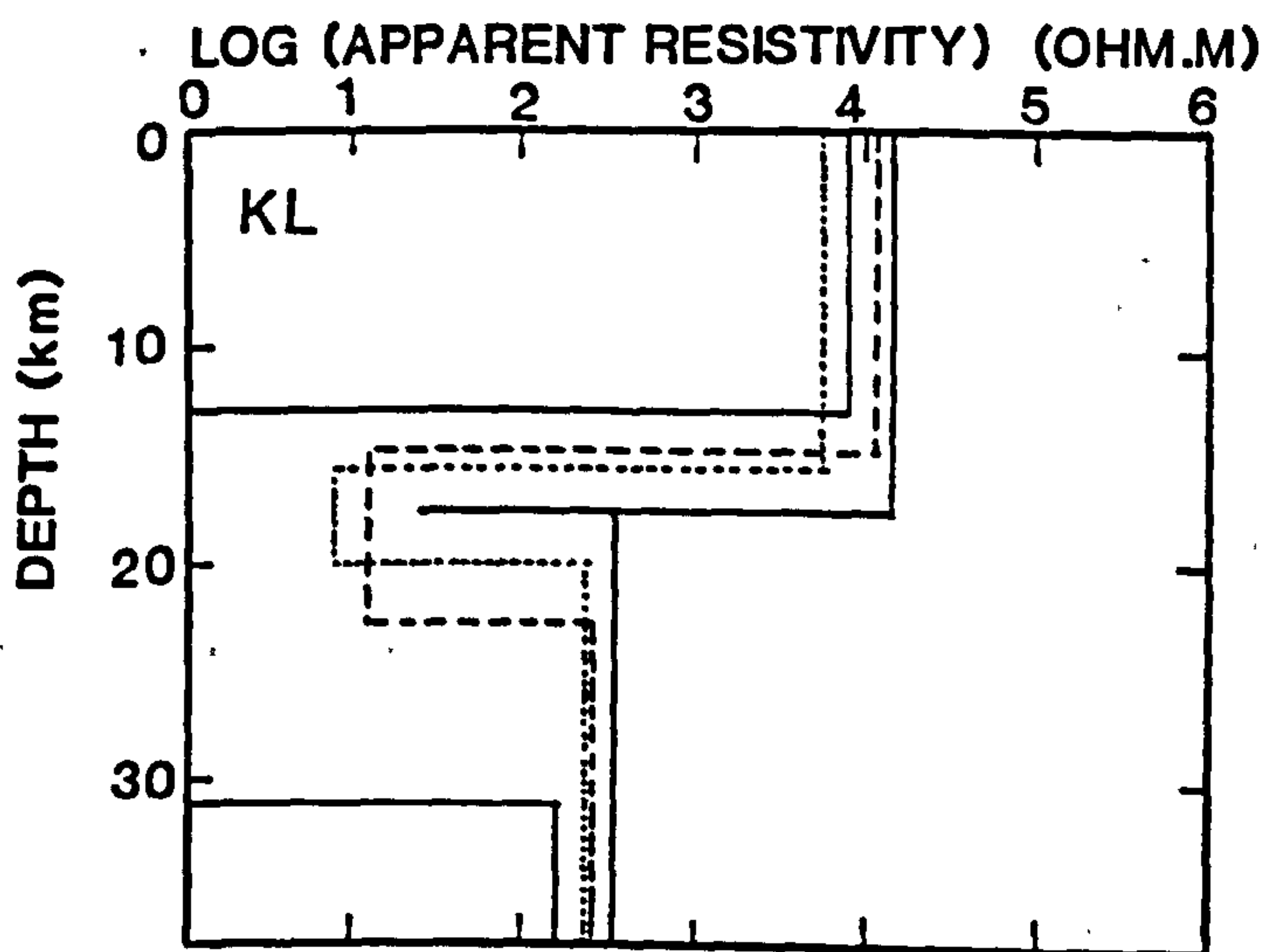
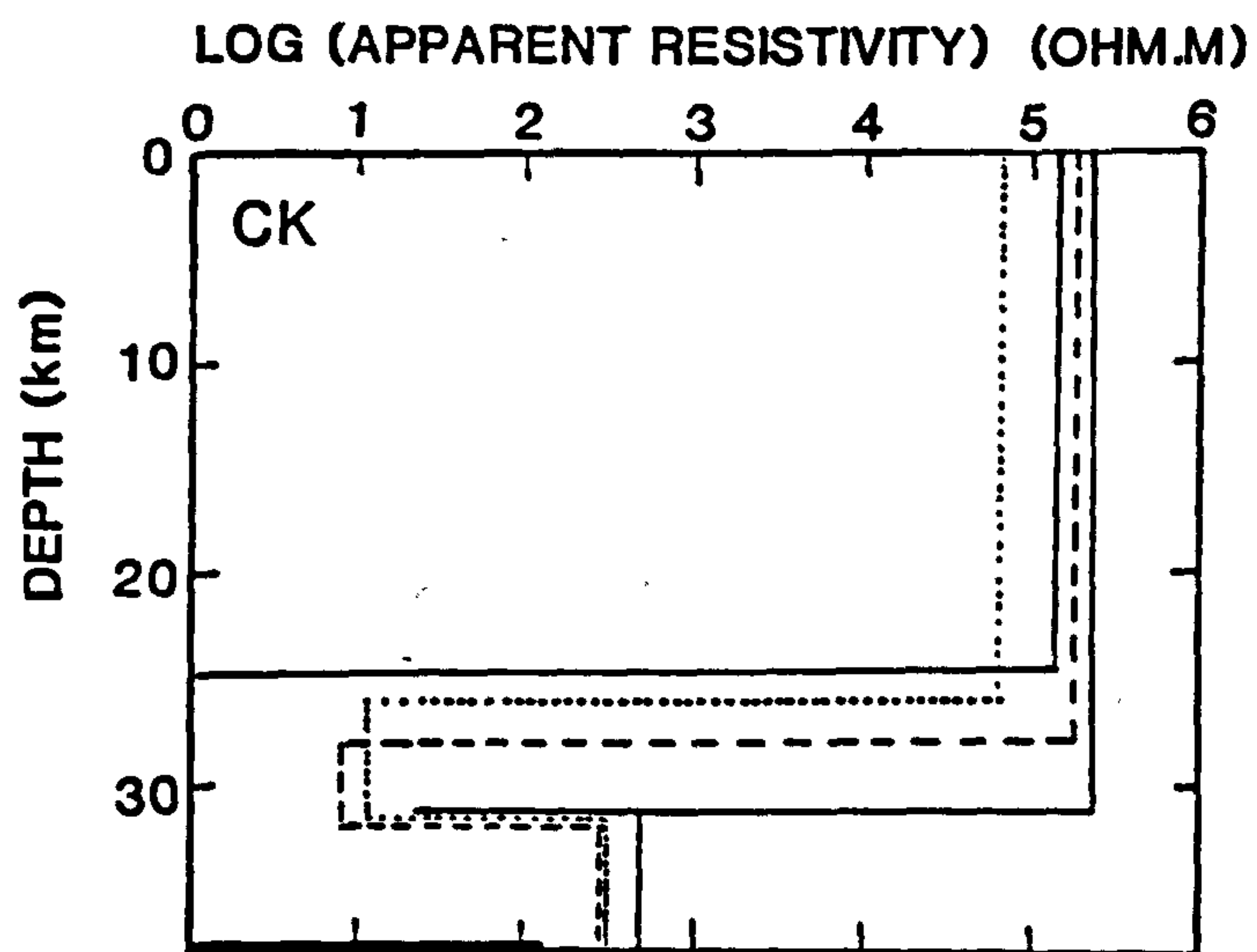


FIGURE F3.14. Resistivity/ depth profiles from MT measurements for three area of England (After Beamish, 1986).

depth due to the effect of overburden pressure on the cracks and pores in the rock. The theory produces predictions two orders of magnitude too low when applied to the mid crust where there is an apparent increase in the rock conductivity. Zones of partial melting have been cited as a possible explanation of this effect but this is only a valid explanation in areas of extremely high heat flow or areas of Tertiary vulcanism. Hydrous mineral conduction and conduction through graphite veins in garnet-graphite granulites have also been suggested as a possible explanations. The most likely cause of the electrical conductivity increase in most localities is the presence of intergranular water but the mechanism by which water occurs in that part of the crust is not known. Two possibilities are that it comes as a release from hydrous minerals during metamorphism of amphibolite facies rocks or it may be carried down to the depths along tectonic zones of underthrusting or at plate boundaries.

Geomagnetic field measurements have indicated a general agreement concerning the overall layering of the crust. The overall picture that is developing from these studies is a crust of about the same thickness as that derived from seismic studies with an intracrustal geo-electrical boundary at a depth of about 16 km which may be the geoelectrical equivalent of the Conrad discontinuity. Some reports suggest that the geo-electrical Moho is indicated by only a small resistivity contrast as electronic conduction is quite important in the upper mantle but the position of this contrast agrees well with values of Moho depth from seismic studies.

3.5. GEOLOGICAL EVIDENCE

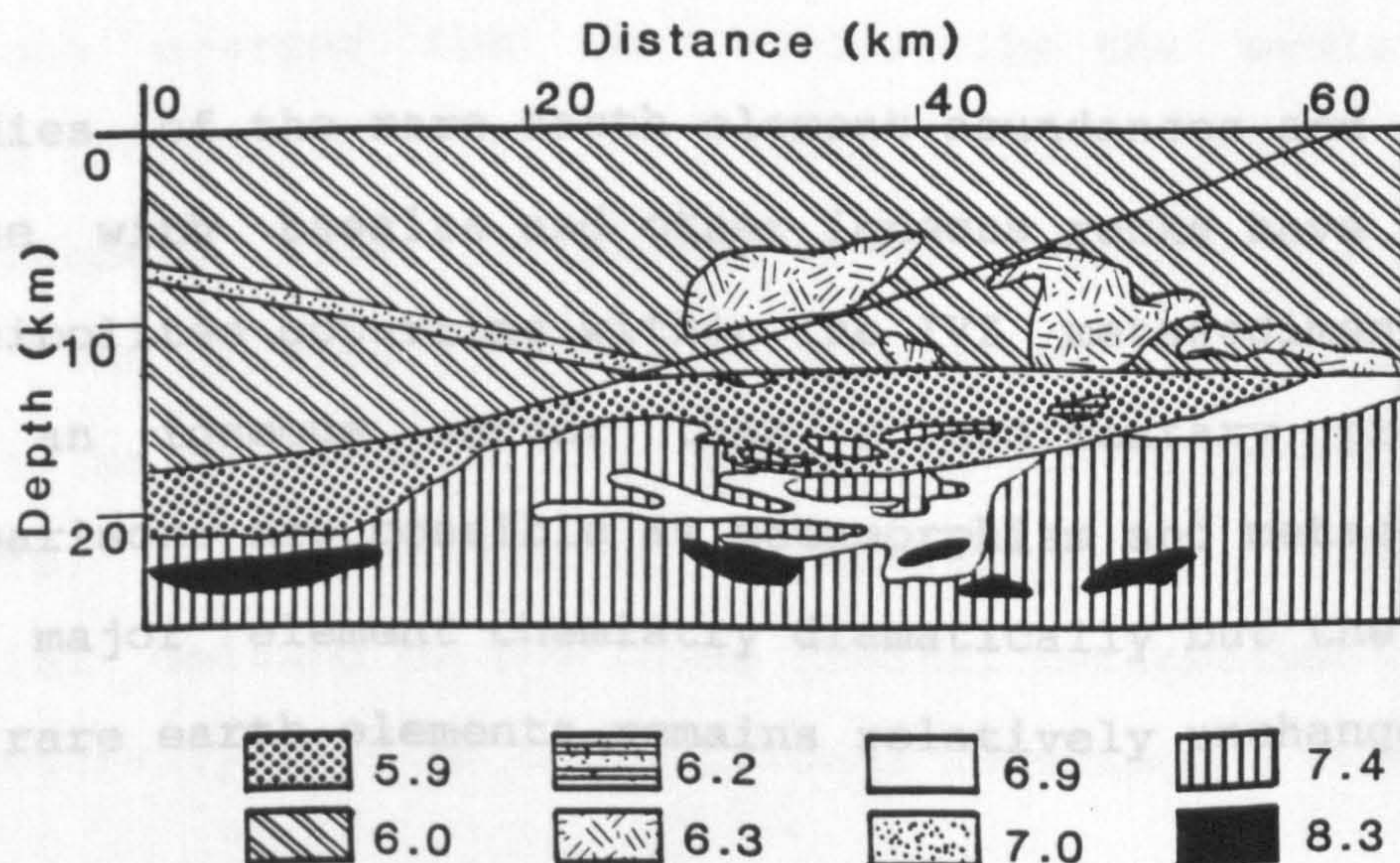
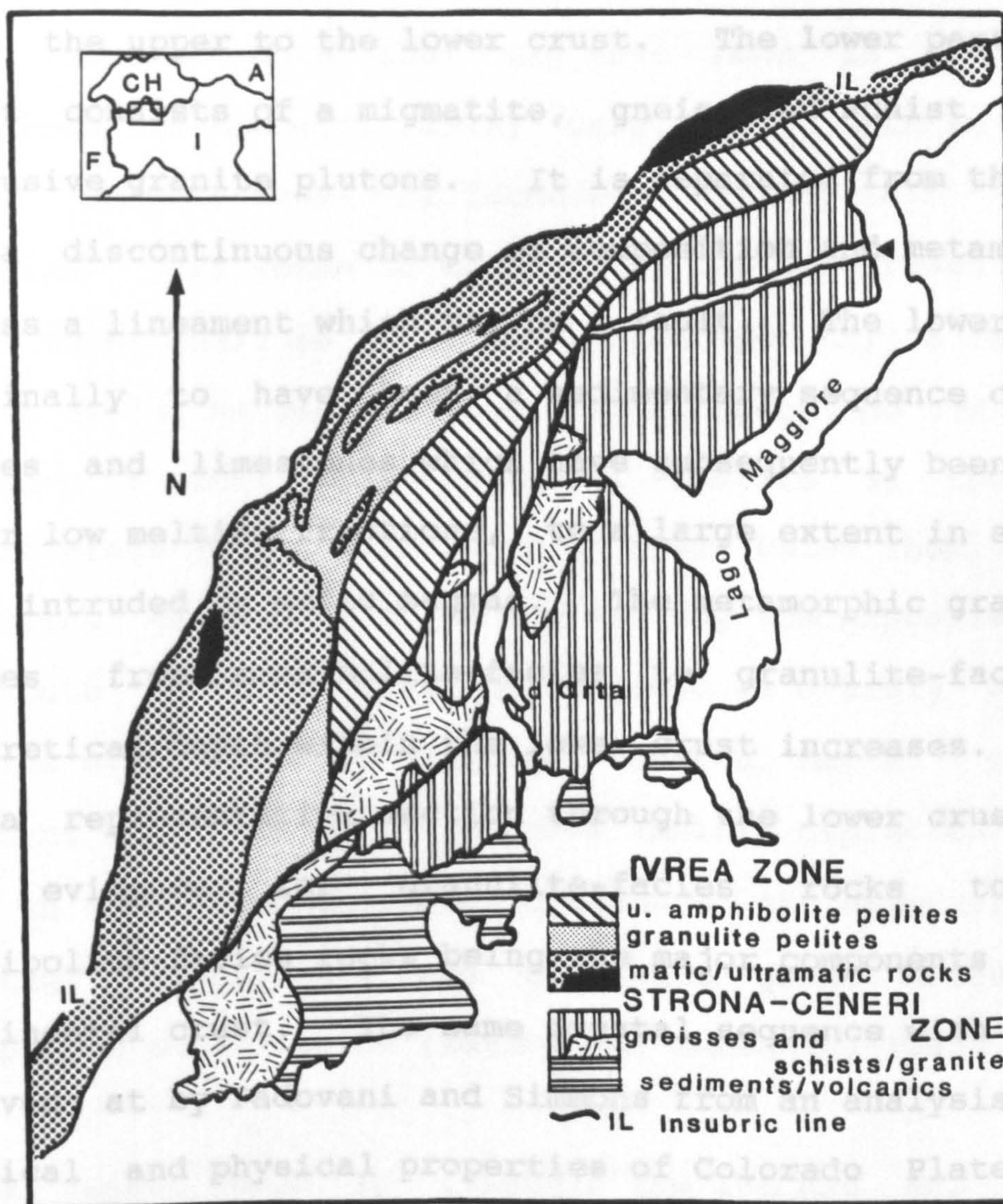
3.5.1. LOWER CRUSTAL EXPOSURES

Crustal cross-sections can give a very useful insight into the chemistry, mineralogy and structure of some regions of the lower crust. Some granulite and amphibolite terrains are of the form that suggest that they are cross-sections of parts of the crust. Accounts of these are numerous (Fountain and Salisbury, 1981; Kay and Kay, 1981). Areas usually quoted are the Kasila Zone, Sierra Leone; Kapukasing Uplift, Canada; Fraser and Musgrave Ranges, W. Australia and the Pikwitonei Subprovince of Canada (Taylor and McLennan, 1985; Percival and Card, 1983).

In North Italy there are some well known zones which seem to show a complete cross-section of the upper and lower crust. The Strona-Ceneri Zone is the exposure of a section of the upper part of the crust and the Ivrea-Verbano Zone is an exposure of a section of the lower crust just below that exposed in the Strona-Ceneri Zone (Fountain, 1976; Fountain and Salisbury, 1981). A general diagrammatic map of this area can be seen as figure F3.15. These zones have been correlated to the Po Basin by seismic velocity data and appear to have been emplaced by thrusting from that region.

The Ivrea-Verbano Zone and the Strona-Ceneri Zone will be referred to as the IVZ and SCZ respectively for the rest of this

FIGURE F3.15. Facies distributions of the Ivrea Zone with a seismic velocity model of the crust that may have produced such a distribution (After Fountain, 1986).



Seismic velocity regimes in metres per second.

review.

The metamorphic grade in the IVZ and SCZ increases continuously from the upper to the lower crust. The lower part of the upper crust consists of a migmatite, gneiss and schist terrain with intrusive granite plutons. It is separated from the lower crust by a discontinuous change of composition and metamorphic grade across a lineament which may be a fault. The lower crust seems originally to have been a sedimentary sequence of sandstones, shales and limestones which have subsequently been depleted in their low melting fractions, to a large extent in some cases and then intruded by mafic magmas. The metamorphic grade of the IVZ ranges from amphibolite-facies to granulite-facies as the theoretical depth within the lower crust increases. If the IVZ is a representative section through the lower crust it adds to the evidence for granulite-facies rocks together with amphibolite-facies rocks being the major components of the lower continental crust. The same crustal sequence with depth is also arrived at by Padovani and Simmons from an analysis of the major chemical and physical properties of Colorado Plateau xenoliths (Padovani and Simmons, 1980).

Studies of the rare earth element abundances and comparisons of these with basalts and other igneous rocks have indicated that amphibolites occurring within the IVZ metasedimentary formation had an igneous rather than a sedimentary protolith. Such comparisons are possible as metamorphism and metasomatism changes the major element chemistry dramatically but the abundances of the rare earth elements remains relatively unchanged and can act

as a memory of the protolith (Sills and Tarney, 1984).

There are many known granulite grade terrains of Pre-Cambrian age that contain rocks that may have been in the lower crust (Glickson and Lambert, 1976; Gray, 1977; Griffin et al, 1978). Large compositional differences occur within this set of terrains. This seems to demonstrate how unrepresentative a lower crust model would be if it were based upon only a few exposures. It is useful to bear in mind that although the IVZ and SCZ seem to give a full cross-sectional exposure of the crust they nevertheless have a full depth of crust below them. This raises important questions as to how and why the exposure evolved to ascertain if it can still be regarded as a truly representative section of the bulk of the lower crust at depth. It is, however, apparent that most of these areas exhibit depletion of K, Pb, U and Th and have undergone a mixture of dehydration, partial melting, amphibolite and granulite grade metamorphism and retrograde metamorphism (Taylor and McLennan, 1985).

In a recent work by Fountain the IVZ and SCZ are used to construct a synthetic reflection seismogram using data from various sources that seek to describe the evolution of these zones (Fountain, preview; Zingg, 1983, 1980; Menhert, 1975; Berckhemer, 1969; Rivalenti et al., 1975). Fountain suggests that the existence of a highly seismically reflective lower crust could be attributed to a combination of underplating by mafic magmas, high grade metamorphism and resulting partial melting of the lower crustal metasedimentary rocks, and structurally induced compositional layering. Compared with the

lower crust the upper crust is seismicly relatively transparent despite its structural complexities because the composition of the upper crustal layers have relatively low acoustic impedance contrasts (Gough, 1986). Fountain recognises the extreme differences in composition between the known areas of lower crustal exposure and suggests that there is considerable variation in the structural nature of the crust. He points out the need for deeper study of this and other lower crustal exposures with special emphasis given to their tectonic history before it is possible to use speculations drawn from lower crustal exposure data to generally describe the lower crust with any degree of surety (Fountain and Salisbury, 1981; Percival and Card, 1983).

Recent work on the IVZ has indicated that the lower crustal rocks not only have a ductile rheology but can also undergo brittle fracture (Brodie and Rutter, 1987). Indeed Brodie and Rutter point out that faulting as a mode of failure in the lower crust may be equally important as ductile flow especially if the rock mass is dominated by high grade metabasalts. They are careful to point out that the mechanism of movement in the high temperature shear zones is not cataclasis but nearer to a localisation of homogeneous flow in the area of 'fault' planes which undergo a high temperature creep process.

3.5.2. XENOLITH EVIDENCE

Kimberlite and nephelinitic magmas have sampled the upper mantle and crust beneath the ancient cratons en route to the surface from their points of origin in the upper mantle and lower crust. Suites of xenoliths of varying mineralogy are found as inclusions in diatremes of kimberlitic and nephelinitic magmas. The study of such suites of xenoliths from various localities can help define the types of mineralogy that are possible at depth beneath these locations.

Xenoliths possess a wider range of chemical and isotopic characteristics than appear in lower crustal exposures. They can be viewed as random samples taken from the Earth's interior and very rapidly transported to the surface. Transport times of the order of one day are usual. This rapid transport enables the rocks to escape massive retrograde metamorphism and avoid long exposures to upper crustal temperatures, pressures and to some extent weathering that has affected the lower crustal cross-sections. They are, however, transported by volatiles and can therefore be altered by them. They are seen as the least equivocal samples of the lower crust to which we have access (Taylor and McLennan, 1985).

The study of xenolith suites in many places favours the hypothesis that there is a compositional discontinuity at the Moho. The bulk of such evidence points to a transition from a mantle of harzburgite and peridotite to a lower crust consisting primarily of granulite-facies rocks (Kay and Kay, 1981).

Dawson has reviewed the upper mantle/crust models that have been based on xenolith suites (Dawson, 1977). There have been several models of the outer layers of the earth based upon xenolith studies. Variously the Moho is believed to consist of three types of transition all of which are broadly similar to a peridotite/granulite-facies transition. The three models are dealt with separately below.

A model based upon the Front Range, Wyoming xenolith suite suggests a transition from basic granulite in the lower crust to a mantle consisting of depleted spinel peridotite with pods of eclogite (Eggler and McCallum, 1974a, b). This model is fairly close to that proposed by Clark and Ringwood (Clark and Ringwood, 1964).

The Kansas xenolith suites suggest a compositional boundary between basic granulite in the lower crust to partly hydrated spinel lherzolite (Brookins and Meyer, 1974).

The xenoliths of the Colorado Plateau seem to suggest more of a hydration boundary between serpentine schist and partly hydrated spinel lherzolite (McGetchin and Silver, 1972). This model is given in figure F3.2. A paper by Padovani and Simmons (1980) describes major element chemical analysis and the measurement of physical properties of Colorado Plateau xenoliths from the Moses Rock dyke and the Mule Ear diatreme. They report that the xenolith suite appears to reflect a crustal sequence with depth that is similar to that found in the IVZ and SCZ. They report an

upper crust of greenschist-facies rocks, an intermediate crust consisting of amphibolite-facies schists, gneisses and occasional granite intrusions and a deep crust that consists of mafic granulite-facies and amphibolite-facies rocks (Padovani and Simmons, 1980).

In all these models the depth of the crust-mantle transition is believed to be at a depth of about 43 km.

Xenoliths from Bournac in the Massif Central, France have also been used to generate a lower crustal composition (Dupuy et al., 1979; Dostal et al., 1981). The lower crustal composition in this region seems to be heterogeneously banded granulite facies rocks with much local variation. The amount of restite appears to be low with most of the remainder being in the form of unmelted granulites but there may be more unsampled restite at depth. This study is in an area with a rather shallower Moho than usual (27 - 28 km). As such the composition may be more typical of more mid-crustal levels. This accounts for the relatively low amounts of restite represented in the xenoliths.

A study of xenoliths from the Lesotho kimberlite pipes (Rogers, 1977; Padovani et al., 1982) has revealed rocks of lower crustal origin consistent with that of residual material following extraction of a partial melt. They are also consistent with rocks of a composition caused by lower crustal metamorphism of cumulates (Rogers and Hawkesworth, 1982).

A study in Spitzbergen (Amundsen et al., 1987) has suggested that

the lower crust consists of mafic granulites with minor amounts of pyroxenites and lherzolites in the lowest parts.

A recent study has shown that selected nodules from one kimberlite pipe in South Australia have P-wave velocities of $6.9-7.7 \text{ km s}^{-1}$ and S-wave velocities of $3.9-4.3 \text{ km s}^{-1}$ (Jackson and Arculus, 1984). They made their seismic wave velocity measurements under pressure and made appropriate corrections for lower crustal temperatures and pressures. The values of seismic wave velocities obtained by Jackson and Arculus coincide with published velocity measurements from southern Australia (Manghnani et al., 1974) and agrees well with claims that the lower crust to a large extent consists of mafic granulites and eclogites as has been suggested by previous seismological and petrological studies.

From a petrological point of view one of the most striking and general results of studies of xenoliths that are thought to come from the lower crust is the overall dominance of mafic granulites, eclogites and amphibolites in the sample set. By using temperature and pressure information inferred from the various methods of geothermometry and geobarometry, the equilibration of minerals suggests that these rocks originate at depths of between 25 and 40 km. Such a composition at this depth implies a relatively high P-wave velocity (greater than or equal to 7.0 km s^{-1}) (Ferguson et al., 1979). Seismological studies of areas suggested by Ferguson et al. have shown agreement of elastic pressure wave values with those obtained from xenolith studies done by Ferguson et al. (Finlayson et al., 1979). These

seismological studies report compressional wave velocities between 7.0 and 7.6 km s⁻¹ in the same depth range.

The inference made by Jackson and Arculus is that the lower crust at least in South-East Australia is composed of or dominated by mafic granulites and eclogites (Jackson and Arculus, 1984). If this is the true composition it is more basic than has often been thought (Kay and Kay, 1981).

In summary it may be said that suites of xenoliths in many areas are of a basic composition (South Africa, Central France, N.W. Africa, W. Germany, S.E. Australia, Colorado and Front Ranges, USA) and many are dominated by basic granulites (Fountain and Salisbury, 1981). Seismic velocities from xenoliths from the depth range 25 - 45 km coincide with those of more basic rocks. This type of composition is not consistent with the more acidic composition suggested by analyses of lower crustal exposures. One argument that has been put forward to explain this is that the naturally less dense, more acidic portions of the lower crust have been preferentially uplifted to form the lower crustal exposures that can be examined on the surface (Taylor and McLennan, 1985). Overall xenolith evidence favours a granulite facies lower crust of a more basic composition than lower crustal exposures support.

3.6. CONCLUSIONS

What broad agreement is there over the structure of the continental crust ? It is probably fair to say the best way to visualise the continental crust is as a complex structure with layering on a large scale and the following features:

1. A high resistivity upper crust of about 16 km thickness.
2. Conrad discontinuity at about 16 km depth traceable by seismics and magnetotellurics but only in some areas.
3. A low resistivity lower crust.
4. Upper crust containing all intracontinental hypocentres, undergoing brittle failure and responding to stress elastically.
5. Lower intra-continental crust aseismic and having a ductile rheology.
6. Upper crust relatively transparent to seismic waves.
7. Lower crust contains many small flat or slightly arcuate seismic reflectors which may be due to layered mylonites or layered intrusions.
8. A reasonably well defined Moho that varies in depth between 30 and 45 km. The Moho is traceable by seismic and magnetotelluric techniques in most areas but there are a few areas in which it is missing. In some localities it has the quality of a first order

discontinuity while in others it seems to be a graded change over many kilometers.

9. Lower crustal rocks are generally thought to be amphibolite and granulite-facies. The granulite facies rocks are preferred to be dry by geochemists as the presence of water would cause retrograde metamorphism despite the fact that dry granulites cannot give the required rise in mid-crustal conductivity that is needed to fulfil electrical observations.

10. There is a considerable lateral variation in the depth and quality of both the Conrad and Mohorovicic transitions.

11. The crust is rather more complex than was at first thought. Complex structuring could be due to extensive deep thrusting, pluton intrusion and underplating. These mechanisms could be of special importance in tectonically active zones and could disturb the overall simple layering so dramatically as to disguise the initial layering.

CHAPTER 4 - SAMPLE DESCRIPTION AND PREPARATION

4.1. INTRODUCTION

Before the electrical conductivity of a rock can be measured it needs to be obtained from the earth and prepared to fit within the high pressure measurement cell. This chapter will briefly treat the way in which the samples were obtained, their description and finally their preparation.

4.2. SAMPLE PROVENANCE

Six of the samples used in this study were obtained by K. Brodie and E. Rutter of the Royal School of Mines, Imperial College, London. The samples were acquired from the Ivrea Zone, N. Italy as blocks from which 25.4 mm cores were drilled. The rock samples used by Brodie and Rutter in their experimental deformation studies come from the same cores (Brodie and Rutter, 1987). For ease of correlation of the two sets of data the labelling system used by Brodie and Rutter has been retained.

The samples are labelled IVD1, IVD2, IVE1, IVE2, MERGOLO21 and MERGOLO22. Their provenance and brief description is given in table T4.1.

Four other rock samples used were collected by S. Brooks from the Lofoten-Vesteralen area of northern Norway in the form of one inch diameter cores. The labelling of these samples was also retained. The exact location from which each of these samples was taken can be found in figure F2.1. in Brooks's thesis



PLATE P4.1. The graphite garnet granulite rock sample
GGRAN in bulk form showing graphite distributed
along the foliations.

(Brooks, 1985) but a brief description is given in table T4.1. These samples are labelled V1, L33B, A8B AND L32.

The final four samples were obtained in bulk form from the Beni Bozere granulite area by Dr A. Nicholas of the Laboratory of Tectonophysics in The University of Nantes, France. One inch cores were drilled parallel and perpendicular to the obvious graphite rich foliations in the rock sample which is shown as plate P4.1. These samples are labelled GGRAN1, GGRAN2, GGRAN3 and GGRAN4. Their provenance and brief descriptions are given in table T4.1.

4.3. SAMPLE DESCRIPTION

This section briefly describes the petrology of the rock samples. Further detail of the optical, Energy dispersive X-Ray and X-Ray Fluorescence analysis is given in sections 4.4 - 4.6. Full descriptions are given as tables T4.2, T4.3, T4.4 and T4.5.

IVD1,IVD2 A pyroxene granulite consisting of
 clino-pyroxene, ortho-pyroxene, plagioclase
 and opaques with minor amounts of biotite and
 hornblende in inclusions.

IVE1,IVE2 An amphibolite consisting primarily of
 amphiboles with associated clino-pyroxene and
 plagioclase.

MERGOLO21
MERGOLO22

Orthopyroxenite (almost monomineralic)
consisting of highly altered orthopyroxene with
minor amounts of olivine, biotite and some
spinel.

V1

Granite consisting of quartz, plagioclase
feldspar and biotite with minor amounts of
amphiboles, sphene and spinels.

L33B

Granodiorite consisting of quartz, perthite and
biotite with minor amounts of amphiboles,
sphene, apatite and spinels.

A8B

Amphibolitic gneiss consisting of hornblende,
pyroxenes, plagioclase feldspar with minor
amounts of biotite and garnet and some olivine,
apatite and spinels.

L32

Amphibolitic gneiss consisting of hornblende,
biotite, plagioclase feldspar, pyroxenes and
some muscovite, olivine, garnet, apatite
and spinels.

GGRAN1
GGRAN2
GGRAN3
GGRAN4

Graphite garnet granulite consisting of large
porphyroblasts of garnet often showing a
rimming of biotite due to retrograde
metamorphism, in a fine granular groundmass of

TABLE T4.1. SAMPLE PROVENANCE AND DESCRIPTION

SAMPLE	AREA	LOCATION	TYPE	FACIES	GRAIN SIZE /mm	ALTER- ATION
IVE1 IVE2	N.ITALY IVREA ZONE	NEAR THE VILLAGE OF NIBBIO, 1/2 WAY UP SECTION	AMPHIBOLITE	AMPHIBOLITE	0.1-0.7	V.LOW
IVD1 IVD2	N.ITALY IVREA ZONE	NEAR THE VILLAGE OF MERGOLLO IN THE LOW- EST STRUCTURAL PART OF THE ZONE	PYROXENE/AMPHI- BOLITE BASIC GRANULITE	GRANULITE	0.1-0.5	V.LOW
MER21 MER22	N.ITALY IVREA ZONE	AS IVE1 AND IVE2	ORTHOPIROXENITE	GRANULITE	0.5-0.8	V.LOW
A8B	N.NORWAY LOFOTEN- VESTERALEN	AUSTVAGOY	GNEISS	AMPHIBOLITE	0.1-0.5	MEDIUM
L33B	N.NORWAY LOFOTEN- VESTERALEN	AUSTVAGOY	GRANODIORITE	-	0.1-0.6	LOW
L32	N.NORWAY LOFOTEN- VESTERALEN	LANGOY	GNEISS	AMPHIBOLITE	0.5-1.5	LOW
V1	N.NORWAY LOFOTEN- VESTERALEN	VESTVAGOY	GRANITE	-	0.01-0.2	LOW
GGRAN1 GGRAN2 GGRAN3 GGRAN4	BENI BOUZERE GRANULITE AREA MORROCO	BB24 NEAR BENI BOUZERE	GRAPHITE-GARNET GRANULITE	GRANULITE	0.05-3.0	HIGH

quartz and plagioclase feldspar. Graphite along veins parallel with the schistosity of the rock. Pyroxenes and spinels in minor amounts.

4.4. OPTICAL MINERALOGY

Thin sections were made of all the samples from rock slices taken as close as possible to that part of the core that was used for the conductivity measurements. An optical polarising microscope was used to make a full examination of the thin sections to identify the major constituents of each sample. The results of this are given in section 4.3. A modal analysis of each rock was done by identification of minerals at a minimum of 750 points but more usually at around 1000 points. The results of the modal analysis are given in table T4.2.

Plates P4.2. - P4.9. show photomicrographs of the samples IVD1, IVE1, MERGOLO21 AND GGRAN3 under polarised light and crossed polars. Plate P4.8., taken at higher magnification than the others, shows a 'thread' of opaque graphite. Most of the graphite in this sample is distributed along planes that are parallel with the schistosity of the rock. Plate P4.8. shows a section through one of the medium sized graphite veins. The schistosity can be seen clearly in Plate P4.1., and Plate 4.9. also shows the sample's granular texture and the way that the schistosity envelopes the large garnet porphyroblasts in the classic helicytic texture.

TABLE T4.2. OPTICAL MINERALOGY

SAMPLE	Qtz	Plag	K-feld	Perth	Bio	Musc	Chl	Epi	Zoe	Cpx	Opx	Ol	Serp	Gnt	Amph	Sph	Apat	Opq
A8B		20.5			1.0					0.2	19.0	0.1		2.0	54.0	0.5	0.2	2.0
L33B	34.0	10.7		52.3	3.5	0.5									1.1	0.5	0.5	0.9
L32	2.0	28.0			35.2					0.8	3.0	0.4		1.0	26.0	0.5	0.5	2.0
V1	57.0			36.0	5.5										0.5	0.2		0.8

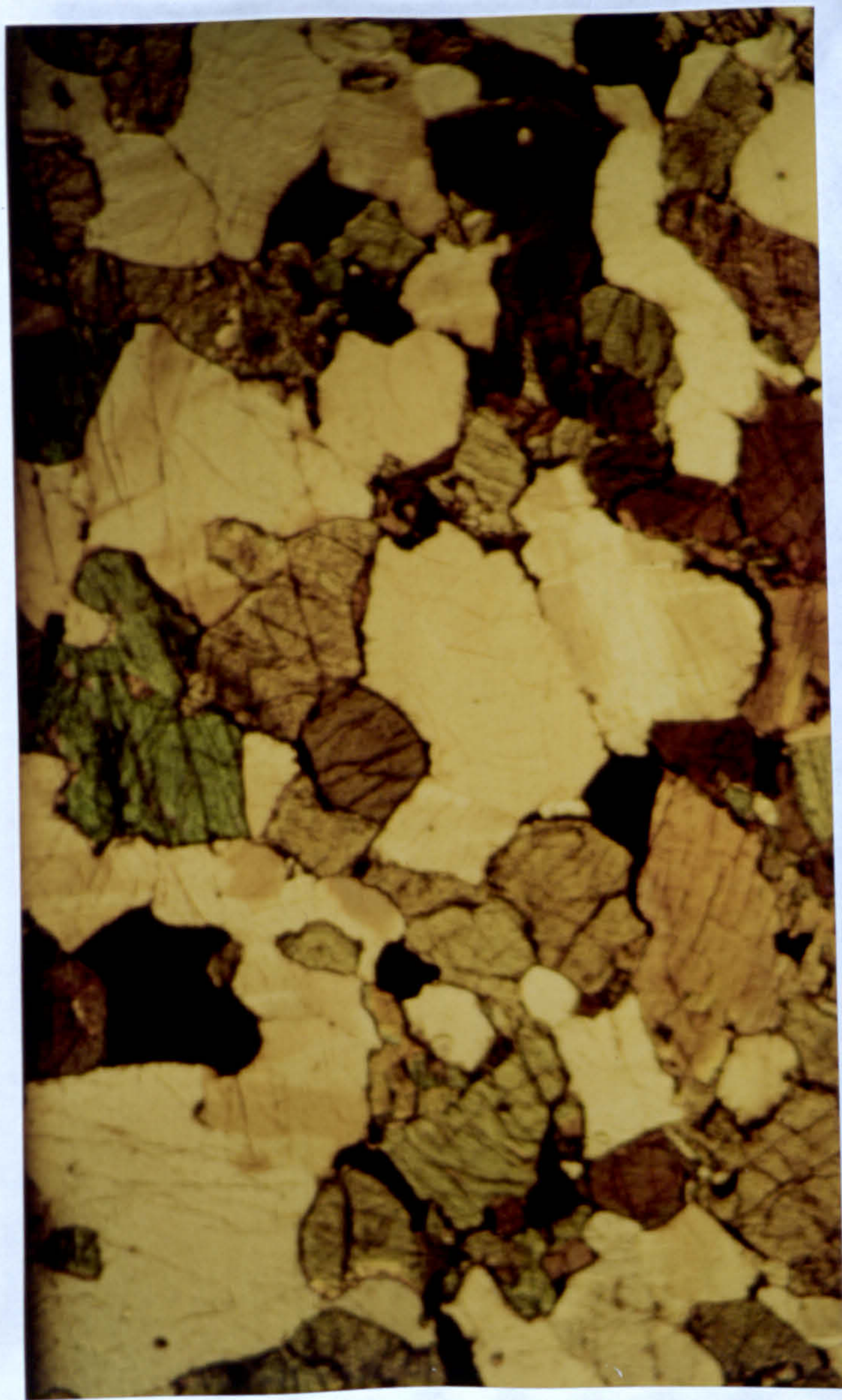


PLATE P4.2. Photomicrograph of sample IVD1 at 25X
magnification in plane polarised light.

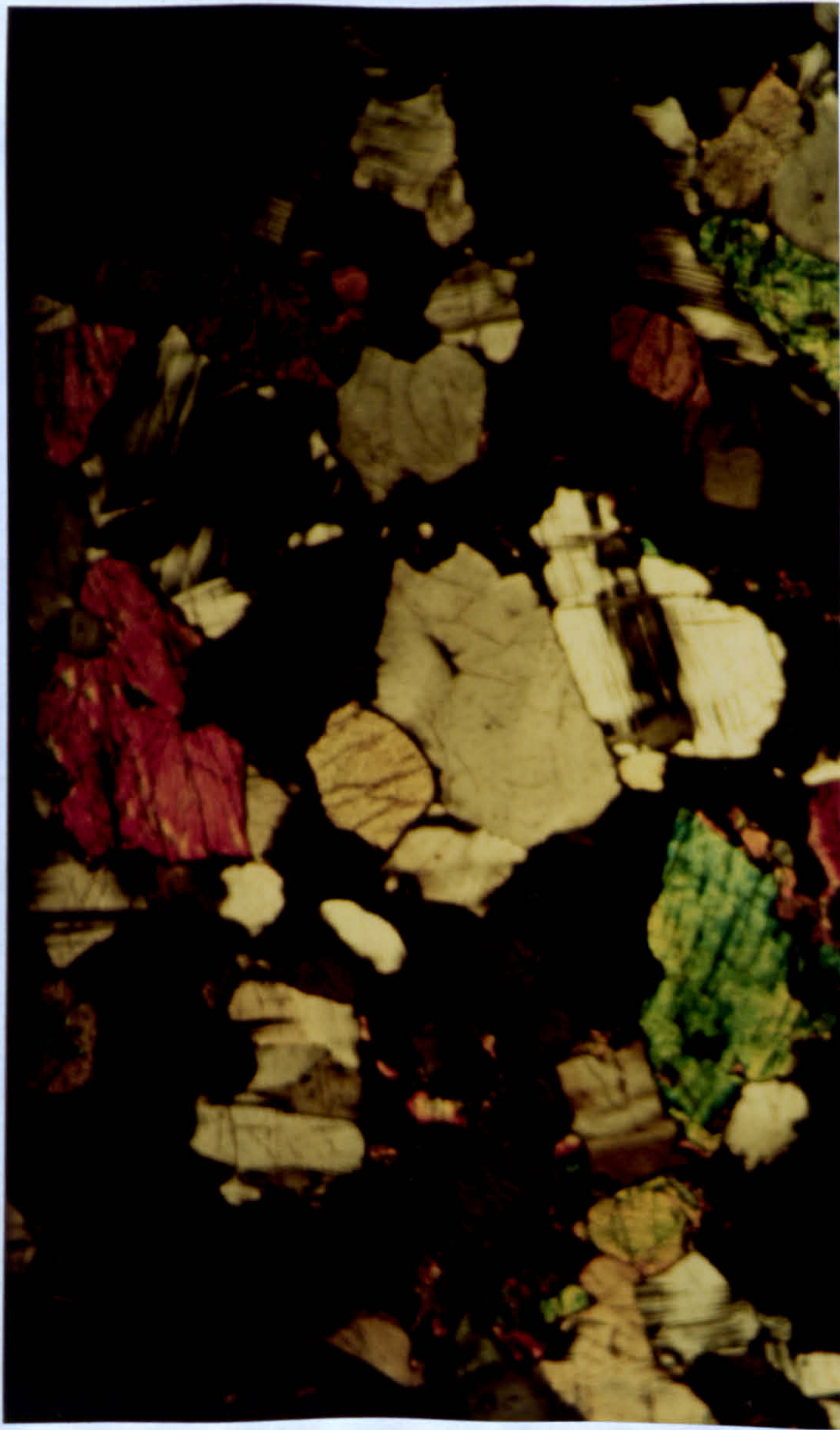


PLATE P4.3. Photomicrograph of sample IVD1 at 25X
magnification through crossed polarisers.

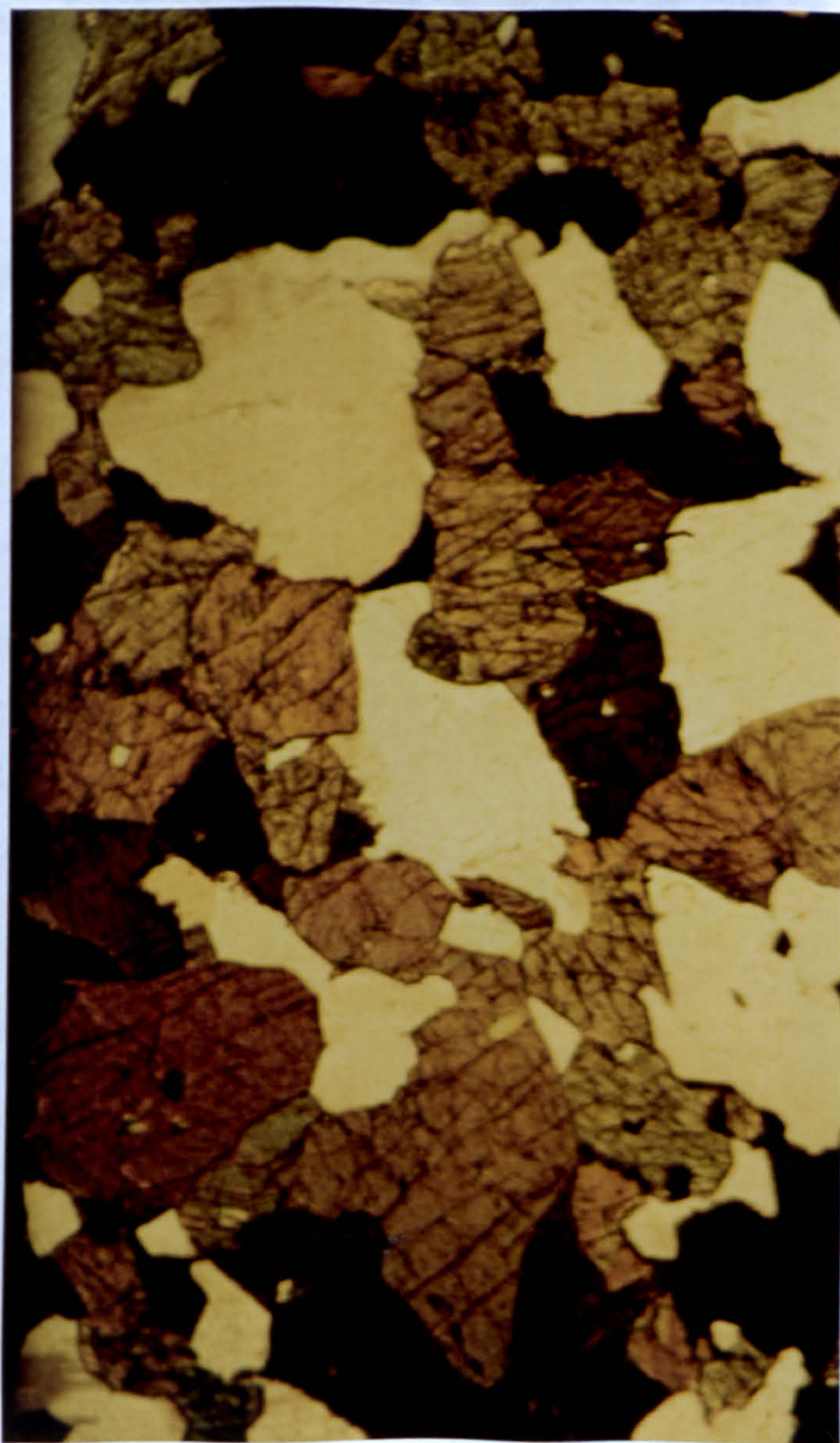


PLATE P4.4. Photomicrograph of sample IVE1 at 25X
magnification in plane polarised light.



PLATE P4.5. Photomicrograph of sample IVE1 at 25X
magnification through crossed polarisers.

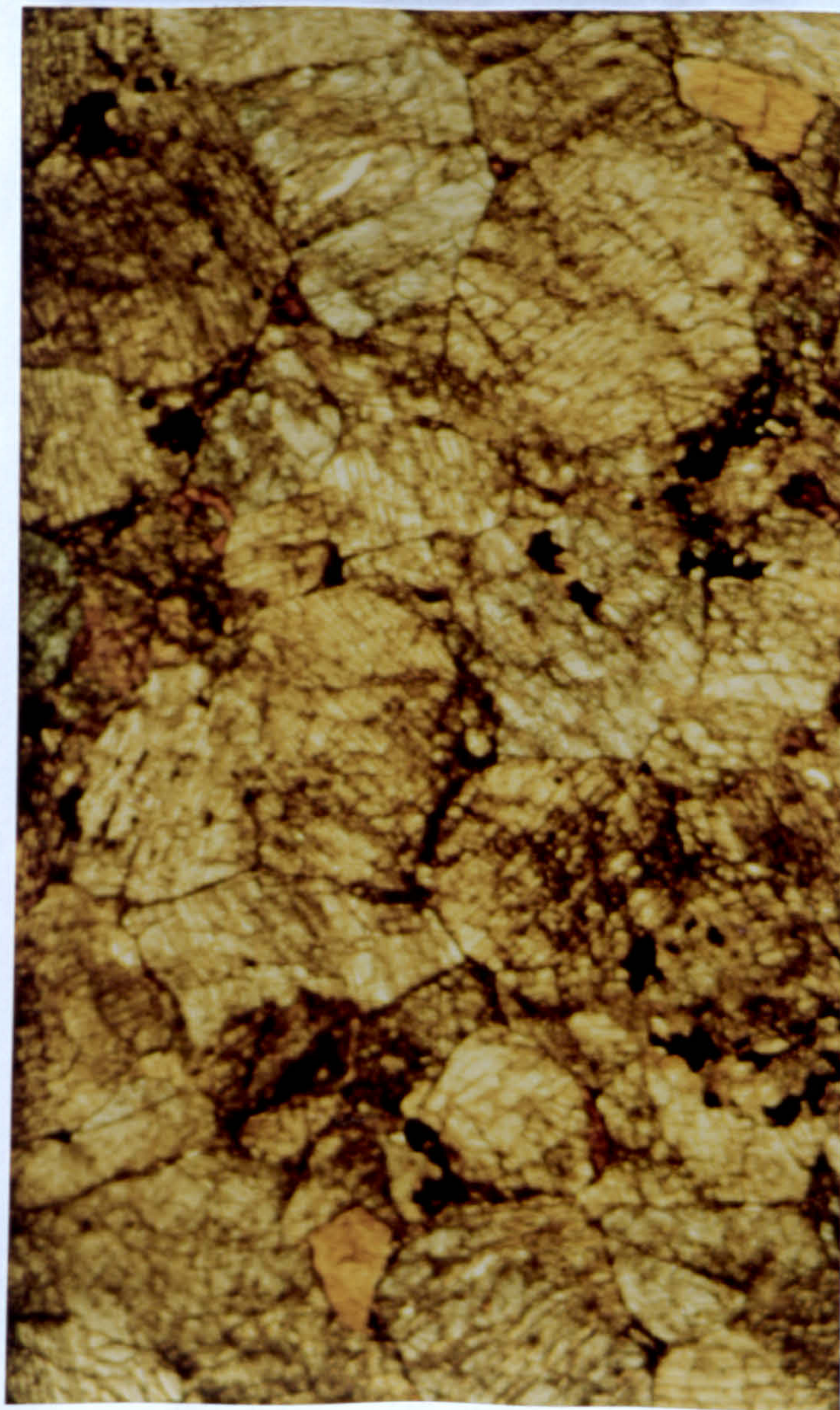


PLATE P4.6. Photomicrograph of sample MER21 at 25X
magnification in plane polarised light.



PLATE P4.7. Photomicrograph of sample MER21 at 25X magnification through crossed polarisers.

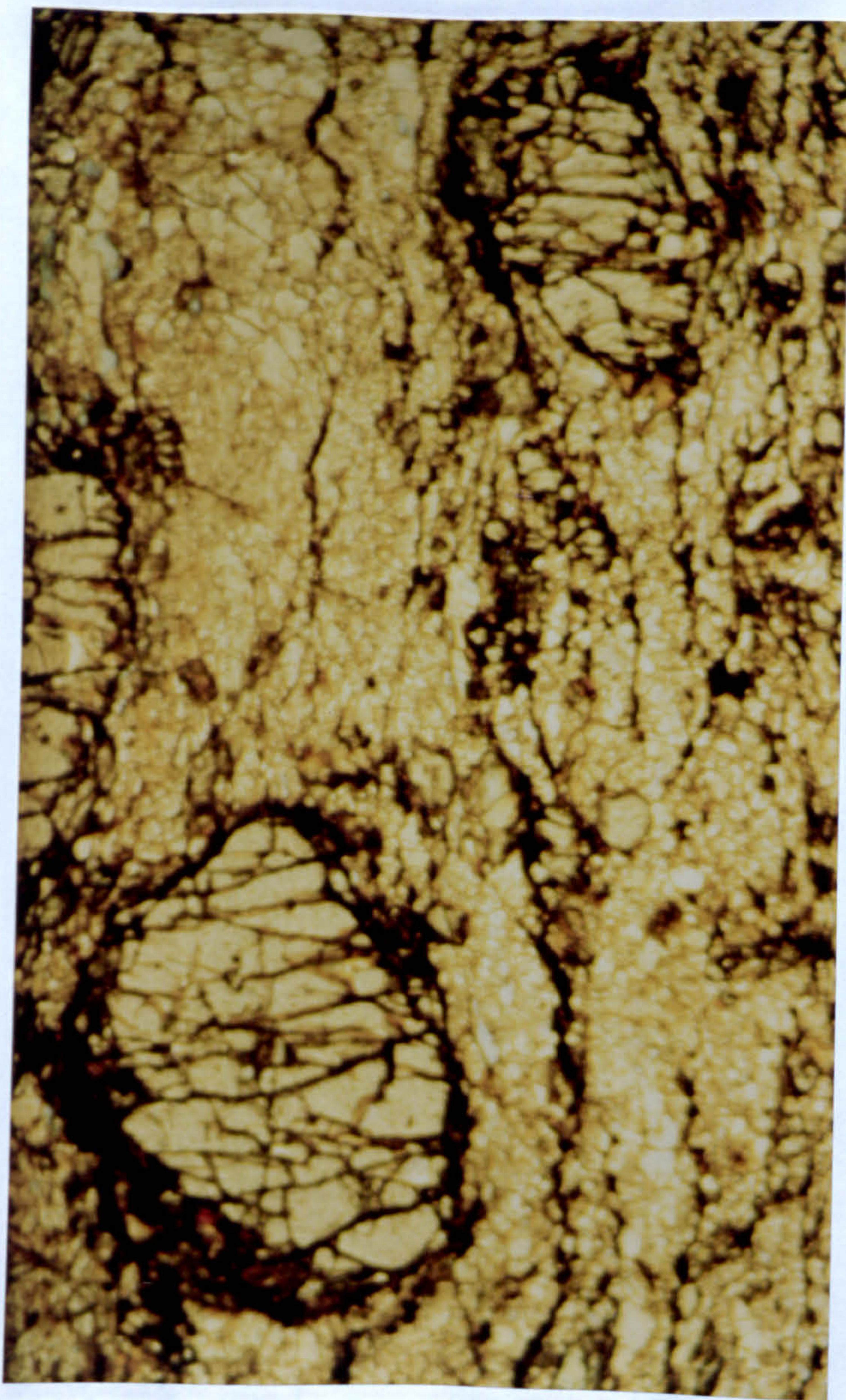


PLATE P4.8. Photomicrograph of sample GGRAN1 at 25X
magnification in plane polarised light.

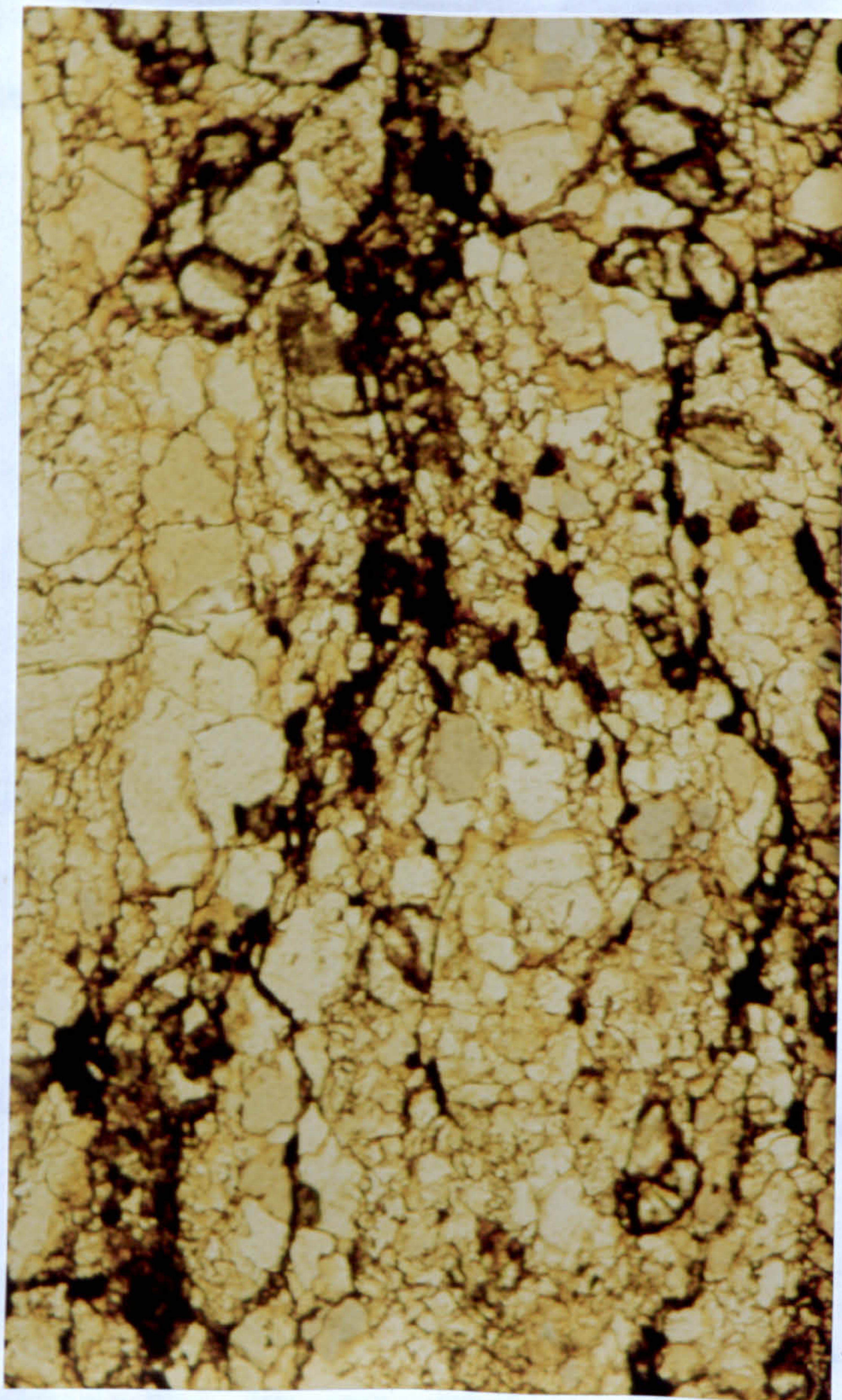


PLATE P4.9. Photomicrograph of sample GGRAN1 at 63X
magnification in plane polarised light.

4.5. X-RAY FLUORESCENCE ANALYSIS

Sections of core from each of the four rock samples IVE2, IVD2, MERGOLO21 and GGRAN4 were taken and prepared for X-Ray Fluorescence analysis. Two types of sample were needed;

- Fused beads, for major element analysis
- Powder Pellets, for minor element analysis

The powder pellets were prepared at UEA in the following way. The rock samples were crushed to a fine powder on a Tema swing mill until the rock flour passed through B.S. 250 mesh bolting cloth. The powder was then dried at 105 °C for eight hours. The powder was mixed with phenol formaldehyde (B.B.Co.-resin R0214) resin, pressed into a tablet using a high pressure uniaxial press and a loading of 15 tonnes and then baked at 110 °C for 30 minutes.

Fused beads were prepared from fine rock powder by the Geology Department of the University of Nottingham.

The X-Ray Fluorescence measurements were done at the N.E.R.C. X.R.F. facility at the University of Nottingham on a Phillips PW1400. The results are shown in tables T4.3. and T4.4. The results shown in table T4.3. do not add up to 100% due to the lack of minor elements in this analysis and a certain small systematic error.

TABLE T4.3. MAJOR ELEMENT X-RAY FLUORESCENCE ANALYSIS

SAMPLE	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	LOI	TOTAL
IVD1 IVD2	48.10	14.33	1.35	12.09	8.26	13.12	2.10	0.08	0.19	0.12	0.48	100.22
IVE1 IVE2	46.72	13.68	1.22	12.07	7.77	14.90	2.24	0.58	0.22	0.10	0.38	99.86
MER21 MER22	51.51	5.08	0.25	11.87	28.35	2.19	0.06	0.00	0.20	0.01	0.15	99.66
GGRAN1 GGRAN2	59.88	20.30	1.21	9.11	2.39	1.65	0.75	2.50	0.12	0.12	2.00	100.03
A8B	68.10	16.2	0.46	3.31	1.44	3.27	5.02	2.03	0.03	0.07	0.44	100.37
L33B	67.2	16.1	0.45	4.01	1.80	3.80	3.90	2.58	0.07	0.03	0.15	100.09
L32	69.08	15.0	0.35	5.20	1.07	3.07	4.09	2.01	0.06	0.10	0.46	100.49
V1	73.00	13.5	0.21	2.56	0.72	1.86	4.02	4.01	0.08	0.02	0.17	100.15

Figures are given in weight percent oxide. LOI = Loss on ignition to 1100 degrees C.

TABLE T4.4. MINOR ELEMENT X-RAY FLUORESCENCE ANALYSIS

SAMPLE	Ba	Ce	Co	Cr	Cu	Ga	La	Ni	Nb	Pb	Rb	Sr	Th	U	V	Y	Zn	Zr
IVD1	44	13	116	229	103	18	< 4	81	2	8	2	114	6	< 1	280	34	74	61
IVD2	47	10	100	235	87	15	< 4	95	3	8	3	125	7	< 1	303	31	69	55
IVE1	INSUFFICIENT POWDER WAS AVAILABLE FOR THIS ANALYSIS																	
IVE2	INSUFFICIENT POWDER WAS AVAILABLE FOR THIS ANALYSIS																	
MER21	< 8	25	164	3121	217	10	< 4	1424	< 1	10	3	10	1	< 1	177	5	76	11
MER22	< 8	21	175	3307	219	12	< 4	1375	< 1	11	4	13	1	< 1	186	4	80	9
GGRAN1	934	81	111	162	50	27	23	67	16	16	78	257	14	< 1	181	47	120	240
GGRAN2	1020	83	101	173	55	29	20	75	21	21	79	278	16	< 1	194	53	127	265
A8B	710	68	80	30	44	15	37	20	6	20	78	595	7	< 1	159	7	59	195
L33B	970	40	39	29	2	20	31	20	4	24	130	693	14	4	48	8	40	151
L32	696	60	87	34	40	14	36	18	4	22	81	611	6	< 1	140	5	57	201
V1	1010	27	30	31	4	26	32	17	6	28	142	721	20	5	39	7	35	142

Figures are given in parts per million.

The mean atomic weight, $\bar{A}$, of each sample was calculated by taking a weighted mean of the major oxide analysis.

$$\bar{A} = \sum_i \left\{ \frac{w_i m_i}{n_i} \right\} \quad \text{E4.1.}$$

where w_i is the fraction of the rock in the form of oxide i

m_i is the molecular weight of the oxide i

n_i is the number of atoms in oxide i

An assumed molecular weight of 22.00 has been used to represent the discrepancy due to the lack of minor elements in the analysis. Mean atomic weights are shown in table T4.3.

4.6. SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS

Rock from near four of the samples (IVD1, IVE1, MERGOLO22, GGRAN1) was taken and examined under a scanning electron microscope. A 'Stereoscan' SEM Type 96113 Mk 2A with a maximum resolution of 0.05 micrometers was used. A Link Systems EDS energy dispersive X-ray analyser, permanently attached to the SEM was used to make a chemical analysis of the sample in bulk, on individual minerals and also to map elements on the surface of the samples.

The nature of the energy dispersive X-ray instrument is such that elements with an atomic number below sodium cannot be detected. The inability to detect oxygen may at first seem a great

disadvantage in analysing most rocks but the certainty that most of the elements in most rocks are in the form of their relevant oxides enables a matrix of oxygen to be assumed. A minicomputer can then be used to compensate for the inability to detect X-ray peaks from the oxygen matrix.

Chemical analysis of the 'whole rock' was done in the following way. The SEM was set to a low magnification (35X), enabling as large an area of rock to be analysed as possible (2.50 mm by 3.00 mm), the area analysed being that shown on the SEM screen during analysis. Six such analyses were done on different areas of each rock sample to maximise the area of rock analysed and reduce the statistical sampling error. The initial output of each analysis is the X-ray energy spectrum of the X-rays produced by bombarding the surface of the sample with fast electrons. Examples of the energy spectra from three of these analyses on three different rock samples are shown in figure F4.1.a,b, and c. The energy spectrum from each analysis was processed by the instruments computer in the following way. The computer was allowed to compare a simulated energy spectrum generated from user picked peaks arising from elements known to be present from the measured energy spectrum. The computer then varied the relative amplitudes of the peaks until the simulated spectrum matched that observed. The background energy was then removed and adjustments made to account for the presence of an oxygen matrix. Such analyses were done on all six of the measurements on each rock. The analyses were averaged to give the chemical analyses given in table T4.5.

TABLE T4.5. ENERGY DISPERSIVE X-RAY ANALYSIS

SAMPLE	Si	Al	Fe	Mg	Ca	Na	K	Ti	S	Mn	Cr	P	Oxide Matrix	TOTAL
IVD1 IVD2	18.726	8.888	6.778	3.308	9.882	2.684	0.098	0.814	0.144	0.126	0.052	0.08	48.446	100.026
IVE1 IVE2	18.592	5.973	7.608	4.637	12.973	2.698	0.305	0.693	0.313	0.202	0.055	0.082	45.866	99.997
MER21 MER22	20.051	3.423	7.527	14.892	2.313	0.268	0.016	0.158	0.115	0.175	0.302	0.043	50.880	100.171
GGRAN1 GGRAN2 GGRAN3 GGRAN4	22.062	10.321	5.824	3.722	2.002	2.122	1.560	0.699	0.323	0.111	0.065	0.090	51.097	99.998

All figures % composition by weight

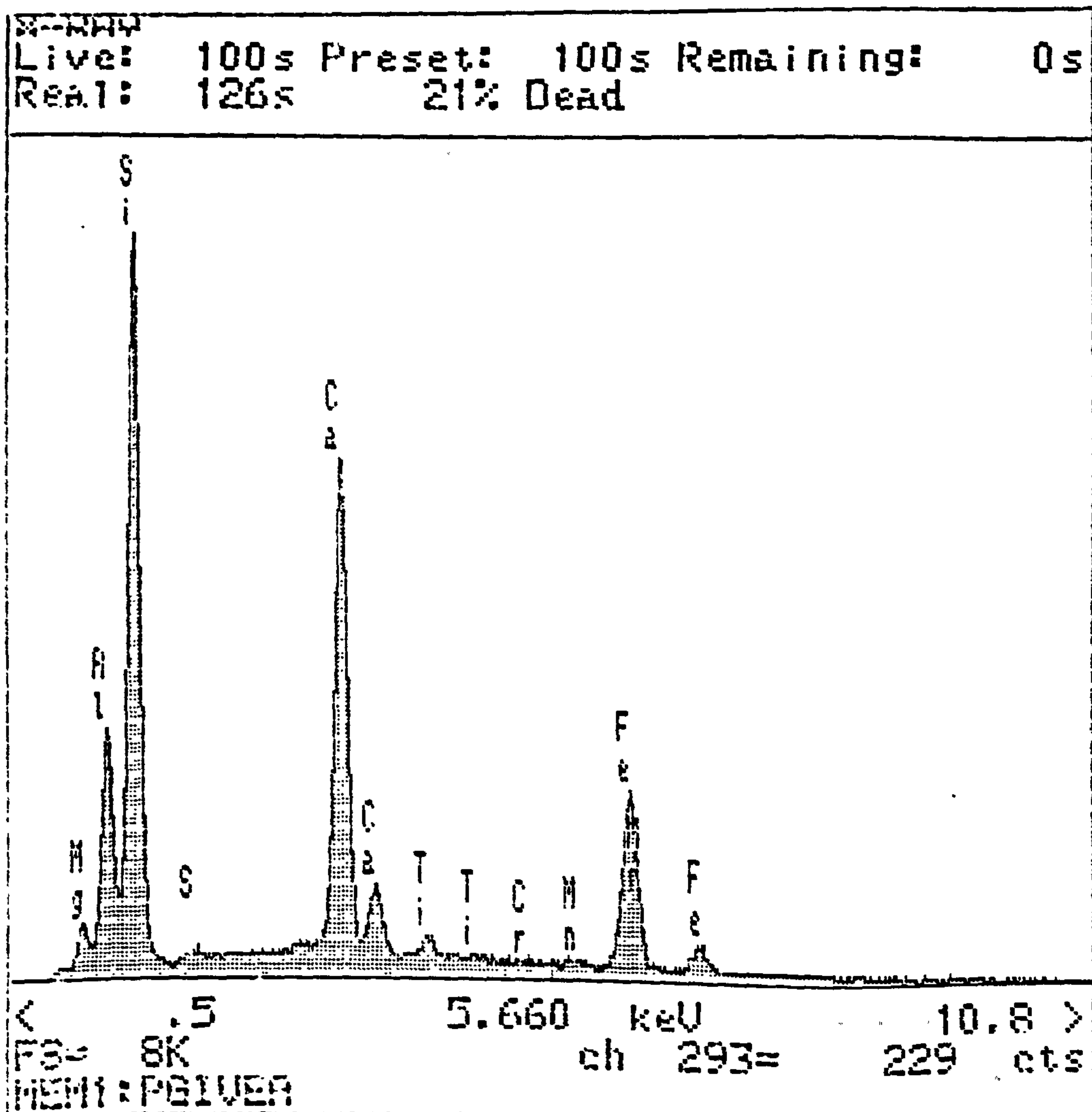


FIGURE F4.1.a. The energy dispersive X-ray spectrum for the basic granulite sample IVE1.

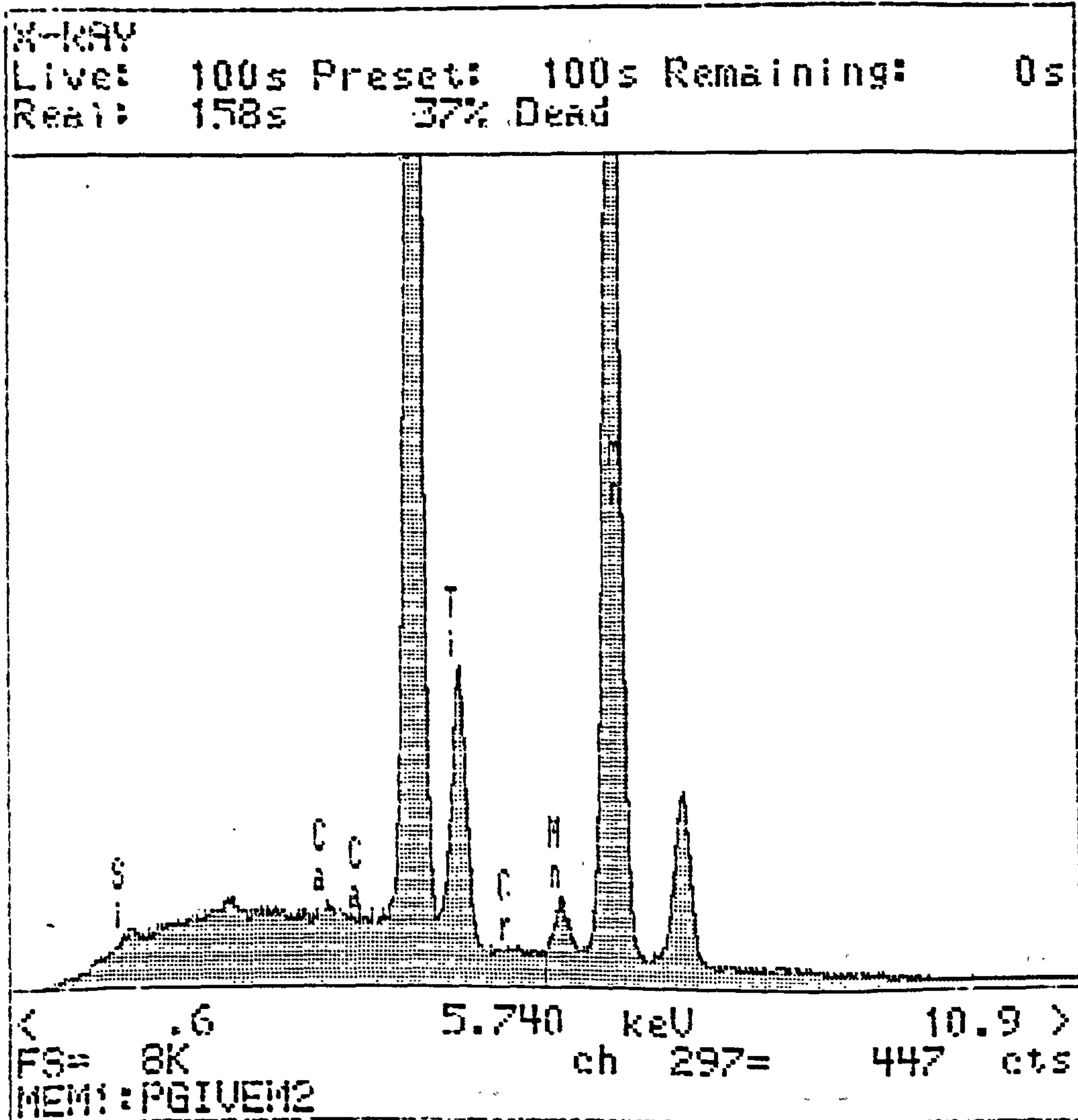


FIGURE F4.1.b. The energy dispersive X-ray spectrum for an Ilmenite grain in the basic granulite sample IVE1. Quantitative analysis of this spectrum has indicated almost pure ilmenite composition by weight with a slight excess of titanium.

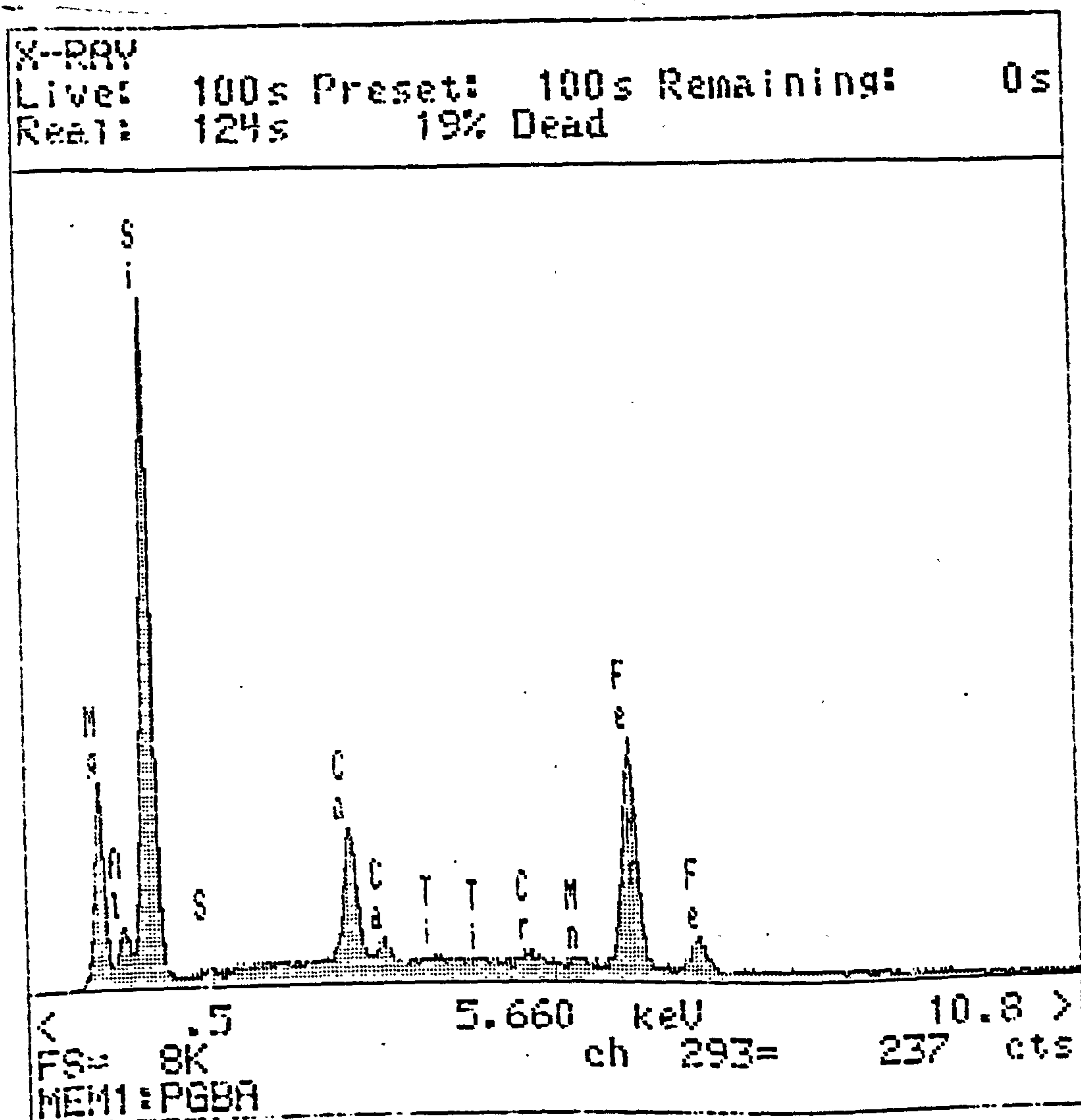


FIGURE F4.1.c. The energy dispersive X-ray spectrum for the orthopyroxenite sample MER21.

The area of analysis can be restrained by increasing the magnification of the SEM. This enables the analysis of single crystals and is therefore a powerful tool in mineralogical work. Comparison of the relative concentrations of elements in a mineral can give valuable information about the extent of solid solution. In this work the technique has been used to confirm the optical mineralogy and obtain the exact chemical composition of various minerals in the rock. Individual analyses have been carried out on a wide range of minerals in these rocks.

Comparison of the chemical composition arising from a range of individually analysed minerals with that of the 'whole rock' as described earlier should enable a modal analysis to be calculated. This needs extra work and remains a non-trivial problem due to the following difficulties;

- Highly multi-mineralic nature of the rocks
- The chemical complexity of the minerals especially those that are hydrous
- Subtle or no differences in proportion of elements in very different minerals due to changes in structure
- Diadochy (solid solution)
- Impurities

Plates P4.10. and P4.11. show scanning electron micrographs of a part of GGRAN1 especially chosen to show an area rich in graphite. It shows a large garnet crystal surrounded by an area of dark graphite and other minerals. Plate P4.10. shows the

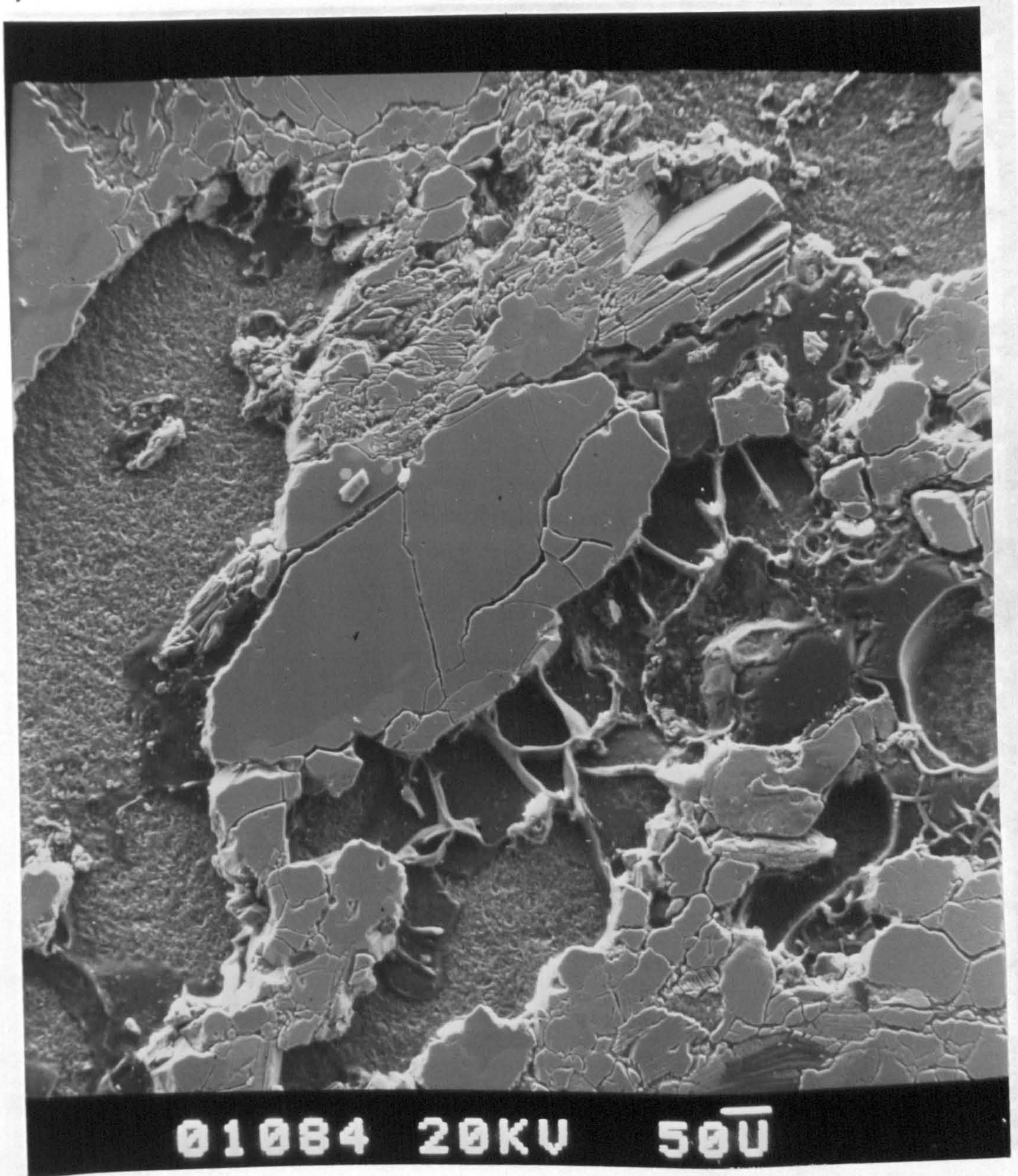


PLATE P4.10. Scanning Electron Micrograph of the graphite granulite sample GGRAN1 in reflection mode showing surface topography.

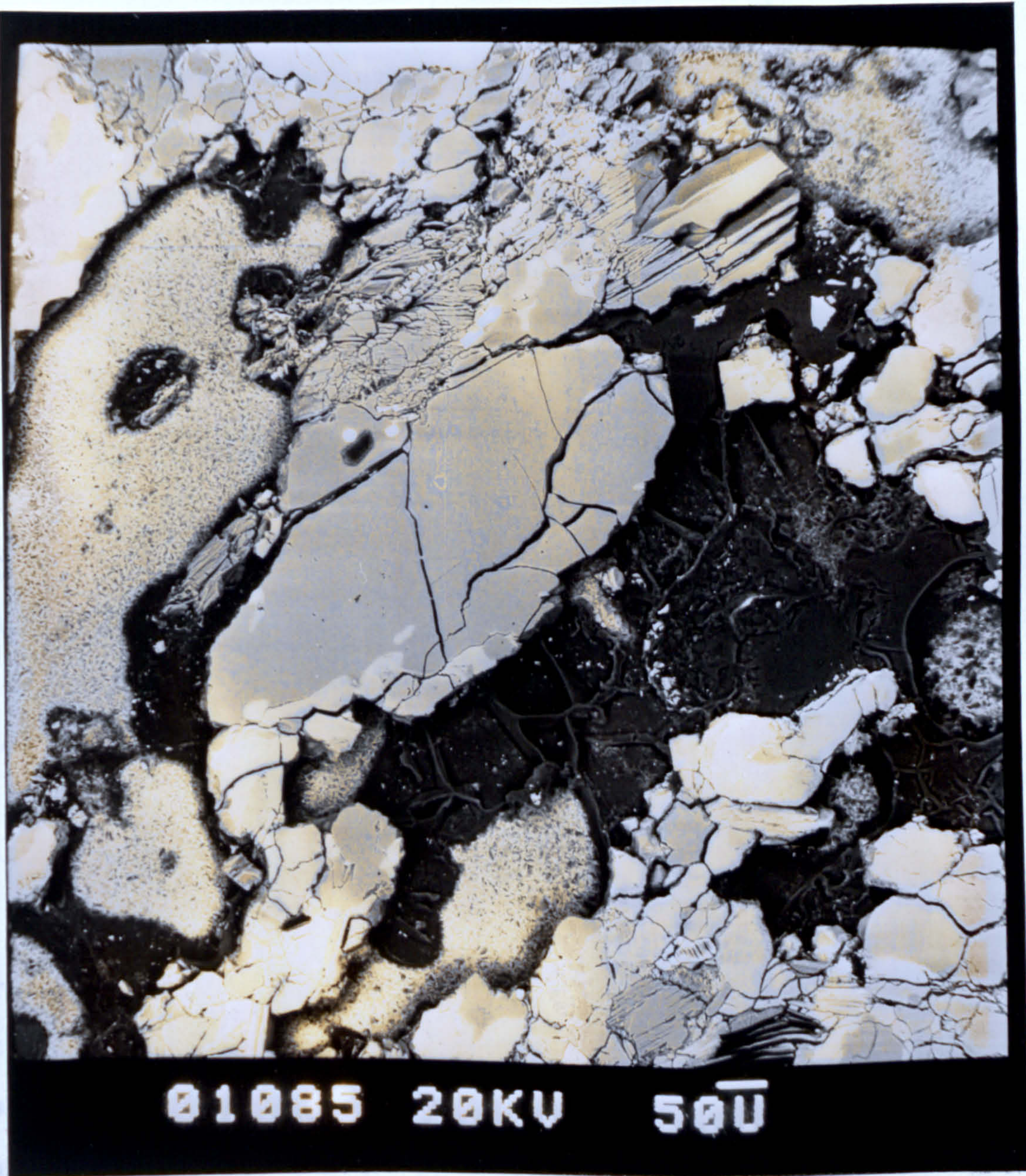


PLATE P4.11. Scanning Electron Micrograph of the graphite granulite sample GGRAN1 in secondary electron emission mode showing differences in mean atomic weight.

surface in reflection mode which delineates surface topography. Plate P4.11. is a secondary electron image. The secondary electron image shows areas of different mean atomic weight as different shades, the higher the mean atomic weight, the lighter the representation. This is particularly useful in the analysis and differentiation of opaque minerals. Spinel minerals have a high mean atomic weight due to the presence of iron, chromium, titanium etc and appear as light areas such as those just above the large crystal in plate P4.11. Graphite has a low mean atomic weight and therefore shows up as very dark. In optical mineralogy both would appear opaque as in plate P4.8. which shows a proliferation of graphite in veins but also some opaque spinels. Development along these lines to enable the weight percent of spinels in a sample to be calculated would be of interest considering the possible importance of this group of minerals in rock electroconductivity.

The energy dispersive X-ray analysis can also be used to construct elemental maps of the scanned area. Plates P4.12. and P4.13. show distributions of various elements in such a map form in the same area as represented in plates P4.10. and P4.11. These distributions are derived from the secondary electron image and the relative concentrations of the various elements at each pixel location into which the image is divided. The image processing ability of the system can be used to image the graphite distribution even though the carbon cannot be detected by the instrument. This was done in the following way: Elemental distribution maps such as those shown in plates P4.12. and P4.13. for all the detected elements were added together. A weighted

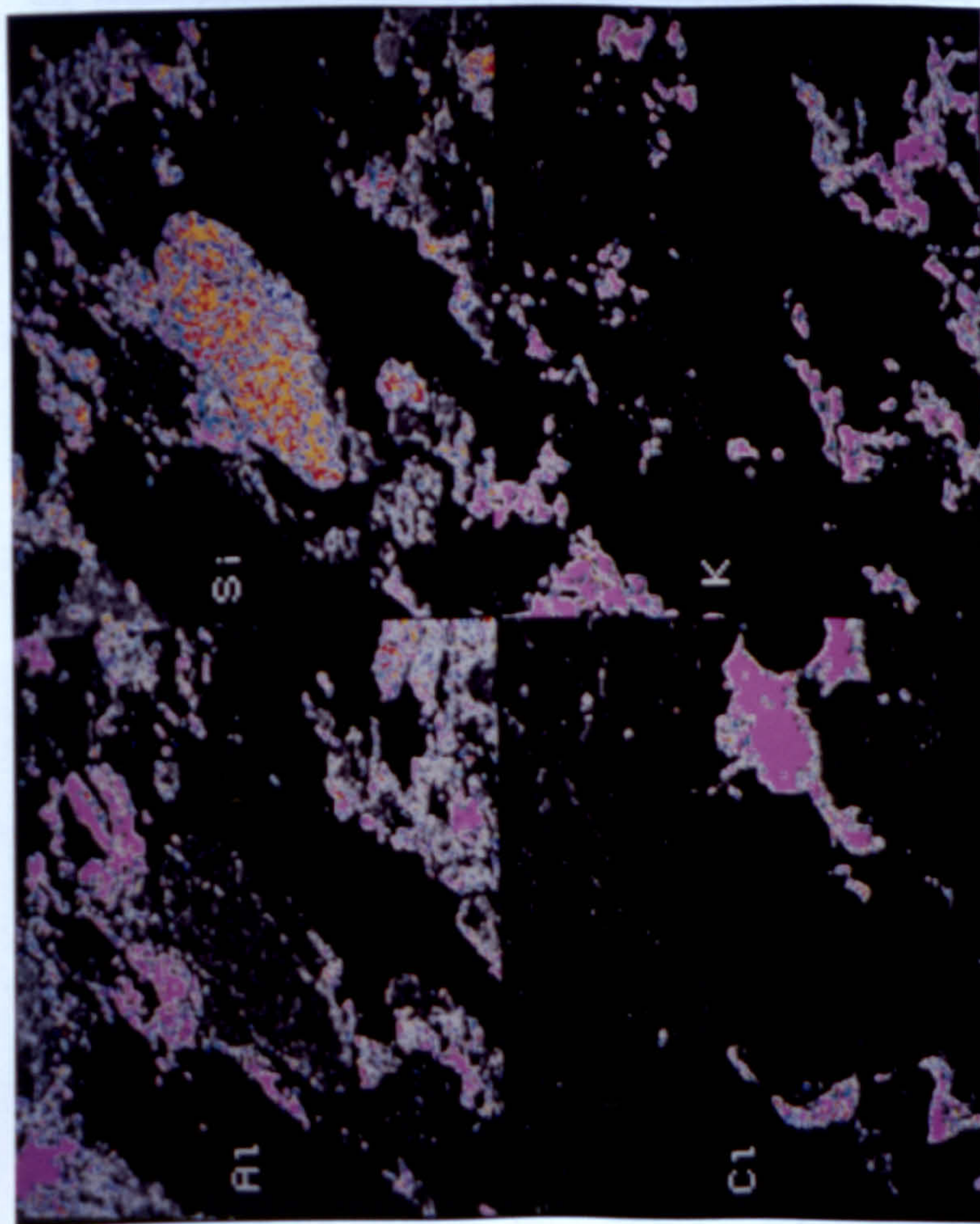


PLATE P4.12. Image processed element maps of the area shown in Plates P4.10. and P4.11. showing the distribution of aluminium, silicon, chlorine and potassium.

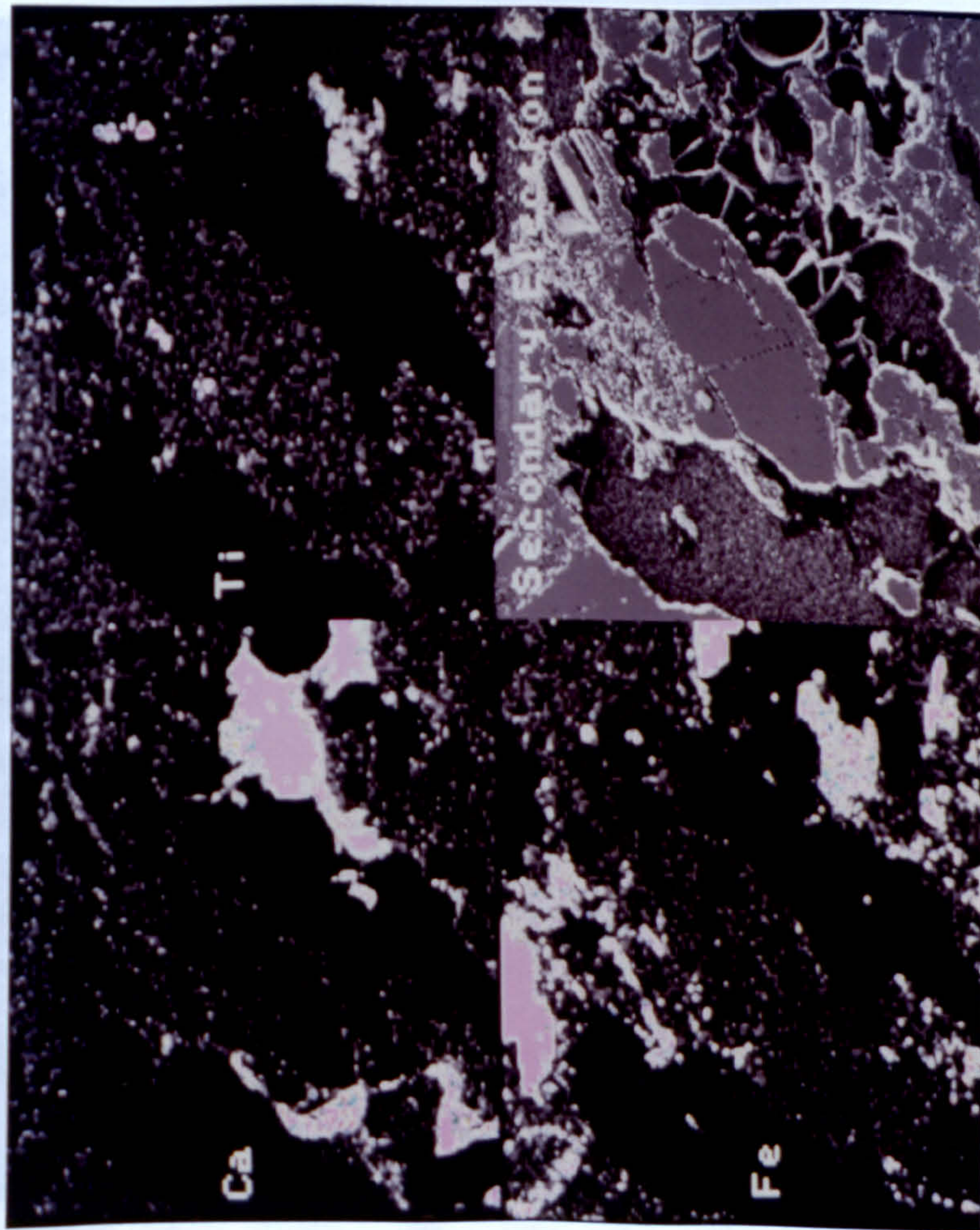


PLATE P4.13. Image processed element maps of the area shown in Plates P4.10. and P4.11. showing the distribution of calcium, titanium and iron and a representation of the secondary electron image of the mapped area.



PLATE P4.14. Image processed carbon distribution. Turquoise - areas of carbon.

negative of this aggregate image was calculated such that all pixels containing values indicating any presence of minerals were inverted to their opposite value and all the pixels blank of detected elements were given a single large value (assigning it a particular colour, in this case turquoise) The image was then overlaid on the secondary electron image to give plate P4.14. In this image all the areas without detected elements (graphite) appear turquoise while all else should appear dark. A white border remains also, this is a measurement artefact from the original secondary electron image. Comparison of plates P4.14., P4.10. and P4.11 enables the areas of graphite abundance to be delineated.

4.7. SAMPLE PREPARATION - GENERAL

The next three sections deal with the preparation and saturation of the samples. This section will deal with the broader aspects of the technique and its application and the following two sections will describe the apparatus designed to saturate the samples in more detail.

Although the concept of sample saturation used by Lee (1977), Evans (1980) and Jackson (1984) has been retained, the system has been significantly improved by upgrading and redesign of the saturating equipment. This has enabled there to be an increase in the effective vacuum in the saturating chamber by three orders of magnitude and also allows much finer monitoring of temperature to help avoid alteration within the sample.

The old system used a simple rotary pump to provide a vacuum of 10^{-2} mbar and a variable transformer to provide current for a heating element. Temperature control was totally manual relying on a mercury filled glass thermometer in a glycerine filled well. Slow heating times were a problem due to the size of the vacuum vessel and the lack of insulation around the heating element that was wrapped around it.

The new system uses a diffusion pump and a rotary backing pump to provide a vacuum of 10^{-5} mbar that is monitored by two gauges. Temperature is automatically controlled by a feedback system designed to hold the vessel at a steady preset temperature. Temperature monitoring is by chromel/alumel thermocouple and platinum resistance transducers. The vessel is heavily lagged resulting in remarkably decreased heat up times.

Rock samples were obtained as 25.4mm diameter cores. They were cut into lengths of about 40mm for ease of handling. Each rock sample was reduced to a diameter of 21.67mm to enable it to fit within the measurement cell. The reduction was facilitated by diamond grinding using water as a coolant and lubricant. The ends of the ground samples were trimmed to 28.60mm long and squared off planely and perpendicular to their cylindrical sides by a Buhler slowly rotating diamond saw using water as the cutting fluid. Each sample was cleaned of any grinding or cutting fluid remaining on its surface. The samples now have the correct dimensions to fit within the measurement cell.

At this point the samples can have their dimensions and weight measured and be loaded into the measurement cell ready for the experiment to progress. Alternatively they can be dried and/or saturated with an electrolyte of known molarity. It is this process which is the more common mode of progression that I will describe next.

Saturating or drying a set of rock samples requires the use of equipment described in more detail in sections 4.8 and 4.9. I will restrict myself to the general method of its use in this section. The rock samples are loaded into the vacuum chamber which is then evacuated. When a steady vacuum has been obtained the heater is switched on. The temperature controller will keep the temperature within the chamber at a steady 80°C. The samples are kept in the chamber at 80°C and under a vacuum of 10^{-4} to 10^{-5} mbar for at least 20 hours. Temperatures higher than 80°C are not used as they introduce the possibility of the rock cracking (Cooper and Simmons, 1977). The combination of a temperature of 80°C and a vacuum is sufficient to ensure effective evaporation of pore fluids. At the end of this period the heater is switched off and the chamber allowed to cool to 40°C or below. When this temperature has been reached the chamber is isolated from the vacuum pumps and water or electrolyte of known molarity can be introduced by opening valves to a reservoir and allowing the liquid to be sucked into the chamber and the pores of the rock as the vacuum is purged. The introduction of the fluid is done slowly to ensure that as much of the volume of the pores as possible is saturated. Initially fluid is introduced making sure that a partial vacuum is left in

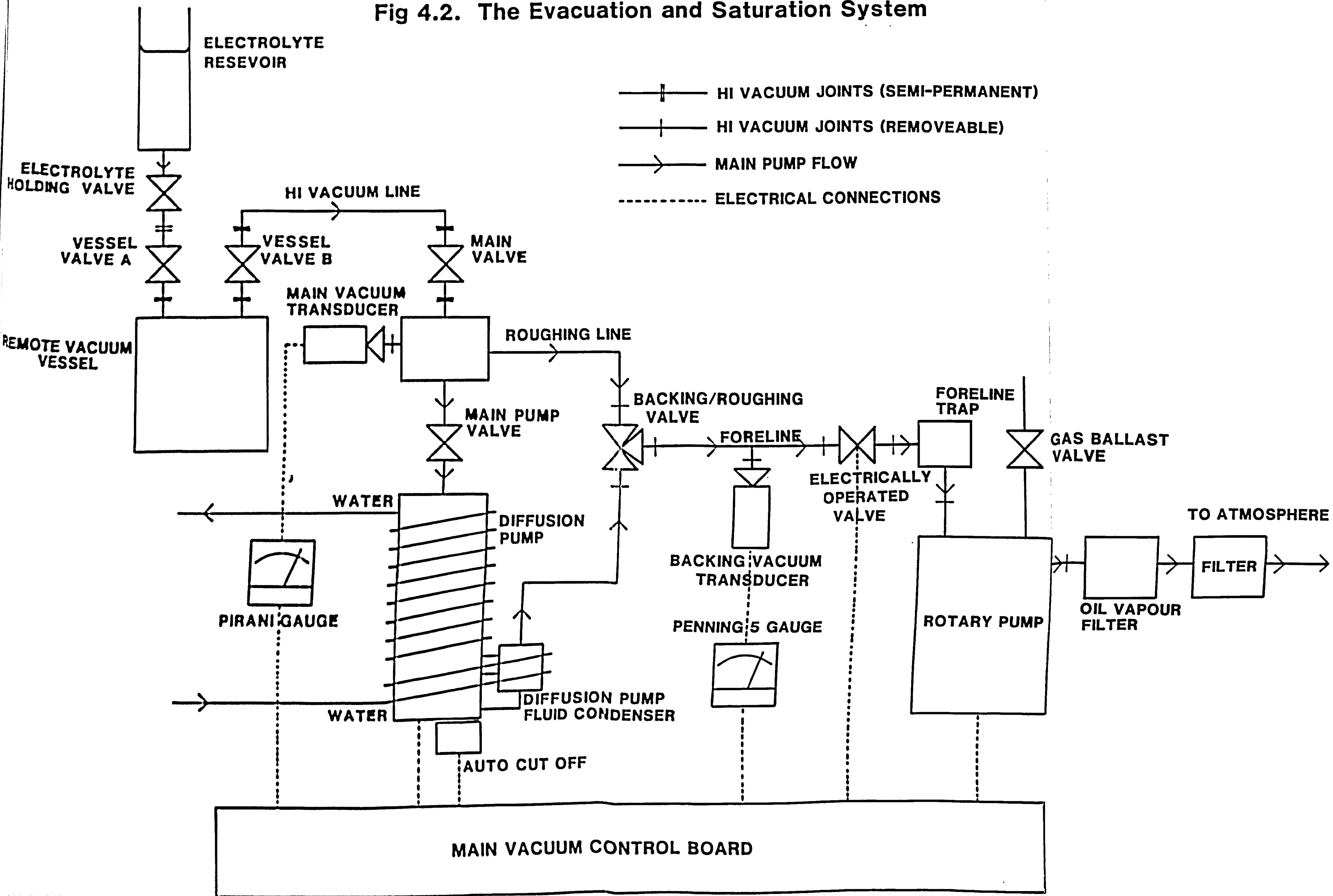
the chamber. If carefully judged sufficient fluid can be let in to cover the samples and yet leave a partial vacuum remaining. The chamber is left in such a state for several hours to allow the fluid to be taken up into all the evacuated pores within the sample. When the remainder of the vacuum has been purged the saturated samples are transferred to a flask where they are kept under the liquid in which they are saturated. Alternatively dried samples can be removed by purging the vacuum with dried air and transferring the samples to a dessicator for storage. Both transfers are done as swiftly as possible with wet or dry gloves or manipulators respectively to avoid evaporation or condensation of water to/from the moist atmosphere depending on the type of treatment the sample has undergone. Particular care was taken not to let dry samples come in contact with breath.

4.8. SAMPLE PREPARATION - THE VACUUM SYSTEM

The vacuum generating apparatus is shown in Figure F4.2. It is based upon the concept of a remote vacuum chamber (Figure F4.3) and a pump system. This design is necessary to isolate the potentially damaging electrolyte saturation fluid from the vacuum pumps and to enable the vacuum chamber to be easily heated and thermally insulated.

The remote vacuum chamber is evacuated by two pumps, a three stage spray-jet diffusion pump and a rotary backing pump. Most of the vacuum pump system has been constructed from parts available from Edwards Vacuum Equipment.

Fig 4.2. The Evacuation and Saturation System



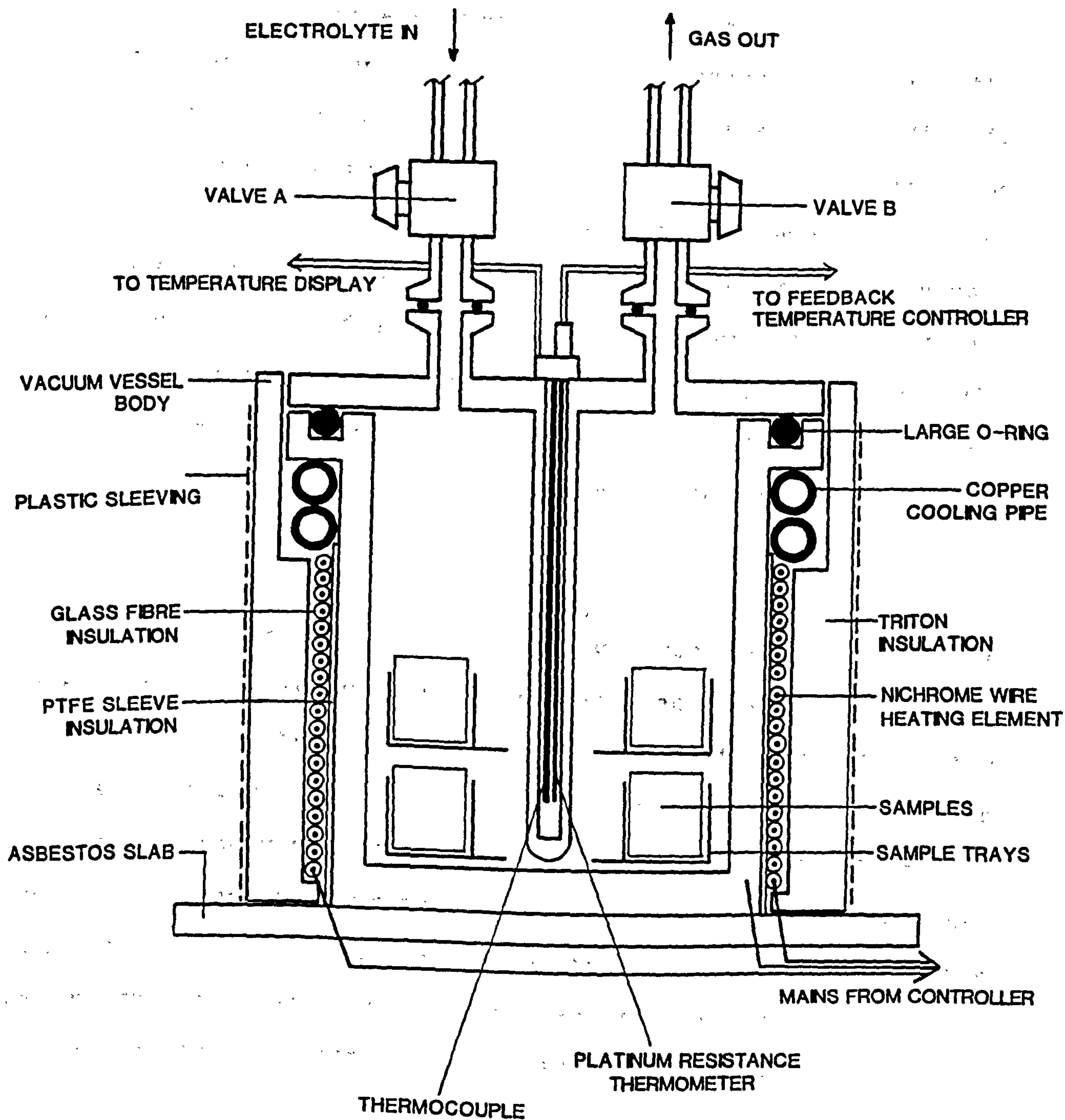


FIGURE F4.3. The remote vacuum vessel.

Evacuation occurs in the following order. The vacuum chamber is loaded with rock samples on sample holding trays. The rotary pump is switched on and used to roughly evacuate the line to the diffusion pump. The rotary pump is then used to roughly evacuate the internal and remote vacuum chambers via the roughing line. At this point the whole system should be at a vacuum of 4×10^{-2} mbar. The system can be set to "full" evacuation when the diffusion pump heater has reached a working temperature. This is done by disconnecting the rotary pump from the roughing line and connecting it to the exhaust of the diffusion pump using the appropriate valves and then opening the valve between the vacuum chamber and the diffusion pump ingress. Gas in the vacuum chamber is initially pumped out by the greater force of the diffusion pump and then removed from the diffusion pump exhaust into the atmosphere by the rotary pump which acts as a backing pump.

The backing vacuum and initial roughing vacua are measured by a Penning 5 backing vacuum transducer head placed on the foreline. The value of the backing vacuum can be read off a gauge on the front panel of the vacuum equipment. Once the diffusion pump is in operation the vacuum chamber vacuum can be read by a Pirani gauge. The Pirani head transducer measures vacua in the internal vacuum chamber which is the same as that in the remote vacuum chamber during evacuation. The vacuum is shown on a meter situated on the front panel of the vacuum equipment.

Three filters have been fitted to the system, a foreline trap to

remove any material that might cause a malfunction of the rotary pump and two rotary pump exhaust filters to remove any oil vapour and noxious gases before the gas is expelled into the laboratory.

Electrolytes can be introduced into the vacuum vessel by isolating the vessel totally from the vacuum pumps and then opening the valve that lets electrolyte solution directly into the chamber.

Re-evacuation requires reroughing of the remote vacuum chamber before the main diffusion pump/vacuum chamber valve can be opened again. The closure of this valve whenever diffusion pump evacuation is not required is not only desirable on safety grounds but also allows both pumps to be kept running while saturation of samples is taking place.

Each time the vacuum chamber is used for saturation it must be dried of all excess fluid before it is used again. Fluid in the chamber or any other part of the vacuum system disables the effective use of the pumps. This is because a vacuum reduces the vapour pressure above a liquid and induces more of the liquid to evaporate. The presence of the introduced vapour reduces the vacuum because it increases the number of molecules in the gaseous state in the vacuum chamber. This variation can be seen on the Pirani gauge and is a useful device for assessing the extent to which rocks have dried out. When the samples are initially inserted into the chamber the vacuum increases rapidly. This rate of increase slowly declines until the vacuum reaches a steady value. The slow approach to a steady vacuum is due to

loosely held water molecules becoming vapour. The pumps then have to remove these before pressure can be reduced further. Further reduction of pressure only causes more loosely held water molecules to evaporate increasing the pressure again until the pumps remove them. In practice the evaporation of such fluid acts to reduce the rate at which the pressure decreases so long as there is fluid to evaporate. After a certain time, usually within two hours the rate of decrease of pressure is almost negligible indicating that all the loosely held water has been removed. If the heating elements around the vacuum chamber are turned on the vacuum suddenly decreases. This is because the conditions of low pressure and temperatures of about 80°C cause more of the rock water to evaporate. If left in this state the pressure will steadily drop until it reaches a steady level less than that obtained previous to heating. This is due to the decrease in the amount of rock water left to evaporate. When the vacuum reaches a steady level again it can be assumed that all the fluid that will vapourise at that vacuum and preset temperature has been removed. If the values of vacuum and preset temperature are sufficient that free water boils it can be expected that the rocks are completely dry though not necessarily anhydrous.

4.9. SAMPLE PREPARATION - THE TEMPERATURE SYSTEM

The temperature control and heating apparatus is shown diagrammatically in figure F4.4. and in more detail in figure F4.5. The feedback temperature controller was designed around a platinum resistance thermometer and two 741 operational amplifiers which drive a solid state relay that switches up to 2 amps through a heating element.

The resistance of the platinum probe changes as the temperature within the central well of the vacuum vessel changes. The variation of the probe resistance causes variations of the potential at point A in figure F4.5. This potential is initially buffered by an operational amplifier. The buffered signal is compared with a preset potential by another operational amplifier which results in the activation, or otherwise, of a solid state relay. If the buffered signal from the probe exceeds the preset potential then the comparator will deactivate the relay, switching off current to the heating elements. Conversely if the buffered signal is less than the preset potential the relay will remain closed allowing current to flow through the heating element. The preset potential is controlled by a ten turn potentiometer with an integral 0 - 999 scale. This enables fine adjustment to be made to the temperature as well as enabling the controller to be calibrated in terms of temperature.

The construction of the heater is shown in figure F4.3. The heater element was constructed of 30 turns of 28 swg nichrome wire threaded in a glassfibre insulating sleeve. The insulated

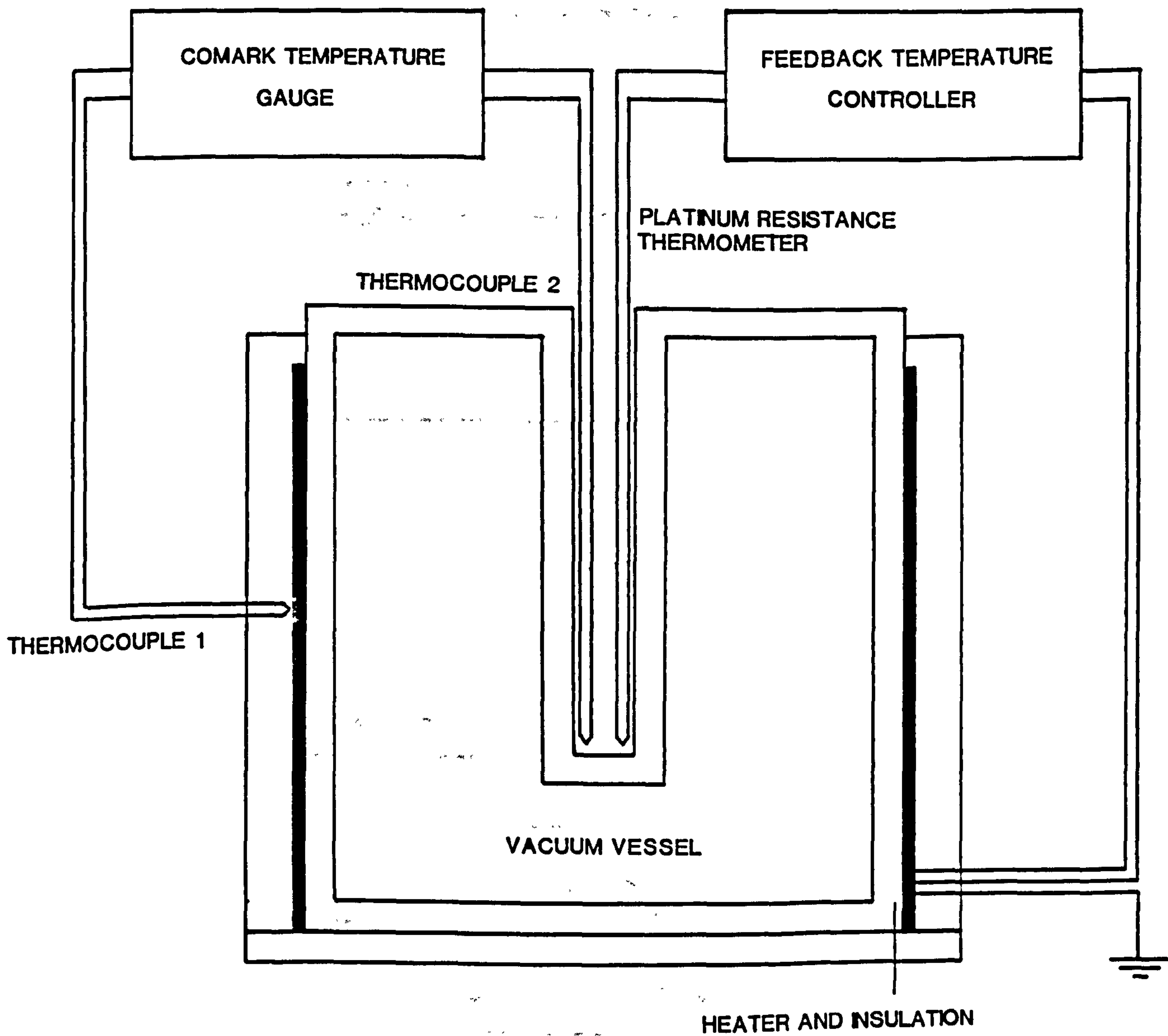
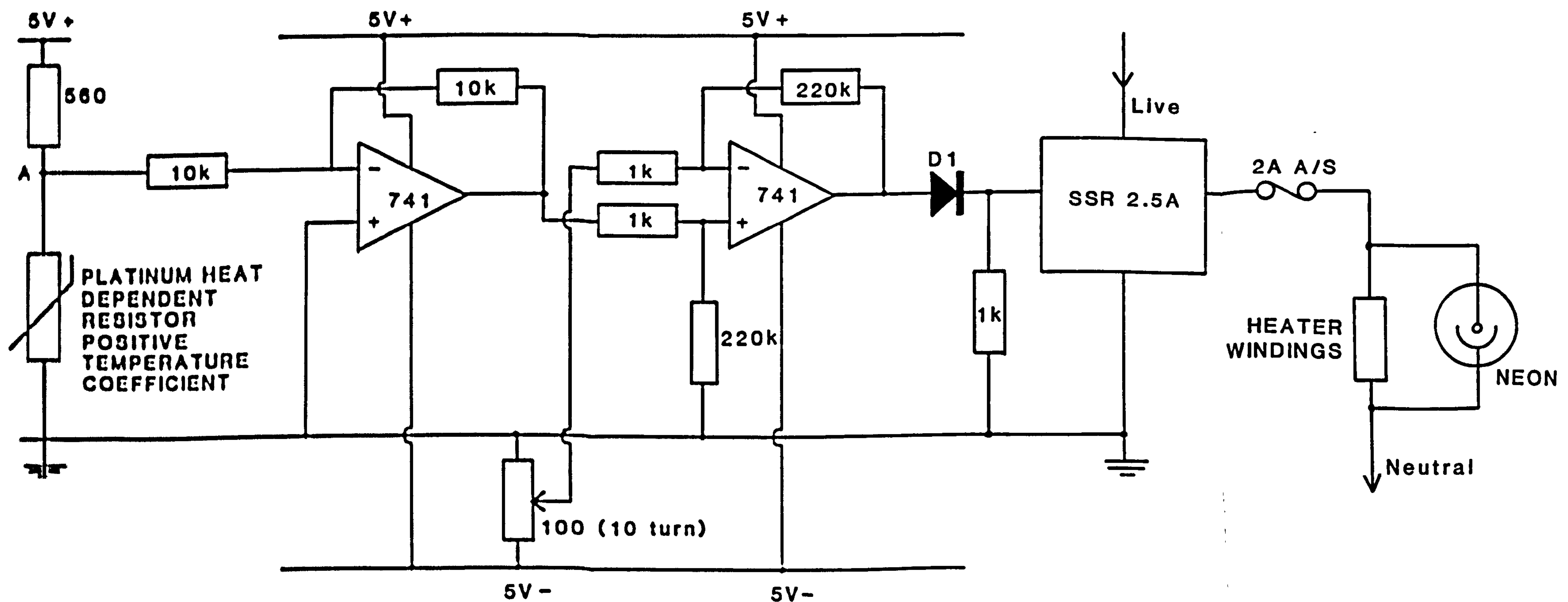
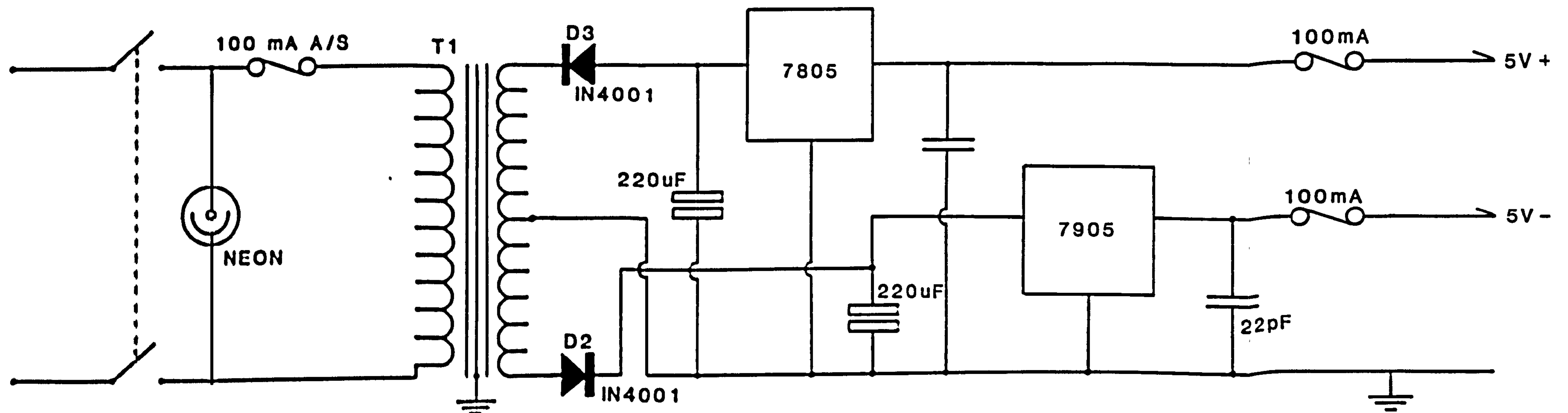


FIGURE F4.4. Schematic diagram of the heating and temperature control circuits of the remote vacuum vessel.

FIGURE F4.5. Circuit diagram of the vacuum vessel feedback temperature controller.



wire was wound onto a thin layer of PTFE, which sleeved the vacuum vessel, and anchored using thermosetting putty. The thin sleeve of PTFE was included as further electrical insulation as it had been noticed that in previous designs there had been breakdown of the glassfibre insulating sleeve and consequent shorting of the heating current across the body of the vacuum vessel after prolonged use. The thickness of this PTFE sleeve was made small in order to retain a high heat transfer towards the interior of the vacuum vessel. The element construction was thermally insulated from the laboratory environment by a double layer of 12.7 mm thick Triton ceramic fibre blanket of a density of 2 kg m⁻² and held in place by polynet plastic webbing. The reduction in heat loss due to the thermal insulation enables the vacuum vessel to be heated up at a much faster rate than was previously attainable. The new heating system is able to reach the required temperature of 80 °C in 8 minutes, a tenfold improvement upon the uninsulated arrangement which consisted of a nichrome winding driven by a variable transformer directly from the mains supply and controlled manually, relying on the reading of a mercury filled glass thermometer.

The main vacuum seal, is provided by a large Nitron 'O' ring. This is sensitive to high temperatures and can decompose. A simple double turn of copper piping was wound around the vessel near the seal. Cooling water circulating through this pipe reduces the temperature in the region of the seal just sufficiently to protect the 'O' ring from temperature degradation.

The temperature in the vacuum chamber well and at the heater element was also monitored using chromel/alumel thermocouples and a Comark digital thermocouple temperature meter which was mounted next to the feedback controller. In normal running mode the internal temperature of the well is kept at around 80 °C. At this 'internal' temperature, the temperature of the heater is about 220 °C which is just below the breakdown temperature of all the insulating materials used.

4.10. SAMPLE MEASUREMENTS

Several routine measurements were made on the samples. These included length, diameter, unsaturated weight, saturated weight and dry weight. The order in which these measurements were made is set out below. Samples of the correct dimensions to fit inside the measurement cell were accurately measured for length and diameter, and weighed on a laboratory balance to give the unsaturated weight, W_1 . The samples were then dried under vacuum for 20 hours. The dry samples were reweighed to give the dry weight, W_2 . At all times it was necessary to be careful to subject the dried sample to as little of the moist laboratory air as possible by storing the dried samples in a dessicator, taking care in their handling, and making the measurements as swiftly as possible. The samples were then subjected to heating under a vacuum again before being saturated with an aqueous fluid. The saturated samples were reweighed to give the saturated weight, W_3 . Before the saturated weight was measured it was necessary to

remove any surface fluid from the sample. During this procedure and the weighing of the sample it was necessary to avoid excess evaporation of the near surface saturating fluid into the atmosphere. This was done by making the measurements as swiftly as possible and manipulating them with gloves dampened in water. At all times, other than whilst they were being weighed, the saturated samples were kept under the fluid in which they were saturated. All weighing measurements were made before the rock was subjected to high pressures as such conditions are likely to alter the porosity of the samples irreversibly (Jackson, 1984).

The porosity of the sample can be obtained from the three weights. Calculations of porosity have been set out in detail by my predecessors, Lee (1977), Evans (1980) and Jackson (1984), and the method will simply be listed in this work in the form of equations E4.2 - E4.5. The original derivations of the formulae may be found in Saad (1969).

Porosity	$\phi = \frac{W_3 - W_2}{V\rho_L}$	E4.2.
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Absorbed Fluid Content	$\alpha = \frac{W_1 - W_2}{W_1}$	E4.3.
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Saturated Bulk Density	$\rho_b = \frac{W_3}{V}$	E4.4.
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Grain Density	$\rho_g = \frac{(1 - \alpha)}{(1 - \phi)} \rho_b$	E4.5.
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where; W_1 is the sample unsaturated weight,
 W_2 is the sample dry weight,
 W_3 is the sample saturated weight,
 V is the sample volume,
 ρ_f is the density of the saturation fluid.

Accurate quantification of errors in the small percentage changes in sample weight due to additional fluid is difficult especially in low porosity crystalline rocks. Jackson (1984) quotes an error of about 10% based on the repeatability of his measurements. Experience with saturating samples leads me to agree with Jackson's observations.

The measurements made on the samples are set out in table T4.6.

TABLE T4.6. WEIGHT MEASUREMENTS AND THE SATURATION STATE OF THE SAMPLES

SAMPLE	INITIAL WEIGHT /g	DRY WEIGHT /g	SATURATED WEIGHT /g	BULK DENSITY kg/m ³ X10 ³	GRAIN DENSITY kg/m ³ X10 ³	POROSITY %	ABSORBED WATER %	SATURATION TYPE	ORIENT- ATION
IVD1	32.361	32.309	32.391	3.082	3.101	0.78	0.16	0.5 M NaCl	-
IVD2	32.539	32.491	32.566	3.084	3.102	0.72	0.15	DD WATER	-
IVE1	32.743	32.681	32.752	3.096	3.111	0.68	0.19	0.5 M NaCl	-
IVE2	33.022	32.959	33.028	3.125	3.139	0.65	0.19	DD WATER	-
MER21	34.752	34.728	34.769	3.299	3.310	0.40	0.07	0.5 M NaCl	-
MER22	35.443	35.422	35.463	3.336	3.347	0.39	0.06	DD WATER	-
A8B	31.470	31.426	31.519	2.999	3.022	0.89	0.14	0.5 M NaCl	-
L33B	26.600	26.568	26.643	2.535	2.550	0.71	0.12	0.5 M NaCl	
L32	32.280	32.212	32.319	3.078	3.103	1.02	0.21	0.5 M NaCl	
V1	27.364	27.337	27.390	2.599	2.610	0.50	0.10	0.5 M NaCl	-
GGRAN1	33.216	33.150	33.243	3.148	3.170	0.88	0.20	0.5 M NaCl	
GGRAN2	33.586	33.515	33.613	3.177	3.200	0.93	0.21	0.5 M NaCl	‡
GGRAN3	33.485	33.421	33.517	3.162	3.185	0.91	0.19	NONE	
GGRAN4	33.545	33.471	33.570	3.182	3.205	0.94	0.22	NONE	‡

WHERE:

DD WATER = Doubly distilled water

|| Indicates that electrical measurements were taken parallel to the planes of foliation of the sample.

‡ Indicates that electrical measurements were taken perpendicular to the planes of foliation.

CHAPTER 5 - HIGH PRESSURE AND HIGH TEMPERATURE APPARATUS

5.1. INTRODUCTION

This chapter describes the experimental apparatus and procedures used to obtain operating conditions up to 200 MPa and 800 °C. It also describes the methods of measuring pressure and temperature and the calibration of these methods. Many of the techniques have been developed over a long period by those who have worked in the gas high pressure laboratory. I am grateful to all who have helped me in this way without knowing it. Some systems such as the pore pressure system have been constructed solely for this experiment.

5.2. THE GAS HIGH PRESSURE SYSTEM

The gas high pressure system provides the confining pressure on the conductivity measuring cell. It also enables the operation of conductivity measuring cells and furnaces in an inert gas atmosphere. High purity Argon gas (99.99+% pure) was used on all measurement runs and high purity Nitrogen was used on all preliminary test runs. An inert gas atmosphere dramatically reduces oxidation of the metal parts of the measurement cell and delicate furnace windings. It also avoids the formation of hydrides and carbides on the refractory metal parts of the measurement cell and minimises contamination of the thermocouples.

The Mark II pressure vessel used in this work and shown in figure F5.1, has a maximum working internal pressure of 200 MPa when its

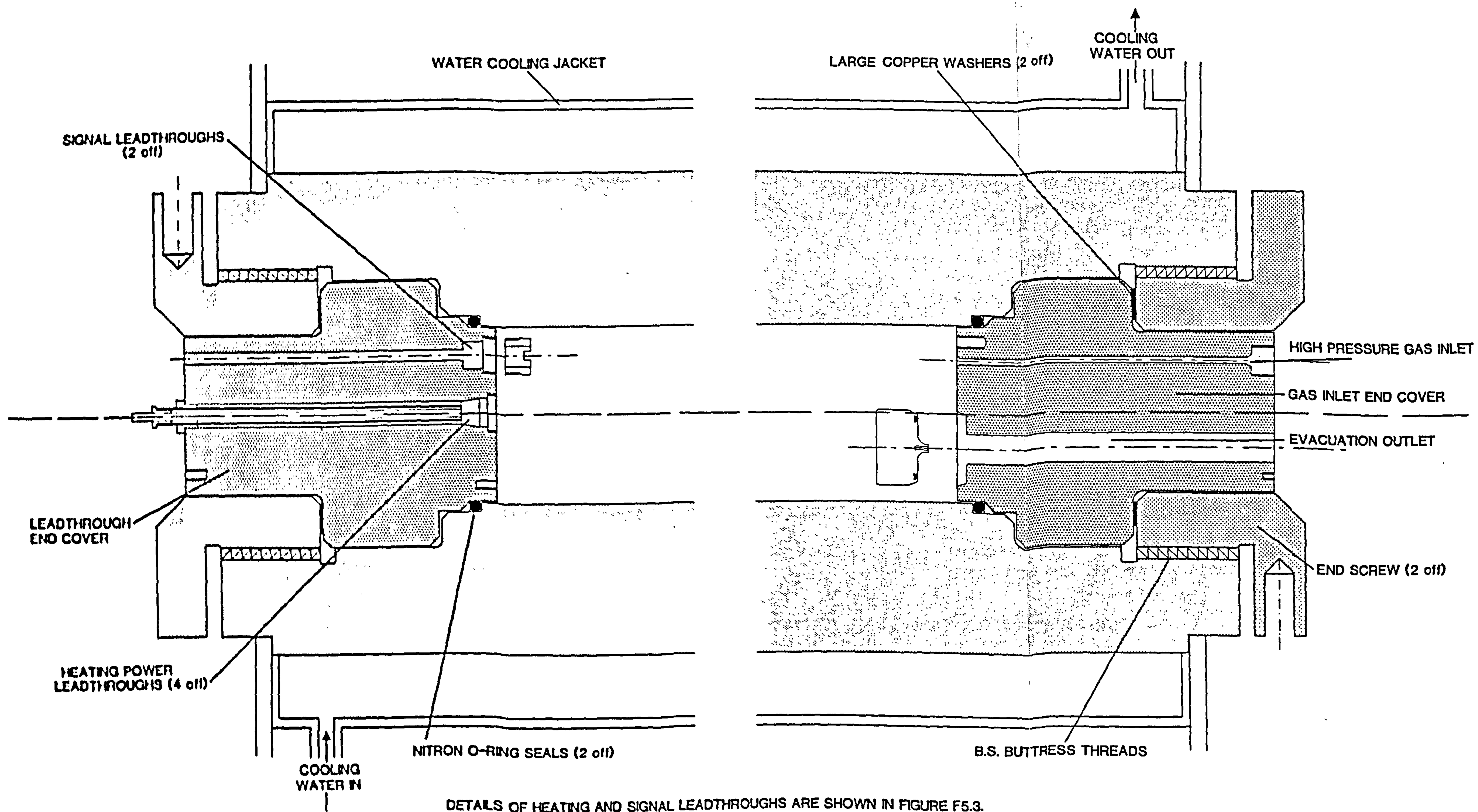


FIGURE F5.1. The 0.2 GPa gas pressure vessel.

external surface is at atmospheric pressure. Its basic construction is a hollow cylinder of Firth Vickers 520B stainless steel. The internal bore diameter is 114.6 mm (4.512") and is 555.6 mm (21.875") long. The confined volume is 5.731×10^6 mm³. The ends of the pressure vessel are sealed with end covers held in place by large nuts made of En 25 steel. The seals are made by two large Nitron 'O' rings between the bore and end cover, one at each end. The entire curved surface of the vessel is surrounded in a jacket that carries circulating cooling water. Plate P5.1. shows the vessel prior to loading with the loading cradle on the left hand side. Tolerances between the endcover and the locking thread are tight and so require the accurate introduction of the endcover and experimental charge to avoid fouling the vessel's bore and threads. Since the endcover is quite heavy it requires the use of a bearing borne cradle. The cradle can be moved small distances in the three orthogonal directions but has to be removed once the endcover has been introduced to enable the locking nut to fit. On the right hand side of plate P5.1. can be seen the vacuum system, described in section 4.5.. This is used to initially purge the pressure vessel of laboratory air by successive evacuation and charging with inert gas before the pressure of the vessel is significantly raised.

All high pressures were generated within a protective steel room, the larger of two available in the high pressure laboratory of the School of Physics in the University of East Anglia. The steel rooms are shown in plate P5.2. These protective rooms were constructed specifically for the protection of personnel against

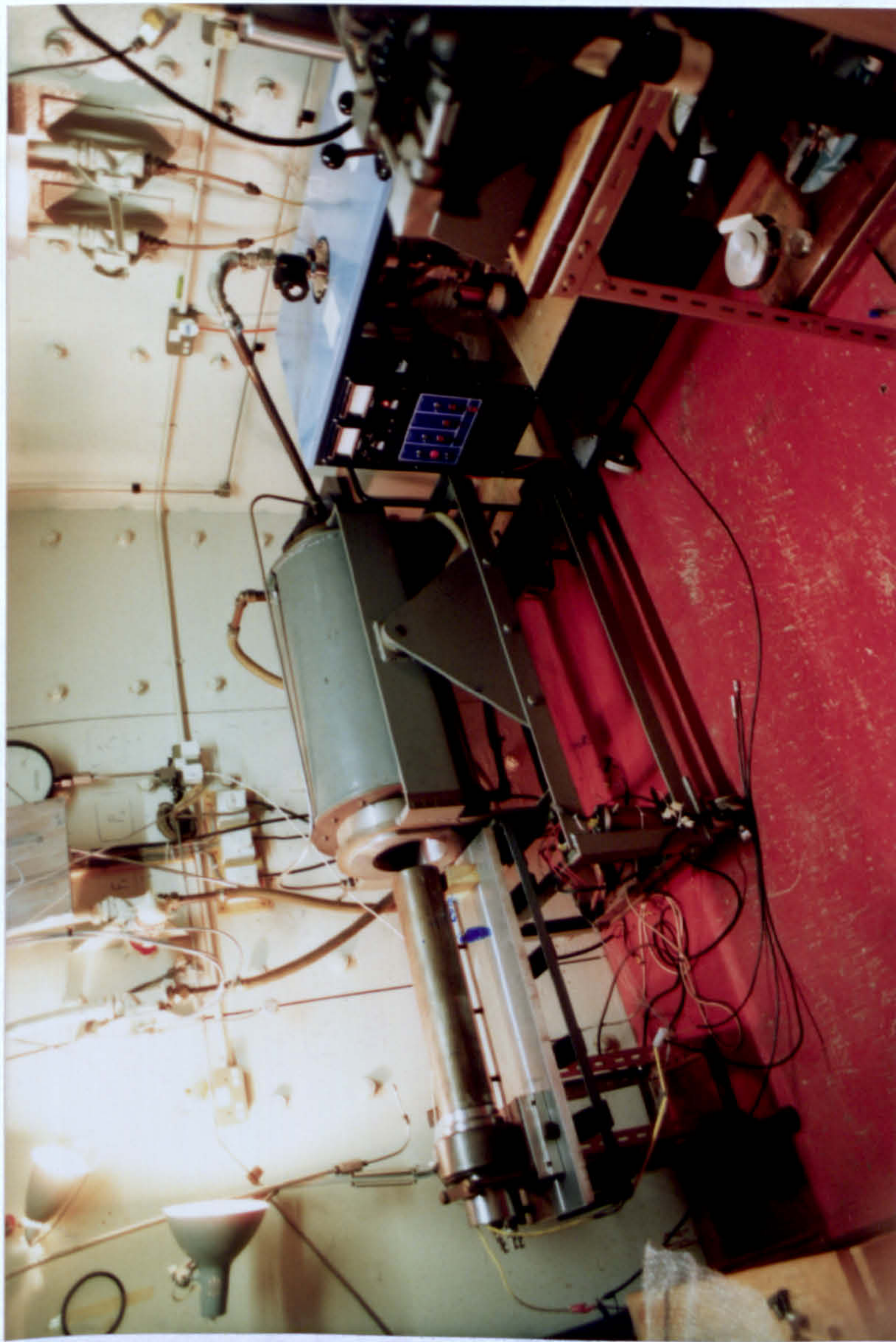


PLATE P5.1. INTERIOR OF THE HIGH PRESSURE SAFETY ROOM SHOWING
THE PRESSURE VESSEL (CENTRE), THE VACUUM PURGE SYSTEM (RIGHT)
AND A FULLY PREPARED EXPERIMENTAL LOAD ON A CRADLE (LEFT)



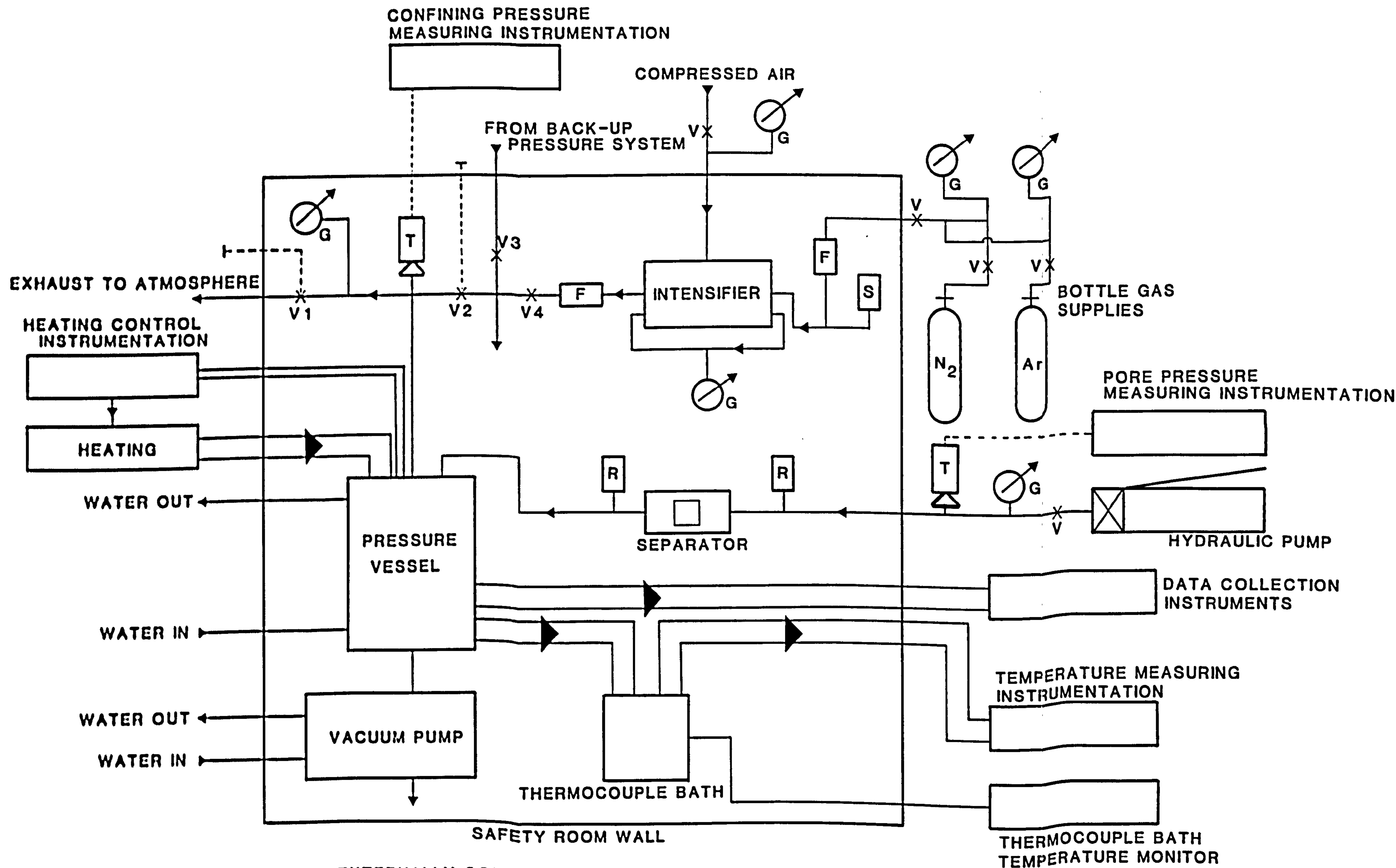
PLATE P5.2. THE HIGH PRESSURE STEEL SAFETY ROOMS IN WHICH THE WORK WAS DONE, HIGH PRESSURE LABORATORY, SCHOOL OF MATHEMATICS AND PHYSICS

blast and flying projectiles in the event of a catastrophic equipment or vessel failure. The rooms have 2" thick steel walls and foundations that are isolated from the main building. The design of the high pressure rooms and the operation of the high pressure system in general was carried out in accordance with the recommendations set out in the High Pressure Safety Code (1975) published by the High Pressure Technology Association (HPTA). The layout of the high pressure system within the larger of the two rooms is shown in figure F5.2. The rooms have been designed to minimise inconvenience in case of failure, maintenance or repair of equipment by enabling pressure to be raised in either room with the compressor from the other. This is done by bypass piping as shown in figure F5.2.

The final gas pressure and inter-stage gas pressure was measured on Bourdon gauges within the safety rooms. These were read from outside the safety rooms when the pressure was raised by using two viewing periscopes. This method was also used to observe the interior of the safety room when the experiment was in progress. Pressure was also measured by the use of strain gauge pressure sensors, these are described in more detail in section 5.5.

Confining pressure was raised by an Aminco compressed air operated diaphragm compressor (46-14021). This is a two stage device that takes gas at bottle pressures of around 100 bar and compresses it to 0.2 GPa. When the compressed air supply is insufficient to raise the pressure further it stalls, cutting in again if the generated pressure falls again providing that the compressed air supply has not been reduced in the meantime. This

FIGURE F5.2. A FLOW DIAGRAM OF THE COMPLETE EXPERIMENT



- V1-V2 EXTERNALLY CONTROLLED MAIN PRESSURE VALVES
V3-V4 ASSOCIATED HIGH PRESSURE VALVES
V LOW PRESSURE GAS VALVES
T PRESSURE TRANSDUCERS
G BOURDON PRESSURE GAUGES
R RESEVIORS FOR ELECTROLYTE AND OIL
S SAFETY VALVE
F INLINE FILTERS

stalling feature has been used to maintain a reasonably steady gas pressure over long periods without the confining pressure dropping dangerously lower than the pore pressure. Even so the equipment has never been sufficiently reliable to leave unattended. The control of gas pressure is effected by the variation of the compressed air pressure and the operation of two valves from outside the safety room. One valve connects the gas compressor to the pressure vessel, the other exhausts the pressure vessel to the atmosphere via an exhaust port outside the laboratory. Both valves can be operated from outside the safety room by means of mechanical couplings.

5.3. LEADTHROUGHS

Three types of leadthrough were required to convey pore pressure, heating current and data lines through the end cover into the pressure vessel. They are shown in figures F5.3a,b,c and will be described in order.

ELECTRICAL INFORMATION LEADTHROUGH Twelve wires were required to carry electrical information from the interior of the vessel. These were four chromel, four alumel and four copper wires and provided for four thermocouples and four conductivity leads. Two of the conductivity leads were used and two kept spare forming a screen. The third electrical connection necessary to the working of the guard-ring measurement configuration was provided by the pore fluid pipe. This will be discussed in greater detail in chapter 6. The twelve leads were assembled into one leadthrough

FIGURE F5.3a High pressure leadthrough for electrical signals.

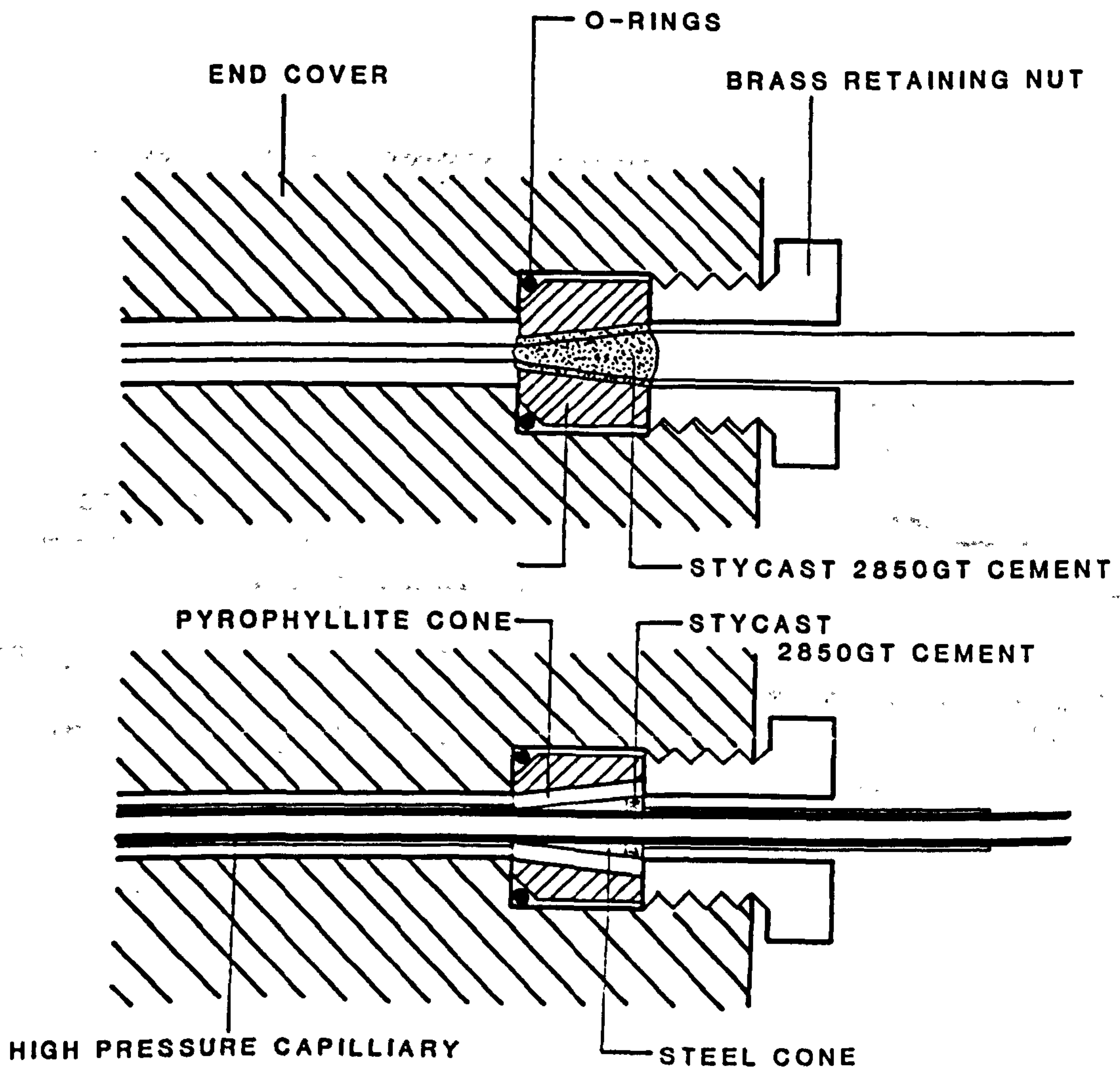
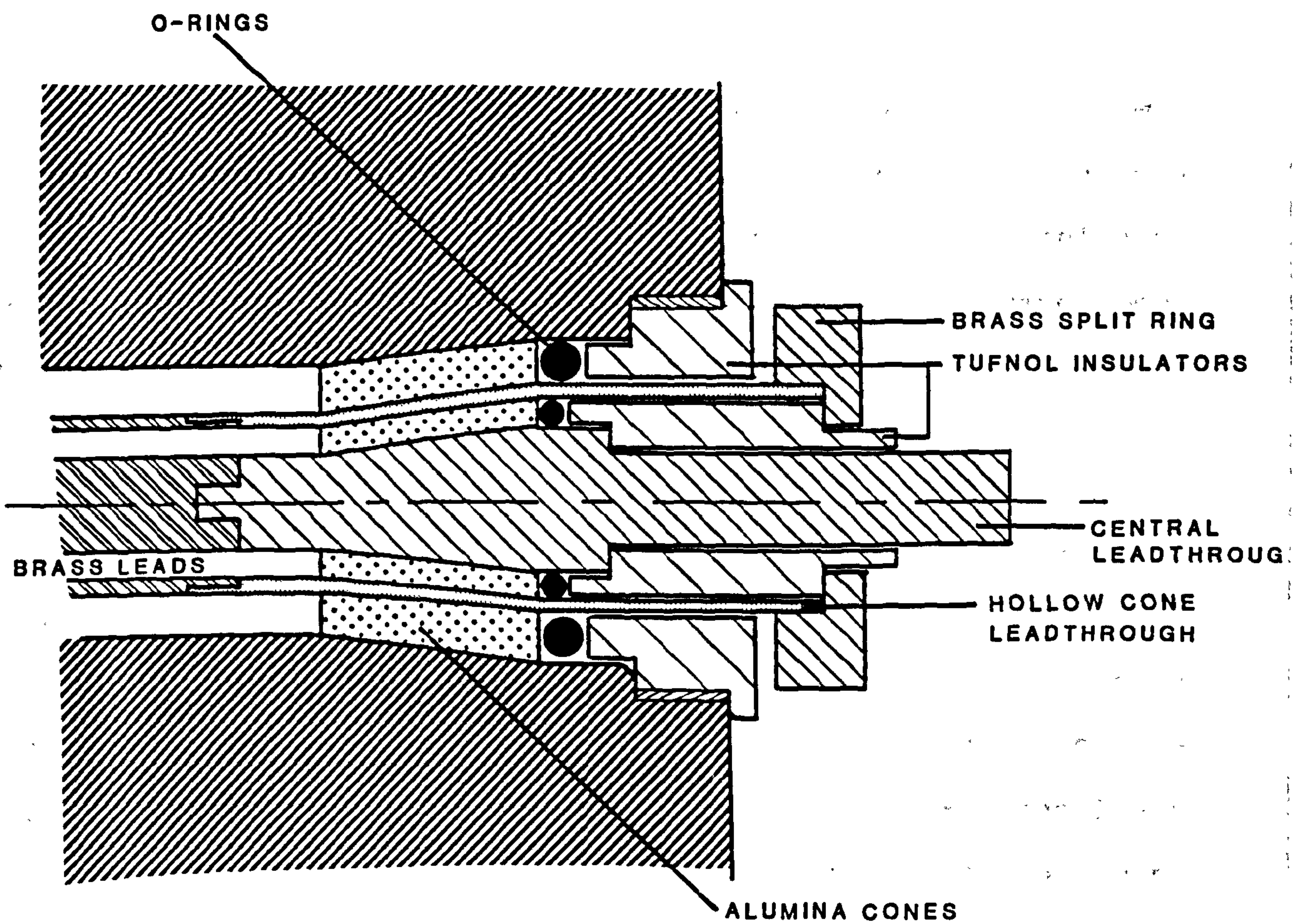


FIGURE F5.3b High pressure leadthrough for pore pressure fluid.

FIGURE F5.3c High pressure leadthrough for heating currents.



based upon the design of Blosser and Young (1962) that has been used in this laboratory for several years and was incorporated into the initial end cover design. Enamelled 28 swg chromel, alumel and copper wires were used. The enamel was stripped from the wires inside the pressure vessel and the wires were insulated with alumina beads, rod and reffrasil. This was done to avoid the degradation of the enamel in hot conditions inside the pressure vessel leading to inaccuracies in the thermocouple readings. It was also for this reason that the wires were stripped with Nitromors instead of using a flame. This design of leadthrough is shown in figure F5.3a.

PORE PRESSURE LEADTHROUGH This type of leadthrough was specifically designed for use with this experiment. It allows the pore pressure to be varied independently of the confining pressure by bringing the pore pressure in from a separate pore pressure raising system. There is a more detailed description of the pore pressure system in section 5.4.

One of the requirements of the pore pressure system is that it should be electrically isolated from the body of the pressure vessel and not earthed in any way. This is because the pore pressure capillary also acts as one of the leads to the conductivity measuring electrodes. The pore pressure capillary was isolated from the end cover by a pyrophyllite cone leadthrough.

The leadthrough was made in the following way; A stainless steel capillary tube made by Aminco, rated at 1 GPa and with an

external diameter of 2.00 mm and a bore of 0.40 mm was brazed into a hardened stainless steel cone. The cone and capillary was then diamond ground into a pyrophyllite cone. The resulting construction was then diamond ground into a leadthrough blank with a cone machined into it as shown in figure F5.3b. The whole construction makes a seal in a similar way to the electrical information carrying leadthrough blanks using a nitron O-ring. The length of capillary tubing in the end cover and external to the pressure vessel was insulated with heat shrinkable plastic sleeving. The capillary inside the pressure vessel was encased in large pyrophyllite blocks and needed no insulation.

FURNACE CURRENT LEADTHROUGHS Three double conductor high current leadthroughs were used. Each had been in place in the end cover for some time and needed only minor servicing before use. Each leadthrough provided live and return paths for one winding, the construction of which can be seen in figure F5.3c. Each leadthrough consisted of a hardened steel cone diamond ground into a ceramic cone. The ceramic cone is diamond ground into a hardened steel cone which is itself ground into another ceramic cone. This whole construction is diamond ground into a cone-shaped bed in the end cover of the pressure vessel. Each ceramic cone was held in place by a PTFE O-ring. In this way two concentric conductors were isolated from each other and the end cover. The leadthrough is shown in figure F5.3c.

5.4. THE PORE PRESSURE SYSTEM

One of the requirements of the experiment was that the pore pressure would be variable independently of the confining pressure. This puts constraints on the experimental design as is discussed in chapters 1 and 6. A variable electrolyte pressure needed to be generated, measured and delivered through the pressure vessel end cover to the measurement cell. The design of the measurement cell combines the pore pressure pipe with the emitter electrode as can be seen in figure F6.7. The emitter must not be earthed due to the internal design of the conductance measuring transformer bridge. This constrains the whole pore pressure system to be isolated from earth and the pressure vessel. The isolation from the pressure vessel has been facilitated by the leadthrough design discussed in section 5.3. The rest of the system is isolated by covering all the high pore pressure piping with Nylon and PTFE piping and mounting all gauges, pumps and separators on wooden stands.

A representation of the layout of the pore pressure system is shown as figure F5.4.

Pressure is raised by the hydraulic pump. Raising the pressure in this way acts upon the separator to produce the same pressure within the electrolyte filled part of the system. The system was designed with filling points for hydraulic oil and electrolyte at high points. The pore pressure was roughly measured on a Bourdon gauge on the oil side of the separator. Accurate measurement of pore pressure was also done with a strain gauge pressure sensor.

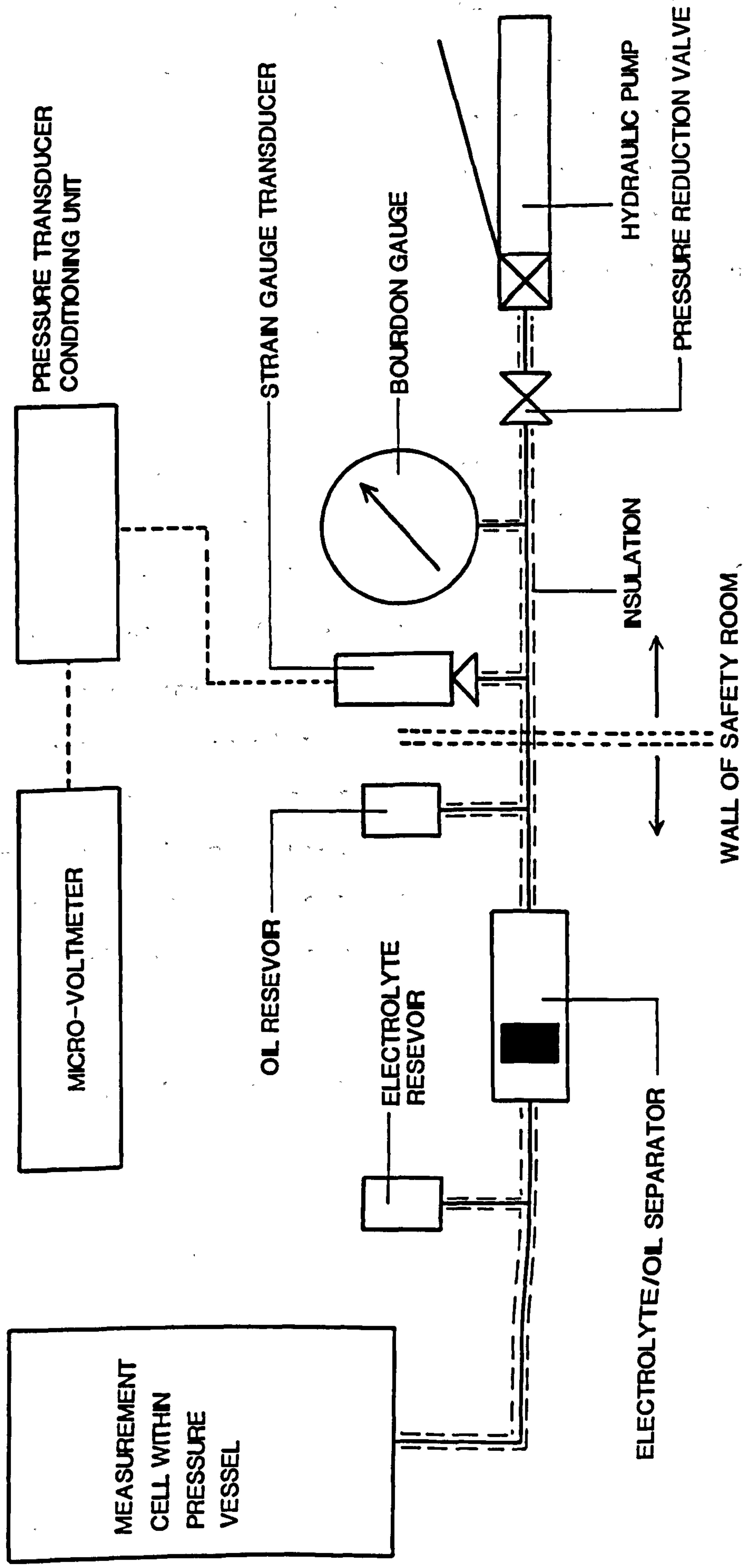


FIGURE F5.4. The pore pressure system.

This will be discussed in more detail in section 5.5. The pore pressure has also been measured on the electrolyte side of the separator to check whether the separator was working well. No significant or systematic difference was found between measuring pressure on either side of the separator so all subsequent measurements were made on the oil side to protect the gauges from attack by the electrolyte solutions.

The hydraulic pump had a one way valve built into it. This valve was loosened to release the pore pressure by allowing the compressed oil to refill the hydraulic pump reservoir. Increasing the volume of the system in this way reduces the pressure of the system and thus reduces the pore pressure. It was found that it was difficult to accurately control the reduction of this pressure, a small movement of the valve causing a drastic reduction in pressure. Another valve was inserted in the system physically close to the hydraulic pump valve to enable a more controlled reduction of pressure.

5.5. PRESSURE MEASUREMENT AND PRESSURE TRANSDUCER CALIBRATION

The pore pressure, gas pressure and interstage gas pressure were all measured using Bourdon gauges. The gas Bourdon gauges were situated within the steel room and viewed with one of two periscopes. The pore pressure gauge was situated next to the hydraulic pump outside the safety room. These gauges were primarily intended as a safety feature to indicate pressure in

the event of a failure of the electrically operated pressure sensors.

More accurate measurement of pore pressure and gas pressure was obtained by the use of Pye Ether BP6 30,000 psi strain gauge pressure sensors. Two transducers were used to measure the gas and pore pressures. Their serial numbers were B3358 HD and B3356 HD respectively. Each transducer was supported by a Pye Ether/Pioden 1720B conditioning unit. This system has a bridge design in which two strain gauges are set opposite the two dummy arms of the bridge. Application of pressure to the transducer produces an out of balance signal of approximately 65 microvolts/MPa. The output from each conditioning unit was fed to two Solartron 7055 microprocessor micro-voltmeters which were programmed to read directly in MPa.

The entire pressure measuring systems - transducer, conditioning unit and micro-voltmeter, were calibrated using a Budenberg dead weight tester before the experiments and again at the end. The calibration relationship was found to be linear to within the quoted 0.02% error (manufacturers specification). The calibration values are shown in table T5.1.

TABLE T5.1. TRANSDUCER CALIBRATION VALUES

TRANSDUCER	BEFORE	AFTER
	micro-volts/psi standard deviation on four calibration runs	
B3358 HD GAS PRESSURE	0.6575 0.0002	0.6585 0.0003
B3356 HD PORE PRESSURE	0.6577 0.0004	0.6590 0.0006

These values suggest that the inherent error in pressure measurement is less than 1%.

Figure F5.5. shows how the various connections for pressure, temperature and conductivity sensing and control of pressure and temperature were made through the safety wall. The numerical codes on the lines indicate the number of connecting lines transferring data or control information.

5.6. THE HIGH TEMPERATURE CONTROL SYSTEM AND FURNACE DESIGN

The furnace used throughout the experiment was designed and made in the high pressure laboratory to enable it to fit closely within the pressure vessel and thermal cladding. The design of the furnace was constrained by the requirements of the experiment and was done using theory given in Carslaw and Jaeger (1959). It will be described first, followed by an outline of the system used to control the temperatures generated by it.

The furnace is shown in figure F5.6. and plate P5.3. It was made by taking a tube of aluminous porcelain 271 mm long with an internal diameter of 32.1 mm and an external diameter of 39.5 mm and painting it with a light coating of alumina cement. The cement was prevented from completely drying by the use of a water spray. Each of three windings was wound on this porcelain former while the cement was still damp. Bare 26 swg nichrome wire was

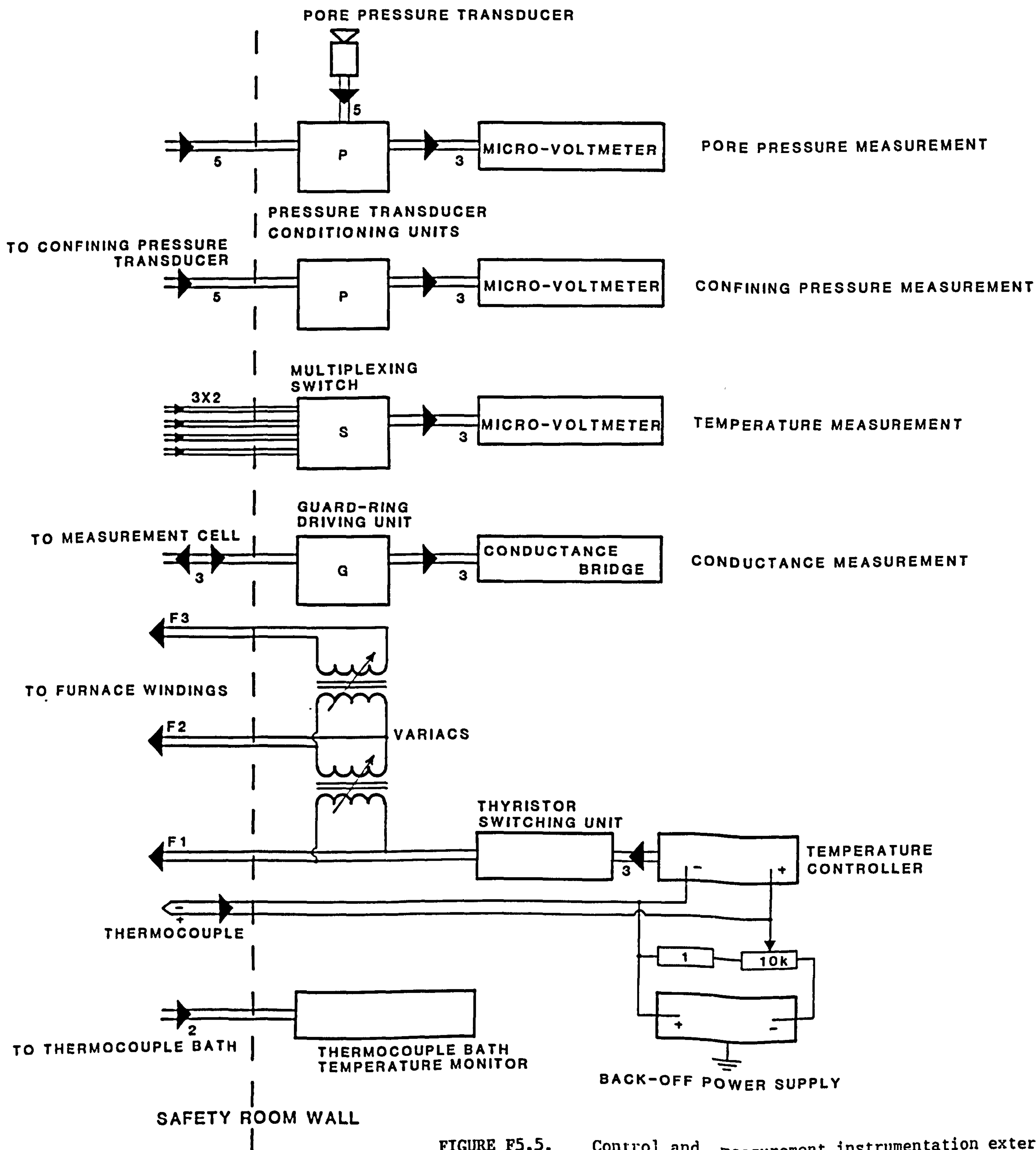


FIGURE F5.5.

Control and measurement instrumentation external to the safety wall.



PLATE P5.3. THE FURNACE AND A FULLY PREPARED EXPERIMENTAL CELL

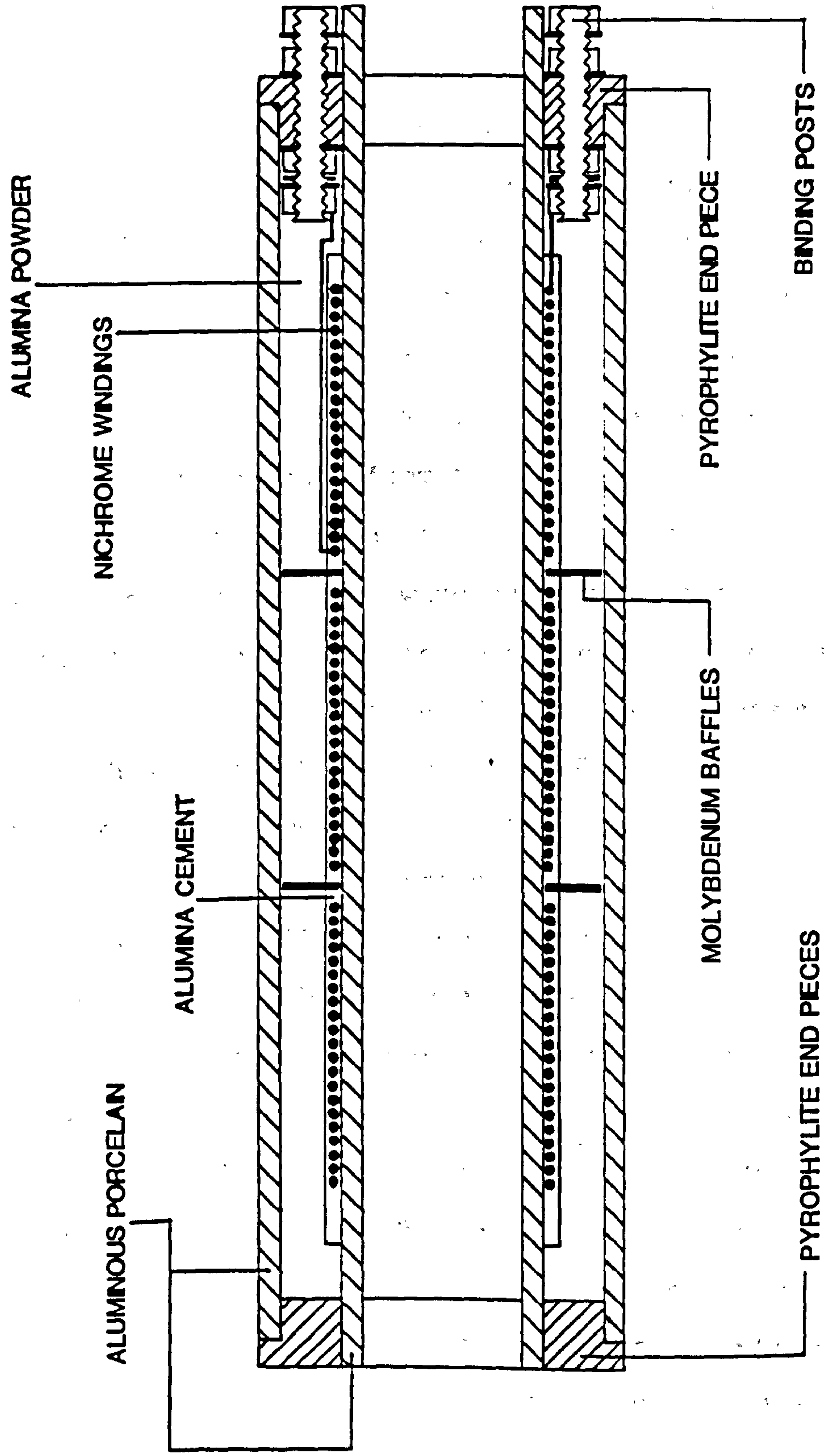


FIGURE F5.6. The furnace design.

used. It was wound at a pitch of about $0.8 \text{ turns mm}^{-1}$ in three independent sections 5 mm apart and about 55 mm long. The power supply to these windings was arranged so that adjacent winding connections were either both positive or both negative to ensure that no arcing would take place within the furnace. The completed windings were tied off using methods described in Seybolt and Burke (1953) and then covered in more alumina cement. The alumina cement helps to space the windings evenly while wet and winding is taking place due to the partitioning of the wires by the small particles of alumina. The alumina cement acts as an insulator when dry. A fired pyrophyllite annular end-piece was carefully drilled to accept six 8 B.A. binding posts for the connection of the furnace leads from the inside of the furnace to its exterior. The end-piece was fitted with its binding posts and attached to one end of the winding/former assembly using alumina cement. All internal connections were made and insulated with thin alumina tubes. The completed bare furnace assembly was tested by attachment to three sources of 0 to 2 amps. The current was slowly increased while the temperature was measured on three thermocouples. The furnace was maintained at around 1000°C (about 2 amps) to allow the cement to completely dry, to test the connections at high temperature and to allow the assembly to de-gas. After 8 hours the furnace was cooled and fitted within an outer furnace tube. This tube was also made of aluminous porcelain, 253 mm long with an external diameter of 62.1 mm and a bore of 51.0 mm. The cavity between the two tubes was filled with fine alumina powder. Two baffles of molybdenum sheet were included between the tubes positioned between each winding taking care not to induce any shorts between the

windings. These help reduce convection currents in the gas that flows within the alumina powder insulator. The construction was finished by fitting another fired pyrophyllite end-piece over the open end. The outer tube and alumina powder filler are thermal insulators. They protect the body of the pressure vessel from the high temperatures generated within the furnace.

The furnace has three separate windings, detailed in table T5.2.

TABLE T5.2. THE FURNACE WINDINGS			
Winding No.	Distance from the end of tube to the beginning of the winding (mm)	Length of winding (mm)	Resistance of winding (ohms)
1	50	55	41.2
2	110	55	40.4
3	170	65	44.3

The furnace was surrounded by pyrophyllite blocks for thermal insulation and support. The pyrophyllite blocks were positioned inside a tubular steel shell which was attached to the lead through end-cover of the pressure vessel. Plate P7.1. shows the assembly half dismantled. The top set of shaped pyrophyllite blocks and upper steel shell were fitted onto the top of this assembly prior to the introduction of the whole into the pressure vessel. A full description of the experimental procedures can be

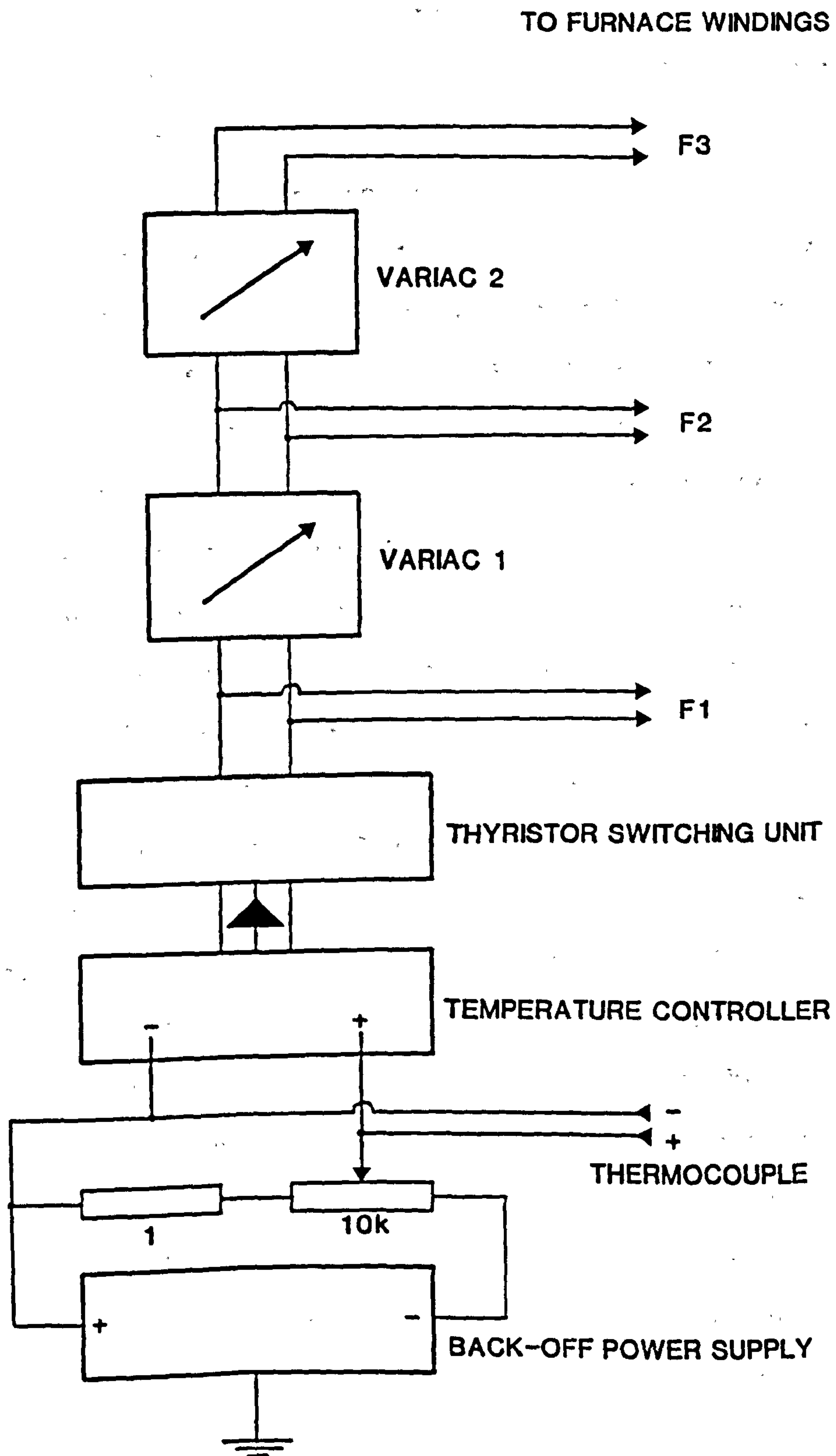


FIGURE F5.7. The heating and temperature control circuits.

found in chapter 7.

The power to each section of the furnace was supplied through a separate variable transformer. Variation of the relative currents through these transformers enables the temperature profile of the central portion of the furnace to be controlled. Most of the space within the pressure vessel is filled with solids as a safety measure to reduce the stored energy of the compressed gas system in case of sudden catastrophic failure of the pressure vessel. Despite this, convection within the gas in the pressure vessel and furnace is very strong. Tilting the vessel into the vertical position and varying the relative heating currents cannot produce a stable constant temperature in the sample region of the furnace but is able to generate a stable low temperature gradient such that the error in the temperature of the sample is minimised. The temperature profile in the sample region of the furnace was measured by four thermocouples (A,B,C and D) at low and high temperature and low (0.02 GPa) and high (0.20 GPa) pressure. The furnace currents were varied to reduce the temperature gradient across the sample under these conditions. The case of minimum temperature differential across the sample is shown in figure F5.8. The position of the thermocouples used in these measurements and for the rest of the experiments is shown in figure F5.9. The minimum temperature gradient ratios of furnace winding currents, shown in figure F5.8. were maintained throughout all the subsequent experiments. All four thermocouples were fitted to all experiments and although thermocouples C and D were used to monitor the sample temperature and provide a feedback signal for the temperature

FIG. F5.8. TEMPERATURE GRADIENTS GENERATED ALONG THE SAMPLE
(MINIMISED BY ADJUSTMENT OF HEATING ELEMENT POWER)

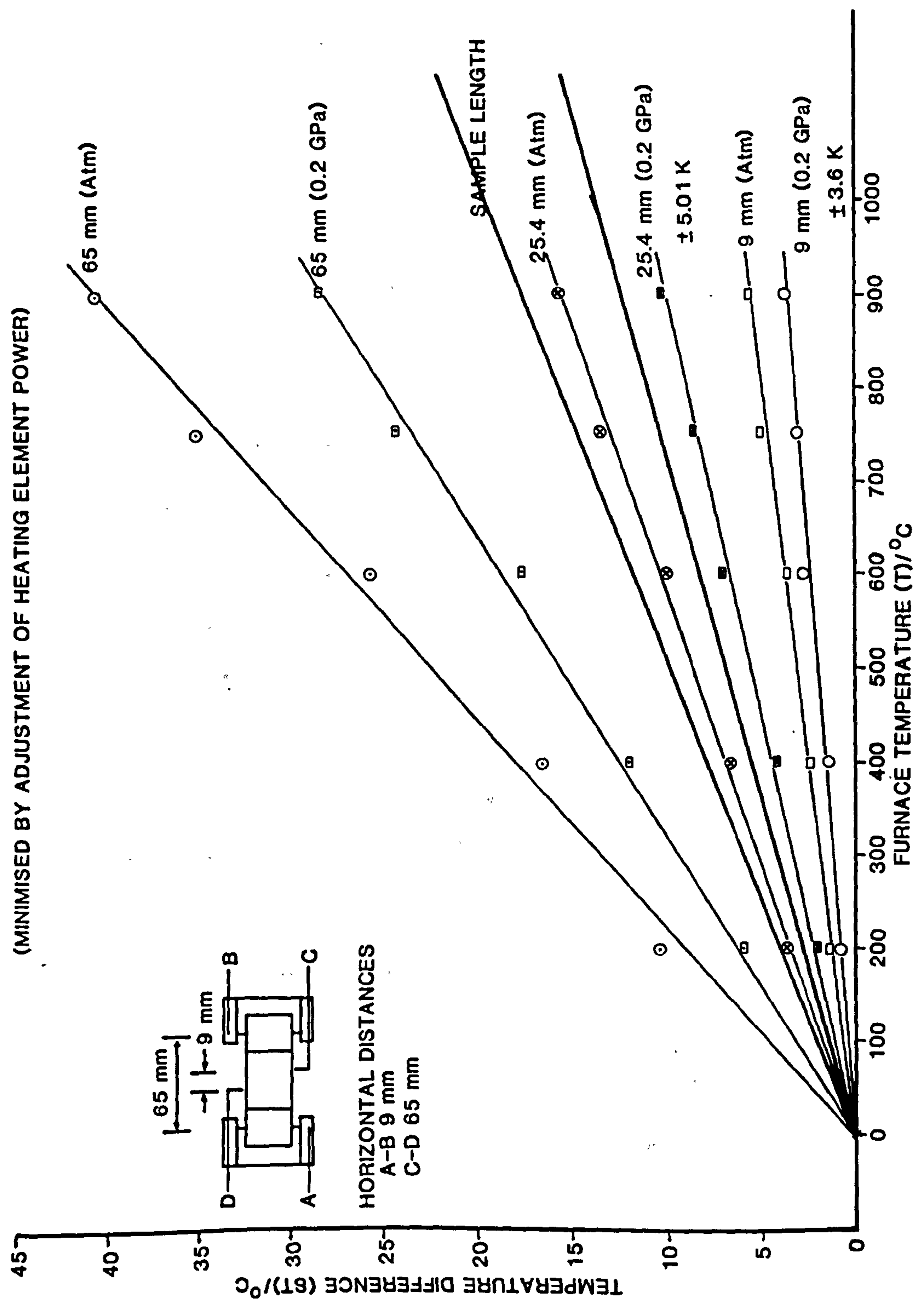
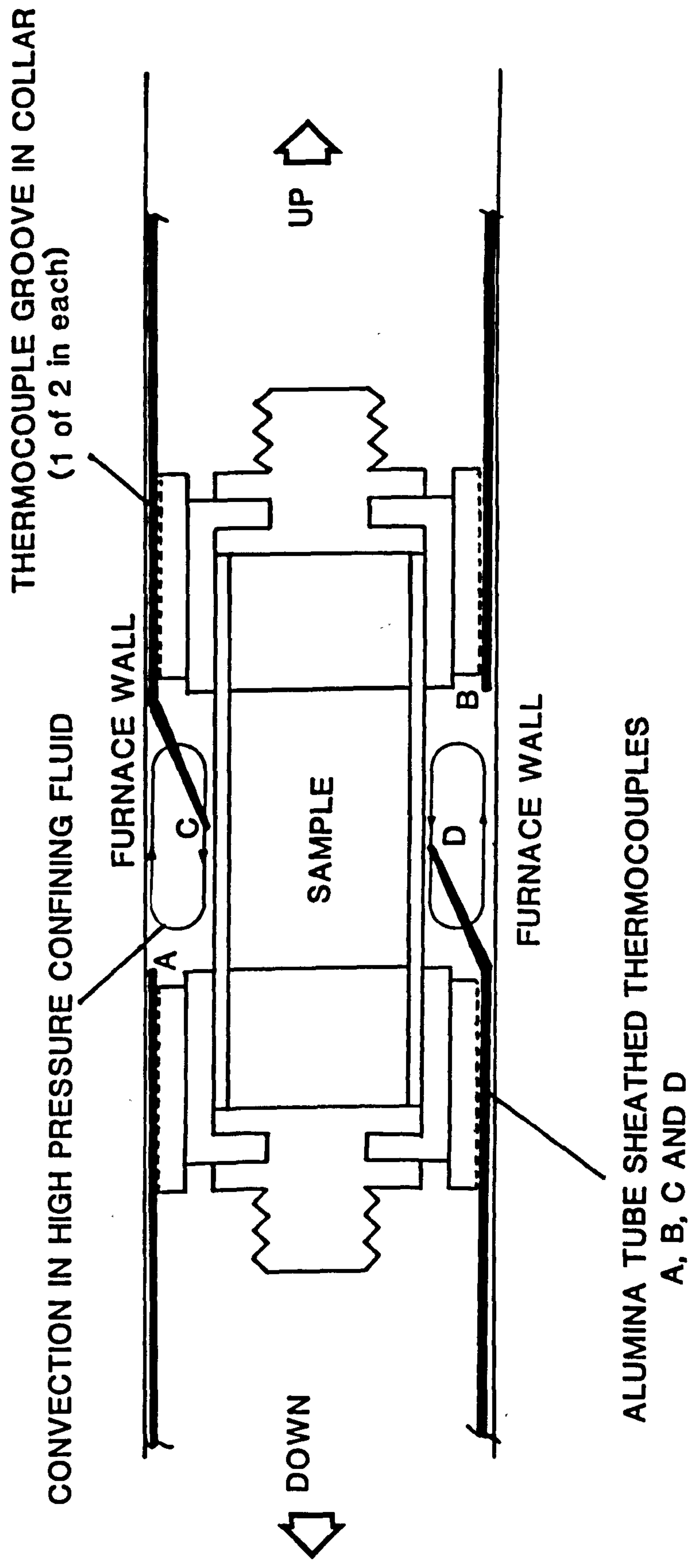


FIG. F5.9. THERMOCOUPLE POSITIONS IN RELATIONSHIP
TO CELL AND FURNACE INTERIOR



controller, periodic checks were made upon thermocouples A and B to check that the temperature gradient had not become excessive for any reason.

The current supply to the variable transformers was switched by a high current thyristor switching unit. The unit was triggered by a West Viscount 3 term temperature controller. The temperature control circuits are shown in figure F5.7. The temperature controller triggers the thyristor unit depending upon how the absolute value, differential value and integral value of an input signal compares with a preset potential. The input signal was derived from thermocouple C (figure F5.9.). The proportional, differential and integral components of control ensure that the temperature control is stable and does not oscillate or wildly overshoot the preset value. The temperature controller accepts signal and preset potentials up to 5 mV. The chromel/alumel thermocouples used in all experiments produce up to around 40 mV at 1000 °C. The apparent problem of too high a signal for the controller to operate was avoided by backing-off the thermocouple potential using a bench power supply and a variable resistor network in such a way that the input into the controller only became positive at thermocouple potentials higher than 30 mV. This allowed preset temperatures between 737 °C and 850 °C to be used. The thyristor unit switched an isolated mains supply. The switched and varied furnace current supplies were transferred into the safety room by shielded cables through roof ports.

5.7. TEMPERATURE MEASUREMENT, THERMOCOUPLES AND THERMOCOUPLE CALIBRATION

All internal temperature measurements were made with 28 swg chromel/alumel thermocouples. Both batches of chromel and alumel used in these experiments were supplied by British Driver Harris Ltd. Four thermocouples (A, B, C and D) were arranged within the pressure vessel as shown in figure F5.9. One of these (C in figure F5.9.) provided an input signal to the temperature controller. Initially all four probes were used to check on the stability and value of the temperature in the cell region of the furnace as has already been mentioned in section 5.6. Subsequently probes C and D were used to measure the temperature and probes A and B were periodically checked to ensure that the steady low temperature gradient remained across the sample.

Inside the pressure vessel each thermocouple wire was stripped of its enamel to minimise degradation and possible thermocouple contamination at high temperature. Each wire was insulated using ceramic beads, alumina tube and refrasil. Externally to the pressure vessel the probe wires were sleeved in PTFE tube, gathered in heat shrinkable tubing, and led to four cold junctions. The cold junctions were made by spot welding and crimping before being immersed in oil in protective quartz glass tubes. Two thermostatically controlled cold junction baths operating at about 45.00 °C were used to immerse the cold junctions. The use of these instead of ice/water immersion vacuum flasks avoids the necessity of opening the safety rooms to refill the vacuum flasks with ice while in the middle of an

experiment. The temperature of the cold junction baths was monitored during the experiment using a 10 channel Comark electronic thermocouple temperature meter. The reading on this meter was calibrated against a standard Pt - Pt 13% Rh thermocouple local standard up to 50 °C. The temperatures of the cold junctions were 45.07 °C and 43.76 °C and had maximum variations away from these values of 0.6% and 0.4% respectively. This variation is negligible. The small difference between the temperatures of the two baths was only taken into account when the temperature profile across the furnace was plotted. This was because during the experimental runs thermocouple C and D were used to measure the temperature and both of these had their cold junctions immersed in the same (second) cold junction bath.

The thermocouple connections from cold junctions to a Solartron 7055 microprocessor micro-voltmeter in the instrument area were made through a screened multicore cable that was passed, with the pressure gauge cables, through a port in the wall of the safety room. The signals from the thermocouples were fed into a multiplexing switch which was used to select which of the thermocouple potentials could be read on the micro-voltmeter. Conversion of these values to temperature was done on a programmable calculator. The potential from thermocouple C was also permanently connected to the temperature controller and backing-off circuits for control of the furnace temperature. The schematic layout of all of these sensors and their cabling is shown in figure F5.2. and figure F5.5.

The batches of enamelled chromel and alumel used in these experiments were calibrated against a quartz glass sheathed Pt -

Pt 13% Rh thermocouple local standard. This local standard thermocouple had been previously calibrated over a range of 273 - 1500 K to within 0.2 K at the National Physical Laboratory. Both the standard Pt thermocouple and a thermocouple made from the required batches of chromel and alumel were placed closely together in a sheath of alumina in a laboratory furnace. The furnace was heated to just over 1000 °C and then left to cool at its own rate. For every 10 °C or so drop in temperature the values of Pt - Pt 13% Rh thermocouple potential and chromel/alumel thermocouple potential were measured. A melting-ice cold junction was used. The voltage measurements were made on a Solartron 7055 microprocessor micro-voltmeter. The calibration data were analysed using linear and non-linear regression to provide a calibration accurate to within 1 °C over the range 0 - 1000 °C.

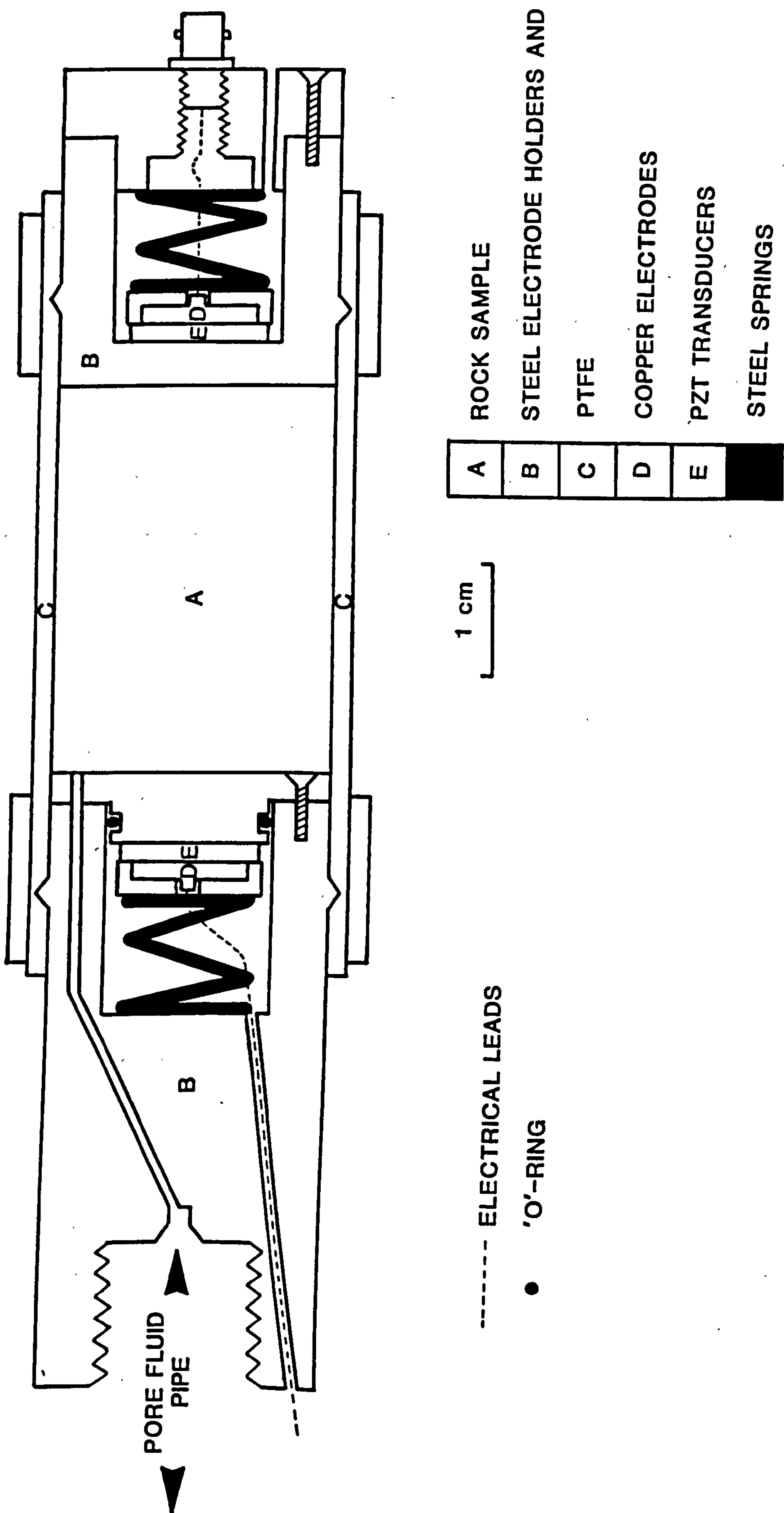
CHAPTER 6 : THE DESIGN OF THE CONDUCTIVITY MEASURING CELL

6.1. INTRODUCTION

This chapter describes the measurement cells that were designed and constructed during the course of this research. The cell has been designed to enable the measurement of electrical conductance of a sleeved rock. The task of enabling electrical conductivity measurements on rocks under simulated mid- and lower crustal conditions in the laboratory has involved a new approach to the design of a measurement cell for the containment of a fluid saturated rock and subsequent independent variation of its pore pressure. This has been entirely due to the lack of a suitable electrical insulator that remains refractory and insulating at temperatures that are thought to exist in the mid- to lower crust while remaining flexible.

The system previously used for making simulated upper crustal measurements of electrical conductivity was designed by Lee in 1974 (Lee, 1977). It could be used to temperatures of 250 °C and with confining and pore pressures up to 0.4 GPa. The Lee cell is shown in figure F6.1. The pressure and temperature constraints were due to the materials from which the Lee Cell, and in particular the sleeve, was constructed. The following sections describe the new cell design, how and why it differs from the Lee Cell and the various tests done on it and its component parts during its design. The Lee Cell is briefly described. Subsequently the various stages of the design of the new cell are described, including some of the design paths that were abandoned. The final cell will be described in detail throughout this chapter.

FIGURE F6.1. THE LEE CELL (After Lee, 1977)



6.2. THE LEE CELL

The Lee Cell, shown in figure F6.1., consists of two stainless steel electrodes which are offered up to each end of a PTFE sleeved cylindrical rock sample. Seals are made between the PTFE sleeve and the electrodes by forcing two compression rings over the PTFE at points where the cylindrical electrodes have a raised annular bump. The Lee Cell was also designed to house piezoelectric transducers to enable value of the seismic velocities to be measured. The cell has a connection for pore fluid in the longer of its two electrodes. It was constructed in two sizes to accept samples of 25.4 mm and 22.2 mm diameters. It has proved rugged and reliable during prolonged laboratory use.

The cell was designed for use in the oil pressure vessels of the Environmental Sciences high pressure laboratory at the University of East Anglia. It was designed for use up to temperatures of 250 °C and confining and pore pressures up to 0.4 GPa. The PTFE sleeving was needed because different fluids were used to transmit different confining and pore pressures and these needed to be isolated from one another. In this respect the Lee Cell (and the new cell) resemble a miniature flexible vacuum vessel that can be inserted within larger pressure vessels. The cells obtain their strength from the strength of the sample they contain. Lee showed that the seals on his cell worked well under the conditions he designed his cell for and that the PTFE remained sufficiently deformable to transmit the confining pressure to the sample (Lee, 1977).

6.3. SPECIFICATIONS

The retention of the axial cell arrangement similar to that employed by Lee, while enabling use up to 900 °C and 1 GPa constrained the new design and choice of materials. The first of the above considerations can only be obtained by the use of an internally heated gas pressure vessel with all its attendant safety implications. The first requirement of the new cell, therefore, was that it should be operable in an high pressure gas environment. The new cell design was also required to fulfill the following specifications:

6.3.1. Pressure Specification

The cell was required to house the sample in such a way that it could be subjected to confining pressures of up to 1 GPa and pore pressures of up to 1 GPa. These parameters were required to be independently variable while measurements of electrical conductivity were made.

6.3.2. Temperature Specification

All parts of the new cell were required to be refractory to at least 900 °C in an inert gas environment.

6.3.3. Stress Specification

The design was required to be such that high stresses would not exceed the yield stress of the material or otherwise cause mechanical failure.

6.3.4. Electrical Insulation

The requirement for certain parts of the cell to be electrically insulating arises from the need to reduce the flow of leakage currents from electrode to electrode around the sample to a minimum. Such leakage currents increase the apparent conductance of the rock sample. This specification was initially important in the case of the cell sleeving but also became important in the specification of the materials for the electrode holders in the later stages of the design.

6.3.5. Pressure Transmissibility

The material making up the sleeve was required to be sufficiently flexible to allow the transmission of confining pressure from the high pressure gas to the sample.

6.3.6. Chemical Attack

All the materials used were required to resist attack and corrosion by a range of agents at all considered working values of temperature and pressure. Potential corrosive agents were considered from the confining pressure medium, sample, pore fluid and interaction of materials making up the cell.

6.3.7. The Use of a Sleeve

The design required the use of a sleeved sample. Sleeving was needed to;

- reduce surface conduction,
- isolate the gas confining pressure medium from the pore fluid transmitting medium,

- enable independent variation of the confining pressure and the pore pressure.

6.3.8. Sealing

The use of a completely enclosed sleeved cell (for the reasons given in 6.3.7.) while retaining a cell design that is relatively simple to load requires the use of seals between the sleeve and electrode holders and between the electrode holders and electrode leadthroughs. Such seals must be sufficiently good to be proof against high pressure gas media at high temperatures and yet be sufficiently easy to put together to enable a relatively swift assembly of the entire experimental cell. This is necessary as the longer the cell assembly takes the more time the saturated pore fluid in the sample has to evaporate.

6.3.9. Machineability

The materials used in this design were, whenever possible, chosen to be as machineable as possible. In some cases, such as that of the alumina electrode holders, this specification had to be neglected so that previously mentioned and more important specifications could be fulfilled.

6.3.10. Price

Whenever possible the cell materials were chosen, taking into consideration the need to perform experiments within as limited a cost as possible.

6.3.11. Availability

Most of the materials used in the construction of the cell were chosen to be available off-the-shelf. In some cases materials were ordered but at no time were materials needed that could not be found off-the-shelf within Europe.

6.4. EXPERIMENTAL DESIGN PARAMETERS

Specification 1 requires the concurrent and independant variation of the confining and pore pressures. The need to use a gas pressure system combined with specification 1 dictated that the sample be completely isolated from the confining pressure transmitting medium. The isolation must be done in some way that the confining pressure can be transmitted through the isolator to the sample. This was enabled in the Lee Cell by the use of a flexible PTFE sleeve.

The Lee Cell PTFE sleeve satisfies all the above specifications except that relating to temperature. Lee designed his cell for use up to 250 °C. The new cell was to work at temperatures up to 900 °C, temperatures at which PTFE and all known flexible insulators decompose or become conductors. The sleeve in the axial cell arrangement needs to be electrically insulating. Failure to fulfil this specification results in large leakage currents and thus gives anomalously large apparent conductance measurements. The lack of any such sleeving material was initially felt to make the use of an axial electrode arrangement infeasible. It was found out, however, that the use of

electronics and a third electrode could reduce and almost nullify the effect of the leakage currents making a metal sleeved axial cell viable. This will be discussed in greater depth in later sections. Firstly the initial avenues of research will be described.

The design was forced to use some conductor as a sleeve. There are several metals which fulfil all the specifications, apart from that about electrical conductivity, several of which were eventually used.

Alternative electrode arrangements were considered. Such arrangements would either use the conducting sleeve as an electrode or limit its leakage effect as much as possible. These arrangements are shown diagrammatically in figure F6.2.

The radial electrode arrangement makes use of the conducting sleeve as one of the electrodes. This arrangement suffers from several problems. Firstly there are leakage currents along the flat faces of the sample. Fine machining of the sample and the material of the endpieces would minimise this but the surface porosity of the sample would ensure that there would always be some leakage current. Secondly every sample would need to be drilled to take a central electrode. This would be an awkward operation to do repeatably and accurately and would be virtually impossible on friable samples. It would also require some way of reliably making good electrical connection between the central electrode and the sample within the drilled hole. Such a connection problem would probably rule out the measurement of dry

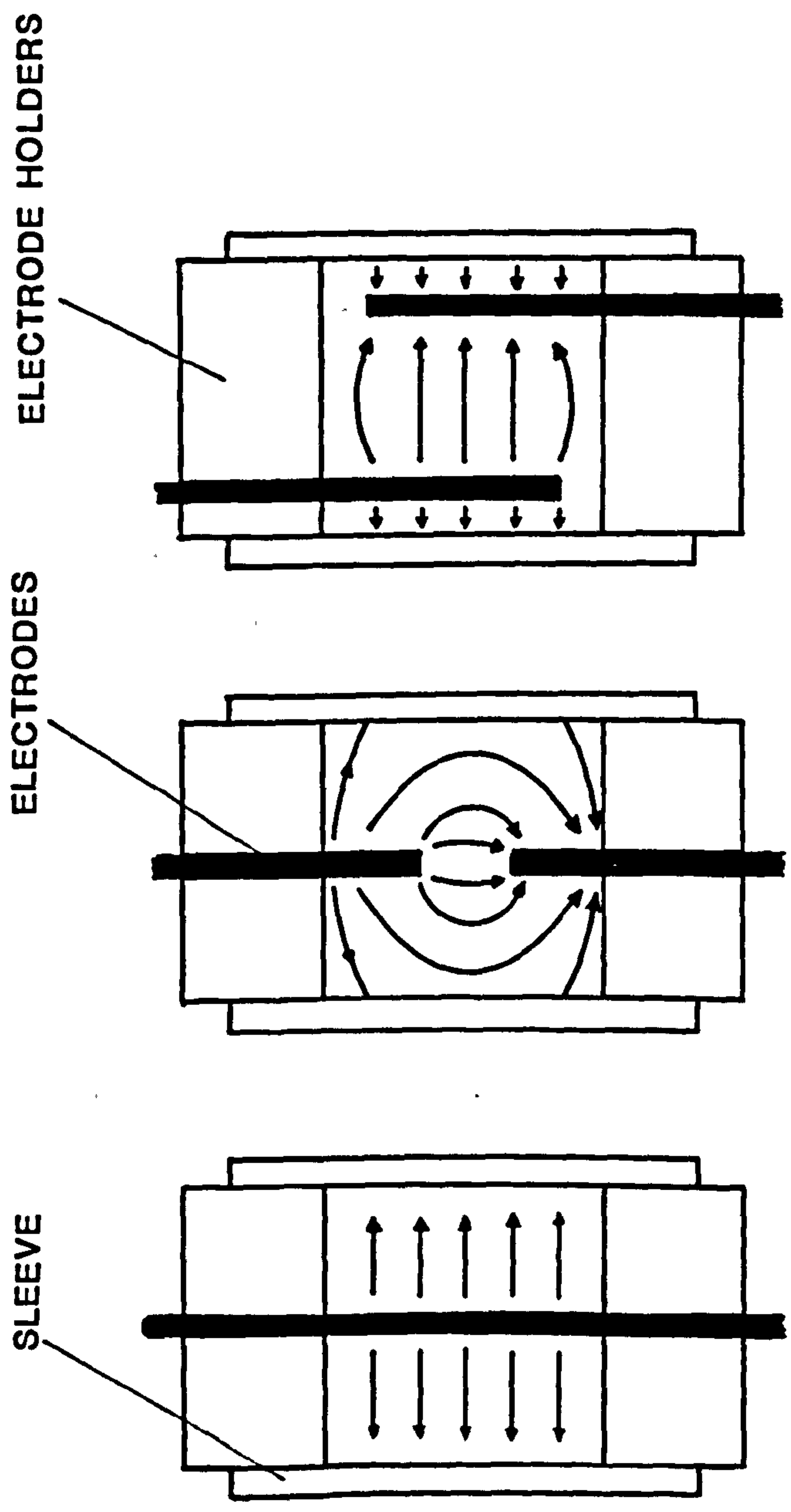


FIGURE F6.2. ALTERNATIVE CELL CONFIGURATIONS SHOWING CURRENT PATHS

samples. One way of ensuring a good connection that was briefly considered was the insertion of a liquid metal into the drilled hole which on solidification becomes the electrode. This was discounted as, a metal that would remain solid at the highest temperatures of the experiment would be very difficult to cast in this way, the very hot metal cooling in the rock would set up such high thermal tangential stresses in the rock that it would split apart and even if it did not fracture the high temperatures would cause local contact alteration. Thirdly, the very complex current paths that give rise to the geometric factor of the cell would need to be modelled by finite element computer techniques or found empirically. The current paths would have a distorted radial symmetry due to the leakage currents on the end faces of the cell. Lack of knowledge of the geometric factor would not allow the conductivity of the rock sample to be calculated from the measured conductance.

Pseudo-axial and pseudo-radial electrode arrangements were briefly considered. These do not make use of the conducting sleeve as an electrode but attempt to limit the effect of a conducting sleeve as much as possible. They both suffer from the same problems as the radial electrode arrangement but if anything are a worse choice because of the reduced but still considerable interaction with the sleeve. In these arrangements the measured volume of rock is also very much reduced.

A further problem that affects all the above arrangements is the need to arrange some leadthrough to supply pore fluid pressure through the end pieces. The addition of such a pipe entry

greatly limits the geometrical possibilities open to the cell designer as well as exacerbating the problem of leakage currents along the flat face of the sample where the pore pressure transmitting fluid would need to be delivered.

More complicated solutions were examined as the 'simple' geometrical designs had failed to fulfil the basic experimental specifications. One of these involves the use of guard-ring electrodes.

Guard-rings have been used to protect electrodes from stray or leakage currents (Cemic and Jansen, 1975). This technique is often used in semiconductor technology but rarely applied to larger scale measurements. It has been used in the measurement of rock conductivities in a few cases (Jackson, 1984). A guard-ring has not been used in the measurement of conductivities of rocks where the leakage currents were large and never in an arrangement with a conducting shunt path such as a conducting sleeve. Thus it was not known whether an actively driven guard-ring could mitigate the effects of a very large leakage current on the apparent conductance. It was proposed to retain the axial electrode arrangement but add a annular guard-ring around the current collecting electrode (collector). The guard-ring electrode would collect the stray current leaking through the sleeve of the cell and dispose of it without it being measured by the conductance measuring instrumentation. It was recognised that if this arrangement worked the measured volume of rock would still have a complex geometrical factor associated with it that would need to be determined empirically. It was also recognised

that electronics would need to be designed that would maintain the instantaneous potential of the guard-ring with respect to the current emitting electrode (emitter) the same as the instantaneous potential of the collector with respect to the emitter and that the end piece electrode holders would have to be made from some electrically insulating material.

The concern over whether the guard-ring system could remove the leakage currents sufficiently to enable valid rock conductance measurements to be made led to provisional experiments to determine the effect of different types of sleeving on a guard-ring and non-guard-ring set-up at normal laboratory conditions. These experiments measured the leakage and sample currents, the efficacy of the guard-ring and acted as a test bed to develop guard-ring driving circuitry. The progress and eventual success of this avenue of research is fully described in sections 6.7. to 6.9.

The adoption of the three electrode guard-ring axial arrangement imposes further constraints upon the cell design. The main constraint is that the end piece electrode holders need to be made of an insulating material.

The immediate design problems fell into nine main areas:

- A. Material for and design of electrodes.
- B. Material for and design of sleeve.
- C. Material for and design of electrode holders.
- D. Material for and design of electrode leadthroughs.

- E. Design of sleeve/electrode holder seal.
- F. Design of electrode leadthrough/electrode holder seal.
- G. Assessment of size of leakage currents.
- H. Testing of the viability of the guard-ring concept in this configuration using perspex and PTFE models.
- I. Design of a guard-ring driver and its testing on the prototype cell and resistor models.

These design problems will be treated in the following four sections. The first four problems will be described in detail in section 6.5. on materials and design. The following two problems will be described in section 6.6. on the design of the cell seals. The last three problems will be dealt with in sections 6.7. and 6.8. which discuss the guard-ring concept in detail and the detailed design of the guard-ring arrangement.

A schematic representation of the early provisional design for the new cell is shown as figure F6.3. It is labelled with the areas needing solving that are listed above.

Once the problems in these areas were overcome, working cells were made, fully tested and calibrated. This is described in sections subsequent to 6.8.

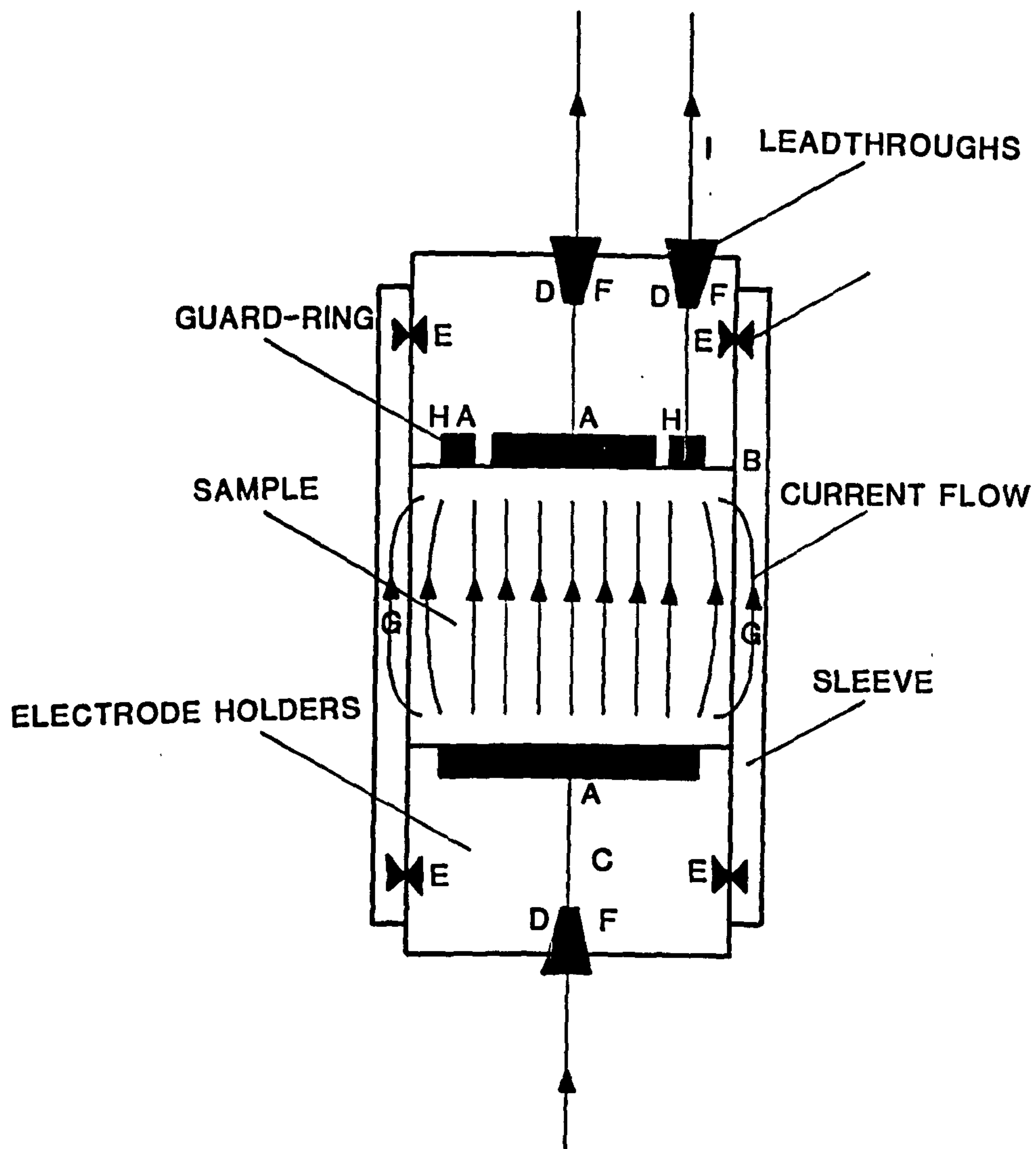


FIG F6.3. CONCEPTUAL DESIGN (omitting pore pressure connections through emitter electrode)

- | | |
|--------------------------|--------------------|
| A ELECTRODES | F LEADTHROUGH SEAL |
| B SLEEVE | G LEAKAGE CURRENTS |
| C ELECTRODE HOLDERS | H GUARD-RING |
| D ELECTRODE LEADTHROUGHS | I GUARD-RING DRIVE |
| E ANNULAR SEAL | |

6.5. MATERIALS AND DESIGN

This section will consider the constraints placed upon the design by the materials available for use, the search for suitable materials and their testing to ensure that they would operate well in the high temperature and pressure environment.

6.5.1. Electrode Holders

A solid block insulator that would fulfil all the specifications outlined in section 6.3. was required. It was realised that a block form of Al_2O_3 might be effective. This material is well known for its extremely good refractory and insulating properties. There was, however, some question over its reaction to stress. Like most ceramics the different forms of solid Al_2O_3 are brittle and although they have considerable compressional strength they have a very low tensile strength. During the course of research the following materials were considered:

- A. Pure single crystal sapphire
- B. Lucalox
- C. Pure recrystallised, extruded and sintered alumina by Thermal Syndicate Ltd (TSL)
- D. Pure recrystallised, extruded and sintered alumina by Morgan Refractories Ltd
- E. Pure recrystallised, cold isostatically pressed alumina by Degussa.

All the materials considered are able to withstand heating to at least 1000 °C. Samples of the various recrystallised aluminas

were heated in a muffle furnace and held at 1200 °C for two days with no visible macroscopic or microscopic changes.

The strength of an electrode holder made from alumina needed to be tested. If a form of alumina were to be used it would need to be 'drilled' to accept electrode leadthroughs and shaped in other ways to allow recess of electrodes. The differential pressures between the inside and outside of such a shaped thick-wall cylinder generate tangential tensile stresses which it was thought might cause failure of the material. A test on a cylinder of single crystal sapphire had been done by Lee and had shown failure at surprisingly low differential pressure. The test involved subjecting the solid sapphire to changes in confining pressure and 'pore' pressure up to 0.4 GPa. The failure occurred at a confining pressure of 0.37 GPa and a pore pressure of 0.15 GPa and involved a large set of fractures in the plane of the cylinder axis developing from the pore pressure inlet of the Lee Cell in which it was tested. There was a corresponding sudden increase in the conductance of the system as this had occurred.

It was thought that a non-porous multi-crystalline insulator such as pure recrystallised alumina (PRA) would have a better response to both compressional and tensile stresses due to the small strains possible along grain boundaries before complete material failure occurs. It is because crack growth can only occur within grains and is stopped when a grain boundary is reached that polycrystalline materials tend to be tougher than those which are monocrystalline. The grain boundaries act as effective damage

limiting horizons. The test done by Lee was repeated on two samples from each of three batches of pure recrystallised alumina from Morgan Refractories Ltd and from TSL.

The PRA from Morgan Refractories Ltd failed in the same mode as the sapphire though at slightly higher effective pressure. These samples were taken and stained with fluorescein in a similar way to which rock samples were saturated with electrolyte in the latter stages of the research (described fully in sections 4.7. to 4.9.). The stained samples were inspected by using a scanning electron microscope and an optical microscope. The width of the stained crack was the same size as the granularity of the alumina (about 10 μ) and appeared to be a slippage rather than an open crack. The cracks on both flat surfaces of both samples were mapped and optical micrographs were taken. No cracking showed on the curved surface of either sample. Both samples were resubjected to high pressure conditions in a repeat of the test. On withdrawal the samples were again examined under the scanning electron microscope and optical microscope and compared with the previous crack maps and optical micrographs. No further cracking was noted. The conductance across the PRA was monitored at all times during the high pressure tests. During the initial test the conductance increased sharply by 650 nS and 830 nS for each sample at values of confining pressure of 0.40 GPa and 0.38 GPa and pore pressure of 0.05 GPa and 0.04 GPa respectively. Although this is much smaller than the conductance jump experienced by Lee in his test on sapphire it is inferred that the sudden increase represents brittle failure of the sample. During the repressurisation tests the conductance

remained constant and very slightly decreased at high effective pressure as one might expect if confining pressure were closing the cracks. The presence of a conducting path through the cracks, however small and seemingly stable of nature, excluded the possibility of using alumina from this batch to make the electrode holders.

The same testing, staining and testing regime was applied to the PRA from TSL. No cracking was noted at any magnification and there was no sudden increase in the conductance.

It was thought that cracking in the first sample may have been due to the following:

- The batch was old and had remained in a laboratory drawer for several years. Brittle alumina stored in this way can acquire many microscopic defects. These may have weakened the PRA by helping to promote cracking.
- The method of manufacture of this and most PRA is by extrusion followed by sintering. Such a technique can leave a small but important isolated porosity (and as was latter found, a small but significant connective porosity) which could also help propagate cracking.

Two new batches of extruded and sintered PRA (ESPRA) were obtained from TSL and Morgan Refractories Ltd. These were subjected to the same tests as done on the previous batches of alumina and showed no cracking.

The SEM and optical examination of all the batches of ESPRA showed surface marks that suggested a significant porosity. It was important to ascertain whether this porosity was isolated or connective. ESPRA with connective porosity is unacceptable as the electrode holders are required to be insulators in the presence of high pressures and a conducting pore fluid. Manufacturers specifications quoted zero connective porosity. The samples of ESPRA were tested for axial gas leakage using a Leybold Hereus Helium leak detector. This instrument operates by applying a high vacuum to one side of a test piece to which a He tuned mass spectrometer is attached. The application of He to the other surface of the test sample by a micro-delivery gas gun tests for leakage points. If a leak is present, the small He atom is pulled through the opening by the pressure gradient. The mass spectrometer detects it and measures a leak rate by measuring the rate of arrival of He atoms. All samples of ESPRA showed a considerable and surprising leakage confirming connective porosity in the direction of the cylinder axis. All the samples were stained with fluorescein and dissected in an axial plane by a slowly rotating diamond saw. Each axial section showed several linear pore paths through the length of the sample. It was realised that the connective porosity was a relic from the extrusion process involved in the manufacture of the ESPRA despite the manufacturers specifications.

PRA is manufactured by another method which stops connective porosities forming. The process involves the packing of pure

alumina powder together under very high pressure before sintering. This is called the cold isostatic pressing method and produces what I will refer to as cold isostatically pressed pure recrystallised alumina (CIPPRA). The technique is guaranteed to produce a completely non-porous product as the high pressures remove all the pore space. Subsequent sintering stabilises the structure.

A batch of CIPPRA was obtained from Degussa (G.F.R.) with a little difficulty. Samples from this batch were tested under high pressure (0.4 GPa) and temperature (1200 °C) and showed no cracking. Fluorescein staining, optical microscope and SEM examination revealed no cracks, microcracks, surface marks, surface porosity or connective porosity in the dissected axial plane. Tests on the He leak detector showed no He leakage. Degussa CIPPRA was adopted as the material for use as electrode holders.

The CIPPRA had been tested to 1200 °C and 0.4 GPa with no failure. Although the subsequent use of the cell has only been to pressures of 0.2 GPa due to the unavailability of a sufficiently high pressure gas vessel, the initial aim was to design a cell to be used at pressures up to 1 GPa. A computer program was written to model the stresses in a machined axial cylinder of CIPPRA up to 1 GPa in the absence of being able to test the alumina up to these pressures. The tangential tensile stress (the highest tensile stress in a cylinder) was calculated and found to be considerably less than the tensile strength of the CIPPRA in the highly unlikely and worst case of pore

pressure; 1 GPa and confining pressure; atmospheric (from manufacturers specifications). In normal use the cell pore pressure will always be less than the confining pressure and hence the cylinder will be in compression. The strength of the ceramic is much greater in the compressive state than in a state of tension.

The resistance of CIPPRA to chemical attack is very good even at high temperatures. No attack by gases, electrolytes or other agents has been noted during the whole of this work. The sealability of the CIPPRA to a metal sleeve is discussed in a later section. The machineability of CIPPRA is poor. It cannot be shaped in any way but diamond grinding. This is a laborious process involving the skimming off of tenths of mm at a time by diamond coated wheels and points. Points are slim cylindrical tools of length considerably greater than their diameter. They are diamond coated and designed to grind small holes by rotating slowly while being lubricated. Over-zealous skimming of the hard CIPPRA strips the diamonds from their bedding and disables the tool. This is a particular problem when grinding small holes with fine points. During the course of this work several sets of points have been stripped in this way. CIPPRA is only available from Degussa in West Germany and is correspondingly expensive.

6.5.2. Leadthroughs

The design of leadthrough seals will be described in section 6.6. This section will be limited to a discussion of the choice of a material from which the leadthroughs could be constructed.

A metallic conductor was required that would be able to transfer signals to the electrodes in an inert gas, high pressure, high temperature environment. Several metals were considered:

- Niobium
- Tantalum
- Titanium
- Platinum

The most important consideration in the choice of a metal for the electrical leadthroughs was that of thermal linear expansivity. It was realised that if the leadthroughs were to be sealed to the ceramic electrode holders over a range of temperatures from 25 °C to 900 °C then it would need to be made of a material with a thermal linear expansivity matching that of the ceramic. CIPPRA has a thermal linear expansivity of $6.9 \times 10^{-6} \text{ K}^{-1}$. Niobium has the closest thermal linear expansivity to that of the CIPPRA of all the available refractory metals ($7.1 \times 10^{-6} \text{ K}^{-1}$). Other materials having a thermal linear expansivity in the useable range are Tantalum, Titanium and Platinum. Niobium was taken as the material of first choice. The niobium has a thermal linear expansivity slightly in excess of that of the CIPPRA. This assures that the seal remains tight as the temperature is raised but that the differential thermal linear expansivity is not so great that large tensile stresses are generated in the CIPPRA. A computer program was written to model the stresses caused by the thermal differential expansion between a thick walled cylinder of ceramic and a plug of metal for various temperatures, internal pressures and external pressures. It was found that the small thermal differential expansion only caused a small increase in

the tangential tensile stress at high temperatures and low pressures. These small tangential stresses were balanced out on application of small values of differential pressure (pressure external to cylinder minus pressure internal to cylinder) which is equivalent to the effective pressure mentioned in chapter 2. Application of greater effective pressures put the ceramic under a compressional regime in which it is very strong.

Niobium leadthroughs were heated in air, H_2 , N_2 and Ar. Slow but extensive formation of a flaky off-white/yellow oxide took place when niobium was heated in air to around 600 °C. The niobium was heated in the other gases to 1000 °C. No mechanical alteration was noted in any of these three experiments. A slight discolouration was noted after heating in H_2 and N_2 due to formation of small amounts of hydrides and nitrides respectively or the presence of oxygen as an impurity in the gases. No reactions were noted in the sample heated in argon. Reaction of the niobium with carbon was noted after the first few experimental runs. In these first experiments the leadthrough was gasketted with copper slip (very finely divided copper suspended in an oil mixture). At high temperatures the oil cracked in a relatively anoxic environment to produce carbon which reacted with the niobium to give the carbide. The subsequent corrosion led to weakness in the emitter electrode leadthrough and buckling of the pore fluid pipe in the electrode holder occurred. This problem was subsequently overcome by using finely divided copper alone as a gasketting material.

Niobium is a metallic conductor and has a resistivity

sufficiently low compared with that of the rock samples to be measured that it can be ignored.

Although a metal, niobium is difficult to machine as it cuts badly and requires patience and skill not to foul the cutting tool. This was a particular problem when cutting screw threads in the pore pressure leadthrough.

Niobium was readily obtained from Goodfellows. The price was high for rods of the metal of the correct radius.

The leadthroughs and the various tests done on them will be discussed further in section 6.6.

6.5.3. Electrode Material

The electrodes were required to sit in the recesses of their respective electrode holders and make good electrical contact with the rock sample. The electrodes had been initially made of stainless steel, though changing to tantalum or platinum had been considered. The important characteristics of the electrode material are that it should not have too high a linear thermal expansivity and it should be resistant to corrosion by electrolytes at high temperatures. Stainless steel is far from ideal in both of these respects. Tantalum has not been available in the correct sizes and platinum is very expensive. Stainless steel electrodes have served sufficiently well but have required changing every two or three experiments and cleaning after each experiment.

The stainless steel electrodes have a thermal linear expansivity which is considerably larger than that of the CIPPRA. The size of the electrode has ensured that the overall increase in thickness of the electrodes on raising the temperature is not sufficient to cause a gap to be formed between the rock and the electrode holder. Such a gap must be avoided to reduce leakage currents to a minimum. In practice the electrodes were made about 1.27×10^{-2} mm thinner than their recesses. This small amount was allowed for to be sure that the electrode did not project slightly from the surface of the electrode holder because of a machining error rather than to allow for differential thermal expansion.

6.5.4. Sleeve

The requirements of the sleeving material have been sufficiently well covered in the previous twenty two pages to limit its discussion here. A copper sleeve was used initially. This type of sleeve worked well in tests up to 0.4 GPa and 250 °C. It transmitted confining pressure to the sample well, showing the imprint of the rock sample on the internal face when cut off the sample at the end of the experiment. It was also sufficiently refractory in an inert gas environment. At temperatures higher than 250 °C the sleeve reacted with the pore fluid. A flaky black deposit of copper oxide was found on the inner surface of sleeves removed from saturated samples after high temperature experiments.

The sleeve material was changed to annealed stainless steel. The stainless steel underwent the same corrosion as the electrodes

but this was at no time sufficient to cause breach of the sleeve or have an effect upon the conductivity of the sample. Sleeves could only be used once as they had to be cut off the samples at the end of the experiment so the corrosion did not have time to build up over several experimental runs. It was found that the stainless steel underwent a subtle change of colour after being heated to high temperatures in nitrogen. No further changes were noted on runs in argon. The colour change may be due to absorption of nitrogen by the stainless steel.

Stainless steel sleeves were used on all but one of the final experimental runs.

6.5.5. Springs

Electrical connection needed to be made between the niobium leadthroughs and the electrodes. The close tolerances of the machining of the electrodes allowed this to be a simple electrical connection. The connection was made in the form of a spring to ensure that good electrical connection was present even at the lowest temperatures.

0.5 mm high temperature molybdenum wire was used to make the leadthrough springs. Construction of the springs was done using manipulators, a die and an illuminated magnifying glass. 0.5 mm wire produces a very stiff spring. The springs lose their stiffness at temperatures above about 500 °C but continue to allow current to flow from the leadthrough to the electrode unimpeded. At the higher temperatures the small differential expansion of the electrodes and the electrode holders and the

presence of a saturating electrolytic pore fluid ensures that good electrode/sample electrical connection is maintained.

6.5.6. Jaws, Restraining Collars and Jaw Holders

These items were all made from stainless steel. They suffered the same tarnishing as the sleeve when heated in N_2 and no such surface changes when heated in Ar as would be expected. These parts are isolated from any pore fluid and hence there was no corrosion from this source. In summary these parts are:

- refractory in Ar and N_2 to 1000 °C
- highly compressively strong
- unaltered by association with Ar, N_2 , Cu and CIPPRA over the range of experimental temperatures.
- machinable, low priced and easily obtainable.

The design of the seal of which these pieces form part will be described in section 6.6.

6.5.7. Electrical Connections External to the Cell

Three connections were made to the cell, one for each electrode. The emitter electrode electrical connection was made coincident with the pore pipe and so need not be discussed further here.

The collector and guard-ring electrode connections were made from molybdenum wire insulated with ceramic beads and refrasil cladding. The collector electrode connection was taken from a screw connector in the extended end of the niobium leadthrough. The guard-ring electrode connection was made by a small screw block connector to a short section of 0.5 mm molybdenum wire that

was inserted into a small hole and crimped into the guard-ring niobium leadthrough cone and insulated with ceramic beads.

6.6. SEALS

6.6.1. The Sleeve/Electrode Holder Seal

A gas tight seal was needed between the metal cell sleeve and the CIPPRA electrode holder. This seal corresponds to the annular seal designed by Lee (1977). The seal designed for this research is annular like the Lee bump seal, but other than this the two sleeves have little in common. In many ways the two seals have opposite sealing philosophies. The Lee seal uses the deformation of a malleable material between a raised bump and a restraining collar whereas the new seal involves the swaging of a thin flexible sleeve into a groove. Parts of this seal were developed in conjunction with Harj Jolly of the School of Mathematics and Physics of the University of East Anglia.

The new seal consists of;

- An annular V-shaped groove in the electrode holder.
- A copper circlet
- Four stainless steel jaws
- A stainless steel jaw holder
- A restraining collar.

and two special tools:

- A wheel tool to swage the sleeve metal into the groove.

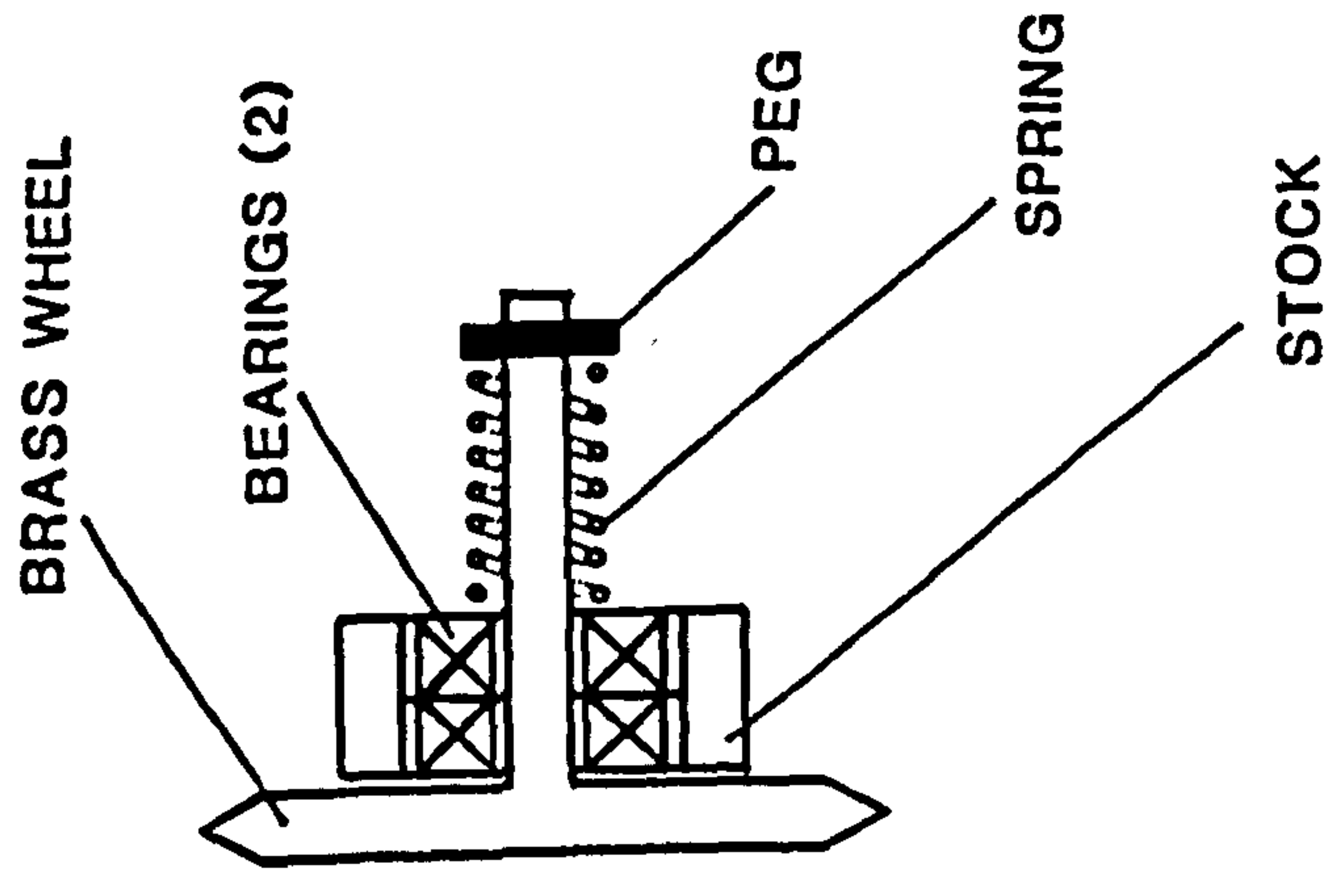


FIGURE F6.4. THE WHEEL TOOL

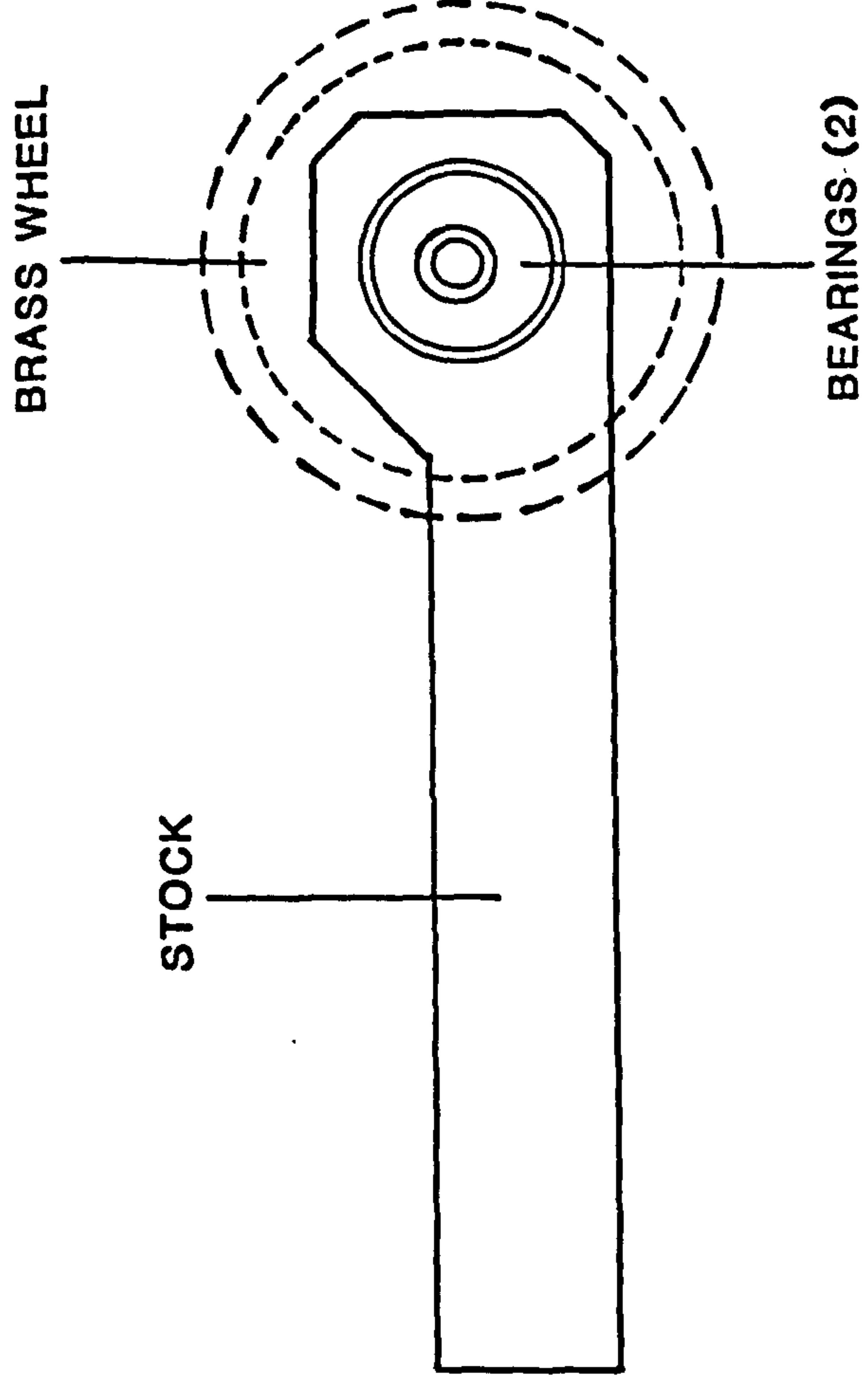
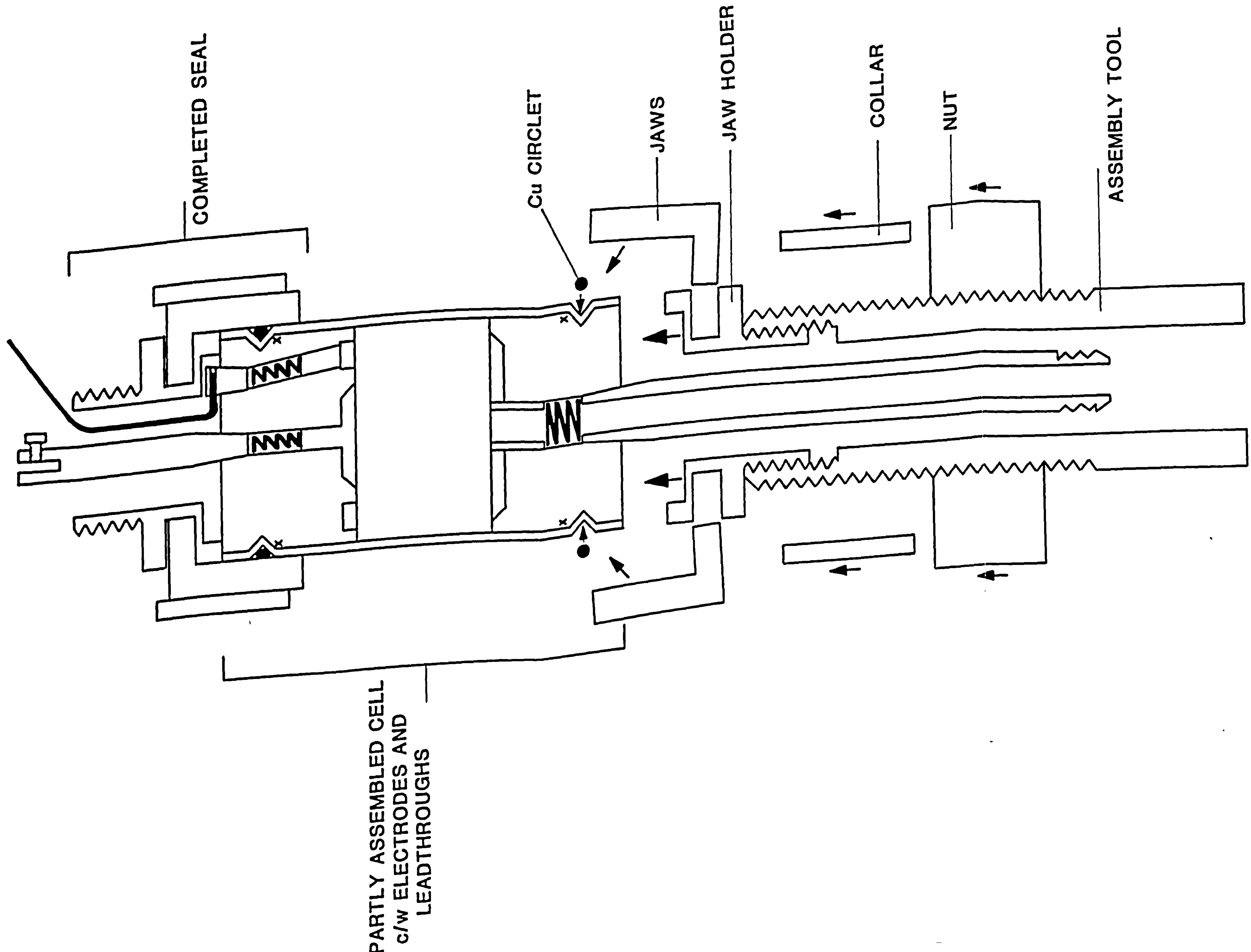


FIG. F6.5. THE ANNULAR SLEEVE SEAL DURING ASSEMBLY.
EXPLODED AND COMPLETED VIEWS



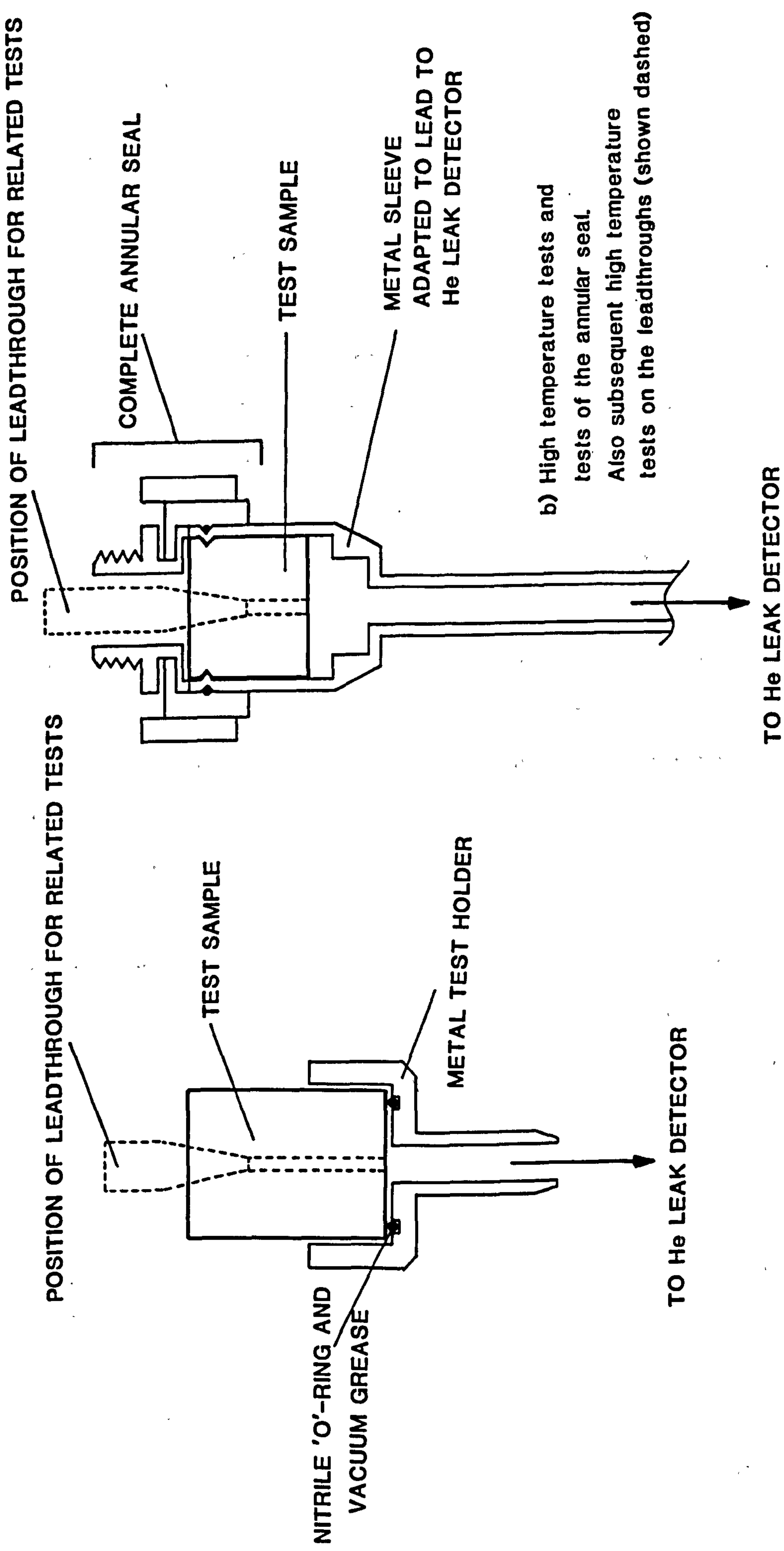
improve the seal at high temperatures. As the temperature is raised the copper circlet expands at a greater rate than the stainless steel parts or the CIPPRA electrode holder. This relative expansion increases the pressure on the sealing area improving the seal.

4. The external face of the jaws and the internal face of the restraining collar are slightly tapered in opposite senses. This allows the gradual increase of the radially compressive force transmitted through the jaws to the seal area as the collar is progressively slid up around the jaws.
5. The assembly tool allows the cell to be held tightly during assembly as well as allowing a large screw thread travelling bolt to push the collar up over the jaws very tightly.
6. The jaws were made in one piece and then cut into four by a slitting saw to ensure symmetry of radial compression arising from jaws that match each other as well as possible.

Leakage tests have been done on test pieces in which a specially adapted sleeve was sealed to a blank piece of CIPPRA. The adapted sleeve was fitted to a helium leak detector as shown in figure F6.6. Tests were done on a simple swaged groove seal and with the complete seal at laboratory temperatures and at 600 °C with the test piece in a muffle furnace in an inert gas environment. Leakage was only found in the case of the simple swaged groove at high temperatures. The full seal design is shown

FIGURE F6.6. SEAL TESTS

a) Low temperature tests for solid blocks and



as part of the full diagram, figure F6.7.

6.6.2. Leadthrough Seals

Three leadthrough seals were produced, one for each of the electrodes. Two of them are solid cones and one is drilled to accept the pore fluid connection. Cone seals operate well in environments where the internal/external pressure gradient is always in one direction. Providing the pressure on the larger surface of the cone (in this case the external cell surface) is greater than that on the inner face (in this case inside the cell) the cone is pushed further into its cone shaped bed and the seal is maintained. The seal is made tight at lower differential pressures by making the included taper angle small (in this case 14° included angle). Such a geometry ensures that the taper is self-locking. The sealing face of the cone and the cone bed are shaped to fit each other by the use of diamond grinding with progressively finer diamond dust/cutting oil mixture.

The initial design for all three seals was a 14° included angle, diamond ground self-locking taper niobium plug in a cone-shaped bed in the CIPPRA with copper slip gasketting material. Helium leak tests were done on these leadthrough seals in a similar way to those done in section 6.6.1. No leaks were found before, during or after raising the temperature to 600°C . The test pieces were dissected along an axial plane and the seal surface was examined; the seal showed no visual signs of degradation. It was postulated that the seal would perform even better at high external pressures relative to the inside of the cell. This seal was used for the preliminary experimental runs. Examination of

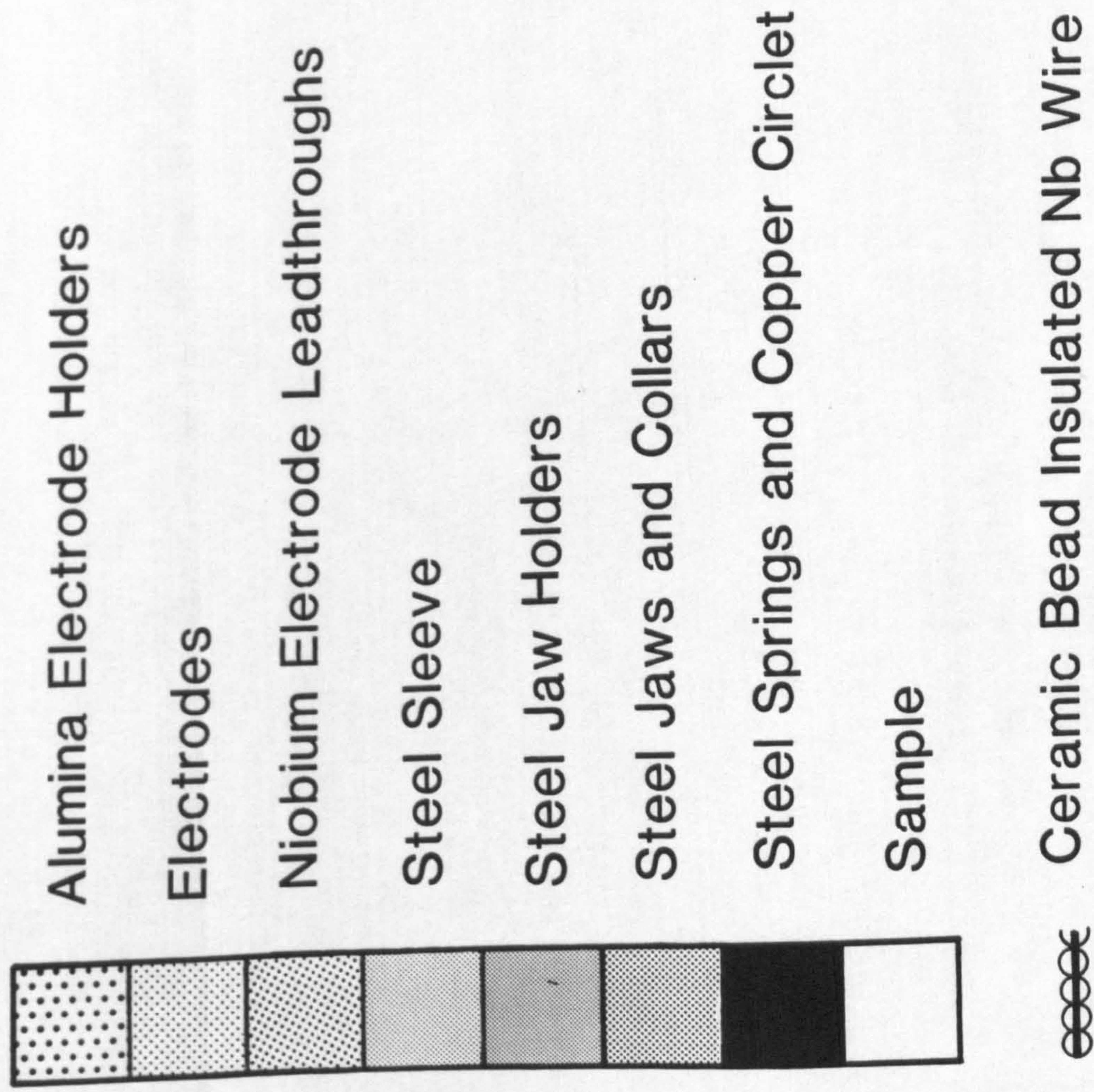


FIGURE F6.7. THE FINAL DESIGN

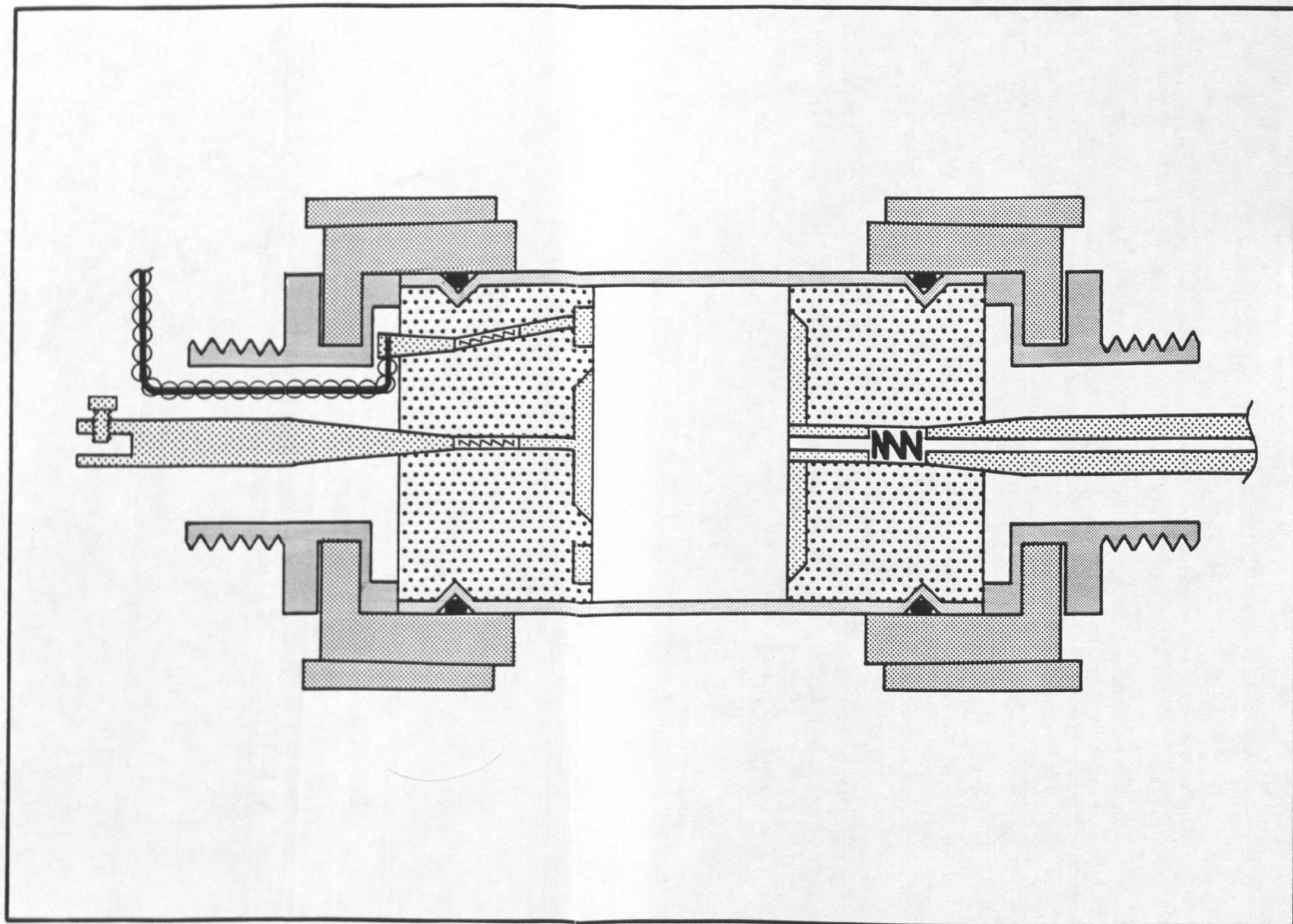




PLATE P6.1. THE FINAL HIGH TEMPERATURE AND PRESSURE EXPERIMENTAL CELL

the seals after these experimental runs showed considerable carbide formation along all the sealed surfaces and some buckling of the sealed surfaces of the emitter electrode/pore fluid leadthrough seal. No leakage had occurred but it was plain that this would have soon been the case. The problem is caused by the particular affinity niobium has for forming carbides whenever in the presence of free carbon. The high temperatures used in the experiments cracked the organic compounds that form part of the copper slip. In the relatively anoxic conditions carbon was formed which subsequently reacted with the niobium causing carbide formation and corrosion. The corrosion especially weakened the thin walled cone end of the emitter electrode/pore fluid leadthrough which was partially buckled and extruded into the CIPPRA central hole. This problem was overcome by subsequently using finely divided copper powder as the gasketing material. The bore of the emitter electrode/pore fluid leadthrough was also reduced to make it stronger at its thin end to avoid extrusion.

Although each seal is of a simple cone design, each leadthrough is different. The collector electrode leadthrough cone is extended about 3 cm from the external face of the electrode holder to allow a screw connection to be made to an external lead to the pressure vessel leadthroughs. The screw connector can be seen in figure F6.7. The guard-ring electrode leadthrough is a simple small cone plug with a low relief on the electrode holder external surface. This surface is slightly recessed to allow a wire connection to be made to the leadthrough through a small hole drilled in the side. Good mechanical connection was then

made by crimping the top of the cone onto the wire using a centre punch. The third leadthrough is the larger drilled emitter electrode/pore fluid leadthrough. This is a drilled cone with a relatively thick wall that is extended external to the cell to become a pore pressure pipe. This pipe is connected to the capillary that runs through the pore pressure leadthrough in the pressure vessel end-cap by a standard high pressure connector.

6.7. THE GUARD-RING - CONCEPT AND INITIAL TESTS

6.7.1. Introduction

Guard-rings have been used for many years to make measurements in situations in which two or three dimensional infinite symmetry can be assumed or approximated (Field, 1954). For example, guard-ring conductance measurements are often used to monitor the thickness of sheets of electrically conducting materials in their manufacture. Commercial probes for this type of application can be bought. Theoretical studies have been done on the shape of current flow in such situations and mathematical models have been generated (Amey and Hamburger, 1949). Guard-rings have, however, fallen out of favour in many areas of scientific research remaining popular only in semiconductor microengineering. They have been used in the measurement of the electrical properties of rocks (Cemic and Jansen, 1975; Will et al, 1979) but only to reduce the surface conduction errors. Such errors can much more efficiently be reduced by good jacketing (Lee, 1977; Olhoeft, 1979b). Guard-rings have never been used to compensate for massive leakage currents through a conductive sleeve as proposed

here.

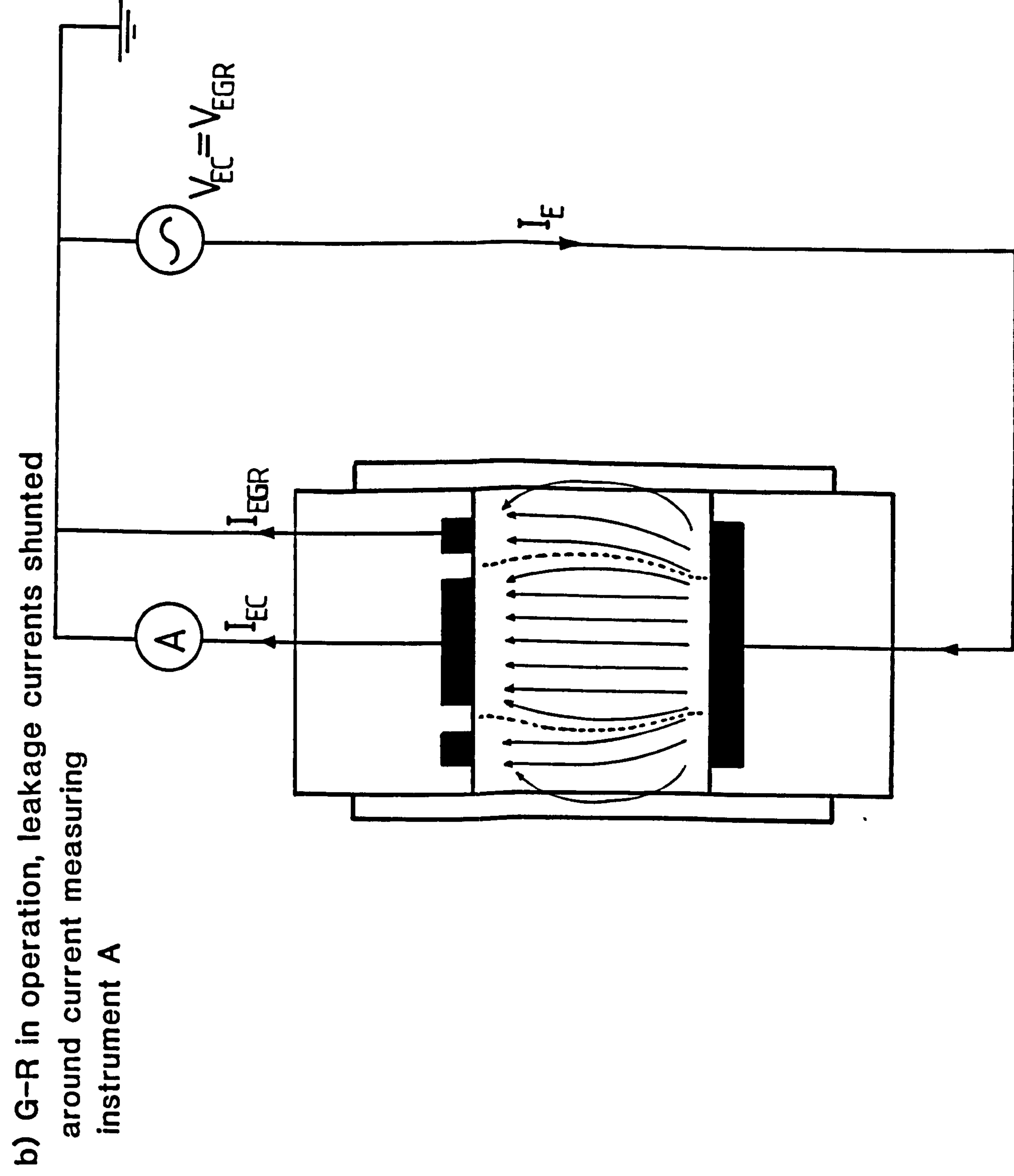
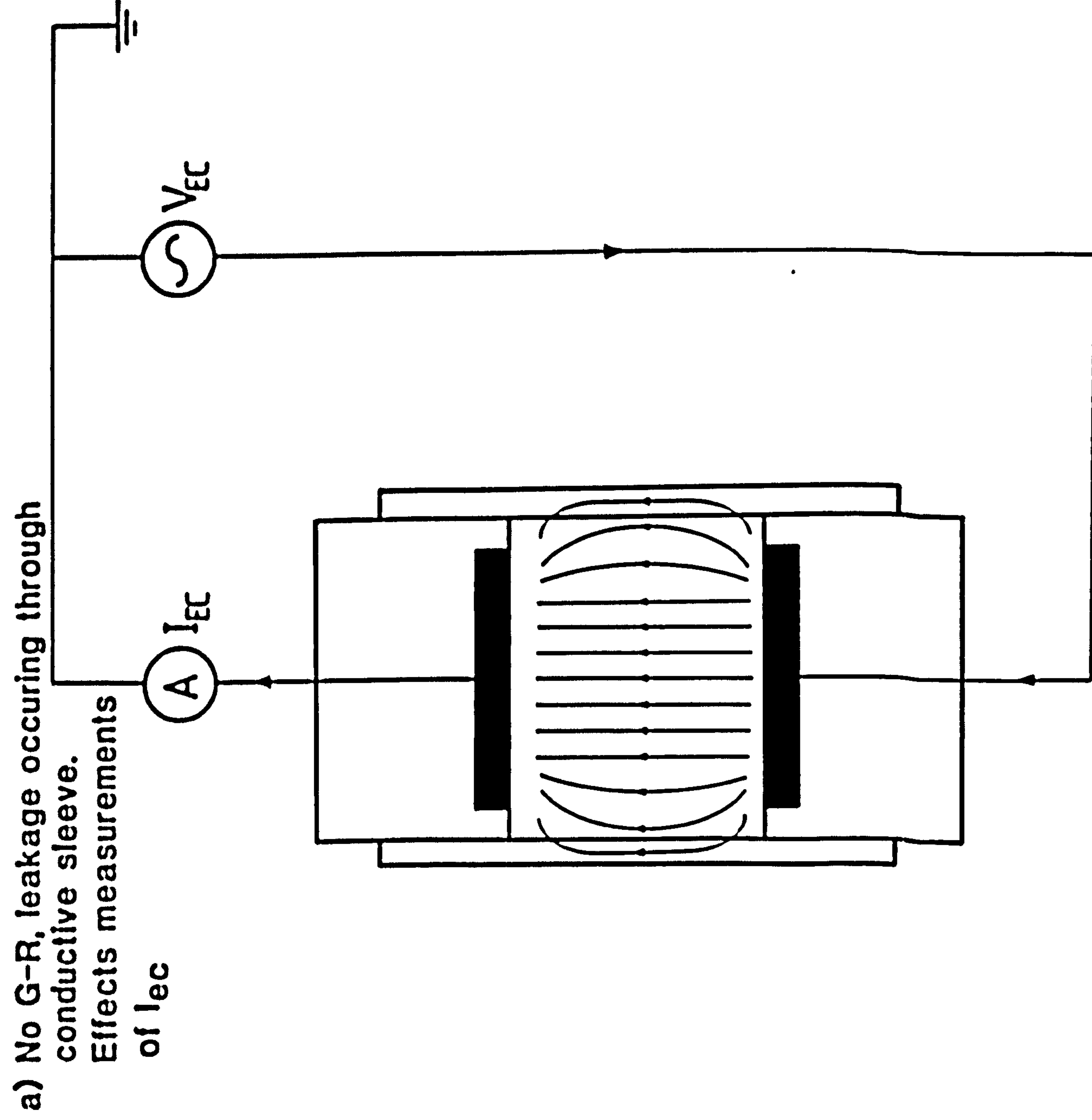
6.7.2. Concept

The next part will describe how the guard-ring works in this particular application with reference to figure F6.8. The current flowing through the system can be classified into two types;

1. current flowing directly through the sample
2. current leaking through the sleeve, along surfaces or through the rock but on a path that is sufficiently distorted by the presence of the conducting sleeve to give rise to an anomalous conductance.

Measurement of conductance involves the generation of a known potential between the electrodes. The current which flows depends upon the effective conductance of the whole sample and cell system and is made up of currents 1 and 2 above. The ratio of the currents 1 and 2 depends upon the conductance of the path from one electrode to the other set up by the rock sample and the leakage and distorted current paths respectively. If one needs to measure the conductance of the rock then one needs to measure current 1 only. Unfortunately current 2 also flows and in the case of the metal sleeved sample is larger than current 1. The arrangement in figure F6.8a. cannot distinguish between these two currents. In this case the measured current is the addition of these two components in unknown ratio. As current 2 is larger than current 1 the conductance of the system calculated from the total current will be significantly lower than that of the rock sample alone. When the conductivity of such a measured rock

FIGURE F6.8. THE GUARD-RING CONCEPT



sample is calculated it too will be lower than the real value. Another complication arises here, as the current paths in the unguarded case are so complicated (figure F6.8a.) and flow through several media takes place, the conductivity so obtained can only be seen as an effective conductivity for exactly that rock/cell system and may not have any real bearing on the conductivity of the rock at all. To make a valid measurement of the conductance of the rock we need to make an accurate measurement of the current flowing through a smaller coaxial sub-cylinder of the rock where the current flow lines are more or less normal to the end faces between a known potential difference. Knowledge of the effective area and length of this sub-cylinder would give a value for the rock conductivity. The problem arises due to the large leakage currents that will inevitably flow along the less resistive leakage pathways to reach the lower potential electrode. These currents need to be gathered and disposed of without affecting the measurement of that part of the total current flow that flows from one electrode to the other through the rock sample. The guard-ring electrode improves the measurements in the following way. The guard-ring electrode has an annular shape and is placed around the collector electrode as shown in figure F6.8b. It is kept at the same instantaneous potential with respect to the emitter electrode as the collector electrode. Therefore no current flows between the collector electrode and the guard-ring despite their close proximity and hence the relatively low resistance between them. If the guard-ring is so connected that it presents an alternative sink for the current (other than the conductance measuring bridge) then the guard-ring will collect all the leakage currents

and currents with grossly distorted paths (all those outside the dotted line in figure F6.8b.) and divert them away from being measured by the conductance measuring equipment. As the guard-ring operates at the same potential as the collector electrode the current path of the currents flowing through the body of the rock can be measured accurately. These currents have paths that form a minimally distorted barrel shape. Calibration of these measurements with measurements taken of the same samples in a insulating sleeve with very negligible leakage currents enables an effective area to be calculated for a barrel shape of current of a given known length. Hence the conductivity can be calculated.

6.7.3. Factors Affecting the Use of a Guard-ring

The individual dimensions of the sample and cell affect the shape of the total current flow pattern. If the ratio of radius of the sample to its length decreases (the sample becoming long and thin), the leakage currents increase at the expense of the current flowing through the body of the rock sample. This is obviously to be avoided. If the ratio increases (the sample is more coin-shaped) the ratio of current through the rock to leakage current will be progressively increased. There comes a point, however, when several factors collude to stop this process acting for the betterment of the experiment. At some point the measurements will not be characteristic of the rock being studied but will depend to an increasing amount on the individual minerals. This depends upon the grain size of the sample being studied. Also as the length of the sample is decreased, the measured conductance is being increased, there will come a time

when the measuring instrument will reach its current measuring limit for any given fixed potential across the sample. The actual values of length and diameter of the sample chosen for this study are discussed later in this chapter. They have been chosen with the above criteria borne in mind, but may not necessarily represent the optimum geometry for samples of this grain size. The size of the samples were kept the same throughout the study so calibrations could be done and rock conductivities calculated. The barrel shape volume through which the measured current flows is difficult to calculate accurately, although a limited number of solutions have been found (Amey and Hamburger, 1949). The current flow problem is further exacerbated by the measurement of essentially heterogeneous rocks with a finite grain size. The determination of the effective area through which the current flows has to be done empirically. The accuracy with which this calibration is done controls the accuracy of the final conductivity measurements. The accuracy depends to a large extent upon the distortion of the central barrel of current flow which in turn depends upon the ratio of the measured current to the leakage current, geometry of the sample, efficiency of the guard-ring driving circuits and the accuracy of the current measuring circuits.

The success of a guard-ring system such as the one described depends upon the efficiency with which it is driven. The electronic driving of the guard-ring circuit rises some very important points. The final circuitry will be described in

section 6.8.

The circuit must be able to generate the same potential with respect to the emitter electrode as the collector has. The collector potential has to be monitored without altering its electrical characteristics. The guard-ring connection must be able to sink a large current with its potential relative to the emitter electrode unchanged compared with that of the collector. A further complication arises due to the need to make measurements using alternating current. The use of A.C. removes otherwise difficult problems of electrode polarisation. The introduction of A.C. also introduces the problems of driving an alternating guard-ring potential in which the instantaneous potential of the guard-ring with respect to the emitter electrode has to be the same as the instantaneous potential of the collector with respect to the emitter electrode. Thus the guard-ring potential needs to be controlled in both amplitude and frequency and be in phase with the collector potential. Research was needed to design a guard-ring driver that would fulfill these requirements.

The use of the guard-ring concept to compensate for the errors induced in electrical conductance measurements by leakage currents has not, to my knowledge, been attempted before. This may not necessarily be surprising as the initial feeling was that such a guard-ring could not possibly sink a large leakage current compared with a small current to be measured, without having a considerable effect upon the measured current accuracy. For any given effective conductance of the whole system and a fixed

potential difference there is a limit to the total current that can flow. If the measured current to leakage current ratio is too small then the measured current may become too small to measure. The effect of altering the geometry as a way of optimising the ratio has already been mentioned. The accuracy of the measuring instrument and the effectiveness of the guard-ring driving circuits also have an obvious role in the ability to make the measurements valid. If, however, the overall ratio of measured current to leakage current is initially too small then the entire concept will not work, no matter how the other factors are optimised. Review of literature produced no further information and the Amey and Hamburger (1949) studies were of no direct use as they assumed a non-conductive boundary condition on the sleeve face. It was thought that the leakage current magnitude would depend upon the conductance of the current path along the end faces of the electrode holder, next to the sample, the sleeve effectively being an electrical short. It was calculated that this may give rise to a measured current/leakage current ratio (I_{EC}/I_{EGR}) of 1:10 although this was later to prove to be too low. Laboratory condition tests needed to be done on prototype cells to ascertain the correct value of the ratio and to discover whether the leakage currents could be collected in the way described above.

Laboratory bench tests were embarked upon to:

1. Develop electronics to drive a guard-ring electrode potential with amplitude and frequency controlled accurately and in real time by the

collector potential.

2. Test the efficacy of such a system on an array of resistors modelling the behaviour of the cell, and on the cell itself.
3. Measure the I_{EC}/I_{EGR} ratio for the conductive sleeved, insulator sleeved, guard-ring and non guard-ring systems and compare it with similar measurements on the modelled array of resistors.
4. Test the extent to which the use of the guard-ring and driving electronics could compensate for the addition of a conductive sleeve by shunting the leakage current away from the current measuring circuits.

6.7.4. Initial Tests on the Prototype Cell and Electronics

Reliable and accurate guard-ring driving electronics needed to be developed before much of the initial tests on the guard-ring cell could take place. The initial work was done with the advice of Dr. G. Turner of the University of East Anglia. The circuits to drive an A.C. guard-ring were initially made up from standard commercial equipment. Many different combinations of these were tried, but all were of a basically similar philosophy; a signal generator with amplitude and frequency either adjusted manually or automatically and synchronisation to the collector output of the conductance measuring bridge. Various modes of synchronisation were tried including triggering both the bridge and the signal generator from an external frequency generator. The potentials between the guard-ring, collector and emitter (V_{EGR} , V_{EC} and V_{CGR}) were monitored at all times using two dual

beam oscilloscopes. It was found that the guard-ring was driven very well in some cases and very badly in others. However well the guard-ring was driven initially it took only a few minutes to become unstable, desynchronise and then fail. It was found that even a slight discrepancy in frequency amplitude or synchronisation caused the guard-ring potential to be badly driven. Some form of circuit was needed that would sense the instantaneous potential amplitude, frequency and phase of the collector and reproduce it for the guard-ring in such a way that any variation in any of the parameters of the collector potential would be automatically compensated for. This requires that the monitoring of the collector potential does not interfere with its operating potential and that the potential generated at the guard-ring would depend only upon that of the collector and not on the amount of current flowing to it. The electronics developed to do this were based around a semiconductor potential sensor and buffer. The circuit was designed around a voltage follower wiring of a 741S operational amplifier. In simple terms it senses the alternating potential generated by the Wayne-Kerr conductance bridge at its collector output. The addition of an extra connection to the collector circuit does not cause error in the conductance reading by shunting current away from being measured in the bridge as the input impedance of the guard-ring driving circuit is very high. The circuits then use the monitored collector potential to generate an identical potential at the guard-ring varying as the collector potential in every aspect. The guard-ring circuitry has, however, a sufficiently small output impedance to enable it to drain any leakage current to earth by-passing the Wayne-Kerr bridge. The size of this

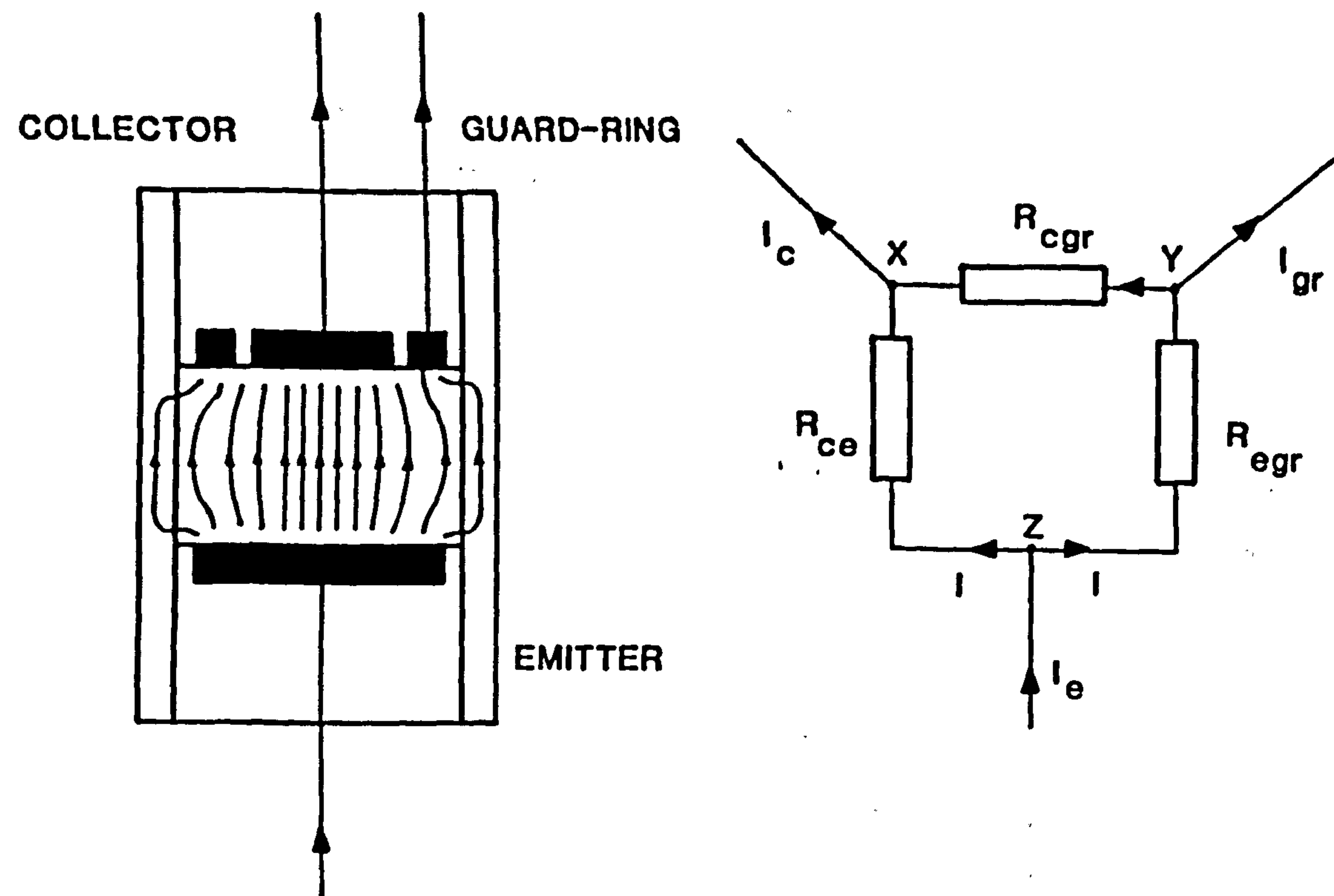
current can be large without significant effect on the potential generated at the guard-ring because of the very small output impedance of the circuitry. The detailed design of the circuit is given in section 6.8.

The guard-ring driving circuits were initially tested on an array of resistors connected to model the expected behaviour of the proposed cell. The resistors were readjusted to more realistic values in the light of subsequent experiments on the cell prototypes and it is these values which are used here. A diagrammatic representation of a prototype cell and the array of resistors used to model its behaviour is shown in figure F6.9. The array has three resistors (R_{EC} , R_{EGR} and R_{CGR}) representing the rock resistance, leakage resistance and collector electrode/guard-ring shunt resistance respectively. Subsequent measurements on a prototype cell showed that;

$$R_{EC} \sim 3 R_{EGR} \sim 30 R_{CGR}$$

R_{CGR} is small due to the proximity of the collector and guard-ring electrodes. I_{CGR} is small, however, due to the negligible instantaneous potential difference between the collector and the guard-ring when the driving circuitry is working. The Wayne-Kerr conductance bridge was used to make conductance measurements on the array without the guard-ring circuits attached, with them attached but turned off and with them operating, as well as on the individual resistors isolated from the rest of the array. The individual measurements of conductance were used to predict the array conductance without guard-ring driver connected (point Y floating). The measured value of array conductance agreed with this figure. Calculations were done to find the currents flowing

FIGURE F6.9. THE MODEL ARRAY OF RESISTORS AND
EQUIVALENT CURRENT FLOW IN A
CONCEPTUAL DIAGRAM OF THE CELL



through each resistor and these were confirmed by insertion of ammeters into the array. I_{EGR} was about three times higher than I_{EC} as expected. The value of the conductance of the whole array with the guard-ring connected but switched off was virtually identical to that obtained when not connected thus showing that point Y can be thought of as floating and the array independent of the guard-ring when the guard-ring is turned off but not disconnected. Both of these values for array conductance were very much greater than the real value of target conductance ($1/R_{\text{EC}}$) as would be expected. The measured value of the array conductance with the guard-ring on was found to be almost the value of the conductance of rock modelling resistor (R_{EC}). The measured value was consistently just less than 1% greater than the real value of the resistor measured in isolation. Monitoring of the potentials showed that V_{EC} was virtually identical to V_{EGR} and V_{CGR} was negligible. Monitoring of currents showed that the current flowing through R_{EC} was defined only by the conductance of that resistor and V_{EC} , that the current through R_{CGR} was negligible and that the current that was flowing through R_{EGR} was around three times that through R_{EC} and being drained to earth through the guard-ring circuits. It was considered that this test proved the effectiveness of the guard-ring electronics when applied to a resistor array model of the conductively sleeved cell although it was recognised that the array oversimplifies the geometric relationships between the various resistances in the real cell. Removal of a large leakage current and its effects on the conductance measurement of a resistor had been demonstrated.

Experiments on the prototype cell were done in the open

laboratory at around 25 °C and atmospheric pressure. The design of the prototype cell differed very little in its overall geometry from the final design that is shown in figure F6.7. The major differences were that the CIPPRA parts of the final cell were initially made out of PTFE in the prototype cell and that no seals were needed. PTFE was used as it would have been unnecessary, expensive and time consuming to construct the cell out of CIPPRA; it was only to be used in laboratory conditions. The tests were done on saturated and dry pyrophyllite. The tests were unsuccessful, lacking any degree of precision. It was discovered that the repeatability of the results was affected by the clamping of the the sample within the sleeve and end-pieces. Two G-clamps were used to clamp the cell axially and three jubilee clips were used to clamp the cell radially in an attempt to improve the precision of the results. The clamping caused the PTFE to flow and deform. The PTFE parts of the prototype cell were replaced by parts made out of perspex. Clamping now produced measurements that were stable and had a high degree of precision, taking into account the differential saturation of the rock sample on different runs. The saturation scatter was overcome by doing a large number of measurements on the same rock after progressive drying and resaturation to allow the average of these measurements to represent some state of average saturation.

The first measurements to be made were those of I_{EC}/I_{EGR} for the conductively sleeved case and the insulating case. The ratio was found to be 1.43 ± 0.02 for the insulating case and 0.36 ± 0.01 for the conductively sleeved case. This last figure was rechecked as it was much higher than expected. Such a high value

of the current ratio is consistent with the supposition that the resistance that controls the leakage current is that due to a current path through the corners of the rock sample to the sleeve instead of along the flat faces of the electrode holders although this is probably an oversimplification. The various resistances and currents were measured and used to adjust the values of the resistor array model. The success of the guard-ring electronics in measuring the conductance of the resistor representing the rock in the model array with these values of resistances, currents and potentials indicated that the guard-ring should be able to shunt the leakage current sufficiently to earth in the cell prototype to allow the conductance of a central cylinder of rock to be measured. The electronics were added to the cell prototype. Measurements of the conductance for one rock sample were taken in the case where the guard-ring was operating, inoperative and unconnected for the insulating sleeved cell, conductively sleeved cell and the unsleeved cell. Many measurements were taken to find the behaviour of the average saturation state of the sample. These measurements showed that the use of the guard-ring circuitry was able to shunt away the leakage current and allow only the rock current to pass through to the conductance measuring bridge. Monitoring of current and potentials showed that I_{EC} was about a third of the leakage current I_{EGR} , I_{CGR} was not negligible but very small and the guard-ring potential was almost identical to the collector potential. Addition of a conductive sleeve instead of an insulating one increased the measured conductance by 2.42 times. The application of the guard-ring electronics reduced this to slightly greater than its unguarded insulating value.

Application of the guard-ring to the insulating sleeved case also produced a small drop in the measured conductance due to the removal of the comparatively smaller leakage currents here. Overall the values of measured conductance of the conductively sleeved rock while measured with a guard-ring technique were within 4% of the true figure obtained from measurements of the sample in a Lee Cell. This error was accepted as being reasonable in measurements of heterogeneous and variable rocks. The increase in error over that obtained upon measurements of the resistor array is most likely partially due to the complex geometry of the current paths in the real cell. The components of the complex current flow in the collector-guard-ring direction in the collector-guard-ring region of the rock sample make the shunt current (I_{CGR}) non zero. A small shunt current flows from the guard-ring to the collector. The effect is to raise the potential of the guard-ring slightly. The collector sinks a small amount of the leakage current which leads to a slight overestimation of the conductance of the rock. The size of the systematic error produced by this effect has been calculated to be 2.7% from measurements of the value of I_{CGR} and $V_{EC}-V_{EGR}$. The systematic error is removed from the measurements by calibration of the cell against the Lee Cell. This calibration, also used to convert the conductance values to conductivity values, is discussed in full in section 6.9.

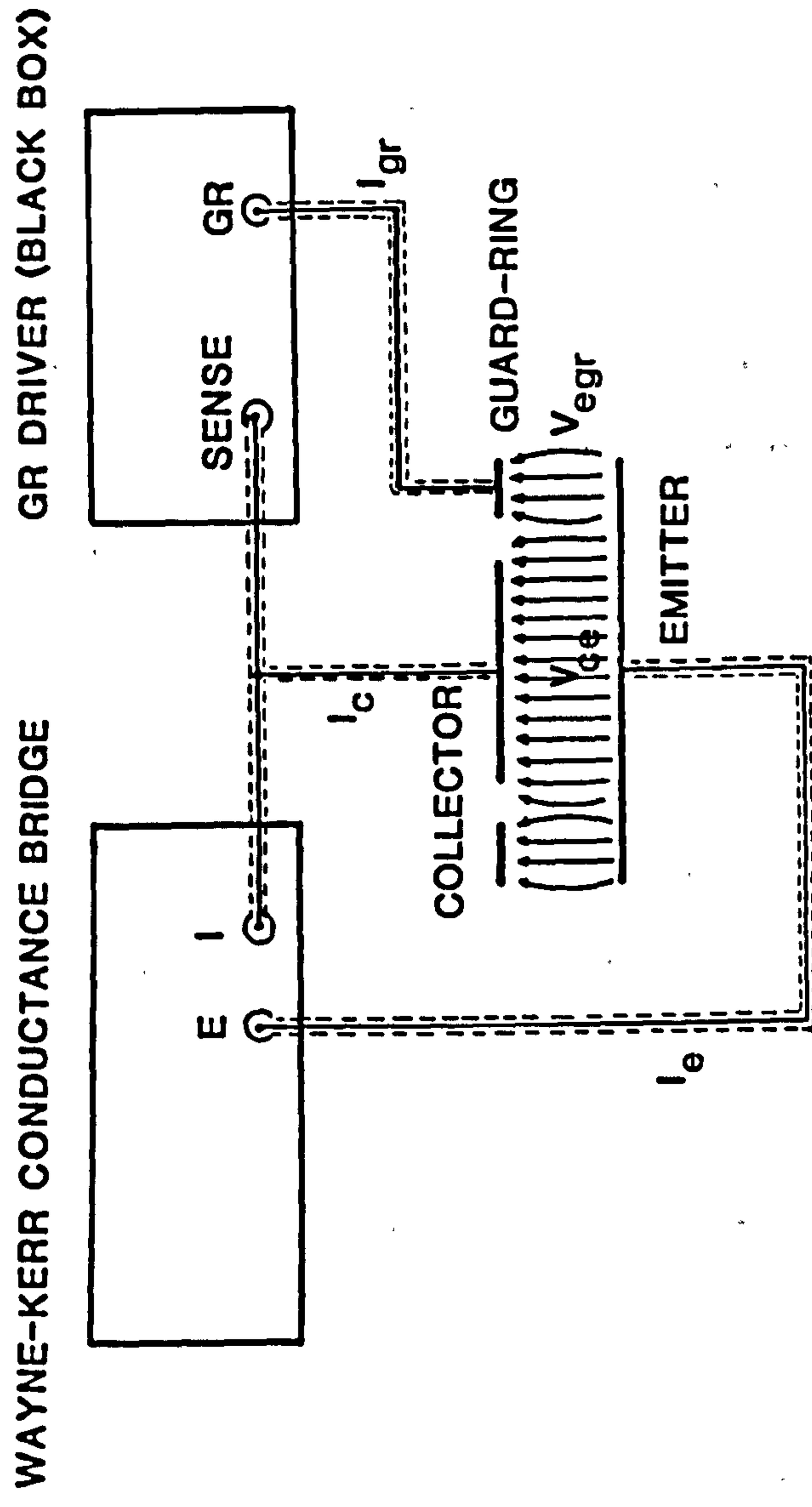
6.8. THE GUARD-RING - THE ELECTRONIC DRIVING CIRCUITS

The final version of the guard-ring driving electronics is a very simple circuit. It is considerably less complicated than an array of synchronised commercial instruments. The last section has dealt with the background of the specifications for the equipment and this section will develop these into a full design.

The circuit can be conveniently described as a black box with a set of operations to carry out. The following discussion will refer to figure F6.10. The requirements are that the box circuits;

1. Generate $V_{EGR} = V_{EC}$
2. Have very high input impedance compared to the rock sample and measurement bridge to ensure that the monitoring of the collector potential does not shunt current away from the measuring bridge and thus artificially lower the measured conductance.
3. Have a sufficiently low output impedance to enable it to sink a large current without that current having significant effect upon the potential of the output.
4. Operate for all values of bridge output potential (20 mV to 20 V peak to peak).
5. Operate at a frequency of 1591.5 Hz (10^4 rad s⁻¹). This is the internal frequency of the Wayne-Kerr conductance bridge without an external driver and the frequency used for all the measurements made in this work.

FIGURE F6.10. EXTERNAL WIRING OF THE GUARD-RING 'BLACK BOX'



All connections and leads are shielded coaxial

6. Be able to sink 100 mA current. This is equivalent to saying that the minimum leakage resistance that the circuit could be used with would be around 200 ohms.
7. Have an adequate slew rate to enable a peak-to-peak swing of 20 V at a frequency of 1591.5 Hz to be faithfully followed. This needs a slew rate greater than around $8 \times 10^4 \text{ V s}^{-1}$ and preferably an order of magnitude greater than this.

The circuit is shown in figure F6.11. The circuit is based around a 741S operational amplifier. This is an esoteric version of the standard 741. The 741S version has been designed specifically to have a better slew rate to fulfil point 7. above. It is wired in a non-inverting voltage follower mode. The amplification of such a circuit is unity. The requirements listed above are all fulfilled by the circuit as follows:

1. Amplification is unity, especially with the ultra stable, low noise 741S.
2. Input impedance of 741S is 10^8 ohms which is improved to 10^9 ohms in voltage follower mode. This compares very favourably with the impedance of around 10^8 ohms in the driest of the rocks that are likely to be measured.
3. The output impedance of the 741S is sufficiently low to sink the required current. It has an output impedance of 200 ohms that improves slightly on wiring in the voltage follower mode. This is very small compared with the impedance set up by a saturated rock of around 125000 ohms and

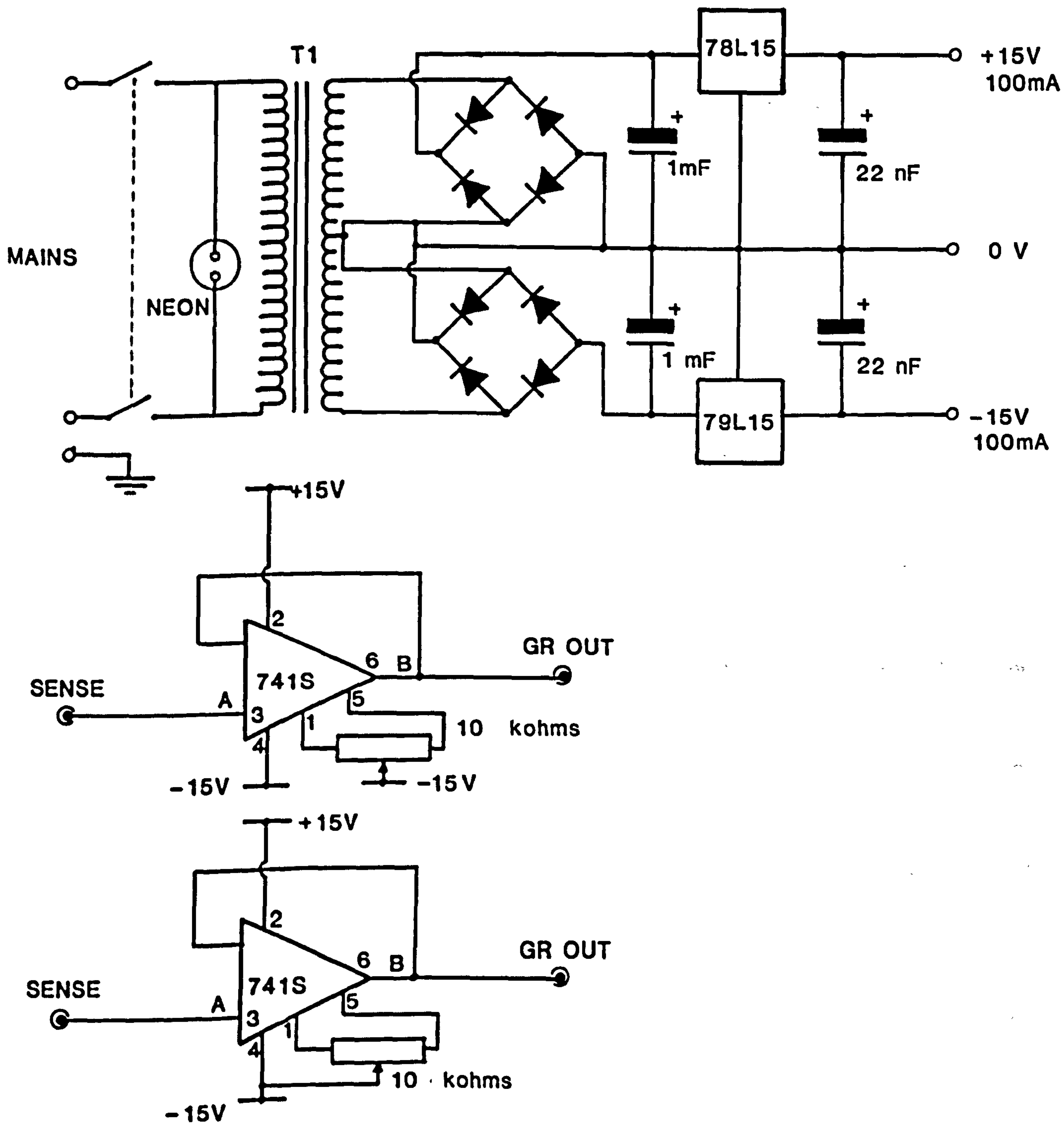


FIGURE F6.11 GUARD-RING DRIVING CIRCUIT

as such will have negligible effect upon the efficiency of draining the leakage current, which will also be passing through a resistance of the same order.

4. The 741S will accept signals up to 30 V peak-to-peak sufficient for this application. The 741S in this application was driven by a supply of 30 V peak-to-peak ensuring that the amplifier operated well within its total voltage range to produce the most stable noise free output signal possible.
5. The limit on frequency depends on the external and internal capacitors of the 741S. All external decoupling capacitors were left out of the circuit design to maximise frequency performance. The internal decoupling capacitors of the Wayne-Kerr bridge were relied upon to decouple the circuit from direct currents. These will not affect the measurements as they have been designed to pass the Wayne-Kerr bridge measuring frequency of 1591.5 Hz. The Wayne-Kerr bridge frequency has a 0.5 Hz error depending upon temperature. This is automatically compensated for by the guard-ring circuits.
6. The maximum output current is defined by the output impedance and the impedance of the leakage routes around the sample. This impedance is always greater than the output impedance of the circuit (200 ohms). The maximum current (for a 20 V peak-to-peak signal) is therefore 50 mA or less

and this is always less than 100 mA that the circuit is capable of sinking.

7. The slew rate of the 741 op-amp is $5 \times 10^5 \text{ V s}^{-1}$. This value is a little too close to the $8 \times 10^4 \text{ V s}^{-1}$ required by the frequency of operation and the maximum voltage swing. A special slew rate version of the 741 was chosen as the op-amp to use. This has a slew rate of $2 \times 10^7 \text{ V s}^{-1}$ which is sufficient to allow the guard-ring circuit to keep track of the voltage changes at the collector electrode. The slew rate of an op-amp is defined by its internal decoupling capacitors.

Figure F6.11 shows the detailed diagram of the guard-ring electronics circuit. The circuit-board was designed with twofold redundancy and an on-board 15 V, 100 mA power supply. The power supply will not be discussed in detail as it is a standard design for a TTL op-amp power supply of this type. The power supply is based around 78L15 and 79L15 voltage regulators.

One of the guard-ring circuits will be described. The collector potential is monitored at point A, which is connected to the non-inverting input pin of the amplifier. The output of the amplifier (on pin 6) is fed directly back into the inverting input of the amplifier. This configuration gives an amplification of unity, the output at pin 6 being identical to that at the non-inverting input (pin 3). The circuit has a very high input impedance and a very low output impedance. The circuit acts as a buffer, the potential at B being always the

same as that at A but with the advantage that A is shielded from changes in the electrical parameters at B. The small 10 kohm circuit-board mounted potentiometer connected across pins 1, 5 and 4 is used to null the direct current output of the amplifier. It was adjusted so that there is zero potential difference between the output and earth when there is zero potential difference between the inputs and earth before measurements were taken. This setting was found to be very stable and did not need rebalancing for the duration of the experiments.

Figure F6.10 shows the final wiring of the guard-ring circuitry in relation to the measurement bridge and the new cell. All connections were made by coaxial cable and plugs except those inside the pressure vessel which were made from home made coaxial cable constructed from wire, thin refrasil sleeving, shield wire and large refrasil sleeving. The conductance of the coaxial connections were removed by balancing the bridge before measurements were taken. The very high input impedance of the guard-ring circuit enabled this to be done in exactly the same way as if the guard-ring circuit was absent from the measuring arrangement.

6.9. THE GUARD-RING - FINAL TESTS AND CALIBRATION

The success of the initial tests indicated that a complete CIPPRA cell should be made, tested and calibrated at high pressures and temperatures in an oil pressure vessel.

The final versions of the cell (shown in figure F6.7.) were constructed. The specific inner dimensions of the cell sample space were:

1. Collector diameter	9.53 mm
2. Emitter diameter	16.52 mm
3. Guard-ring inner diameter	12.71 mm
4. Guard-ring outer diameter	16.52 mm
5. Sample diameter	21.60 mm
6. Sample area	366.4 mm ²
7. Sample length	27.80 mm
8. Sample Volume	10.19 cm ³
9. Guard-ring/collector	1.59 mm
10. Guard-ring/sleeve	2.54 mm

A range of tests and calibrations were done on the finished cell. These are described below.

A cell was loaded with a saturated sample of pyrophyllite. The cell was inserted into an oil pressure vessel and cycled to a pressure of 0.4 GPa. It was left at this pressure for an hour to ensure that the metal sleeve was tightly pressed against the sample allowing no passage of electrolyte between the sample and the sleeve. On removal two small annular steps on the surface of the sleeve could be discerned where the sleeve had been pressed onto the electrode holders and sample of slightly different diameters. This observation was taken as a sign that the sleeve was transmitting the confining pressure to the sample well. After removal the cell was left on the laboratory bench for five days to discover if the cell was prone to any conductance drift

from, for example, small amounts of evaporation. Drift was found to take place at a negligible rate (1.2×10^{-6} S hr⁻¹). There was also a temperature variation noted. The change in laboratory temperature from around 23 °C during the day to 13 °C at night caused a related variation in the conductance of around 25×10^{-6} S, the higher conductance occurring at the higher temperature as would be expected. Temperature variations of the laboratory air would not cause problems in the final experiment as the temperature was well controlled at all times.

Another cell was loaded with a saturated sample of pyrophyllite. The cell was inserted into an oil pressure vessel and cycled up to an effective pressure of 0.4 GPa and a temperature of 250 °C. The cell was left at high temperature and pressure overnight. The cell was examined on cooling and removal from the pressure vessel. No leaks were noted during or after the experiment. The cell retained its physical integrity and no cracks or corrosion were noted.

A loaded cell was heated to 900 °C in an inert gas environment in a muffle furnace and left there for three hours. The cell was examined on cooling and removal. No cracking or corrosion was noted. A slight discolouration of the metal parts was observed.

The main test and calibration are now described. It has been shown that the arrangement so far described can measure the conductance of an axial sub-cylinder of the sample of unknown volume and geometry without the effect of the leakage currents flowing through a conducting sleeve and in the rock outside the

sub-cylinder. This information is of no practical use without knowledge of the geometry of the sub-cylinder from which the rock conductivity can be calculated. The geometry of the sampled volume of the rock of a given conductivity, defined here by physical and electrical boundaries, affects the conductance measured. The conductivity of the rock can only be calculated if this geometric factor can be found. The geometric factor for these measurements is also a transformation from conductance to conductivity. The geometric factor has units of m^{-1} as the transformation from conductance to conductivity requires the multiplication of the measured conductance by some cell geometry-defined measure of length and division by some cell geometry-defined measure of cross-sectional area. The complex current paths make these cell characteristic length and area unobtainable directly without large scale computational analysis. A complicating factor is that these characteristic values are interdependent. The geometric factor can be found for a defined size of sample by comparing measurements made in the cell and in a cell in which the geometric factor is easily numerically calculated. Such a method has been used in this case by using the Lee Cell. An effective area can be calculated from the derived geometric calibration factor by assuming that the effective length is the same as that of the sample. The value of the effective area is a useful measure of how much current is passing through the sleeve of the cell and how much through the sample itself. The effective area of a sample sleeved in a conducting material in the absence of a guard-ring is larger than the actual sample area, this is because the effective area is the measure of that area a sample would need to have to return the

measured conductance. The use of a guard-ring removes these external currents and hence reduces the effective area to a value in which only current with relatively undistorted paths through the sample are used in the conductance measurement. The effectiveness of the guard-ring can therefore be judged by the value of the effective area given in table T6.1.

In this case the geometric factor is found empirically by comparison of measurements on the rock samples with those taken in well controlled geometric circumstances of the Lee Cell. The Lee Cell has a simple geometry in which, if leakage currents are neglected (a valid assumption with PTFE sleeving at high pressure) the volume over which the conductance is measured is the whole of the rock sample. Thus the conductivity can be easily calculated in the Lee case. Calibrations of the new cell were done against conductivities obtained by measurement of the same samples in the Lee Cell over a range of pressures and temperatures for two different rock types. Two rock types were chosen as it was thought that the porosity of the rocks may affect the size of the current sampled sub-cylinder in the new arrangement and hence the conductivity generating calibration. The two rocks were;

- | | | |
|-----------------------|---------------|-------|
| 1. Pyrophyllite | High Porosity | 6.90% |
| 2. Amphibolite Gneiss | Low Porosity | 0.08% |

Porosities have been obtained by measuring saturated and dry sample weights in a method described in chapter 4.

A sample of pyrophyllite was saturated with 0.5 M NaCl. It was loaded into a Lee Cell cycled up to 0.4 GPa and left there overnight. Conductance monitoring was continued throughout. Subsequent cycling gave a repeatable post pressure transient set of conductance measurements. The sample was removed from the high pressure environment and loaded into the new cell. The new cell was pressure cycled up to 0.4 GPa and left overnight in the same way. Conductance measurements were taken during this and on subsequent post transient cycling. In the latter case the transient is very small, most of it already having been removed while in the Lee Cell. These measurements were repeated for resaturations of the sample. The same experiments were done for the other sample of pyrophyllite but in this case measurement in the new cell was followed by measurement in the Lee Cell. The experiments were done in both orders to determine whether the short term compressional history of the rock would affect the calibrations. The great homogeneity of the pyrophyllite sample makes an intercomparison of the two rocks feasible. It was found that the post transient conductance values changed little whether measured in the Lee Cell first or the new cell first. The average values of the calibration factors from these two measurements were taken. Saturation state had a greater effect upon the measurements. Average saturation state conductances were calculated from multiple experiments on dried and resaturated rocks.

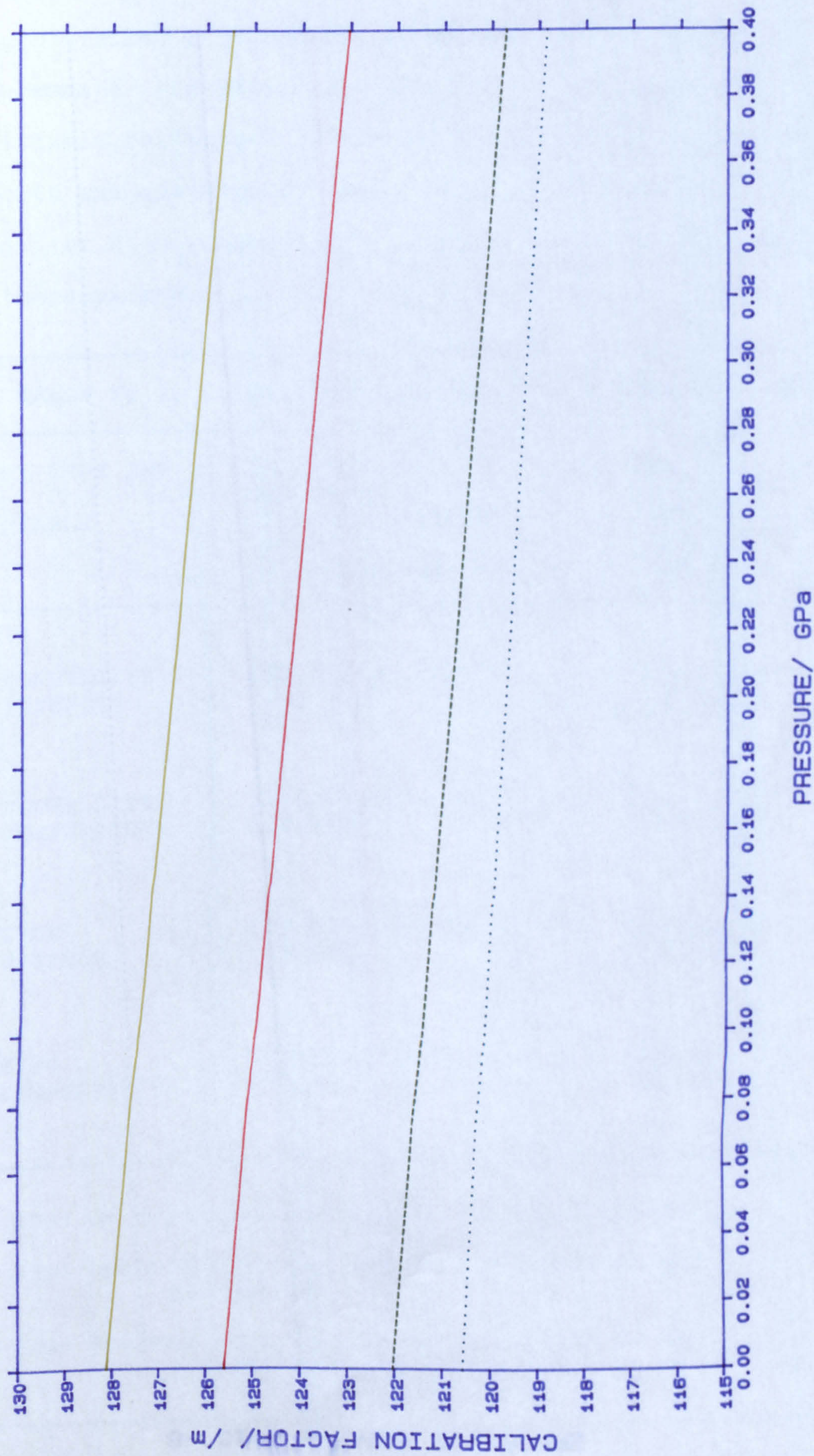
These experiments were repeated for the amphibolite gneisses. Such experiments gave similar values of effective area as the pyrophyllite samples indicating that although porosity had some

effect on the relative size of the leakage to measured current flows, it was not great. Small porosity variations between metamorphic rocks could be ignored. The amphibolite gneiss calibration values were used to calculate the conductivities of all the samples in the main experiment.

Experiments were also done to compare the conductance of different samples of the same two types of rock in each of the cells in high pressure post transient conditions and temperatures up to 240 °C. The samples were handled carefully and multiple measurements were taken whilst varying the order the samples were put in each of the cells to ensure that the average saturation state of the sample was measured and that the cell order had no effect on the overall result.

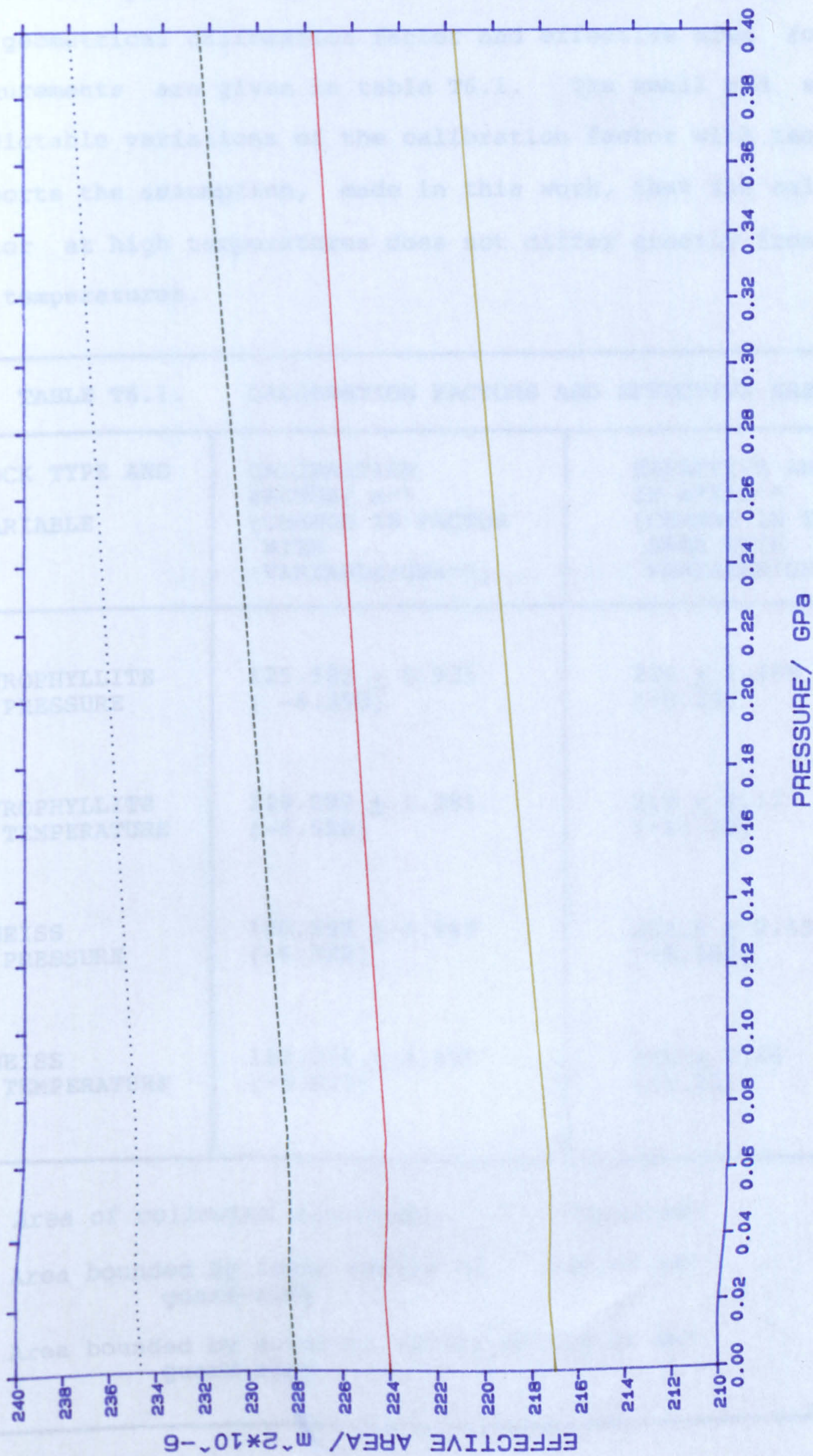
All the data from these experiments were collated and used to produce a new-cell-conductance to conductivity calibration curves for a range of temperatures, pressures and the two types of rock. The variations are shown in figure F6.12. and F6.13. The variations with temperature and pressure are so small that single figures have been used to calibrate the data and the small error combined with the overall error of the experiment. These figures can be used to calculate the effective area of the flat face of the sub-cylinder sampled by the measured current flowing through the centre of the rock sample. The effective area is the area that a cylinder of rock of the calculated conductivity and of the fixed length would have if it were to give a measure of conductance observed. It would be expected that this effective area would be slightly larger than the area of the collector

FIG. F6.12. VARIATION OF CELL CALIBRATION FACTOR
WITH PRESSURE AT DIFFERENT TEMPERATURES



- PYROPHYLLITE AT 25 degrees C
- PYROPHYLLITE AT 250 degrees C
- AMPHIBOLITE GNEISS AT 25 degrees C
- AMPHIBOLITE GNEISS AT 250 degrees C

FIG. F6.13. VARIATION OF CELL EFFECTIVE AREA
WITH PRESSURE AT DIFFERENT TEMPERATURES



- PYROPHYLLITE AT 25 degrees C
- PYROPHYLLITE AT 250 degrees C
- AMPHIBOLITE GNEISS AT 25 degrees C
- AMPHIBOLITE GNEISS AT 250 degrees C

electrode due to a certain amount of barrel shaped distortion of the current paths. This is in fact the case. The final values of geometrical calibration factor and effective area for these measurements are given in table T6.1. The small and seemingly predictable variations of the calibration factor with temperature supports the assumption, made in this work, that the calibration factor at high temperatures does not differ greatly from that at low temperatures.

TABLE T6.1. CALIBRATION FACTORS AND EFFECTIVE AREAS		
ROCK TYPE AND VARIABLE	CALIBRATION FACTOR/ m ⁻¹ (CHANGE IN FACTOR WITH VARIABLE/GPa ⁻¹)	EFFECTIVE AREA IN m ² X10 ⁻⁶ (CHANGE IN EFFECTIVE AREA WITH VARIABLE/GPa ⁻¹)
PYROPHYLLITE + PRESSURE	125.585 ± 0.935 (-6.390)	224 ± 1.489 (+9.23)
PYROPHYLLITE + TEMPERATURE	128.097 ± 1.281 (-6.550)	217 ± 2.17 (+11.72)
GNEISS + PRESSURE	120.569 ± 4.943 (-4.322)	234.6 ± 9.45 (+8.546)
GNEISS + TEMPERATURE	122.034 ± 4.637 (-5.823)	228 ± 8.66 (11.22)
Area of collector electrode71.33 mm² Area bounded by inner radius of guard-ring126.88 mm² Area bounded by external radius of guard-ring214.34 mm²		

CHAPTER 7 : EXPERIMENTAL PROCEDURE

7.1. INTRODUCTION

The previous three chapters have described the samples, their preparation, the high pressure and temperature systems and the design of the experimental cell. This chapter will draw all of these various aspects together in a description of the experimental procedure. It will start with a brief note on safety then will describe the loading of the experimental cell and progress chronologically.

7.2. SAFETY

The safety aspects of the experimental procedures were borne in mind at all times during the preparation and running of experiments. The recommendations set out in the High Pressure Safety Code (1975) published by the High Pressure Technology Association (HPTA), the Health and Safety Executive (HSE) and internal high pressure laboratory practices were followed at all times. This required the constant supervision of high pressure apparatus during high pressure cycles, the complete closure of safety rooms during experiments, strict adherence to set pressure raising and lowering procedures and the use of full body protection.

There are two safety aspects of running of experiments that require some further elucidation. The nature of the design of the experimental cell and the independant generation of pore pressure raises the possibility that the confining pressure could

drop below the pore pressure due to some gas leakage or problem with the gas compressor. If this happens then the design of the experimental cell would be dangerously stressed, the stainless steel sleeve would be put under a high tensile stress and would explode. Although this is not extremely dangerous it would be a considerable set-back to the experiment and would probably entail a complete rebuild of furnace and experimental cell.

The other aspect although more unlikely is more catastrophic in its implications. The furnace is efficient enough to reach 1000°C with a current of around 2 amps in a well ventilated laboratory. The furnace requires less current when it is well lagged, such as it is when within the pressure vessel. The temperature controller and thyristor can control up to 5 amps. If it were to fail in such a way that it introduced more current into the furnace windings than required then the furnace has the ability to reach temperatures much higher than 1000°C. There would come a time, before the nichrome windings degraded, when the temperature of the body of the pressure vessel would be dangerously raised despite the internal pyrophyllite thermal insulation. Such a raising of the temperature of the pressure vessel body drastically reduces its tensile strength and a catastrophic failure at its maximum design pressure becomes theoretically possible. The internal thermal cladding fulfils a secondary, but no less important a role, in that it fills as much of the internal space of the vessel as possible. This reduces the amount of confined and compressed gas and hence reduces the confined energy of the system. Despite this the confined energy of the remaining volume of enclosed gas suddenly expanding still

represents a considerable hazard.

The above two aspects of safety required that constant supervision of the relative pressures and the maximum temperature was kept during the use of high pressures and temperatures.

7.3. EXPERIMENTAL CELL LOADING

The loading of the experimental cell takes about ten minutes. Although this procedure is more complex than the loading of the Lee Cell, it remains fast enough to avoid excessive evaporation of pore fluid from the rock sample.

All the handling of the cell pieces during and after the loading of the cell was done using plastic gloves. The loading was begun by setting out all of the cleaned cell pieces on a clean bench covered in sterile paper. The alumina guard-ring electrode holder assembly was checked by depressing the guard-ring and collector electrodes to check that the compression of the supporting springs was sufficient to allow the electrode elements to withdraw completely into their recesses in the electrode holder yet stand out 1 mm when not in compression. The electrical continuity between the electrode leadthroughs and the electrodes was checked. The rock sample was taken and offered up against the guard-ring electrode holder assembly making sure that the motion compresses the springs that hold the guard-ring and collector slightly out of the holder. The rock should now be flush with the electrode holder ensuring a good contact between

the electrodes and the sample. The combination of electrode and sample was slid carefully into one end of a stainless steel sleeve until the external end of the electrode holder was flush with the end of the sleeve. The friction between the sample, the electrode holder and the sleeve holds the sample and electrode holder together against the compression of the springs. The emitter electrode holder assembly was taken and the emitter electrode slightly depressed to check that the electrode supporting spring was compressible enough that the electrode retreats into its recess in the emitter electrode holder. It was also checked that the spring would hold the electrode about 1 mm proud of the electrode holder when not in compression. The electrical continuity between the electrode leadthrough and the emitter electrode was checked with a multimeter. The emitter electrode holder assembly was carefully slid into the open end of the sleeve until completely against the sample compressing the springs and ensuring good contact between the emitter electrode holder and the sample. In this state the sample is fully enclosed and the rate at which pore fluid evaporation will take place is very much reduced.

The half assembled cell was sealed by the following procedure. The half-assembled cell was mounted in a collet in a lathe and set to rotate slowly. The stainless steel sleeve was swaged into the groove in the electrode holder at the projecting end of the cell using a specially designed wheel tool. This tool is shown in figure F6.4. The same procedure was done for the other end of the cell. The swaging makes the initial seal by crimping the metal sleeve around the shoulders of the ceramic groove. This

seal is 'fixed' by the fitting of the four jaw clamping arrangement described in chapter 6. The fitting of the clamping arrangement was done in the following way. A jaw holder was taken screwed into a specially prepared assembly tool and locked there by the use of a locking screw. This arrangement can be seen in figure F6.5. The tool was mounted vertically in a vice. The semi-assembled cell was mounted vertically on the jaw holder as also shown in figure F6.5. A circlet of copper wire was laid in the swaged groove in the stainless steel sleeve. A circlet of wire of this cross-section is about twice the size that would sit comfortably in the groove. Four matched jaws were taken and arranged around the cell by locating their positioning ridges in the annular recess in the jaw holder. The jaws are stable in this position and sit in a way that they fall slightly more open than the circlet in the groove in a similar way to which the jaws of a clutch pencil are when not quite gripping the pencil lead. The jaws were brought together compressing the copper circlet and clamping the annular seals by the sliding a retaining collar up around the jaws. The action of screwing a large nut along the assembly tool to force the retaining collar tightly over the jaws and thus crushing the copper wire in the groove and making the seal gas tight is helped by the external taper on the jaws and corresponding taper on the internal face of the retaining collar. The cell and completed seal were removed from the assembly tool by unlocking the retaining screw. The seal at the other end of the cell was then completed in exactly the same manner.

The cell was then loaded, fully prepared and ready for location in the furnace and pressure vessel. Although the loading of the

experimental cell seems lengthy and overcomplex it is necessary to enable a gas tight ceramic/metal seal and can be completed in less than ten minutes by a practiced researcher.

7.4 CHARGING THE PRESSURE VESSEL

This section describes the arrangement of the cell and furnace in the pressure vessel and the connection of thermocouples and data lines. The experiment was supported on a bed of pyrophyllite blocks within the pressure vessel. A long semi-tubular shell, the length of the pressure vessel, was screwed onto the the leadthrough endcover of the pressure vessel. This case was filled with sculptured pyrophyllite blocks. The blocks were shaped to accept the furnace and the electrical leads. The bottom set of blocks were drilled to convey the furnace current leads to the furnace and their upper surfaces were grooved to convey thermocouple and electrical conductance data lines to both ends of the furnace. The furnace current lines were laid permanently in their holes. The arrangement of the pressure vessel charge at the begining of each experiment was as shown in plate P7.1. The bottom blocks and furnace leads are in place, thermocouple and data lines are attached to the leadthroughs but not attached to the cell or laid in their ducts, the furnace and cell is not in place and the pore pressure connection remains unattached. All preparation of the pressure vessel charge was done on the stand shown in plate P7.1. on the laboratory bench.



PLATE P7.1. THE EXPERIMENTAL LOAD AT THE BEGINNING OF PREPARATION

The preparation of the experimental charge occurred in the following order. The thermocouple and conductivity leads were held aside by a clamp and stand. The loaded experimental cell was taken and primed with pore fluid by filling the pore pressure connection using an hypodermic needle. The pore pressure leadthrough pipe was also primed with pore fluid in the same way and temporarily sealed with small rubber bungs. The experimental cell pore pressure pipe was attached to the pore pressure leadthrough pipe after the removal of one of the small rubber bungs by using an Aminco high pressure pipe connector. The furnace was carefully slid over the experimental cell taking care not to foul it. The diameter of the experimental cell was designed to be such that it is a close fit within the furnace. The furnace was slid so far over the experimental cell that the guard-ring connection end of the cell almost emerged from the furnace end. In this position the connections to the guard-ring and collector leads on the cell were easily accessible. The guard-ring and collector leads were connected to the relevant leads leading through the pressure vessel endcover and hence out into the laboratory. The furnace was slid a little backwards to ensure that the cell occupied the central portion of the furnace within the area covered by the central winding. The length of the pore pipe, in relation to the dimensions of the recessed bed in the pyrophyllite blocks, was designed to be such that the furnace would only snugly fit within the sculptured cradle prepared for it in the blocks when the cell was in the correct position within the centre of the furnace. When the furnace is fitted into its cradle the Aminco high pressure pipe connector is outside the furnace and high temperature region. Once the

furnace was in place the conductance leads were fitted into their ducts in the pyrophyllite blocks. Two alumina sheathed thermocouples were introduced into each end of the furnace in such a way as to fit into the grooves machined for them in the restraining collars of the experimental cell as shown in figure F5.9. All the thermocouple leads were carefully laid in the grooves and ducts prepared for them in the blocks. The two accessible furnace current leads were connected to the furnace. This is the stage the preparation of the charge had reached in plate P7.2. The charge was covered with the upper set of pyrophyllite blocks and then secured with the upper part of the steel shell and a steel end-piece, which was screwed into place on the end-cover and around the steel end-piece. The charge was rotated on its stand through 180° and the bottom half of the steel shell removed. Five or six pyrophyllite blocks were removed to enable the remaining four furnace current leads to be connected to the furnace. The blocks were then replaced, the shell replaced, screwed down and the charge rotated into its original position.

The experimental charge was transferred from the bench stand to the cradle attached to the pressure vessel. The cradle was adjusted to allow easy introduction of the charge into the pressure vessel. The charge was slid into the pressure vessel. A large copper gasket was positioned around the end-cover projection. The cradle was dismantled and removed from the pressure vessel. The pressure vessel was tightly sealed with a large end nut and the swung up into a vertical position. The pore pressure system was fully primed using a pore fluid filler

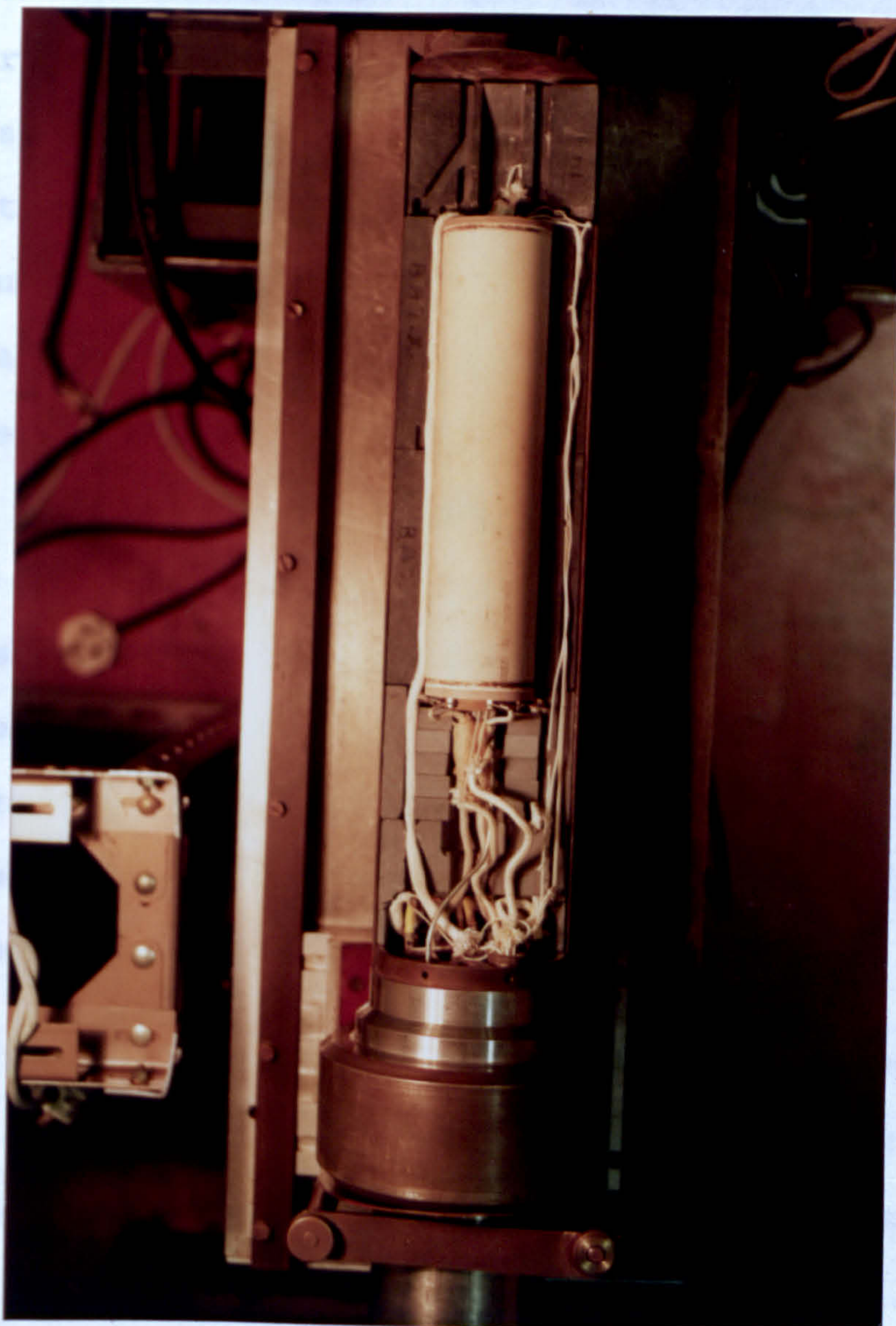


PLATE 7.2. THE HALF COMPLETED EXPERIMENTAL LOAD

reservoir and connected to the, now unbunged, pore pressure leadthrough pipe. The gas pressure pipe was connected to its attachment on the gas pressure inlet end-cover of the pressure vessel. The evacuation port was attached to the evacuation system and the cooling water was turned on. The heating current leads were connected to the external nipples of the furnace current leadthroughs. The thermocouple cold junctions were inserted into the cold junction baths and the eight thermocouple leads were connected to the cables leading out of the safety room to the thermocouple multiplexing switch. There are three conductance leads, two (the guard-ring signal and collector signal) use the signal leadthrough to pass through the pressure vessel endcover, the third (the emitter signal) passes through the endcover in the pore fluid pipe and is tapped from the pipe to a connection block. All the conductance leads were connected to shielded coaxial cables by the junction block. The coaxial cables were led out of the safety room to the guard-ring controller and conductance bridge. The cold bath monitoring thermocouples were inserted into the cold baths. All the various connections had then been made.

The fully connected pressure vessel was purged of air by three or four consecutive cycles of evacuation and application of uncompressed bottle argon. Each cycle of purging consisted of opening the evacuation valve, evacuation, closure of the evacuation valve, opening of the valves for the application of the bottled gas and then closing them again. When this had been done three or four times the pressure vessel was considered to be full of the inert gas that was to be used and ready to continue with the experiment.

7.5. EXPERIMENTAL PROCEDURE

The experiments progressed in the following manner; The gas confining pressure was increased to 0.2 GPa in steps of 0.02 and 0.04 GPa. At each increment the experiment was left for the conductance values to settle down. A conductance reading was taken only when two readings taken ten minutes apart were within 2.5% of each other. At first this took up to a hour but became less as the pressure was increased. After each reading the confining pressure was increased another increment. The experiment was left at a confining pressure of 0.2 GPa overnight. Conductance readings were made as the confining pressure was decreased in steps in a similar way to the increase. The 2.5% criterion was applied but in this case the time lapse before it was obeyed was minimal. Conductance measurements were made while the gas confining pressure was re-increased to 0.2 GPa in 0.02 and 0.04 GPa steps. This was done to check on the repeatability of the conductance curve obtained while decreasing the confining pressure. Again the 2.5% criterion was applied and once again obeyed within a few minutes.

The confining pressure was held at 0.2 GPa. The pore pressure was increased to the required amount in increments of 0.04 and 0.05 GPa. At each value the system was left to equilibrate until the Lee criterion, defined in chapter 8, was obeyed before a measurement of conductance was taken. The pore pressure was

raised to 0.18 GPa in some experiments and left there. In other experiments the effect of the functional dependance of the electrical conductivity of the pore fluid solution as a function of temperature and pore pressure upon the conductance of the saturated rock was to be studied. In these experiments the pore pressure was raised to 0.1 GPa and left while the conductance was investigated as a function of temperature. After this had been done the pore pressure was increased and then the temperature variation measurements repeated.

Once the required confining and pore pressures had been applied, the sample was ready to undergo the measurement of conductance at raised temperatures. Two methods of obtaining these measurements were used. The first involved setting the temperature controller to around 825 °C and allowing the furnace to freely increase the temperature up to this value. Although conductance measurements were made during this rise it was realised that the relatively swift raising of temperature in this uncontrolled way would render these measurements invalid. This is due to the thermal lag with which the centre of the rock sample will have. The effect on pre-transient measurements on raising temperature is a depression of the measured conductivity. The representation of this effect can be seen in the data and is discussed in chapter 9. The temperature controller was used to hold the sample at high temperature for about 12 hours to allow any transient drop to occur but also allowing the whole sample to reach a stable constant temperature. The temperature controller was switched off and the sample was allowed to cool with help from the circulating cooling water supply to the pressure vessel.

Conductance measurements were made every 50 °C or so as the sample cooled. All conductance measurements made while temperature was undergoing a change were made in the following way. The thermocouple readouts were monitored until the temperature was roughly at the value at which the conductance was required to be measured. The thermocouple temperature reading was taken immediately followed by the conductance reading. The thermocouple reading was remeasured, followed by remeasurement of the conductance and finally a last reading of the thermocouple reading. All these measurements were made as quickly as possible. It was found that all five measurements could be taken in about 30 seconds. The three temperature values were averaged and the two conductance measurements were averaged to give the final values of conductance for each value of temperature. The cooling rate at high temperatures was 0.016 °C s⁻¹. This decreased to around 0.0098 °C s⁻¹ at the lower temperatures. The thermal lag of the centre of the sample at these values of cooling rate are 4.96 °C and 2.03 °C respectively. These values are of the same magnitude as the temperature error in the temperature measurement and have been shown to have a negligible effect on the accuracy of the conductance/temperature measurements compared to the errors in the direct temperature and direct conductance measurement. Furthermore, comparisons with conductance measurements/temperature made in the second way confirm the validity of the measurements made in this way. This will be discussed in more detail later on in this section. Once the sample had cooled to around 100 °C the temperature controller was activated again and the process of conductance/temperature measurements was repeated as before except that the sample was

left at high temperatures for a shorter length of time to allow the temperature to become constant throughout the sample. A longer period of time was not necessary as the sample was already in its post-transient drop state and calculations of rock thermal conductivity indicated that a length of time of only 90 minutes was required to equilibrate the temperature throughout the whole sample. No conductance measurements were made during these post-transient temperature increases. Three successive temperature cycles were usually done in this way.

The second method of making conductance/temperature measurements has the advantage that it defines the experimental temperature conditions much more accurately and is therefore a more valid experimental procedure. It has, however, the considerable disadvantage that it requires a larger length of time to take each measurement, sufficiently longer to interfere with the viability of such an experiment. Several experimental runs were done combining measurements made in the second way with the method previously described. This was needed to show that the faster method was not significantly different from the better characterised second method. The second method differed from the first in that post transient drop conductance measurements were made during a well controlled increase in the temperature as well as during a controlled decrease in temperature. The temperature control was effected in the following way. The temperature was increased to a preset temperature and left for 60 minutes for the temperature of the sample to equilibrate before a measurement was taken. The temperature was then increased to the next temperature repeating the procedure. Control of temperature

during temperature decrease was done in a similar way leaving the sample temperature to stabilise on a series of steps.

The advantages of the free cooling method of making measurements is that its speed is much greater than the second method, a factor of some importance when the full experimental runs required several days and nights to complete. The requirement for supervision of the equipment at all times during which high temperatures and pressures were used combined with the length of time of each experimental run made it necessary to save time whenever possible.

Some sample runs were closed down after the temperature cycling. Some sample runs were continued by making conductance measurements during low temperature cycling of confining pressure to enable the conductivity in as completed a post-transient state as possible to be obtained.

Other samples were cycled up to a high temperature and maintained at this temperature while conductance measurements were made during the cycling of the confining pressure and pore pressure. These measurements were made to enable high temperature values of conductance to be made as a function of confining pressure. It was hoped that these measurements would enable a high temperature isothermal extrapolation of pressure to 1 GPa to be made in an attempt to make up for the practical problem of a lack of a 1 GPa gas pressure vessel.