# Development of Cylindrical Laminated Methanol Steam Reforming Microreactor with Cascading Metal Foams as Catalyst Support

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**Abstract**: In this study, the cascading metal foams were used as catalyst supports for constructing a new type of cylindrical laminated methanol steam reforming microreactor for hydrogen production. The two-layer impregnation method was used to load the Cu/Zn/Al/Zr catalysts, and the ultrasonic vibration method was then employed to investigate the loading performance of metal foams with different types and thicknesses. Furthermore, the effect of the type of catalyst placement, pores per inch (PPI) and foam type on the performance of methanol steam reforming microreactor was studied by varying the gas hourly space velocity (GHSV) and reaction temperature. Compared with two other types of catalyst placement studied, the microreactor containing catalyst-loaded metal foams without clearance cascading (3×2) showed the highest hydrogen production performance. When the PPI of the metal foam was increased from 50 to 100, both the methanol conversion and the $H_2$ flow rate gradually increased. Our results also showed that a microreactor with Cu foam as a catalyst support exhibits increased hydrogen production and higher stability than those of a microreactor with Ni foam.

**Keywords:** Microreactor; Methanol steam reforming; Catalyst support; Metal foams; Hydrogen production

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# 1 Introduction

Hydrogen has received extensive attention as an energy source because it has a higher combustion heat value and pollution-free characteristics [1]. In recent years, with the development of proton exchange membrane fuel cell (PEMFC) technology, the expectations have risen that hydrogen will become a significant clean fuel in the near future [2-3]. However, hydrogen also has several obvious disadvantages such as a low boiling point, high flammability, and difficulty of transport and perfusion, which hinder the popularization and application of PEMFC. Currently, the online hydrogen fuel processing systems involved the microchannel technology, which is characterized by compactness, high efficiency, and low cost, and can convert liquid hydrocarbon fuel to hydrogen for PEMFC in transient operation [4-5]. Microchannel reactors can be integrated with PEMFC, which does not increases the volume and weight significantly, while the fuel cells can use the existing hydrogen fuel system to provide a continuous supply of hydrogen, which is an important foundation for the construction of effective fuel cells. Therefore, the development of efficient microreactors for hydrogen production is crucial to meet the requirements of various applications [6].

In general, the traditional microreactors for hydrogen production are classified into three types—parallel, curved, and micro-pin-fin arrays—and fabricated by means of MEMS, laser machining, electrical discharge machining, and traditional machining technology [7-12]. The catalyst is usually coated by means of impregnation as well as chemical and physical vapor deposition. However, the catalysts coated on these

structures exhibit a series of disadvantages such as low adhesion, ease of shedding, and high machining cost [13-15]. The microreactors for the methanol steam reforming reaction with porous metal materials as catalyst support are characterized by short reaction flow path and low pressure drop, and the catalyst support has a three-dimensional porous structure and a large specific surface area, such that the catalyst can be coated more evenly to form a microstructure catalyst [16]. Thus, porous materials show potential as a new generation catalyst supports of microreactors for hydrogen production. Metal foams [17-20], metal fiber porous materials[21-24], and porous ceramics [25] have all been successfully used in various chemical reaction systems.

Metal foams are commonly made by means of a direct foaming of metals, in which mass production may be implemented easily with low cost [26]. Metal foams are made up of mesh pore and have a polyhedral shape exhibiting high specific surface area, low density, and large pore size, and have been widely used as catalyst support materials [27]. Roy et al. developed a methane reforming reactor for hydrogen production with a Pd-Rh metal foam catalyst structure; they found that it demonstrated excellent catalyst stability and activity, and could be widely applied in the processing of solid oxide fuel cell[18]. Aartun et al. coated a Rh catalyst on the surface of aluminum foam by means of impregnation, and then was successfully employed for the partial oxidation and steam reforming reaction of propane [19]. Yang et al. prepared a Co-W-B-P/Ni catalyst on the surface of a nickel surface using ultrasonic assisted electrodeposition, and showed that the catalyst can be used in

the hydrolysis process of ammonia borane with a remarkable hydrogen production efficiency due to its high catalytic activity and recyclable properties [20].

In this study, the cascading metal foams were used as a catalyst support, and Cu/Zn/Al/Zr catalysts were loaded using the two-layer impregnation method. The loading performance of the metal foams and the stability of the catalysts were investigated. The effects of different types of catalyst placement, pores per inch (PPI), and foam type on the reaction performance of methanol steam reforming microreactor for hydrogen production were also studied.

## 2 Experimental

### 2.1 Cylindrical microreactor

Fig. 1 shows a diagram of the cylindrical laminated methanol steam reforming microreactor for hydrogen production used in this study. The microreactor consists mainly of an evaporation chamber, a reaction chamber, heating cartridges, a thermocouple, and inlet and outlet tubes. Three grid trays were used in the evaporation chamber to increase the amount of water vaporization and the flow uniformity. A mixture of methanol and distilled water (mole ratio of 1:1.3) was pumped into the evaporation chamber through the inlet tube with an injection pump [28]. After the mixture evaporated in the evaporation chamber, the reformate gas reacted in the presence of the catalyst in the reaction chamber at a temperature range of 200℃ to 400℃ which was controlled by the heating cartridges.

The types of catalyst support placement in the reaction chamber are shown in Fig.2. In the first type, a ring gasket with a thickness of 2 mm was set between three

pieces of metal foams (each with a thickness of 2 mm), which were placed in a manner here referred to as clearance cascading (clearance value=2mm), as shown in Fig. 2a. In the second type, three pieces of metal foam (each with a thickness of 2 mm) were placed without clearance cascading, as shown in Fig. 2b. In the third type, metal foams each with a thickness of 6 mm were placed directly in the reaction chamber, as shown in Fig.2c. The hydrogen production performance associated with each of the three types of catalyst placement was measured as a function of gas hourly space velocity (GHSV) and reaction temperature.

## 2.2 Catalyst coating and characterization

The procedure for processing the catalyst support was divided into the following four steps: cutting the metal foams, cleaning the surface, catalyst coating, and catalyst activation. First, the metal foams were cut into circular plates by wire cut electrical discharge machining. Before coating, the metal foams were cleaned with ethanol in an ultrasonic bath to remove any organic substances. After drying, the metal foams were reduced in an atmosphere sintering furnace at temperature of $400℃$. Then, the two-layer impregnation method was employed to load the catalyst on the metal foams. Each 2 mm and 6 mm metal foam was loaded with 0.3 g and 0.9 g of catalyst, respectively. Then, the catalyst supports were sufficiently impregnated in the catalyst precursor solution ($Al_2O_3$ and nitrate solution) and were dried in an oven at 100 °C for 15 min. The above steps were repeated until the precursor solution was completely consumed, such that metal foams loaded with Cu/Zn/Al/Zr catalyst with two-layer catalyst structure were obtained [29]. After the coating process, in order to

activate the catalyst effectively, the catalyst supports were calcined at 400 °C for 120 min under a $N_2$ flow rate of 100 mL/min. The supports were then calcined at 300 °C for 60 min under a mixture of $N_2$ (100 mL/min) and $H_2$ (50 mL/min) to complete the activation of the catalyst. The microstructures of the catalyst supports were observed with scanning electron microscopy (SEM) (Hitachi SU70, Japan).

The performance of the catalyst coating was evaluated by ultrasonic vibration. The metal foam-coated catalysts were first immersed in distilled water, then placed in an ultrasonic vibration bath (No: KQ5200DB) with an input power of 200 W for several min at 24°C. After the ultrasonic vibration procedure, the metal foams were dried in an oven, and then weighed using an electronic balance [23].

### 2.3 Methanol steam reforming test

Fig.3 shows a schematic diagram of the testing system for the methanol steam reforming microreactor for hydrogen production. The system consists of a fuel tank, injection pump, evaporator, reformer, thermostat, data collector, condenser, dryer, gas chromatograph, computer, mass flow controller, and soap bubble flow meter. During the experiment, the temperature of the methanol steam reforming microreactor for hydrogen production was controlled by a thermostat. The mixture of methanol and water (mole ratio of 1:1.3) was pumped into the evaporation chamber with the injection pump; after evaporating, the mixture reacted in the reforming chamber. Next, the hydrogen-rich gas passed through the 0°C cold trap to condense any remaining unreacted methanol and water. An on-line gas chromatograph (No: GC1690) with a TCD detector was then used to analyze the composition of the hydrogen-rich gas. The

flow rate of the hydrogen-rich gas was determined with a soap bubble flow meter [23].

Methanol conversion $X_{MeOH}$ and H2 flow rate $v_{H_2}$ can be calculated by the following

formula [30]

$$X_{MeOH} = \frac{F(y_{CO} + y_{CO_2})}{22.4 \times v_{MeOH,in}} \times 100\% \qquad (2)$$

$$v_{H_2} = F \times y_{H_2} \qquad (3)$$

Where F is the normal flow rate of effluent gas, $y$ is the volumetric fraction, and

$MeOH,in$, is the molar flow rate of methanol fed into the microreactor.

## 3 Results and Discussion

### 3.1 Analysis of catalyst coating performance

Fig.4 shows SEM images of the 100 PPI Cu and Ni foams coated with Cu/Zn/

Al/Zr catalysts. It is clear that the Cu (Fig. 4a) and Ni (Fig. 4b) foams had relatively

uniform interconnected three-dimensional pore structures, and in both these foams the

catalyst could coat evenly between the skeleton and pores to form a three-dimensional

network catalyst with interconnected structure (as shown in Fig. 4 c). In addition, it is

worth noting that the catalyst coating on the surface of the Cu foam still had

considerable microscopic structure (as shown in Fig. 4c and 4d), while the Ni foam

showed evidence of had the phenomenon of catalyst accumulation (as shown in Fig.

4e and 4f). Thus we conclude that the Cu foam is beneficial to coat copper-based

catalyst. In addition, the flower shaped structures on the surfaces of the metal foams

not only significantly increased the specific surface area of the catalyst support, but

also improved the catalyst loading efficiency.

Fig. 5 shows the effective catalyst loading of three samples of 2-mm-thickness

and one sample of 6-mm-thick 100 PPI Cu and Ni foam. As shown in Fig.5, the effective catalyst loading after the two-layer impregnation method was about 0.9 g. After ultrasonic vibration, the effective catalyst loadings of three samples of 2 mm thickness and one sample of 6-mm-thick Cu foam were higher than those of the corresponding samples of Ni foam. Furthermore, it was found that after 1 min of ultrasonic vibration, the effective catalyst loadings of the 6-mm-thick Cu and Ni foams decreased significantly. By comparison, the three samples of 2-mm-thick Cu and Ni foam exhibited higher effective catalyst loadings. After 2–3 min of ultrasonic vibration, the three samples of 2-mm-thick Cu and Ni foams still exhibited considerably higher catalyst loadings comparing with 6-mm-thick foam. This is mainly due to the fact that the interior pores of a catalyst are difficult to coat, and part of the catalyst coating remains on the surface of the metal foams when catalysts are coated on the thicker metal foams. Therefore, the catalyst is more likely to detach from the surface during ultrasonic vibration for 6-mm-thick foam.

### 3.2 Effect of catalyst placement on hydrogen production

Fig.6 presents the methanol conversions and $H_2$ flow rates of Cu foam with different types of catalyst placement as a function of GHSV at 300 °C. It can be seen that as GHSV increased, the $H_2$ flow rate rose gradually, but the methanol conversion decreased. This is because a higher GHSV causes a decrease in the residence time of the reactant such that a fraction of the reactant cannot sufficiently carry out the reforming reaction on the surface of the catalyst, which reduces the methanol conversion. However, the overall amount of reactant which participated in the

reforming reaction increased with increasing GHSV, and therefore, the $H_2$ flow rate gradually increased [31]. Fig.7 shows the methanol conversions and $H_2$ flow rates of Cu foams with different types of catalyst placement as a function of reaction temperature. At a GHSV of 16.2 K·mL/g·h, it is easily seen that with an increase in reaction temperature, the methanol conversion and $H_2$ flow rate rose gradually. This observation can be attributed to the fact that the methanol steam reforming reaction is endothermic; therefore, within a certain temperature range, a higher temperature can increase the catalyst activity and thereby increase the methanol conversion and $H_2$ flow rate [31].

The microreactor with a catalyst support without clearance cascade placement (T2) showed remarkably high methanol conversion and $H_2$ flow rate when compared with the other two types. The possible reason for this result is that gaskets set between Cu foam plates (as in T1) will extend the reforming reaction cavity length, which causes uneven heating and impedes heat and mass transfer, such that the performance of the microreactor for methanol steam reforming deteriorates [32]. However, when 6-mm foam Cu is selected as the catalyst support (T3), the low effective catalyst loading (as shown in Fig.5) leads to poor reaction performance. Therefore, metal foams without clearance cascading placement (T2) are recommended as the best catalyst supports for methanol steam reforming microreactors for hydrogen production. Comparing to the Shen's microchannel foam reactor [33], it is found that our developed cylindrical microreactor also exhibited higher methanol conversion.

### 3.3 Effect of the porosity (PPI) on hydrogen production

The methanol conversions and $H_2$ flow rates of Cu foams with different PPI as a function of GHSV are shown in Fig. 8. Fig. 9 shows the methanol conversions and $H_2$ flow rates of Cu foams with different PPI as a function of reaction temperature. Considering Cu foams with 50, 80, and 100 PPI used as catalyst supports without clearance cascading (T2) under the same GHSV and temperature conditions, it can be seen that the methanol conversion and $H_2$ flow rate increased significantly with an increase in PPI, and the Cu foam with 100 PPI showed the best performance for hydrogen production. This result can be attributed to the fact that foam metal with lower PPI has greater porosity and pore size, which will cause low pressure drop and decrease of the residence time of the reactant and easy the catalyst loss during the reaction process thus resulting in a drop in hydrogen production [34].

*3.4 Effect of the foam type on hydrogen production*

The methanol conversions and $H_2$ flow rates at 300 °C of 100 PPI Cu and Ni foams as catalyst supports are shown in Fig. 10. With increasing GHSV, the methanol conversion and $H_2$ flow rate of the Cu foam were higher than those of the Ni foam. This result is mainly due to the fact that the effective catalyst loading and catalyst coating strength of the Cu foam are higher than those of the Ni foam (as shown in Fig. 5). Fig. 11 shows the methanol conversions and $H_2$ flow rates of 100 PPI Cu and Ni foams as catalyst supports as a function of temperature with a GHSV of 16.2 K·mlL/g·h. It is easily seen that the Cu foam showed a significantly higher methanol conversion and $H_2$ flow rate, which is attributed to the fact that the thermal conductivity of the Cu foam is 89 W/mK and that of the Ni foam is 31 W/mK [26],

such that the heat transfer in the Cu foam will be more efficient and the hydrogen production performance will be better than that for the Ni foam.

### 3.5 Catalyst stability test

Stability is an important factor when measuring the reaction performance of microreactor. In the process of measuring the stability of the reaction performance in this study, 100 PPI Cu foam was selected as a representative catalyst support without clearance cascading (T2) at a reaction temperature of 300 °C and a GHSV of 16.2 K·mL/g·h. From Fig.12, it can be seen that with continuous measurement over 10 h, although the methanol conversion and $H_2$ flow rate decreased slightly, the $H_2$ flow rate remained above 0.3 mol/h. The Cu foam coated with Cu/Zn/Al/Zr catalyst by means of the two-layer impregnation method without clearance cascading (T2) showed remarkable stability for hydrogen production, and also showed no obvious inerting or sintering effects.

## 4 Conclusion

Cascading metal foams as catalyst supports were used in a new type of cylindrical laminated methanol steam reforming microreactor for hydrogen production, in which Cu/Zn/Al/Zr catalysts were coated using the two-layer impregnation method. The reaction performance of the methanol steaming reforming microreactor for hydrogen production was analyzed for different types of catalyst placement, PPI, and foam types of the catalyst support. This study showed that due to its high thermal conductivity and superior catalyst coating performance, 100 PPI Cu foam without clearance cascading exhibits the best hydrogen production performance

and stability when compared to the other metal foams in this study. The cylindrical laminated methanol steam reforming microreactor for hydrogen production developed in this study can be used as a reliable on-line source of hydrogen for PEMFC and hydrogen power electronic devices, and may have the potential for broad applications.

## Acknowledgments

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**Figure captions**

Fig. 1: Diagram of cylindrical laminated methanol steam reforming microreactor for hydrogen production.

Fig. 2: Diagram of the three types of metal foam placement in the reforming chamber: (a) T1: N = 3, D = 40 mm, H = 2, S = 2; (b) T2: N = 3, D = 40 mm, H = 2, S = 0; (c) T3: N = 1, D = 40 mm, H = 6.

Fig. 3: Schematic diagram of the testing system for the methanol steam reforming microreactor for hydrogen production.

Fig. 4: SEM images of 100 PPI Cu and Ni foams loaded with Cu/Zn/Al/Zr catalyst: (a) Cu foam×45; (b) Ni foam×45; (c) Cu foam-loaded catalyst×45; (d) Cu foam-loaded catalyst×2K; (e) Ni foam-loaded catalyst×45; (f) Ni foam-loaded catalyst×2K.

Fig. 5: Effective catalyst loading: (a) Cu foam; (b) Ni foam.

Fig. 6: (a) Methanol conversion; (b) $H_2$ flow rate of Cu foam with different types of catalyst placement as a function of GHSV.

Fig. 7: (a) Methanol conversion; (b) $H_2$ flow rate of Cu foam under different types of catalyst placement as a function of temperature.

Fig. 8: (a) Methanol conversion; (b) $H_2$ flow rate of Cu foams with different PPI as a function of GHSV.

Fig. 9: (a) Methanol conversion; (b) $H_2$ flow rate of Cu foams with different PPI as a function of temperature.

Fig. 10: (a) Methanol conversion; (b) $H_2$ flow rate of Cu and Ni foams as a function of GHSV.

Fig. 11: (a) Methanol conversion; (b) $H_2$ flow rate of Cu and Ni foams as a function of temperature.

Fig. 12: Stability of Cu foam-coated catalyst without clearance cascading: (a) methanol conversion; (b) $H_2$ flow rate.