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Thermodynamic Analysis of Temperature and Pressure on Carbon Deposition for Methane Reforming at Low Temperature in Micro-combustor

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ABSTRACT

Aimed at problems caused by carbon deposition in the micro-combustor, such as catalyst deactivation and channel block, based on the technology of methane-wet air autothermal reforming and the effects of hydrogen and methane conversion, the influences of temperature and pressure on carbon deposition below 973K are discussed with thermodynamic analysis method in this paper. Results show that for a definite feed gas composition, carbon deposition adds with increasing temperature firstly, and then decreases. Reaction pressure is suitable to maintain at 1atm. Moreover, the increasing methane mass flow, decreasing air and steam mass flow can lead to expansion of carbon deposition temperature region exists, also lead to the amount of carbon deposition increase and the temperature peak of carbon deposition shift to higher temperature segment. Under the research conditions that methane mass flow is 6.6g/h, reaction pressure is 1atm, air-methane ratio and steam-methane ratio are respectively 2 and 1 in the micro-combustor, the temperature range of carbon deposition production is at 680~850K. The largest carbon deposition is occurred and its mass fraction is 0.66% when the reaction temperature is at 785K, also the methane conversion rate and the mass fraction of hydrogen are approximately 53.43% and 2.37% respectively.

Keywords: Micro-combustor; Methane; Low temperature; Pressure; Autothermal reforming; Carbon deposition; Thermodynamic analysis

NOMENCLATURE

A	molar of air in feed gas (mol/h)
B	molar of stream in feed gas (mol/h)
C_p	heat capacity at constant pressure ($J \cdot mol^{-1} \cdot K^{-1}$)
M_{CH_4}	mass flow of methane in feed gas (g/h)
N	molar of methane in feed gas (mol/h)

P	pressure (atm)
P^θ	standard atmospheric pressure
T	temperature (K)
ΔG^θ	standard gibbs free energy (kJ/mol)
ΔH^θ	standard enthalpy (kJ/mol)
a、 b、 c	constants on heat capacity
k_i	equilibrium constants of reaction
n_i	molar of each component at equilibrium (mol/h)
	mole ratio of air to methane
	mole ratio of stream to methane

INTRODUCTION

With the researches on micro-combustion power plan in depth, more and more attention has been paid to carbon deposition caused by methane reforming as well as maintaining efficient and stable combustion. The carbon deposited could bring many problems to devices, such as channel block, increasing pressure-drop, affecting fluid distribution, and active area of catalyzer reduces by them [1]. At present, the study of carbon deposition on methane reforming mainly focus on the preparation method of catalyst with different core activity for different temperature in large space above 1100K and also the effect of the amount and species of carbon deposition on the reforming catalyst activity in the fixed-bed or fluidized bed [2-6]. However, there are few researches about the characteristics of carbon deposition in the micro-channels with low temperature (especially less than 973k) and low fuel flow.

It is known that methane-wet air premix catalytic reforming in the micro-chamber is a feasible method to maintain stable combustion [7]. Under a certain condition,

methane partial oxidation coupling with steam reforming can effectively reduce the carbon deposition, and a little hydrogen produced by reforming reaction can improve ignition and burning obviously [8-9]. Thus it is necessary to study the characteristics of carbon deposition during autothermal reforming in the micro-combustor, especially for the influence of temperature and pressure on the limits of deposition which bases on effect of hydrogen. The objective is to analyze the deposition behavior and find out its key factors of methane autothermal reforming at low-temperature.

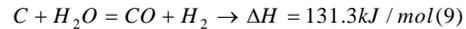
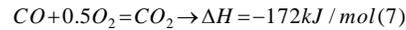
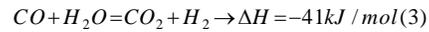
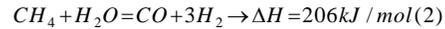
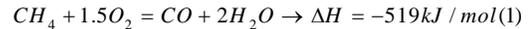
Ahmed S et al. [10] presented an ideal autothermal reforming reaction and analyzed that it represents steam reforming reaction when oxygen-methane ratio is 0, which is strongly endothermic reaction; when the oxygen-methane ratio increases, the needed heat reduces in the reaction; when the ratio is 0.44, the heat in the reaction is to minimum. Usually, the methane autothermal reforming reaction is in an exothermic condition (the oxygen-methane ratio is >0.44). So, carbon deposition for the autothermal reforming of methane and wet air in micro-combustor below 973K is discussed by thermodynamic analysis in this paper based on the feed gas ratio mentioned above. The influences of temperature and pressure on carbon deposition, effect of hydrogen and methane conversion under lean oxygen as the emphases of this article are researched theoretically. The objective is to analyze the deposition variation of methane autothermal reforming at low-temperature for further experimental study and to offer theory and technology support for developing the technology of micro-chamber catalytic reforming combustion.

1 SYSTEM OF CARBON DEPOSITION FOR METHANE REFORMING IN MICRO-COMBUSTOR

The structure features of micro-combustor are that channel is small (at magnitude of mm), resident time of gas is short and internal specific surface area is large. Furthermore, feed gas often do not yet react and then flow out which bring about low efficiency. However, resident time is inversely proportional to the mass flow and reasonable flow is the basic elements for the study. In recent years, the developed micro-engine of which volume is in the order of 1cm^3 can produce about 10~100W power, and the consumption mass flow of fuel is selected 7g/h in general [11]. On this basis, the initial mass flow of methane is chosen as 6.6 g/h for analysis in this paper.

The autothermal reforming of methane and wet air in micro-combustor is a complex reaction system. In the system, the heat emitted by the partial oxidation reaction is higher than that absorbed by methane steam reforming. The two processes can be divided into two steps and also can be joined together. For millimeter channel, Jiao et al [12] shows that space reaction could occur in the dimensions and surface catalytic reaction is restrained to a certain extent. Now without taking into account the wall effect of catalyst and NO_x , just the effect of gas composition, temperature and pressure involved in thermodynamics is considered. the main reactions

recommended by Christensen [13] in addition with carbon deposition reaction is described as follow:



Visibly, the substances participating in one reaction is appeared in other reactions at the same time. When the balance of the system is acquired, compositions in each reaction should have reached equilibrium. Although there are many reactions, only a group of independent reactions needs to be discussed. The so-called independent reaction is the one that can not be obtained by linear combination of other reactions in the group. If a group of independent reaction is selected, other reactions in the system can be acquired from this group reaction by linear combination [14]. Analyzing this balance system, there are CH_4 , H_2O , CO , H_2 , CO_2 , O_2 , C and N_2 , which the eight compositions are from four elements of C, H, N, O. Thus the system has four independent reactions (number of components - number of elements = 8 - 4 = 4). Reaction (1) is partial oxidation of methane, reaction (2) and (3) respectively is steam reforming reaction (SMR) and steam gas shift reaction (WGS) respectively. The three reactions are the main reactions accepted by many scholars for the study of methane autothermal reforming. In addition, due to pyrolysis reaction (5) of methane mainly occurs above 1100K, (1), (2), (3) and (4) are chosen as independent reactions to conduct the following thermodynamic equilibrium calculation and other reaction can be obtained by the linear combination of the four reactions.

2 THERMODYNAMIC CALCULATION METHOD

According to the group of independent reaction, equilibrium constant expressions of reactions can be found. Then due to one component attending reactions should meet all the independent reactions balance, each gas equilibrium composition can be acquired. Finally the change content of carbon deposition can be learned.

2.1 Reaction equilibrium constant calculation

The function of thermal capacity and temperature are as following, a , b , c is shown in table 1 [15]:

$$C_p = a + b \times 10^{-3}T + c \times 10^5 T^{-2} \quad (10)$$

$$\text{From } d\Delta H^0(T) = \Delta C_p dT \quad (11)$$

$$\Delta H^0(T) = \Delta aT + \frac{1}{2}\Delta b \times 10^{-3}T^2 - \Delta c \times 10^5 T^{-1} + I_0 \quad (12)$$

Substitute $\Delta H^0(298)$ to (11), the I_0 can be acquired:

$$\text{Because: } \frac{d \ln k^0}{dT} = \frac{\Delta H^0(T)}{RT^2} \quad (13)$$

$$d\Delta G^0(T) = -RT \ln k^0 \quad (14)$$

$$\text{So: } \ln k^0 = -\frac{I_0}{RT} + \frac{\Delta a}{R} \ln T + \frac{\Delta b \times 10^{-3}}{2R} T + \frac{\Delta c \times 10^5}{2RT^2} - \frac{I_K}{R} \quad (15)$$

Tab.1 Thermodynamic constants of each substance under 298K and atmospheric pressure

components	ΔG^0 (kJ/mol)	ΔH^0 (kJ/mol)	a	b	c
CH ₄	-50.79	-74.9	12.426	76.693	1.423
H ₂ O	-228.75	-241.8	29.999	10.711	0.335
CO	-137.3	-110.5	28.409	4.100	-0.46
H ₂	0	0	27.28	3.264	0.502
CO ₂	-394.4	-393.5	44.10	9.037	-8.535
O ₂	0	0	29.957	4.184	-1.674
C	0	0	0.084	38.911	-1.464

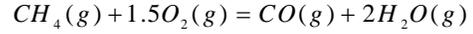
$\ln k$ can be acquired by $\Delta G^0(298) = -RT \ln K_{298}$, and then it is substituted to Equ.(12) to calculate the integral constant I_K , the relationship of reaction equilibrium constant and temperature can be obtained (Equ.(16)). Equilibrium constants of reaction (2) ~ (4) are expressed by k_2 , k_3 and k_4 respectively.

$$\begin{cases} \ln k_2 = -22736/T + 8.158 \times \ln T - 4.437 \times 10^{-3} \times T \\ \quad - 4.28 \times 10^3 \times T^{-2} - 26.219 \\ \ln k_3 = 5726/T + 1.56 \times \ln T - 0.15 \times 10^{-3} \times T \\ \quad - 0.476 \times 10^5 \times T^{-2} - 16.0813 \\ \ln k_4 = 20874/T - 1.52 \times \ln T + 2.39 \times 10^{-3} \times T \\ \quad - 0.546 \times 10^5 \times T^{-2} - 13.13342 \end{cases} \quad (16)$$

2.2 Characteristic model of equilibrium component in reforming reaction

Supposing that in feed gas: molar of methane, air and steam is $N(\text{mol/h})$, $A(\text{mol/h})$, $B(\text{mol/h})$ respectively; reaction pressure is P . Moreover, defining that mole ratio of air to methane is $\alpha = A/N$ and steam to methane is $\beta = B/N$. The compositions at balance in the system are shown in table 2.

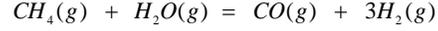
Firstly, methane is partially oxidized with lean oxygen, oxygen is consumed completely:



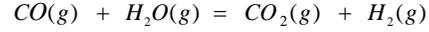
Initial	N	$0.21A$	0	B
balance	$N - 0.14A$	0	$0.14A$	B

Assuming that in reaction (2), the transformation amount of CH₄ is $x \text{ mol/h}$, the transformation of CO is $y \text{ mol/h}$ in (3),

and the transformation of CO is $z \text{ mol/h}$ in (4).



balance	$N - 0.14A - x$	$0.28A + B - x$	$0.14A + x$	$3x$
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balance	$0.14A + x - y$	$0.28A + B - x - y$	y	$3x + y$
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balance	$0.14A + x - y - z$	$0.5z + y$	$0.5z$	
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Table 2. The molar of each component at equilibrium

Component	H ₂	CO	C	CH ₄
n_i (mol/h)	$3x+y$	$0.14A+x-y-z$	$0.5z$	$N-0.14A-x$
Component	CO ₂	H ₂ O	N ₂	Total (not C)
n_i (mol/h)	$y+0.5z$	$0.28A+B-x-y$	$0.79A$	$N+B+1.07A+2x-0.5z$

In the paper, conversion rate of CH₄ is regarded as $[(0.14A+x)/N] \times 100\%$. And the mass fraction of components is expressed as [the quality of one component/ the total quality of system] $\times 100\%$.

The equilibrium constant can be expressed by the partial pressure of components when the reaction system reaches balance. The results are show below:

$$\begin{cases} k_2 = \frac{(0.14A+x-y-z)(3x+y)^3}{(N-0.14A-x)(0.28A+B-x-y)(1.07A+N+B+2x-0.5z)^2} \times \left(\frac{P}{P^0}\right)^2 \\ k_3 = \frac{(y+0.5z)(3x+y)}{(0.14A+x-y-z)(0.28A+B-x-y)} \\ k_4 = \frac{(y+0.5z)(1.07A+N+B+2x-0.5z)}{(0.14A+x-y-z)^2} \times \left(\frac{P}{P^0}\right)^{-1} \end{cases} \quad (17)$$

According to the size feature of the micro-combustor [11], one basic parameter of methane mass flow is $M_{\text{CH}_4} = 6.6(\text{g/h})$. Besides, when the oxygen to methane ratio is 0.44 which is mentioned above, the corresponding air-methane ratio and steam-methane ratio are about 2 and 1 respectively. This article focuses on the influences of temperature and pressure at the certain feed gas ratio on carbon deposition. Therefore, the other basic reaction parameters are chosen as: air-methane ratio is =2, steam-methane ratio is =1, the inlet temperature which is the premix temperature of feed gas is equal to the reaction temperature. Then the results about k_2 , k_3 and k_4 from Equ. (16) at certain temperature are substituted to Equ. (17). MATLAB as a facility is used to solve this equation. Consequently, all the equilibrium compositions of system can be obtained at different temperature, pressure and feed gas ratio based on the basic parameters. And the rule of carbon

deposition with the change of temperature and pressure is observed.

3 RESULT AND DISCUSSION OF THERMODYNAMIC ANALYSIS FOR CARBON DEPOSITION

3.1 Influence of temperature

Table 3. Mass fraction of carbon deposition in balance at different temperatures

T (K)	C (%)	T (K)	C (%)
635	/	823	0.460
673	/	847	0.079
680	0.0016	850	0.016
723	0.337	873	/
773	0.640	923	/
785	0.658	973	/

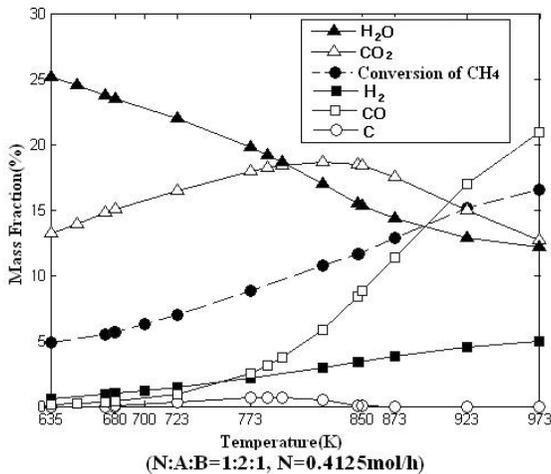


Figure 1. Influence of temperature variation on the components in equilibrium

Thermodynamic equilibrium calculation below 973K is done according to the above conditions. The result of carbon deposit is shown in table 3. It can be seen that carbon deposit reaction of methane autothermal reforming in micro-channel mainly occurs in 685-850K. Out of this temperature range, the thermodynamic equilibrium system without carbon deposition is constructed again, in which reaction (4) is not considered. Fig.1 shows that the influence of temperature on equilibrium components. It shows that the amount of carbon deposition firstly increases and then decreases with the rise of temperature. The appearance of carbon deposition does not work remarkable effect on the trend of methane conversion and hydrogen, which both increases with temperature rise. Taking 680K for example, the mass fraction of carbon deposition is 0.0016%, the mass fraction of reacted methane and hydrogen is 5.68% and 0.99% respectively. The carbon deposition reaches the maximum of 0.66% at 785K, while 9.29% and 2.37% for the reacted methane and hydrogen, also the methane

conversion rate is 53.43%. When temperature reaches 850K, Carbon deposition reduces to 0.016%, reacted methane and hydrogen keep on increasing to 11.73% and 3.41% respectively.

The change of CO₂ shows an increase-then-decrease trend, the maximum of which achieve 18.31% at 823K. And the amount of water has been declining. Though the amount of CO increases, the changing speed is different according to temperature. At low temperature, its increase is not obvious. However, when it reaches the temperature at which there is the peak of carbon deposition, CO increase sharply.

The main reason is that WGS and carbon deposit reaction are both micro-exothermic reactions and their equilibrium constants are bigger especially temperature below 823K than SMR reaction, as shown in table 4. It means that the generated CO from methane oxidation mainly takes part in reaction (3) and (4) at lower temperature. So C, H₂ and CO₂ all increase firstly and the amount of carbon deposition reaches its peak at approximately 785K, while CO increases slowly because of massive consumption. As the increase of temperature and heat in the system, SMR gradually plays a major role, thus the reacted methane and the amount of hydrogen continuously increase. The information can be found in table 4 that with temperature increase, the equilibrium constants of reaction (3) and (4), especially reaction (4), rapidly decrease. Temperature gradually becomes the main factor restricting the above two reactions. All these factors result in the decrease of C and CO₂, while CO increases sharply because of the quick SMR reaction and the restriction of reaction (3) and (4).

Table 4. Equilibrium constant at different temperature

T(K)	k_2	k_3	k_4
573	5.92E-08	36.17	2801670.7
673	5.15E-05	10.79	13003.8
773	0.008101	4.50	249.2
873	0.4081	2.33	12.17
973	9.2308	1.41	1.14
1023	34.883	1.14	0.42

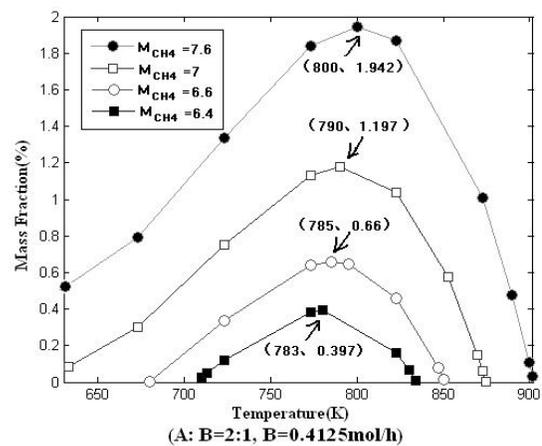


Figure 2 Carbon content vs temperature under different mass flow of methane in equilibrium

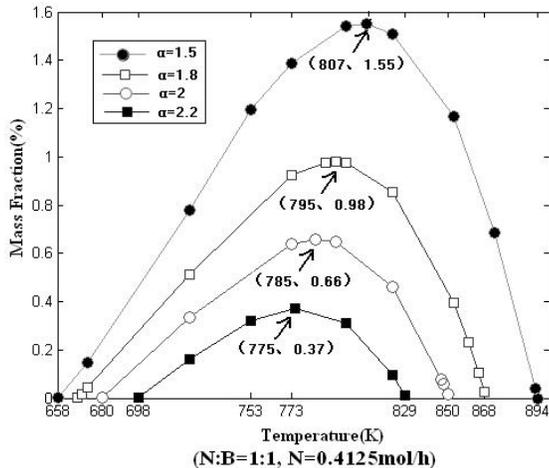


Figure 3 Carbon content vs temperature under different air condition in equilibrium

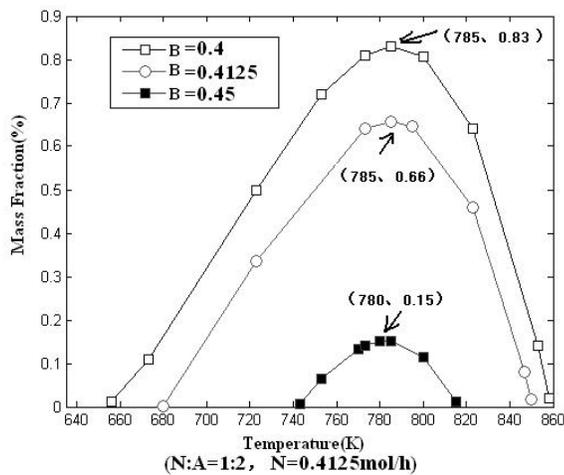


Figure 4. Carbon content vs temperature under different steam condition in equilibrium

The mass fraction of carbon deposition at different temperatures (below 973K) is also researched through changing mass flow of methane, air and steam, as shown in Fig. 2-4. The carbon deposition all firstly increases then decreases with temperature rising and it always appears in a certain temperature range. In the condition of only changing mass flow of methane, carbon deposition appears between 710K and 835K when M_{CH_4} is 6.4g/h; the temperature range is 633-870K when M_{CH_4} is 7g/h. If only change the amount of air, the related temperature range is 658-895K when α is 1.5; when α changes to 2.2, the temperature is about 700-830K. If only the amount of steam is changed, the temperature range appears in 743-815K and 656-860K respectively when B is 0.4mol/h and 0.45mol/h. The main reason is that heat of system aggregates with the increase of temperature at low temperature, and the amount of carbon appears and increases. But it turns to decrease at a certain point with temperature increasing, which means the deposit capability of the system

gradually disappears. The explanation is that the restriction of temperature on reaction (4) is enhanced, which can be seen from equilibrium constant, and the forward direction reaction is weakened.

From fig.2-4., the following phenomenon can be seen with increase of methane or decrease of air and steam. The temperature range in which carbon deposition appears extends and the amount of carbon deposition increases. And the corresponding temperature of peak carbon deposition shifts to higher. All these can be seen from the cases below. When the mass flow of methane increases from 6.4g/h to 7g/h, the maximum mass fraction of carbon deposition rises from 0.397% to 1.194%, and the corresponding temperatures of which are 783K and 790K. For the change of air carbon ratio from 2.2 to 1.8, the maximum value of carbon deposition mass fraction changes from 0.37% (at 775K) to 0.98% (at 795K). The peak value of carbon deposition mass fraction increases from 0.15% (at 780K) to 0.83% (at 785K) with the change of steam flow from 0.45mol/h to 0.4mol/h. Nevertheless, no matter what the feed gas ratio mentioned above in the micro-channel, the temperature range at which the most carbon deposition appears is always between 750 K and 800K.

According to the analysis above, when temperature is below 973K, the carbon deposition caused by CO disproportionation reaction only appears in a certain temperature range and gradually disappears at higher temperature. The reason here is that methane cracking in high-temperature is ignored, the rise of temperature is harmful for disproportionation reaction, which is an exothermic reaction. So reaction temperature for carbon deposition removal is very important on the research of methane autothermal reforming in micro-channel at low temperature.

3.2 Influence of pressure

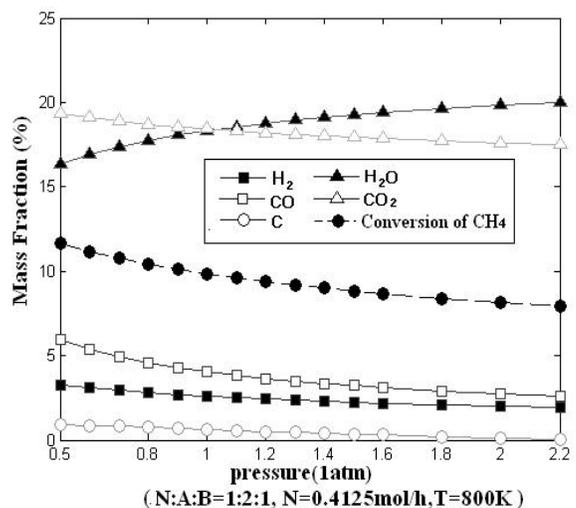


Figure 5. Influence of pressure variation on the components in equilibrium

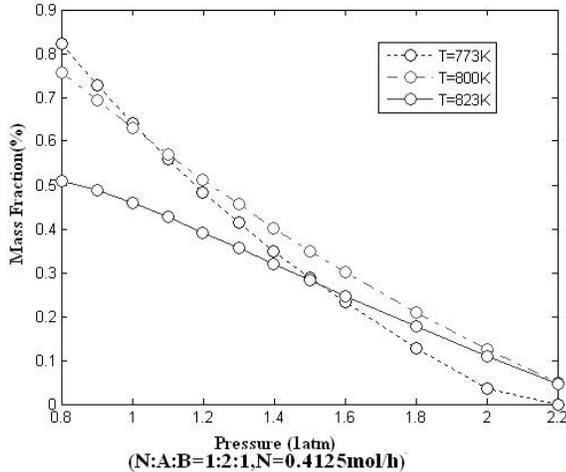


Figure 6. The change of carbon content with pressure variation under different temperature in equilibrium

The influence of reaction pressure on thermodynamic equilibrium component at 800K is shown in fig.5. It can be seen that the amount of H₂O tends to increase and others in the figure all tend to decrease, and among which the change of methane conversion and hydrogen is relatively quickly. At 0.8atm, the mass fraction of reacted methane, hydrogen and carbon are respectively 10.43%, 2.81% and 0.76%. The values of the three above parameters are 8.99%, 2.30% and 0.40% at the pressure of 1.4atm. Also it can be seen that the changing rate of these components becomes smaller gradually as pressure rises. The change of mass fraction of reacted methane, carbon and hydrogen are 1.44%, 0.51% and 0.36% respectively as pressure increases from 0.8 to 1.4 atm. However when pressure changes from 1.4 to 2atm, the corresponding reductions are 0.86%, 0.30% and 0.28%.

From the perspective of chemical equilibrium, it is a volume-increase reaction. High pressure would lead that the reaction tends to occur toward the volume-decrease direction and the decline of methane conversion, which is harmful to the occurrence of reforming reaction. At the same time, the amount of H₂O increases, while H₂ and CO are reduced. Then the C and CO₂ decrease because of the reduction of CO leads to WGS and carbon deposit reaction carry toward reverse direction. Besides, it can be seen in fig.6 that carbon deposition always declines with pressure increase and just the higher the temperature the more slowly decreasing of carbon deposition.

In the two charts mentioned above, it can be seen that increasing pressure can reduce the amount of carbon deposition. But the effect of pressure on carbon deposition is not obvious, while the amount of reacted methane and hydrogen decrease faster with pressure increase. Thus the reaction pressure in micro-channel should not too be high. Choosing an atmospheric pressure which is easy to operate not

only could minish carbon deposition but also promises a certain methane conversion and hydrogen yield.

4 CONCLUSIONS

Temperature plays an important role in carbon deposition caused by methane reforming in micro-combustor, especially when the temperature is below 973K. With the temperature increase, the amount of carbon deposition increases firstly and then decreases. Moreover, the change of CO₂ shows an increase-then-decrease trend and the amount of H₂O has been declining. Though the amount of CO increases, its increase is not obvious at low temperature. But when it reaches the temperature at which the peak of carbon deposition arrives, CO increase sharply.

Increase of methane mass flow or decrease of air and steam leads to expansion of carbon deposition temperature region and the amount of carbon deposition. Also it can be found that the temperature peak of coking shifts to higher temperature segment. When the methane mass flow rate is maintained around 7g/h, no matter what kind of feed gas proportion in micro-combustor, the largest amount of carbon deposition appears between 750~800K.

Increasing pressure can reduce the production of carbon deposition, however the mass fraction of hydrogen and methane conversion also decrease. Moreover, the effect of pressure on deposition is not obvious. Thus it is proper to keep an atmospheric pressure in micro-channel

Under the research conditions that mass flow rate is 6.6g/h, reaction pressure is 1atm, air-methane ratio and steam-methane ratio are respectively 2 and 1 in the micro-combustor, the temperature range which carbon deposition exists is at 680~850K; it is up to the largest carbon deposition mass fraction of 0.66% when temperature is at 785K, which methane conversion rate and mass fraction of hydrogen are approximately 53.43% and 2.37% respectively.

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