고온용 니켈 나노필터 제조에 있어서 세라믹 코팅 효과에 관한 연구

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The effect of ceramic coating on nickel nano-filter for hot-gas filtration

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1. Introduction

The product gas from solid fuel gasification process consisting of hydrogen, carbon monoxide and hydrocarbons can be used for fuel in molten carbonate fuel cell (MCFC) and gas turbine or raw material for hydrocarbons synthesis, i.e. methanol, liquefied petroleum gas (LPG) and gasoline etc.. However, apart from the main gas components, the product gas from the gasifier contains several impurities like particles, tars, alkalis (Na, K), nitrogen compounds and sulphur compounds etc.[1-4]. Especially, particles can result in plugging, abrasion of downstream equipment and environmental pollution. Even though micron size particles have a large portion analyzed by mass distribution, when analyzing the number of particles rather than mass distribution, it is obvious that a large portion has sub-micron particles. Thorough gas cleaning of the gasification gas is therefore essential for reliable and environmentally sound operation[5]. Furthermore, high temperature gas cleaning is one of the most promising technologies because it can potentially achieve substantial energy savings and provide means for a more effective whole process optimization. It means that the installation of a high temperature gas cleaning filter can protect the following installation such as condensation, heat exchangers and/or wet scrubbers.

There are different kinds of filter media. While fine pore size ceramic media is available at high temperature [6, 7], the use of ceramic filter has limitations, i.e. week mechanical strength, long-term microstructural instabilities at operating condition such as temperature and vapor, and permeability limitation. On the other hand, the metal filters has achievable potential advantages such as good handling, mounting and sealing properties and durability to withstand thermo-mechanical shocks and stress caused by system vibration[8]. Unfortunately, metal filter could not provide high efficiency for the ultrafine particle in sub-micron size to introduce to the solid fuel gasification process. In previous study, we developed nickel nano-filter which can clean nano-sized particles from air with very high efficiency [9].

The aim of this study is to increase sustainable temperature of nickel nano-filter that separates ultrafine particles from gas with high efficiency. Gas permeation flux test via pressure drop was carried out with air at room temperature. The nickel nano-filters were characterized with SEM and mercury porosimeter.

2. Experimental

2.1. Fabrication of nickel and alumina modified nickel filtering media

15g of pure nickel powder which has purity of 99.9% and particle size of 2.0 -10.0 ㎛ (average 5 ㎛) and alumina coated nickel powder with aluminum nitrate solution by incipient wetness impregnation method were compressed without binder in metal cylindrical mold with 50 mm in diameter using a home made press under pressure of 42 MPa. The compressed nickel filters (designated CN series) and alumina coated nickel filter (designated AC series) were further heat treated in vacuum furnace filled with high-purity hydrogen at the temperature range of 450-900℃ and 1100-1400℃ respectively.

2.2. Gas permeation and particles filtering test

Gas permeation test was conducted with air at room temperature. Air was introduced by mass flow controller and the pressure was controlled by digital pressure regulator. The air permeation flux was measured by a digital soap-bubble flow meter.

3. Results and discussion

3.1. Filter properties and air permeation flux

Heat treatment helps filter have mechanical strength and thermal resistance. However, small sized metal powder is prone to be sintered at high temperature. This causes the porosity, average pore diameter and total pore volume of filter to decrease and the air permeation flux too. In order to clarify the effect of heat treatment temperature, CN and AC series nickel filters were treated at the temperature range of 450-900℃ and 1100-1400℃ respectively. As shown in Fig. 1, the solid volume fraction of CN and AC series filters increases with increasing heat treatment temperature. Since the sintering of nickel powder took place from 600℃ (CN042060), the undulation surface of nickel powder became smooth ones. Furthermore, above 700℃ (CN042070), the pore fraction drastically decreased with an increase in the heat treatment temperature. On the other hands, AC series filter showed very stable structure up to 1200℃ (AC042120). At 1300℃ (AC042130), filter partly melted down and some pores were plugged. At 1400℃ (AC042140), the great part of filter melted down and the most of pores were plugged.

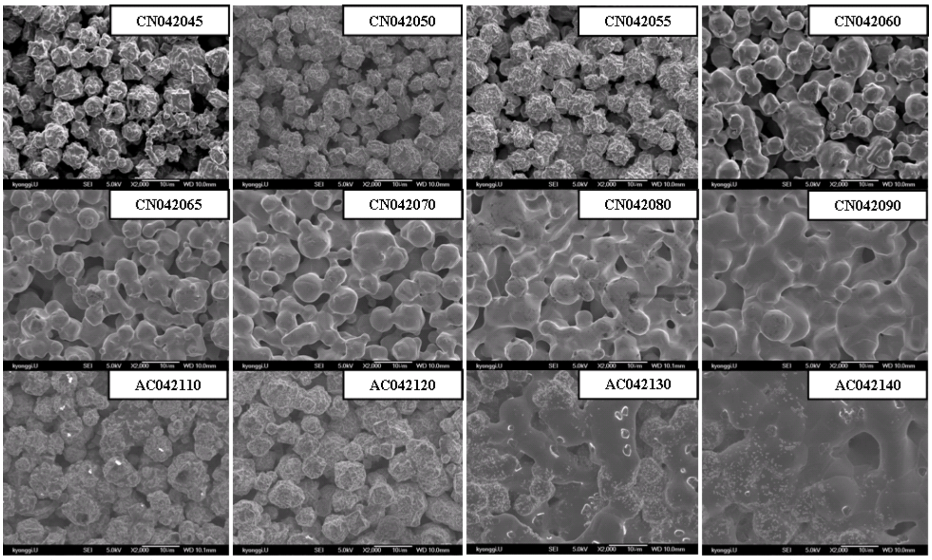

Fig. 1. The surface SEM images of CN and AC series nickel filter.

Fig. 2 is the porosity and average pore diameter along with the heat treatment temperature of CN and AC series filters. As shown, the porosity of CN series increased along with the heat treatment temperature range from 450 to 500℃. And then it decreased with increasing the heat treatment temperature. On the other hand, the average pore diameter of CN series filters increased along with temperature range from 450 to 550℃ and then decreased with increasing the heat treatment temperature from 550 to 900℃. The surface sintering of nickel powder at 550℃ extends the pore size and it seems to cause the increase of porosity and average pore diameter. However, above 550℃, nickel powder melted down and the pores were plugged resulting the decrease of porosity and average pore diameter. On the other hands, the porosity of AC series filters decreased with increasing temperature and the average pore diameter increased along with temperature range from 1100 to 1300℃ and than drastically decreased at 1400℃. From SEM and mercury porosimeter analysis, it can be insisted that alumina coating on the surface of nickel powder prohibited metal sintering, melting down and pore plugging.

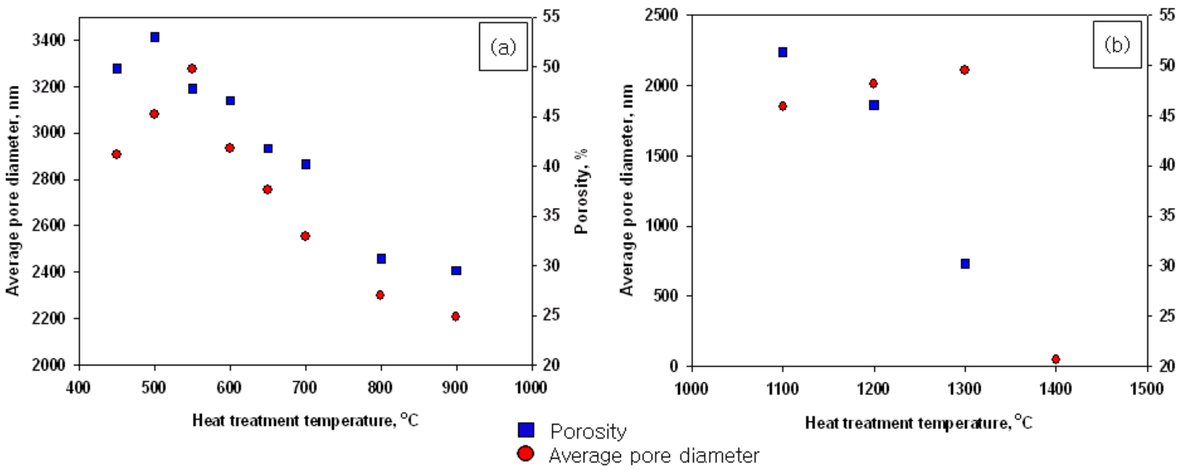

Fig. 2. The pore properties of CN (a) and AC (b) series nickel filters.

Fig. 3 shows the effect of the heat treatment temperature on the air permeation flux. It can be seen that the air permeations of CN042045 and CN042055, which were treated at 45 0℃ and 550℃ resfectively, were almost equal. CN04250 which was treated at 500℃ had largest air permeation flux. It can be explained by porosity of Fig. 2 where porosity of CN042055 is larger than any other filters. Though, above 700℃, air permeation drastically decreased, the air permeation decrease rate was small up to 650℃. It means that the CN series filter can withstand up to 650℃. On the other hands, the air permeation flux of AC series filter were little bit lower than CN series filter. It can be explained by the average pore diameter in Fig. 2. As shown in Fig. 2, while the porosity was similar to the CN series filter, the average pore diameter was smaller than CN series filter. However, the air permeation flux of AC series filter showed very good stability up to 1200℃ and similar to the CN series filters whose heat treatment temperature were below 650℃ considering average pore diameter rate.

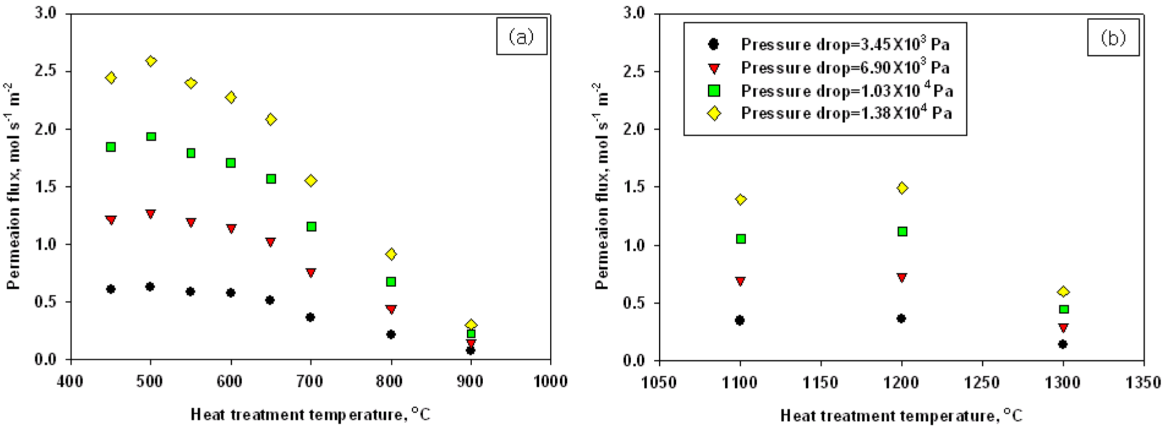

Fig. 3. The effect of heat treatment on air permeation flux: (a) is CN and (b) is AC series.

Conclusions

Nickel filters were successfully made by nickel powder pressing process. It was clarified that the air permeation flux depended on porosity and average pore diameter. And the porosity and average pore diameter were controlled by heat treatment temperature. Since the nickel powder which has mean-size of 5㎛ was prone to be melted at high temperature, the nickel filter could not withstand over 700℃. However, the alumina coating on the surface of nickel powder prohibited the metal sintering and the enclosing of the pores of the nickel filter. The alumina coated nickel filter could withstand up to 1200℃.

We hope that the developed nickel filter not only could be applied to the high temperature process such as solid fuel gasification for reliable and environmentally sound operation of the following process, but also provide energy savings and a more effective whole process optimization.

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