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Fabrication of porous nickel–copper alloy with controlled micro-sized pore structure through the Kirkendall effect

Linping Yu [a, b], Yao Jiang [a, *], Yuehui He [a], Xinli Liu [a], Huibin Zhang [a]

[a] State Key Laboratory of Powder Metallurgy, Central South University, Changsha 410083, PR China
[b] School of Chemistry and Biology Engineering, Changsha University of Science & Technology, Changsha 410114, PR China

HIGHLIGHTS

- The mono-pore size and asymmetric porous Ni–Cu alloy are conveniently fabricated by powder metallurgy.
- The Kirkendall effect plays an important role in the pore generation and evolution process of porous Ni–Cu fabrication.
- The mono-pore size porous Ni–Cu alloy shows an excellent corrosion resistance in the hydrofluoric acid at 65 °C.
- The mono-pore size porous Ni–Cu alloy filter greatly simplifies the treating of phosphorus slag in the phosphorus producing.

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ABSTRACT

To solve the mirofiltration problem in harsh environments, the porous Nickel–Copper (Ni–Cu) alloy is fabricated by powder metallurgy. Homogenous skeleton and abundant interconnected pores microstructure are found in the as-sintered porous Ni–Cu alloy. Meanwhile, it maintains an adequate mechanical strength under high temperature up to 900 °C. Corrosion kinetics indicates that the corrosion resistance of porous Ni–Cu is superior than porous nickel in the same concentration of hydrofluoric acid. Compared with conventional phosphorus slag purification practices, the described porous Ni–Cu filters enable the superior-yield of phosphorus effectively and have the potential to decrease the energy consumption and simultaneously ensure an environmental friendly process in phosphorus industry.

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1. Introduction

Three-dimensional interconnected porous metals with high strength, good flexibility and excellent machinability have been widely applied in fine chemicals [1], biomaterials [2], environment engineering [3], etc. Some fabrication technologies of the porous metals, including the metal powders and fiber sintering method [4,5], the organic metal salts decomposition and sintering [6], the de-alloying method [7] and so on, have been developed to fabricate various porous metals. However, it is a great challenge to prepare an asymmetric porous metal membrane as a filter combining both high filter fineness and large permeation flux, which will have a wide range of application in present production, especially in the severe serving environments. For porous metals, both the large number of interconnected pores with the controllable pore size and the precise chemical composition are crucial. The bulk Nickel–Copper (Ni–Cu) based alloys exhibit both the excellent corrosion resistances in various acid, alkali and salt solutions [8,9], especially in the solution containing fluoride ion [10], and typical electrochemical performance, which made it could be applied in hydrometallurgy, nuclear industry, chemical catalytic synthesis [11], hydrogen producing [12], fuel cell industry [13] and other industries. Thus, it is very important to develop the Ni–Cu based alloys to fabricate the special novel porous membrane with high performances, which might bring about the revolutions of some traditional industries.

About two million tons of yellow phosphorus, in which the traditional hydrometallurgy [14,15] with the huge precipitation tank for the sufficient dissolution of the yellow phosphorus separating from phosphorus slurry in the hot water more than 65 °C is utilized, has been produced in China annually. The current yellow

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* Corresponding author.
E-mail address: jiangyao@csu.edu.cn (Y. Jiang).

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phosphorus production results in a great deal of energy consumption, water consumption and considerable environmental pollution. A large number of separation and purification technologies of porous membrane filtering for the phosphorus production, mainly replacing the current fabric filtering, have not been realized due to the existence of fluorine associating phosphorus ore [16].

It is well-known that there are a large number of pores formed in the diffusion couple heated at elevated temperature if the counter-diffusion coefficients of the components exists a significant difference, which is called the Kirkendall effect [17–21]. A great different counter-diffusion coefficient of Nickel and copper makes it to be a typical diffusion couple to confirm this effect [19,22], which enlightens on that the fabrication technology of the porous Ni–Cu based alloys reactively synthesized by the mixed Ni and Cu and other alloying elements powders. Based on the pore-forming mechanism of the Kirkendall effect, a new fabrication technology of porous Ni–Cu alloy with controllable pore size has been developed. As anticipated, this new porous Ni–Cu alloy not only has homogenous pore structure but also maintains the excellent corrosion resistance property of bulk Ni–Cu alloy.

For all the bulk Nickel–Copper (Ni–Cu) based alloys, the system with composition of Ni:Cu = 2:1 exhibits an excellent corrosion resistance in both the reductant and the inoxidable acid. Sometimes minor additives enable this alloy to meet with different mechanical and machining requirements while the chemical stability is not affected at the same time [9,10]. To avoid the influence of impurity elements to the diffusion and pore forming process, a binary composition of 67 wt.%Ni–33 wt.%Cu is determined for developing the corresponding porous structure materials in this research. The evolution of the pores, the characteristics of the diffusion process during the sintering at elevated temperatures, as well as the mechanical properties of the sintered porous Ni–Cu are demonstrated in detail. In addition, the chemical stability of the prepared porous Ni–Cu in hydrofluoric acid has also been explored preliminarily. A pilot scale test firmly shows the great prospect of the porous Ni–Cu for applying in purification process in phosphorous industry.

2. Experimental procedures

2.1. Preparation of porous Ni–Cu alloy

Nickel and copper were of highest purity (99.9 wt.%) commercially available (Jin Chuan Co.). They were mixed to obtain a uniform mixture of Ni-rich (67 wt.%Ni–33 wt.%Cu) composition. The median diameter for Ni and Cu copper are 45.0 μm and 37.0 μm, respectively. The mixing tanks have filled with dried nitrogen to avoid the oxidation.

Cylindrical compacts with dimensions of Φ30 mm × 2.0 mm are cold pressed at uniaxial pressure of 100 MPa in air. Sintering is performed in the furnace in vacuum of 1.0×10^{-4} Pa. The compacts are buried in alumina balls for further uniform heat conduction, the heating rate is controlled in 5 °C per minute, at temperatures of 500, 600, 700, 800, 900, 1000, 1100 and 1150 °C for durations of 1 h followed by furnace cooling to room temperature.

2.2. The composition and pore structure analysis

In the process, porous Ni–Cu alloy samples are prepared for microstructural analysis and measurement of the mechanical properties. The permeate flux, maximum pore size of the porous Ni–Cu alloy are measured by bubbling test [23] with pure N_2 as the flow medium. The pore size distribution is determined by the use of mercury intrusion (Pore Master 33GT, Quantachrome Instruments). Archimedes principle is employed to measure the open porosity of porous samples.

The phase and crystal structure are analyzed by XRD (D/max 2550VB with Cu Kα radiation). The morphologies of the materials are characterized by SEM(JSM-6360LV, 20 kV). The elemental distributions of Ni–Cu samples are also analyzed by the area scan of energy dispersive spectroscopy (EDS). The three dimensional X-ray diffraction microscope (Xradia VersaXRM-500) gives the morphology of internal pore structure of as-sintered porous Ni–Cu material. The tensile strength and bending strength of porous NiCu alloy are measured using an Electric Servo-hydraulic Material Test system (INSTRON 3369). The strain rate of hot tensile test is 1.0 mm/min.

Elemental composition of sintered porous Ni–Cu alloy is measured by ICP, after wholly dissolved with 50% (volume ratio) nitric acid.

Three samples are run for each measurement of the porous parameters, the mechanical strength, and the corrosion resistance in hydrofluoric acid.

2.3. Preparation of asymmetric porous Ni–Cu alloy

Raw materials for coating films are the carbonyl-nickel powder and the argon atomized copper powder with purities above 99.9 wt.%, with the median particle size of 4.0 and 9.0 μm, respectively. Well blended fine powders together with 10 wt.% PVB in ethanol gel are made into slurry, which is spread on the surface of semi-finished porous Ni–Cu substrate, with thickness controlled within 100–200 μm. The Ni–Cu substrate with fine mixture coating is dried at 70 °C in vacuum oven (255 Pa) for 3 h, and then sintered in the vacuum furnace. The mixing and first part of sintering procedure of the semi-finished porous Ni–Cu substrate are the same as mentioned in subsection 2.1, while the sintering ends at 700 °C. After the fine powders have adhered on the substrate, sintering continues, while the heating rate is controlled in 2 °C per minute avoiding the crack of coating, at temperatures of 800, 900, 1000, 1100 and 1150 °C for durations of 1 h. The slow enough heating rate and the durations at different temperatures can ensure the completely decomposition of PVB, otherwise the cracking will happen in the coating layer.

2.4. Corrosion test in hydrofluoric acid

Immersion test of porous Ni–Cu with average pore size of 5.2 μm is carried out in 6.0 mol/L HF solution (65 °C), porous Ni with the same average pore diameter is used for comparison. Inductively coupled plasma atomic emission spectrometry (ICP) is adopted for determination of ions concentration corroded from porous Ni–Cu immersing in hydrofluoric acid. The open circuit potential of porous electrodes versus time in the hydrofluoric acid is measured at multi-channel electrochemical work station by three electrode system. Electrodes are sealed by polyvinylidene fluoride (dissolved with 1-Methyl-2-pyrrolidinone), the apparent working area is 1 cm^2. The reference electrode and auxiliary electrode are saturated calomel electrode and platinum, respectively.

3. Results and discussion

3.1. The composition and general morphology of porous Ni–Cu

The X-ray diffraction analysis (XRD) results of the compacts sintered in vacuum furnace at different temperatures duration for 1 h at a certain heating rate of 5 °C/min are shown in Fig. 1a. It can be seen that the alloying between Ni (JCPDS: 65-0380) and Cu (JCPDS: 65-9743) component is continually proceeding with increasing the sintering temperature. There is a single homogenous

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