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Porous Ni-Cr-Mo-Cu alloys fabricated by elemental powder reactive synthesis

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Abstract

Fabrication of porous Ni-Cr-Mo-Cu alloys is available through Ni, Cr, Mo and Cu elemental powder reactive synthesis at the sintering temperature of 1150 °C. The pore structures, including swelling behavior, open porosity, pore size and viscous permeability of the porous Ni-Cr-Mo-Cu alloys are systematically investigated. The results revealed that the parameters of pore structure firstly increased, and then decreased with the increase of the sintering temperature. When the sintering temperature rose to 1000 °C, the volume expansion, open porosity and viscous permeability of porous Ni-Cr-Mo-Cu alloys reached the peak value of 8.0%, 41.6% and 17.2 μm², respectively. In addition, it was confirmed that the pore structure evolution was based on three stages, that is, in the low temperature section before stearic acid decomposition, the formation of pores was initiated by the volatilization of pore-forming agent and the interstitial pores of green compact; when the temperature was lower than 1083 °C, the increase of open porosity was mainly due to the Kirkendall effect caused by solid partial diffusion; while, when the temperature is within the range of 1083 °C, the shrinkage behavior at high temperature and a small amount of metal solution formed by the unreacted Cu led to decrease in pore structure parameters.

1. Introduction

Due to the three-dimensional interconnected pore structure, porous materials are widely used in the field of separation and filtration, fluid blending, gas distribution and split-flow process. Among all porous materials, the porous alloys play an important role, especially in the industries with severe environment due to their excellent fluid permeability and high strength, and the advantages in machining and welding performance [1, 2]. It is well documented that the Kirkendall effect may lead to the formation of pore in the materials [3, 4]. Because of Kirkendall effect, powder metallurgy has the opportunity to make use of either elemental or prealloyed powders to fabricate porous alloys. Due to the advantages of near-net-shape formation, lower sintering temperature and adjustable pore structure, powder metallurgy is widely used in the preparation of porous alloys [5–7].

So far, many researches have been carried out on porous alloys for their unique properties. The porous Fe-Al intermetallics was reported by Gao et al [8, 9], which was realized through elemental powder reactive synthesis with Fe and Al, the open porosity reached 41% at the temperature 1000 °C. Yao Jiang [10, 11] studied the performance of porous Ti-Al alloys prepared by elements reaction synthesis systematically, and found that the Ti-Al porous alloys exhibited excellent performance in terms of corrosion and oxidation resistance at elevated temperatures. Besides, porous NiAl prepared via reactive sintering technology by Dong et al [12] also demonstrates good pore structure parameters. However, several strong exothermic peaks of Fe-Al, Ti-Al and Ni-Al porous alloys appeared during the sintering process, which are accompanied by the occurrence of severe exothermic phenomenon, leading to the collapse of the pore structure of porous materials and severe material...
deformation. Some studies have shown that porous materials prepared by combining multiple mixed element powers can slow down the exothermic reaction to a certain extent, and keep the pore structure intact [13–15]. Wu et al[13, 16] prepared the porous Ni-Cr-Fe alloy with the Cr content changed through powder metallurgy, and the results showed that the increment of the Cr content could slow down the exothermic reaction, and keep the shape of the porous alloy intact. However, the exothermic phenomenon still exists, and the process for the control of temperature rise is complicated. Therefore, it is of great significance to develop new porous material characterized by low heat dissipation, stable sintering process and controllable pore structure, and they have the same excellent pore structure parameters, which can be used in complex environments. Nickel-based Hastelloy alloy (Ni-Cr-Mo-Cu alloy) shows excellent properties, which is widely used for pipeline transportation, corrosion-resistant valves and key components of aircraft carrier shafts and pumps in the environment of hydrofluoric acid, hydrochloric acid, alkali salts and reducing acid media. However, comparing with previous works on the fabrication of porous Ni-Cr-Mo-Cu alloy, few reports are made on the preparation of porous Ni-Cr-Mo-Cu alloy without any pore former. And the pore forming mechanism of porous Ni-Cr-Mo-Cu alloy with elemental powders has not been reported as well.

In this paper, porous Ni-Cr-Mo-Cu alloy was fabricated with the Cr content varying from 10% to 35% through reactive synthesis in vacuum furnace. The effects of various parameters, including composition of the initial powders, temperature, phase constitution and pore structure of porous Ni-Cr-Mo-Cu alloy are systematically investigated. Besides, the pore structure forming mechanism is also explored.

2. Experimental procedure

In the present work, powder mixtures of Nickel (99.9% pure, 500 ~ 800 mesh), chromium, molybdenum and copper (99.9% pure, 200 ~ 400 mesh) were selected as the starting material (the composition were (75%–x)Ni(10%+x)Cr10%(Mo5%Cu, x = 0, 5%, 10%, 15% and 20%, mass fraction) to fabricate porous Ni-Cr-Mo-Cu alloys. Extra 5% stearic acid was added into the mixture as a binder for the shaping of green compact. The powders were mixed for 24 h in a polyurethane jar with zirconia balls used as the mixing balls at the ball/powder ratio of 1:2. After that, the dried mixtures were cold-pressed into green compacts with the dimensions of 40 × 10 × 2 mm under the pressure of 160 MPa. And then the as-pressed discs were sintered in a vacuum furnace at the vacuum degree of 1.2 × 10⁻³ Pa. To study the effects of sintering temperature on phase transition and pore structure evolution, the sintering temperature was increased from 550 °C to 1150 °C at the increasing interval of 150 °C, and the holding time of 2 h.

The phase constitution was detected by x-ray diffraction (XRD: Dmax 2500VB) at the scan rate of 4° min⁻¹ and the step size of 0.02°. The pore morphology was characterized by scanning electron microscopy (SEM, FEI Nova Nano 230). The open porosity was measured by the Archimedes’ method. Besides, the differential thermal analysis (DTA, NETZSCH STA 409C, Germany) was conducted to analyze the thermal responses of the reactive synthesis sintering reactions, which was used to determine the nature of the reactions (endothermic or exothermic) of the various reactions at the heating rates of 15 °C min⁻¹.

3. Results and discussion

3.1. The influence of Cr content on porosity of porous Ni-Cr-Mo-Cu alloys

Figure 1 shows the open porosity with different Cr contents in the porous Ni-Cr-Mo-Cu alloys under the final temperature. It can be seen that the open porosity increased with the increase of the content of Cr, besides, the open porosity reached 34.3% at 30% Cr, and 34.4% at 35% Cr respectively. In order to study the influence of the diffusion and migration rate of each atom on the pore structure, we carried out theoretical calculations on the impurity diffusion.

The temperature dependence of the diffusivity can be expressed by the diffusion coefficient D, and the diffusion rates of different elements at the final sintering temperature can be calculated [17]. The dominant elements that lead to the formation of pores can be summarized as follows:

\[ D = D_0 \exp (-Q/RT) \]  \hspace{1cm} (1)

Where, \( R \) represents the gas constant with the value of 8.314 J/(mol · K), \( Q \) refers to the activation energy (J mol⁻¹), and \( T \) is the thermodynamic temperature. The data of diffusion rates in different metal elements are listed in table 1. It can be seen that the diffusion rate of Cr atoms in Cu atoms is the largest, reaching \( 8.22 \times 10^{-13} \text{m}^2\text{s}^{-1} \) at 1150 °C [18]. This is due to the fact that Cu changes from solid to liquid, and the diffusion rate of solid-liquid is faster than that of solid-solid. The diffusion rate of Cr atoms in Cu atoms is followed by the diffusion of Cr atoms in Ni atoms at the diffusion rate of \( 1.61 \times 10^{-14} \text{m}^2\text{s}^{-1} \). Cr atoms play an important role in the formation of pores, which will migrate around Ni and Cu atoms to increase their volume,
resulting in macroscopic volume expansion of the porous alloy. At the same time, pores will be formed in situ of Cr atoms due to the diffusion and migration of Cr. When the content of Cr reaches 30%, the porosity is saturated, and the continuous increase of the content of Cr has little effect on the porosity. A similar situation also occurs in Ti-Al \[10, 19, 20\] with the result in consistent with the change trend of open porosity.

3.2. DSC Curves in Ni-Cr-Mo-Cu reactive synthesis

In the system with 95% \((\text{55}\% \text{Ni} + 30\% \text{Cr} + 10\% \text{Mo} + 5\% \text{Cu}) + 5\% \text{stearic acid}\), as can be seen from the result of DSC curve (figure 2), there are seven endothermic peaks within the range from 30 °C \ (~ 1150 °C). The first endothermic peak appeared at 64 °C when the reaction of melting of stearic acid occurred. While the second one appeared at 239 °C which is near the boiling point of stearic acid, and also the volatilization temperature of stearic acid. The endothermic peaks appeared at 419 °C and 539 °C stand for the reaction of mass solid solution of Mo atoms and the reactions of \(2.88\text{Ni} + 1.12\text{Cr} \rightarrow \text{Cr}_{1.12}\text{Ni}_{2.88}\) respectively, which is in accordance with our previous work \[21\]. The endothermic peak appeared at the temperature of 817 °C represents the reaction of Ni and Cu, that is, the reaction of \(\text{Ni} + \text{Cu} \rightarrow \text{NiCu}\), which is coincident with the work of Yu \[16\]. The last two exothermic peaks appeared at 972 °C and 1083 °C correspond to the reactions of solid solution of unreacted Cr atoms \[13, 22\] and the melting of unreacted Cu atoms, respectively. Therefore, the continuous heating method (continual temperature increase without holding time in the entire heating procedure) with high heating rate may promote the solid-state diffusion and the reaction among Ni, Cr, Mo and Cu to form thicker diffusion barriers. Consequently, the original shape of sintered compact discs of porous Ni-Cr-Mo-Cu alloys may be preserved.

3.3. Pore structure parameters of porous Ni-Cr-Mo-Cu alloys in sintering procedure

Porous Ni\(30\%\text{Cr}10\%\text{Mo}\5\%\text{Cu}\) alloy was selected to be analyzed at different temperatures in this and the following chapters due to the important role of Cr element in pore formation. Figure 3 shows the expansions (axial, lateral and volume) as a function of the final sintering temperature with the stepped heating method. As can be seen, between 550 °C and 1150 °C, all expansions increase with the increase of the sintering temperature.

![Figure 1. Effect of Cr content on the open porosity of porous Ni-Cr-Mo-Cu samples.](image)

| Diffusion element | Base element | \(D_0\left(10^{-4}\text{m}^2\cdot\text{s}^{-1}\right)\) | \(Q/(\text{kJ}\cdot\text{mol}^{-1})\) | \(T/\text{K}\) | Diffusion rate/(\(\text{m}^2\cdot\text{s}^{-1}\)) |
|-------------------|--------------|-----------------|-----------------|------|-----------------|
| Cr                | Ni           | 8.52            | 292.1           | 1423 | \(1.61 \times 10^{-14}\) |
| Cr                | Mo           | 1.88            | 339             | 1423 | \(6.76 \times 10^{-17}\) |
| Cr                | Cu           | 0.337           | 195             | 1338 | \(8.22 \times 10^{-13}\) |
| Mo                | Ni           | 1.3             | 283             | 1423 | \(5.31 \times 10^{-15}\) |
| Mo                | Cr           | 0.00027         | 242.8           | 1423 | \(3.30 \times 10^{-17}\) |
| Cu                | Ni           | 0.61            | 255             | 1423 | \(5.21 \times 10^{-15}\) |
| Ni                | Cu           | 2.7             | 236.6           | 1349 | \(1.86 \times 10^{-13}\) |

Table 1. Diffusion rate of metal elements in other elements in porous NiCrMoCu alloys.
firstly and then decrease. And the volume expansion, axial expansion and radial expansion reach their ultimate values of 6.8%, 2.9% and 1.35%, respectively. Figure 3 (b) displays the variations of the open porosity as a function of the sintering temperature with the stepped heating method. As can be seen, the open porosity increases firstly and then decreases with the increase of the sintering temperature within the measurement range. The largest open porosity reaches 42.8% at the sintering temperature of 850 °C. Careful analysis of volume expansion and open porosity shows similar growth trends with the sintering temperature. Since the sintering temperature is lower than 550 °C, the formation of pores is attributed to the interstitial pores of green compact, and the pores are formed by the volatilization of pore-forming agent, and the skeleton is formed. Within the sintering temperature range of 550 °C ∼ 850 °C, the axial, radial, volume expansions and the open porosity maintain an obvious growth. This is attributed to the fact that the diffusion rate of atoms increases with the rising of sintering temperature, which caused Kirkendall effect to become intense, thus making great contributions to the subsequent growth of volume expansion [23]. In the temperature range of 850 °C ∼ 1000 °C, the volume of the sample expands slightly, while the open porosity decreases in a small range. The pores are continuously created by the Kirkendall effect, and the volume and open porosity are further increased. However, at 1000 °C, that is, near the melting point of copper atoms, the volume of the sample shrinks in the high temperature section, closure pores increase. Therefore, the sample volume remains unchanged, however, the porosity decreases slightly under the co-effect of the two factors. When the sintering temperature reaches 1150 °C, the volume and the open porosity of the sample decrease. At this temperature, the metallic solution is formed by the melting of Cu atoms in pore canal, in the meantime, the volume of the sample shrinks and the open porosity is further

![Figure 2](image1.png)

**Figure 2.** DSC profiles of specimen with the nominal composition of Ni-Cr-Mo-Cu produced by reactive sintering.

![Figure 3](image2.png)

**Figure 3.** (a) Axial, lateral, and volume expansion ratios of porous Ni-Cr-Mo-Cu alloys sintered at different temperatures; (b) The open porosity as a function of the sintering temperature.
decreased due to the surface tension. In addition, in this paper, the particle size of Ni powders is very small, which is rich in surface energy. During the sintering process, the reduction of the surface energy become one of the main driving forces for sintering, leading to partial densification of the material [19, 24]. Therefore, the expansion rate of the porous Ni-Cr-Mo-Cu alloys decreased at the final temperature.

As a porous medium, viscous permeability plays an important role in filter performance. According to the standard of DIN EN ISO 4022:2006 [25], gases are more suitable than liquids for the determination of the viscous permeability coefficient \( \psi_v \). For a laminar flow regime, the incompressible fluid flow in a porous medium is governed by Darcy’s law, in which the permeability can be evaluated using the following formula:

\[
Q = \frac{\varphi v A}{\eta e} \Delta p
\]

where, \( Q \) refers to the volume flow rate, \( A \) represents the cross-sectional area \( (2.83 \times 10^{-4} \text{ m}^2) \), \( e \) is the thickness of the sample, \( \eta \) is the dynamic viscosity of the fluid \( (N_{22}, \eta = 1.766 \times 10^{-5} \text{ Pa·s at room temperature}) \), and \( \Delta p \) refers to the pressure drop from the entrance to the exit of the sample. As shown in figure 4(a), the curves based on the pressure drop versus the volume flow rate were experimentally determined. The plots of \( Q \) as a function of \( \Delta p \) at different temperatures, are linear, and can be demonstrated with a straight line. This linearity indicates that the flow was in the laminar flow regime, and Darcy’s law was applicable to the samples. The viscous permeability coefficients were calculated from the slope of the line between \( \Delta p \) and \( Q \), with the results listed in figure 4(b). It can be seen that the gas viscous permeability of porous Ni-Cr-Mo-Cu alloys increased with the increase of the temperature. Within the temperature range from 550 °C to 1000 °C, the viscous permeability coefficients changed from 3.89 µm² to 17.2 µm², and then dropped down to 11.1 µm² at the sintering temperature of 1150 °C, which is sufficient for the applications of solid-to-gas separation. Within the range of 550 °C ~ 1000 °C, with the increase of the temperature, the diffusion rate of metal elements accelerated, and the pores were connected gradually, resulting in an increase in air permeability. Subsequently, the porous alloy was partially densified, and the air permeability was decreased.

Figure 5 shows the SEM images at various sintering temperatures. It can be seen that at 550 °C, the pores are mainly composed of the piled pores of the original powder. While when the temperature rises to 700 °C, partial fusion is formed between the metal particles and other particles. When the temperature further rises to 850 °C ~ 1000 °C, the porous framework was basically formed, besides, the porosity and the pore size increased obviously. Metallurgical bond is formed through diffusion reaction between particles. When the sintered body reaches the final temperature of 1150 °C, all the powder particles reacted completely, and the surface of the solid part is mostly smooth without cracks, pore aperture shrinks. Besides, the number of pores decreases.

3.4. Synthesis process of porous Ni-Cr-Mo-Cu alloys

In this study, the sintering processes were carried out between 550 °C and 1150 °C, therefore, the resultant phases for the sintering at different temperatures can be clearly understood. The phase identification was performed by XRD and binary alloy phase diagrams of Ni-Cu and Ni-Cr. Figure 6 shows the results of porous Ni-Cr-Mo-Cu alloys sintered at 550 °C, 700 °C, 850 °C, 1000 °C and 1150 °C, respectively. As can be seen, for the porous alloys sintered at 550 °C, only pure Ni, Cr, Mo and Cu phases were detected, indicating that there was no reaction or detectable reaction between mixed elements powers. When the sample was heated to 700 °C, two phases of Ni (Cr, Cu) and Cr_{1.12}Ni_{0.88} were observed [26]. The diffraction peak of Mo phase was broadened, and
the results illustrated that the unreacted Ni reacted with Cr to synthesize Cr\textsubscript{1.12}Ni\textsubscript{2.88} \[24 \]; besides, a part of amorphous alloy of the metal of Mo was formed \[27, 28 \]. As the temperature further increased to 850 °C, the samples consist of Mo, Ni (Cr, Cu), Cr\textsubscript{1.12}Ni\textsubscript{2.88} and NiCu. The diffraction peak of Mo phase was further broadened. When increasing the sintering temperature to 1000 °C, the Mo phase disappeared, which indicates that, near the temperature of around 1000 °C, Mo has transformed into an amorphous alloy completely, and Ni reacts with Cr to generate Cr\textsubscript{1.12}Ni\textsubscript{2.88} continuously. When the temperature reached 1150 °C, the main phases are the same as it is at 1000 °C.

It can be seen from the phase analysis that three major phase transformation and pore forming processes exist. In the meantime, from the analysis of the previous XRD phase, it is known that before it reached 550 °C, the compound reaction of element powders had not yet occurred. At this time, the pores are mainly composed of the pores remaining in the green compact, and those generated when the forming agent is removed. Compared with the phase transformation from Ni phase and Cr phase to Cr\textsubscript{1.12}Ni\textsubscript{2.88} in most cases, the first stage is the transformation of Mo phase to the amorphous alloy. According to the theory reported by He et al \[29 \], different metal elements reacted to produce new compounds, and a large number of pores formed at the original

**Figure 5.** Surface morphologies of porous Ni-Cr-Mo-Cu alloys at different sintering temperatures: (a) 550 °C; (b) 700 °C; (c) 850 °C; (d) 1000 °C; (e) 1150 °C.
positions. Therefore, it is reasonable to believe that the porosity increment is more notable at the elevated temperature of 700 °C. As the temperature continued to rise, the remaining Ni and Cr continue to react to form Cr$_{1.12}$Ni$_{2.88}$, and Mo phase continued to transform to amorphous state until 1000 °C [26]. Therefore, the above factors resulted in a slight increase in open porosity. At the elevated temperature of 850 °C, the stage is characterized by the reaction between Ni and Cu to form NiCu, and the reaction would also produce pores. When the temperature reached 1000 °C and 1150 °C, no new phases are produced.

### 3.5. Pore formation mechanisms of porous Ni-Cr-Mo-Cu alloys

The pore evolution of the porous Ni-Cr-Mo-Cu alloy was studied by SEM in the back scattered electron (BSE) imaging mode. Figure 7 shows the comparisons of compositions and pore morphologies of compact discs sintered at different temperatures. As illustrated in figure 7(a), the dark gray, slight gray substrate and the silver smooth spheroid are Cr, Ni (Cu) and Mo, respectively. It can also be seen from figure 7(b) that the significant change in the microstructure of the sample lies in the appearance of bright gray smooth layers with a thickness of ∼10 μm which are corresponding to Cr$_{1.12}$Ni$_{2.88}$. And the analysis is proved by the EDS detected results. It is of interest to note that these layers, in fact, can act as the inert powders for preventing the SHS in our quaternary alloy system [9]. As the sintering temperature increases to 850 °C, the bright gray regions become obvious, which are adjacent to the Ni atoms, it indicates that the chromium reacts with nickel to form Cr$_{1.12}$Ni$_{2.88}$ compounds. So, it is critical that the compact discs must experience the stepped heating process to obtain sufficiently thick layers during the sintering. In addition, we can see some large pores appeared in figure 7(c). Since there are almost no such obvious large pores below 700 °C, these pores should be caused by the Kirkendall effect for the different intrinsic diffusion coefficient [30]. When the sintering temperature increases to 1000 °C, a further growth of bright gray regions is observed, which is corresponding to the Cr$_{1.12}$Ni$_{2.88}$ compounds, the light white region is Mo, and the black ones are corresponding to the pores. At the same time, the number and size of pores

![Figure 6. XRD patterns of porous Ni-Cr-Mo-Cu alloys with different final sintering temperatures.](image-url)
also change, the number of pores increases and the size decreases. These pores must be caused by the sintering. At this temperature, the intrinsic diffusion coefficient of different elements become large and Kirkendal effect become intensified. So, the number of pores distributed in figure 7(c) increases. However, because of the shrinkage behavior of porous materials at high temperature [31], the size of pores reduces. When the sintering temperature reaches 1150 °C, the light gray regions increase further and become uniformly dispersed, and the morphology is poriferous with leak tight structure. In addition, the dark gray regions pass off on the whole, which means that most of Mo has been incorporated into the Ni matrix. In the final temperature, the shrinkage behavior is further intensified. The open porosity descents. Furthermore, the capillary force formed after sintering higher than 1080 °C may be caused by the liquid Cu since the temperature exceeds the melting point of Cu, which causes the open porosity of the material decreased further. From the discussion given above, the BSE results are consistent with the variation of pore structure parameters.

Figure 7. SEM–BSE images and EDS analysis of porous Ni-Cr-Mo-Cu alloys with different final sintering temperatures: (a) sintered at 550 °C, (b) 700 °C, (c) 850 °C, (d) 1000 °C, (e) 1150 °C, respectively.
4. Conclusion

The systematic study of the microstructures (phase identification and pore structural parameters) and the permeability of synthesized porous Ni-Cr-Mo-Cu alloys leads to the following conclusions. (1) Porous Ni-Cr-Mo-Cu alloys have been successfully fabricated through elemental powder reactive synthesis with the stepped heating method. (2) The pore evolution in the porous Ni-Cr-Mo-Cu alloys is attributed to three stages: in the low temperature section, the formation of the pores is caused by the volatilization of interstitial pores and interstitial holes existing in green compact; in the middle temperature section, the increase of open porosity is due to the formation of Cr1.12Ni2.88 and NiCu intermetallic compounds, and the Kirkendall effect based on solid partial diffusion; in the final temperature, the decrease of open porosity is related to the melting of unreacted Cu atoms and the shrinkage behavior at high temperature.

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Data availability statement

The data that support the findings of this study are openly available at the following URL/DOI: https://doi.org/10.1088/2053-1591/abb562.

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