Double-pore structure porous Mo–Si–B intermetallics fabricated by elemental powder metallurgy using NH₄HCO₃ as pore-forming agent

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Abstract

The multiphase porous intermetallic compounds Mo₃Si-Mo₅Si₃-Mo₅SiB₂ of a double-pore structure has been successfully fabricated by combining the in situ reaction synthesis with the pore-forming agent method. The effects of NH₄HCO₃ content and size on porosity, pore diameter distribution, permeability, and compressive strength were investigated systematically. The results show that: with the NH₄HCO₃ increasing from 0 to 60 vol%, the total porosity increases from 46.6% to 73.2%, the big pores volume increases from 2.3% to 69.4%, the gas permeability increases from 5.34 × 10⁻⁷ 1/(min · cm² · Pa) to 1.74 × 10⁻³ 1/(min · cm² · Pa), and the compressive strength decreases from 392 MPa to 14.8 MPa; on the other side, with the NH₄HCO₃ size increased from 48 μm to 230 μm, the parameters of this porous intermetallics changed slightly except to the significant increase of big pores diameter. An exponential equation of $\sigma_c = \sigma_s (1-\rho)^{1/0.254}$ based on generalized mixture rule (GMR) has been put forward to quantitatively describe porosity-compressive strength behaviors. Results from this study indicate the potential applications in various filtration environments by tailoring the shape, contents, and size of the pore-forming agent.

1. Introduction

Porous intermetallic compounds have been widely used in many fields in the last two decades, such as filtration, catalyzing and sound insulation [1]. Porous Mo–Si–B intermetallic compounds are a promising gas/liquid separation material owing to their good mechanical properties, excellent corrosion resistance and outstanding oxidation resistance at high temperature [2]. Mo–Si–B alloys have been exhaustively studied as elevated-temperature structural applications since Nowotny et al investigated the first Mo–Si–B phase diagram at 1600 °C [3]. What’s more, the MoSi₂, Mo₅Si₃, Mo₅SiB₂ and Mo₅SiB₂ intermetallic compounds have the higher melting point and good property of sulfur-resistant, which is extremely important for engineering applications [4].

Nowadays, various technologies have been developed to synthesize porous intermetallic compounds, such as conventional elemental powder metallurgy [5–10], pore-forming agent additive [11–14], combustion synthesis [15–22], impregnation method [23], and freeze-casting technology [24]. Elemental powder metallurgy was the most common way to synthesize micro-size porous Ti–Al [5, 6, 25], Fe–Al [7], and Ni–Al [8] system intermetallic compounds by utilizing the Kirkendall effect, which is particularly suitable for obtaining open-cellular pore structure. The unidirectional porous TiAl-Mn intermetallic compounds with the fully lamellar structure were been successfully produced by extrusion of elemental powder mixtures and reactive sintering of the extruded powder mixtures [9]. Nickel aluminide foam and porous titanium composite were fabricated by chemical reactions [26]. Super-light Fe–Al, Ti–Al, and Ni–Al porous materials with high porosity even more then 80% were successfully produced applying a novel processing route basing on using carbamide as pore-
forming agent [11, 12, 14]. This technology usually applies to the fabrication of ceramic foam with macro-pore. The high porosity porous Ti–Al, Fe–Al, Al–Cr and Ni–Al intermetallics were also prepared by a rapid process of and thermal explosion reaction [15–18, 20]. Highly porous Ni–Cr–Al alloy was produced by the polymeric foam replication method combined with a reaction sintering process [23]. Obviously, the porosity, pore size and other pore structure parameters of porous materials are limited to the preparation methods. Thus, it is significant to choose the method which structure parameters can be controlled broadly while relatively good mechanical properties can be obtained. The space holder method is an easy way to produce highly porous intermetallic compounds by using various pore-forming agents. Adjusting the pore structure parameters accord to the industrial application, that can be achieved by combining the pore-forming agent additive method with elemental powder metallurgy.

In this paper, the double-pore structures of highly porosity porous Mo–Si–B (MSB for short in this paper) intermetallics was fabricated by elemental powder metallurgy method used NH₄HCO₃ as pore-forming agents. The phase composition, porosity, pore size distribution, microstructure and mechanical properties of the porous MSB intermetallic compounds were investigated.

2. Experimental

The raw materials using in this study are as follows: the elemental Mo, Si and B powders with the purities of 99.9%, and mean particle sizes 2.5, 5.8 and 12 μm respectively; The commercial NH₄HCO₃ particle with irregular shape was selected as pore-forming agent, in order to adjust the pore parameters in sintered samples, the NH₄HCO₃ particle were sieved with different d₅₀ ranging from 48 to 230 μm. The pore-forming agent NH₄HCO₃ was decomposed into NH₃, CO₂ and H₂O with low-temperature heating, and these decomposition products were quickly expelled from the sintering furnace with the flowing argon gas.

The nominal composition of Mo–28Si–8.5B (at%) were employed in this paper. The elemental powders were mixed together in absolute alcohol by using a planetary ball mill with zirconia milling balls. The slurry was milled for 3 h at a rotation speed of 300 rpm, followed by vacuum drying at 60 °C for 8 h. Before mixing the NH₄HCO₃ with Mo–Si–B powders, the pore-forming agent should be sifted to effectively avoid agglomeration. And then, the pore-forming agent was added by mixing the MSB powders and NH₄HCO₃ in different content ratio. The green bodies were prepared into a disc of φ13 mm × 10 mm by a uniaxially cold process under a die pressure of 300 MPa. The cold-pressed compacts were firstly heated to 350 °C in a slow heating ramp of 2 °C min⁻¹ to prevent the NH₄HCO₃ decomposition rapidly result in forming the macro cracks, and then held at this temperature for 1 h to remove all decomposition products of pore-forming agent, then heated to the temperature of 1600 °C at a rapid heating rate of 10 °C min⁻¹ and held at this temperature for 2 h. The sintering process was conducted in a tube furnace with argon gas at the flow rate of 25 ml min⁻¹.

The phases present in the samples were determined by x-ray diffraction (XRD, DMAX–RB, Rigaku) with CuKα, radiation (at a scanning rate of 10 °/min). The total porosity and open porosity were measured by the vacuum impregnation method based on the Archimedes principle. The pore size distributions were measured by mercury porosimetry (AutoPore IV 9500, Micromeritics). The pore morphology was characterized by scanning electron microscopy (SEM, SUPRA-55, Zeiss, operated at 20 kV) equipped with an energy dispersive x-ray spectroscopy (EDXS) system with the elemental analysis. The permeability of porous samples was tested by using Darcy's equation (1):

\[ ΔP = \frac{Q}{L} \frac{\eta}{AK} \]  

where \( ΔP \) is the pressure drop from entrance to exit of the sample, \( Q \) is the flow rate, \( \eta \) is the dynamic viscosity of the fluid, \( L \) is the thickness of the sample, \( A \) is the cross-sectional area of the sample, and \( K \) is Darcy’s permeability. The tested samples were cut into φ13 × 3 mm, and the \( n \) value is 1.75 × 10⁻³ Pa s in this study. The compression testing was tested by the universal metal material testing machine (CMT–4305, MTS, operated at a compress speed of 0.05 mm min⁻¹) with the cylindrical samples of φ3 × 6 mm. The value of compressive stress was calculated according to the maximum stress during the compression process, five parallel experiments were performed on each kind of sample.

3. Results and discussion

3.1. Phase composition analysis

Figure 1 shows the XRD pattern of the porous Mo–Si–B specimen with 50 vol% NH₄HCO₃ pore-forming agent, which was sintered at 1600 °C. The XRD pattern shows that Mo₅Si, Mo₅Si₃, and Mo₅SiB₂ are the main phases, which conform to the Mo–Si–B system phase diagram under the nominal composition in this paper. And few
weak peaks from SiO$_2$ are both identified in XRD spectra with/without NH$_4$HCO$_3$. The formation of SiO$_2$ phase is unavoidable owing to the milling process in addition to sintering may introduce oxide [27]. Comparing to the Mo–Si–B alloys, the oxidation resistance of the pseudo in situ Mo–Si–B–O alloy that was improved in the temperature range between 727 °C and 1127 °C [28]. The pre-formed SiO$_2$ is beneficial to the oxidation resistance by reducing the incubation stage of formation of continuously protective borosilicate layer. And the NH$_4$HCO$_3$ began broken down into ammonia gas, carbon dioxide, and water at 36 °C, and this process was done completely at 60 °C. Therefore, the pore-forming agent was not detected in the XRD pattern. The XRD pattern with NH$_4$HCO$_3$ after complete decomposition (held on at 350 °C for one hour) and the XRD pattern of the raw materials without NH$_4$HCO$_3$ also presented in the figure 1. By contrast: firstly, SiO$_2$ is not present in XRD pattern of mixed elemental powders; at a temperature higher than 100 °C, the water vapor decomposed by NH$_4$HCO$_3$ was expelled out tubular furnace with argon gas. Which indicating that the formation of SiO$_2$ is irrelevant to the decomposition of NH$_4$HCO$_3$. Many literatures of dense Mo–Si–B structural materials indicate that SiO$_2$ is introduced from the small amount of absorbed oxygen, SiO$_2$ and MoO$_3$ which are formed in the surface of Si and Mo powder particles, respectively. As a result, the SiO$_2$ accumulates and grows at the grain boundary, eventually forming a substantial amount that is sufficient for XRD detection.

3.2. Microstructural characteristics

Figure 2 shows the optical and SEM microscope of the section profile of the samples, which were sintered at 1600 °C with 0 vol%, 50 vol% NH$_4$HCO$_3$ (the average size is 86 μm). In this paper, the pores with a diameter of 0.5–10 μm are defined as small pore, and the pores with a diameter of 10–300 μm are defined as big pore. The smaller pores with the pore size concentrate on 1–10 μm as shown in figures 2(a) and (b), which originates from the space between element powder particles and Kirkendall effect as having been exhaustive studied in the Mo–Si interdiffusion system [29]. The total porosity and open porosity occupied 46.6% and 33.8%, respectively. When the content of NH$_4$HCO$_3$ is 50 vol%, a highly porosity porous MSB microtopography of the double-pore structure as shown in figures 2(c) and (d), the big pores which apparently originates from the removed pore-forming agent particles. The small pores act as the interconnected channels between the big pores can be observed as marked in figure 2(c), which guarantees an open-cellular structure between the big pores. Another part of big pores is connecting each other directly, and merging into larger pores, as it can be seen in figure 6(a), which is helps improve the permeability of the porous MSB intermetallics significantly.

The cross-section microstructure of porous MSB intermetallics with 50 vol% NH$_4$HCO$_3$ of different particle diameter as shown in figure 3, a typical double-pore structure is obtained. Apparently, the outline of the big pores perfectly replicates the irregular morphology of the pore-forming agent. As the particle size increase from 48 to 230 μm, the diameter of big pores increased, and more and more big pores are connected.

3.3. Porosity and pore structure parameters

Figure 4(a) shows the porosity of the porous MSB intermetallics with different contents of pore-forming agent NH$_4$HCO$_3$ ($d_{50} = 48$ μm). It is obvious that both the total porosity and open porosity increase monotonically as...
Figure 2. Optical (a), (c) and SEM (b), (d) microscope of the section profile of the porous samples without (a), (b) and with (c), (d) NH$_4$HCO$_3$.

Figure 3. The microstructure porous MSB intermetallics with 50 vol% NH$_4$HCO$_3$ of different particle diameter: (a) 48 μm; (b) 86 μm; (c) 160 μm; (d) 230 μm.
the increasing of the pore-forming agent contents. Comparing the sample without pore-forming agent, the porosity of the sample with 60 vol% NH$_4$HCO$_3$ roughly doubled. The effects of pore-forming agent particle size on the pore size distribution of porous MSB intermetallics with 50 vol% NH$_4$HCO$_3$ as shown in figures 4(b) and (c). The feature of double-pore structure can be seen in figure 4(b), the histogram and its imaginary line trend show double peaks structure compare to the sample without pore-forming agent. The peaks of small pores concentrate on the same size of 1–5 μm, while the peak position of big pores shifts to right with the increase of pore-forming agent volume. The higher content of pore-forming agent, the greater the probability of the big pores merging into larger pores, which result in this phenomenon. As it shows in figure 4(c), with the increase of pore-forming agent from 0 to 60 vol%, these small pores occupy 97.7%, 54.5%, 40%, and 30.6% in pore volume; these big pores occupy 2.3%, 45.5%, 60%, and 69.4% in pore volume, respectively. With the pore-forming agent contents increase, the cumulative pore volume increased and the specific surface area decreased, as shown in figure 4(d). This indicates that the porosity is sensitive to the volume content of big pores, while the specific surface area is sensitive to the volume content of small pores.

Figure 5 shows the effects of pore-forming agent size on porosity and pore structure feature of porous MSB intermetallics with 50 vol% NH$_4$HCO$_3$. As can be seen, with the increase of pore-forming agent particle size in the range of 48–230 μm, the total porosity and open porosity change slightly. Therefore, it can be considered that the particle size of NH$_4$HCO$_3$ has no significant influence on the porosity of porous MSB intermetallics. Obviously, a double-pore structure was obtained, and the bimodal imaginary lines of pore diameter distribution are also shown in figure 5(b). With the particle size of the pore-forming agent increases from 48 to 160 μm, the peak position of big pores ranges from 10–50 to 150–200 μm correspondingly. The pore structural characteristics of the big pores are entirely dependent on the size and morphology of the pore-forming agent particles. As it shows in figure 5(c), with the increase of the pore-forming agent from 48 to 160 μm, these small pores occupy 39.7%, 40.4%, and 39.3% in pore volume; these big pores occupy 60.3%, 59.6%, and 60.7% in pore volume, respectively. This further indicates that the volume of big pores is independent of particle size, but depends on the contents of the pore-forming agent. It is helpful to regulate the dual-pore structure by adjusting the parameters of the pore-forming agent. The effects of particles size on cumulative pore volume and specific surface area are shown in figure 5(d), the cumulative pore volume increases slightly, corresponding to the characteristics of porosity which as shown in figure 5(a). While the specific surface area decreases slightly with
the increasing of pore size. In general, the larger the pore diameter, the smaller the specific surface area of the porous material.

3.4. Permeability
Figure 6 shows the effects of the pore-forming agent on the penetrability of porous MSB intermetallics. As can be seen in figure 6(a), the penetrability and penetrability coefficient both increased with the increase of NH$_4$HCO$_3$ contents. As for single-pore structure, the gas permeability increases almost linearly when the porosity increased [30]. However, it should be noted that while the pore-forming agent content is less than 40 vol%, the permeability growth is extremely slow; once the pore-forming agent content exceed 40 vol%, the permeability induces exponential growth. That is because when the content of the pore-forming agent is greater than a certain value, the big pores in the double-pore structure begin to connect with each other, which is shown in figure 2(c). The interconnected big pore greatly improves the possibility, and it will no longer be limited to the small pore structure. Figure 6(b) shows the permeability of the porous MSB intermetallics with different particle sizes of 50
vol% pore-forming agent, the permeability and permeability coefficient increased slightly with the increase of NH$_4$HCO$_3$ size, and the values remain at the same exponential level. As we discussed on figure 3, the larger the size of the pore-forming agent, the more interconnected big pores, which provides a larger aperture for the gas permeation process. In conclusion, the permeability of double-pore porous MSB intermetallics is a complex problem, which is determined by porosity, pore diameter, pore diameter distribution and other factors.

### 3.5. Compressive strength

In order to future evaluate the effect of the pore-forming agent on the porous MSB, the relationship between the mechanical property and the content of NH$_4$HCO$_3$ was investigated using compressive strength as a criterion. Table 1 shows the dependence that the pore-forming agent exhibits a predominant influence on the compressive strength, more pore-forming agent and less mechanical strength. Especially at 40 vol% relative to 0 vol%, the compressive strength was steeply decreased from 329 to 87.3 MPa. Once the volume content of NH$_4$HCO$_3$ reaches to 60%, its compressive strength drops sharply to 14.8 MPa. Compared with single-pore structural porous materials, the strength of double-pore structure porous materials is usually lower due to the existence of stress concentration at the pore walls and struts between big pores. The ultra-light double-pore structures of TiAl is ranges from 5.07 MPa to 19.2 MPa [11]. Figure 7 shows the feature that the pore-forming agent exhibits a positive effect on the strain-stress behaviors: the true strain increased slightly with increasing of NH$_4$HCO$_3$ volume fraction, despite the brittle fracture as present in true stress-strain curves. These results indicate that the double-pore structure is beneficial to elevate the strain capacity, and markedly reduce the mechanical strength of porous MSB.

Regarding porous materials, the mechanical properties depend on not only the porosity but also the pore-geometry, including the shape, size, and distribution (continuity and connectivity) of the pore [31–36]. A great deal of research work attempts to reveal the effect of pore on mechanical properties of porous materials. O’Kelly et al established three main approaches of composite theory, cellular solids and minimum solid area (MSA) to determinate relationship between mechanical and porous structure [37]. Among them, the MSA can be easily calculated for idealized structures, that is, regular stacking of uniform (spherical, spheroidal, cylindrical or cubic) pores in a continuous solid medium. A simple equation which has been widely used in evaluating mechanical properties of porous materials can be considered as developing MSA model, which can be expressed...
by the following equation

\[ \frac{M}{M_0} = \exp(-bp) \]  

(2)

where \( M \), measured mechanical porosities (such as hardness, Young’s modulus, and strength, et al) of porous materials; \( M_0 \), mechanical porosities of a density specimen with zero porosity; \( p \), the volume fraction of the porosity; \( b \), a constant that a coupling result of all the pore structure features include the size, geometrical shapes. The natural logarithm value of \( \ln(M/M_0) \) decreases linearly with the porosity, and \( b \) equal to the slope. The application of this equation is premised on \( p \leq 0.4p_c \), where \( p_c \) is the critical porosity that corresponds to the percolation limit of the solid phase [36], and this equation fails when \( p \) is equal to \( p_c \). What’s more, equation (2) displays an evident disadvantage: the boundary condition that \( M \) should be equal to zero when \( p \) is equal to 1 is not satisfied [38]. Theoretically, equation (2) can be used for only low values of porosity \( (p \leq 0.3) \).

A similar empirical equation relation to describe the dependence of strength on porosity has been proposed by Dutta et al [31].

\[ \sigma = \sigma_0(1 - p)^m \]  

(3)

where \( \sigma \), measured strength of porous materials; \( \sigma_0 \), the strength of a density specimen with zero porosity; \( p \), the volume fraction of the porosity; \( m \), a constant which is same as \( b \) in equation (2). Unlike conditional equations (2), (3) satisfied the boundary condition \( \sigma = 1 \) at \( p = 1 \). However, equation (3) assumes the crack length independent of porosity, and the specific fracture energy, \( \gamma_i \), is proportional to the elasticity modulus. This equation shows better accuracy in predicting the strength of porous ceramics [39], especially in porous Si3N4 [31]. But there are still somewhat limitations when equation (3) extended to other porous materials.

Ji et al [36] proposed a simple but elegant mixture rule for providing a unified description of the physical properties of polyphase composites in terms of component properties, volume fractions, and microstructures, which was named as generalized mixture rule (GMR). Actually, some of the important rules of properties can be unified by the GMR that expressed as:

\[ M_i^f = \sum_{i=1}^{N} (V_i M_i^f) \]  

(4)

where \( M \) is a specific property, \( V \) is the volume fraction of component, the subscripts \( i \) and \( f \) represent the \( i \)th phase, and the composite consisting of \( N \) phases, respectively. \( J \) is a constant which is mainly controlled by the shape, size distribution, and distribution (continuity and connectivity) of the phases. For porous materials, which can be considered as a special class binary system consisting of a strong phase of the solid framework and a weak phase of air. Equation (4) can be expanded as:

\[ M_i^f = (1 - V_w) M_s^f + V_w M_w^f \]  

(5)

Where subscripts \( s \), \( w \) represents the strong phase, weak phase, respectively. For the pores phase, its mechanical properties equal to zero, that is to say, \( M_s \) equals to zero. In order to reflect the effect of porosity on mechanical properties, taking \( V_w \) as the volume fraction porosity (\( p \)), equation (5) can be sampled as:

\[ \frac{M}{M_i} = (1 - p)^{1/J} \]  

(6)

\[ M_i^f = (1 - p)^{1/J} \]  

(5)

\[ M_i^f = (1 - p)^{1/J} \]  

(6)

In order to quantitatively analyze the influence of pore structure on mechanical property of porous MSB, the porosity versus \( \sigma/\sigma_i \) curves were fitted out based on the GMR, as shown in figure 8. Therefore, the pore shape factor parameter \( J \) of double-pore structure porous MSB be determined by equation (6), which was calculated to be 0.254 as shown in figure 8(a). An empirical relational expression (compressive stress versus porosity) of porous MSB with the pore-forming agent of average size 86 \( \mu \)m is obtained as follow:

\[ \sigma_c = \sigma_i(1 - p)^{1/0.254} \]  

(7)

\[ \sigma_c = \sigma_i(1 - p)^{1/0.254} \]  

(7)

Ji et al [36] presented a higher \( J \) value of 0.287. The empirical relational expression can be written as

\[ \sigma_c = \sigma_i(1 - p)^{1/0.287} \]  

(8)

J is the parameter that depends on the geometrical shape, spatial arrangement, orientation and size distribution of pores, and in turn on the materials and the fabrication method and the value of \( J \) should lie in the range from \(-1 \) to \( 1 \) (except to \( 0 \)), giving a wide range of properties at a given relative density or porosity. What’s more, the smaller value of \( J \) means the porosity has a greater effect on the mechanical. And a lower \( J \) value will be produced by intergranular, continuous, channel pores cavities; a higher \( J \) value can be caused by intragranular, isolated and rounded pores [36]. When \( J \) equals 1, the pore structure of porous materials has no stress concentration, such as long cylindrical or hexagonal pores aligned parallel to the stress direction. Obviously, the pore size distribution
was changed by the addition of the pore-forming agent and resulting in a higher stress concentration at the skeletons between big pores. The pore shape factor $J$ of porous ceramics, such as Al$_2$O$_3$, MgO, SiC, Cr$_3$C$_2$, and Si$_3$N$_4$, ranges from 0.2 to 0.3 \cite{40}. In comparison, the double-pore structure porous MSB intermetallics has a higher $J$ value, it helps to maintain adequate strength under high porosity, and it is important for application.

4. Conclusions

The double-pore structure porous Mo–28Si–8.5B intermetallics with porosity varying from 58\% to 73\% were investigated by using NH$_4$HCO$_3$ as a pore-forming agent. the size of small pores and big pores center on 0.1–10 and 10–300 $\mu$m, respectively. The big pore microstructure parameters can be easily regulated by control of the content and particle size of the pore-forming agent. The resulting porosity-compressive strength behaviors of porous MSB intermetallics are generally well-described by an exponential equation based on GMR. And the specimens without NH$_4$HCO$_3$ exhibit a higher value of pore shape factor $J$ than those with double-pore structure. Therefore, the strength tends to become insensitive to big pore size, especially at a higher level of porosity. This paper implies a possibility to fabricate the porous Mo–Si–B intermetallics with suitable pore characteristics to meet the diversified application requirements by a concise control of the size, the shape, as well as the size distribution of starting NH$_4$HCO$_3$ particles.

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