Original article

Porous NbAl$_3$/TiAl$_3$ intermetallic composites with controllable porosity and pore morphology prepared by two-step thermal explosion

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**ABSTRACT**

Porous NbAl$_3$/TiAl$_3$ with adjustable porosity was prepared by the thermal explosion (TE) mode of combustion synthesis (CS). This was achieved by using NaCl as temporary space holder, which was completely removed from specimens before TE reaction. The combustion image of this process provided the visual evidence for the observation of TE. The temperature profile, course of reaction as well as the effect of NaCl content on the phase composition, pore morphology and open porosity of porous NbAl$_3$/TiAl$_3$ were investigated. The results showed that the addition of TiH$_2$ prevent the samples from cracking and deforming, and in situ synthesis of NbAl$_3$/TiAl$_3$ intermetallic composites. The interconnected channel was formed when using NaCl as space holder, which made the porous NbAl$_3$/TiAl$_3$ an open cellular structure. The open porosity can be improved from 48% to 79% by adding 70vol.% NaCl particles. The small pores (<20μm) in porous NbAl$_3$/TiAl$_3$ originated from the intergranular pores in green compact and the decomposition of TiH$_2$, whereas the large pores (200–300μm) were attributed to the replication of NaCl particles. This leachable space holder route provides a flexible way to adjust the pore morphology and porosity to multifarious applications.

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1. **Introduction**

As a highly promising engineering material in a wide range of industrial field, porous metals has received extensive attention due to its excellent performance such as the superior toughness, energy absorption capability during impact, and high specific surface areas. However, the poor corrosion resistance, and insufficient oxidation resistance at elevated temperatures limit their practical applications in many fields, for instance, the high-temperature filtration and catalytic support in harsh circumstances [1]. Recently, porous intermetallic, especially the aluminides of transition metal such as NbAl$_3$ and TiAl$_3$ have attracted widely attention [2,3]. The

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combination of metallic and covalent bonds in this porous intermetallics material provide sound mechanical properties with low density and excellent oxidation resistance at high temperatures, which makes them as the potential candidates for application in heat exchangers, gas/solid–solid separation, and catalysis [4–7].

Most of the research proposed to date have focus on the reactive sintering to fabricate porous intermetallics [8,9]. In general, this procedure based on the Kirkendall effect, holding samples at 550–650 °C for 2–3 h, followed by high-temperature sintering at about 1100 °C with a heating rate of 1–5 °C min⁻¹. However, the lower heating rate and long holding time are essential for the formation of Kirkendall pores, which means that a long-time and high-energy consumption process. As a promising alternative, combustion synthesis (CS) provide an pioneering method for preparing porous intermetallics with a shorter processing time, more energy efficient, and larger productivity [10]. CS characterized by two reaction mode, viz. self-propagating high-temperature synthesis (SHS) and thermal explosion (TE) [11]. In SHS mode, the reaction is initiated at one end and proceeds through the whole sample by the propagation of combustion wave. In this case, the porous intermetallic made by SHS mostly accompanied by the deformation and multiphase structure owing to the large temperature gradient [12]. In contrast, the TE mode involves a volumetric reaction that takes place simultaneously once the entire sample has been heated to the ignition temperature, and the sample maintain original shape free from cracking or deformation. Recently, the fabrication of porous intermetallics, ceramics, and alloys through TE has been reported [13–15].

Although the TE method is efficient and straightforward, it does not enable a favorable regulation of morphology, volume fraction and dimension of the pores, owing to the extremely short duration of TE. To tackle this problem, a space holder method using leachable temporary templates has been successfully developed. This process mixes the space holder with reactant, and then removes it to manufacture products with a tailored pores. The porosity of sample was controlled by the content of added space holder particles. Simultaneously, the pore shape and pore size inherits the characteristics of the space holder particles. Up to date, various types of space holders have been utilized to fabricate metal foams or porous intermetallics. Mondal et al. [16] fabricated the bio-medical titanium-foams with 89% porosity by using ammonium bicarbonate (NH₄HCO₃) and acrowax as space holder. However, the carbon element and TiO₂ was detected in porous product due to the decomposition of acrowax and organic binder. Moreover, the extra procedure needed to remove space holder during sintering process provide the challenges in the aspects of time and cost. Karczewski et al. [17] made the porous FeAl intermetallic foams with porosity up to 40% via tartaric in situ foaming technique. The sample with added tartaric shows a more regular distribution of pores and exceptional filtration performance. Nevertheless, the trace of oxygen and carbon indicates that the unreacted tartaric was surviving after the combustion of this pore former.

In comparison to the ammonium and organics, the NaCl has came into notice due to the low-cost and environmentally-friendly. Furthermore, the NaCl can be easily removed from compact, which is unlike the residue of organic can react with matrix during sintering at high temperature [18]. Currently, there is no definitive method to remove NaCl space holder, one of which is to dissolve NaCl particles by water leaching after sintering. However, the flow of molten NaCl during sintering destroys the structure of the pores, and it is more difficult to remove NaCl after sintering [19]. Another way is to volatilize the NaCl particles from compact because of the high vapor pressure of NaCl (1 kPa at 987 °C), which poses a negative effect on the equipment [20]. Therefore, the appropriate removal of NaCl is a key factor during the manufacturing process.

In view of the previous studies on preparation of porous intermetallic, the process often takes a insufficient efficiency and high-energy consumption, as well as the uncontrollability of pore structure. Therefore, the present study focus on the fabrication of porous NbAl₅/TiAl₃ intermetallic with tailored pores by a facile TE reaction. NaCl particles were used as space holder and removed by water leaching before sintering. The temperature and combustion image was recorded during the TE reaction, and the effect of NaCl content on the phase composition, pore morphology and porosity of the products was investigated.

## 2. Experimental procedure

### 2.1. Materials and process

The raw materials used in the present study are shown in Table 1. Nb, Al, and TiH₂ powders were weighed in a weight ratios of (Nb–50 at. % Al):TiH₂ = 9:1 (10 wt. % TiH₂). The powders were wet-mixed using ethanol in a planetary ball mill (QM-ISP2-CL, China) for 4 h (450 rpm) and completely dried in an oven at 50 °C for 12 h. Then the Nb–Al–TiH₂ powder mixture and NaCl particle was manually mixed in an agate crucible, to prepare green compacts with 0, 30, 50, and 70 vol.% fraction of NaCl. A few drops (6 wt.% of Nb–Al–TiH₂/NaCl) of ethanol was added into the mixture to obtain sound blend of metal powder and NaCl particle. The Nb–Al–TiH₂/NaCl mixture was then uniaxially pressed under a pressure of 200 MPa to form compacted cylindrical discs of 16 mm diameter and ~2 mm height. Subsequently, NaCl particles were thoroughly removed from green compacts in deionized water at temperature of 60 °C for 4 h before sintering, and the water was replaced every half an hour. Afterwards, the Nb–Al–TiH₂ samples were dried in an air oven for 12 h. For comparison, Nb–Al green compacts without TiH₂ and NaCl was also made in same procedure.

The schematic diagram of the experimental setup is shown in Fig. 1. Reactions of the Nb–Al and Nb–Al–TiH₂ were carried out in a tube furnace (OTF-1200X, China) under high vacuum (5.0 × 10⁻⁴ Pa), at a heating rate of 10 °C min⁻¹ to

<table>
<thead>
<tr>
<th>Powder</th>
<th>Particle size (μm)</th>
<th>Purity (%)</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nb</td>
<td>45</td>
<td>99.3</td>
<td>OTIC</td>
</tr>
<tr>
<td>Al</td>
<td>38.5–74</td>
<td>99.9</td>
<td>Aladdin</td>
</tr>
<tr>
<td>TiH₂</td>
<td>45</td>
<td>99.0</td>
<td>Aladdin</td>
</tr>
<tr>
<td>NaCl</td>
<td>200–300</td>
<td>99.8</td>
<td>Aladdin</td>
</tr>
</tbody>
</table>

Table 1 – Properties of the initial reactant powders.
900 °C for 30 min. A thermocouple (WRe3–WRe25, with a frequency of 100 Hz) of 0.1 mm diameter was placed between the two samples to monitor the temperature of the sample, which was then used to obtain the temperature vs. time profile. Moreover, the combustion image of TE was recorded with a CMOS camera (HDR-CX900E, SONY) at a speed of 100 frames s⁻¹.

2.2. **Product characterization**

The differential scanning calorimeter (DSC) was detected on a differential thermal analysis instrument (Netzsch STA449F3, Germany) to determine the nature of the TE reactions, a constant flow of argon (50 ml min⁻¹) was maintained during heating to protect the samples against oxidation, and the samples were heated at a constant rate (10 °C min⁻¹) up to 1000 °C. Phase identification was carried out by X-ray diffraction (XRD) on a Bruker D8 Advance diffractometer with a Cu target (λ = 0.15418 nm). The microstructure was characterized by optical microscopy (Olympus) and scanning electron microscopy (SEM, Quanta 250) combined with energy dispersive spectrometry (EDS, Uantax 400) for elemental analysis. The open porosity and density of the samples was determined by the immersion method based on the Archimedes principle, according to the following equations:

\[ P_{open} = \frac{M_{im} - M_{air}}{\rho_{oil} \cdot V} \times 100 \]  
\[ \rho = \frac{M_{air}}{V} \]

where \( M_{im} \) is the mass (g) of sample filled with vacuum pump oil, \( M_{air} \) is the dry mass of the sample, \( \rho_{oil} \) is the density (g cm⁻³) of the vacuum pump oil, \( V \) is the external volume of the sample, \( P_{open} \) is the volume fraction of open porosity (vol.%)

of the sample and \( \rho \) is the density of the sample. The volume change of the sample was determined by the measured dimension before and after sintering process.

### 3. Results and discussion

#### 3.1. Characteristics of thermal explosion (TE) process

Fig. 2 shows the temperature evolution profiles and the various steps of thermal explosion (TE) of the Nb–Al and Nb–Al–TiH₂ samples at a linear heating rate of 10 °C min⁻¹. The TE reaction involves two important parameters, the ignition \((T_{ig})\) and combustion \((T_c)\) temperatures [11], as shown in Fig. 2a. An interesting feature of Nb–Al system is that TE occurs there at a temperature almost 200 °C higher than the melting point of Al, which is distinct from most other TE reaction system, for instance, Fe–Al [21] and Ti–Al [22]. In the early stages of the TE process, the temperature of the sample slowly increases with the furnace temperature at a constant rate. When the curve shifts upward and the temperature reaches \(T_{ig} (876.7 \degree \text{C})\), the exothermic reaction starts instantaneously. Subsequently, the temperature abruptly increases to the combustion peak \((T_c, 1368.5 \degree \text{C})\), corresponding to the occurrence of the TE process. At the end of the TE reaction, the released heat is not sufficient to maintain the temperature of the sample, and the temperature immediately drops, along with the furnace temperature. Fig. 2b shows the combustion images of Nb–Al sample, which remained unchanged till the temperature reached 667 °C when a liquid phase formed and exuded rapidly. These liquid droplets were subsequently sucked back into the compact at the initiation of combustion and the whole compact turned red hot, and the non-uniform deformation was appeared owing to the exuded-sucked process.
Subsequently, the temperature increased rapidly to $T_c$ and the whole sample became white hot leading to further volume expansion and the crack covered the surface. However, for the Nb–Al–TiH$_2$ compact, there are two reactions during heating process as shown in Fig. 2c. The first reaction followed closely the melting point of Al, and the latter was reacted at about 818.1 °C. These two reactions can be defined in following discussion. Although the compact was expanded slightly, it should be noted that the original cylindrical shape was maintained, no surface cracks or compact deformation.

Fig. 3a shows the differential scanning calorimeter (DSC) profile of Nb–Al compacts at a heating rate of 10 °C min$^{-1}$. An endothermic peak at 664.6 °C and an exothermic peak at 909.8 °C corresponding to the melting of Al and the reaction of Nb–Al, respectively. However, for the Nb–Al–TiH$_2$ specimen, there are two endothermic peaks and two exothermic peaks (Fig. 3b). The first endothermic peak at 508.4 °C is the decomposition of TiH$_2$, and a mass loss (0.26%) in TG curve can be observed. Meanwhile, Fig. 3c shows the XRD pattern of Nb–Al–TiH$_2$ specimen at 600 °C (Point A in Fig. 3b), the Ti phase was formed, which further confirmed the decomposition reaction of TiH$_2$. The second endothermic peak at 661.3 °C due to the melting of aluminum, and followed immediately by an exothermic peak at 672.7 °C corresponding to the TE reaction of Ti–Al [22]. The XRD pattern of Nb–Al–TiH$_2$ specimen at 760 °C (Point B in Fig. 3b) shows the Nb and Al are main phase, accompanied by minor TiAl$_3$ phase owing to the reaction of Ti and Al. The NbAl$_2$ was formed when the sample heating to 900 °C (Point C in Fig. 3b), as shown in Fig. 3c. Therefore, the exothermic peak at 821.1 °C is the TE reaction of Nb and Al, which consistent with the results of temperature evolution profile.

Previous study shows that the combustion reaction of Nb–Al is triggered by the dissolution–precipitation process [23,24]. However, according to the Nb–Al phase diagram, the solubility of Nb in Al is almost near zero and is very limited even in liquid Al. And from the published data, the solubility of Nb in liquid Al is about 0.02 wt.% at 700 °C, increasing to 0.06 wt.% at 800 °C [25]. These data indicate that the higher temperature will be required for the ignition of thermal explosion in Nb–Al system. Moreover, the difficulty in wetting the Nb particle surface by liquid Al could be other factor hinder the reaction, which is confirmed by the exudation of liquid Al in Fig. 2b, when the temperature higher than 660 °C. In the present study, the ignition temperature of Nb–Al was reduced from 876.7 °C to 780 °C by adding TiH$_2$ powder, and the combustion temperature was reduced from 1368.5 °C to 818.1 °C. Thus, not only did the TiH$_2$ act as a stabilizing agent of Al, a promoter of the thermal explosion, but it also served as a diluent in combustion to prevent the synthesized product from cracking and deforming.

3.2. Product phase analysis

The XRD patterns depicted in Fig. 4 identify the formation of NaAl$_3$(JCPDS No. 13-0146)/TiAl$_3$(JCPDS No. 37-1449) intermetallic composite with different content of space holder, suggesting that the NaCl content have no significant effect.
on the phase transition during heating process. It should be noted that no traces of NaCl were detected in the sintered products, confirming the complete removal of NaCl particles by the water leaching process. Simultaneously, TiH$_2$ was completely decomposed and full involvement of Ti from TiH$_2$ in the synthesis of TiAl$_3$. Moreover, a substantial of unreacted Nb (JCPDS No. 35-0789) were identified in the products. Combine with the results of DSC test, the sequence of the formation of NbAl$_3$/TiAl$_3$ intermetallic composite can be summarized as follows:

\[ \text{TiH}_2 \rightarrow \text{Ti} + \text{H}_2 (450 - 550 \, ^\circ\text{C}) \]  
\[ \text{Ti} + \text{Al} \rightarrow \text{TiAl}_3 (673 \, ^\circ\text{C}) \]
\[
\text{Nb + Al} \rightarrow \text{NbAl}_3(821^\circ\text{C})
\]

Fig. 5a shows the micrograph of the sample heated to 900 °C for 30 min. The sample consisted of dark region corresponding to the pores, and together with dark gray and light gray phase dispersed around the bright core regions. EDS analysis showed that the composition of light gray region (point A) is about Nb–74Al–5Ti (at.%), corresponds to NbAl\(_3\) phase and solid solution Ti atomic. The bright core (point B) corresponded to about Nb–6.81 at.% Al, which was identified as the Nb(Al) solid solution according to the Nb–Al phase diagram, and this results is in agreement with XRD patterns showing the presence of unreacted Nb in the product. As can be seen from Fig. 5a, the Nb is encapsulated by NbAl\(_3\) phase like a core–shell structure was obtained through the TE reaction, indicating the incomplete conversion of Nb and Al. Milanese and Pis'menskaya [26,27] pointed out that the final product of NbAl sample is composed by the polyphasic structure, and the dissolution–precipitation mechanism produces the formation of NbAl\(_3\) precipitates around the Nb grain cores, which was appeared in the present study. The dark gray region (point C) was identified as TiAl\(_3\) phase, dispersed around the Nb–NbAl\(_3\) core–shell structure. The previous study shows that the sub-compound constituents plays a constructive role in the improved ductile/compression strength of intermetallic matrix. Venkateswara et al. [28] suggested that the presence of TiNb phase enhances the fracture resistance of TiAl at 650–800 °C, similar to previously reported results at room temperature. Similarly, Shu et al. [29] fabricated the Ti\(_2\)AlC/TiAl composites, showing that the average ultimate compression strength of the Ti\(_2\)AlC/TiAl composite is 179 MPa higher than that of the TiAl without sacrificing ductility.

Furthermore, the specimen was immersed in an uncured epoxy resin, then the resin was cured. After polishing, it was found that the pores were filled with the resin, showed the feature of interconnected pores (open pores).

### 3.3 Porous microstructure

The SEM images of fracture surface of sintered specimen was shown in Fig. 6. The low-magnification image (Fig. 6(a)–(d)) shows a noticeable difference in cross-section of the porous monolithic with various volume fractions of NaCl. For the specimen without NaCl, a foam-like pore structure is observed, with a relatively uniform porosity across the whole specimen, which is an inherent characteristic of the TE reaction process [30,31]. However, a number of large pores was formed with the addition of NaCl particles, which are marked by red circle in Fig. 6b. Moreover, the large pores exhibit the characteristic shape of the original NaCl particles, and the pore size distribution ranged from 200 µm to 300 µm. Note that the large pores are not found in the specimen without NaCl, indicating that the large pore was inherited from the original NaCl particles, although it was removed by leaching.
process before the TE reaction. This results suggest that the possibility of realizing tailored pore structure can be obtained by selecting appropriate shape and size of space holder. For instance, in present study, the pore shape (cubic) and pore size (200–300 μm) of product was designed by the NaCl particles (Fig. S1 in the Supplementary Information), and the numbers of this type of pores was increased with the increase of NaCl content.

Fig. 6(b)–(d) shows that the shape of large pore became more and more circular with increasing NaCl content. The more NaCl particles added into the specimen, the more agglomeration of pores was appeared, as shown in Fig. 6c. In consideration of the shape of original NaCl particles is cubic-like rather than circular, the rounded shape of large pores is probably owing to the deformation and crush of NaCl particles, during the cold pressing procedure.

The high-magnification SEM images (Fig. 6(e)–(h)) shows the small pores with size about 20 μm randomly dispersed throughout the whole sample without NaCl, and embedded in pore wall of large pores. On one hand, the large number of original pores existed in the green compacts, due to the non-dense packing of powder particles. This original pores further growth during the TE reaction, which plays an important role in the source of small pores in the product. On the other hand, the addition of TiH2 contributed to the formation of the small pores. Yeh et al. [32] showed that the number of pores increased with the increase of TiH2 in porous Ti5Si3 and Ti5Si3/TiAl intermetallics, and demonstrated that the hydrogen gas released from decomposition of TiH2 is responsible for this result.

Furthermore, Fig. 6(e)–(h) shows the pore window was appeared in the specimens with added NaCl particles, and the number of pore window increases with increasing volume fraction of NaCl particles. Owing to the difference in compressibility between the Nb–Al–TiH2 powder and NaCl particles, motion and redistribution of NaCl particles arise during the cold-press procedure, resulting in the contact area formed between the NaCl particles. The more NaCl particles, the easier it is to connect any two or more individual particles. After leaching process, the interlinked NaCl particles dissolve, then a shared pore window is existed between two contiguous large pores. Hu et al. [33] suggested that the percentage of pore windows increased with the amount of NaCl added, leading to the open/overall porosity ratio increased. In this study, the large pores are interconnected by pore windows, showing great potential for applications in heat exchange and filtration [34].

The optical metallographic images of specimens is presented in Fig. 7. Based on the above discussion, the golden region can be identified as the NbAl3/TiAl3 intermetallics skeleton, and the black region represent the pores. For the sample without NaCl, the pores distributed uniformly in the whole cross section, which consistent with the observation of SEM image in Fig. 6a. The pores shape are irregular, and most of pores are open pores. Two types of pores existed in the sample with added NaCl particles, large pores derived from the replication of NaCl particles and small pores embedded in pore walls. The small pore shows a dual function which not only elevates the open porosity of samples, but also facilitates the connectivity of large pores. With the increase of NaCl particles, the distribution of large pores become more uniform, and the pore walls become thinner. Moreover, the interconnected channel (marked by dotted red line) was formed by the connection of pores, which leads to an open pore structure in the porous material.

3.4 Expansion behavior and porosity

Fig. 8(a) and (b) illustrates the macroscopic images of green compact after water leaching and sintered products by adding 0, 30, 50, and 70 vol.% NaCl particles. After sintered at 900°C for 30 min, specimens with different volume fraction of NaCl revealed an noticeable macroscopic dilating; however, in all cases, specimens maintained the integrated cylindrical shape with no compact deformation or collapse. On the sample surface, the pores can be visible to the naked eye, and the number of pores rise with the increase in NaCl content. In addition, Fig. 8c shows the expansion ratio (radial, axial, and volume) as a function of volume fraction of NaCl. The volume expansion ratio is about 41–42%, and the radial and axial dimensions
also showed a similar trend, indicating the content of NaCl particles has no distinct effect on the expansion behavior. The XRD (Fig. 4) patterns demonstrates that the porous NbAl3/TiAl3 possessed the same phase, and the combustion image shows the reaction of Ti and Al leads to the expansion of specimens. Therefore, it can be concluded that this dimensional change behavior is most likely a consequence of the heat released during the TE process. Jiao et al. [22] reported that the volume expansion ratio up to 69.3% of Ti–Al intermetallic prepared by TE, and ascribed it to the abrupt change in sample temperature. Fig. 9 shows the open porosity and density of sintered products. The open porosity was measured to be 48.2% in specimen without NaCl, which mainly attributed to the small pore evolved from original pores in green compact and the decomposition of TiH2. With addition of NaCl particles, the open porosity of the synthesized product with 30, 50, and 70 vol.% NaCl is 62, 70, and 79%, respectively. It shows that the porosity of the product can be well designed by the content of NaCl particles, which plays role as a pore former. Correspondingly, the density decreased from 3.1 to 1.1 g cm\(^{-3}\) with increasing volume fraction of NaCl, showing an opposite trend to the open porosity. Moreover, the alterable porosity of porous NbAl3/TiAl3 suggests that the space-holder route not only impact on pore morphology but also provides a flexible way to adjust the porosity to multifarious applications.
4. Conclusion
The fabrication of porous NbAl3/TiAl3 intermetallic composites with controllable pore morphology and porosity was conducted by thermal explosion using NaCl spacer holder. Not only did the TiH2 powder used in Nb-Al system as a stabilizing agent of Al, but also as a diluent of TE reaction to prevent the synthesized product from cracking and deforming. The ignition temperature (Tig) and combustion temperature (Tc) decreased from 876.7°C/TiAl3 suggests that the space-holder route not only impact on pore morphology but also provides a flexible way to adjust the porosity to multifarious applications.

4. Conclusion
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Conflicts of interest
The authors declare no conflicts of interest.

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Appendix A. Supplementary data
Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jmrt.2019.05.007.

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