



Microstructure and evolution of (TiB₂+Al₂O₃)/NiAl composites prepared by self-propagation high-temperature synthesis

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Abstract: (TiB₂+Al₂O₃)/NiAl composites were synthesized by self-propagation high-temperature synthesis, and their phase compositions, microstructures and evolution modes were studied. The microstructures and shapes vary with the TiB₂+Al₂O₃ content in the NiAl matrix. TiB₂ particles take a great variety of elementary shapes such as white bars, plates, herringbones, regular cubes and cuboids. These results outline a strategy of self-assembly processes in real time to build diversified microstructures. Some TiB₂ grains in sizes of 2–5 μm are embeded in Al₂O₃ clusters, while a small number of TiB₂ particles disperse in the NiAl matrix. It is believed that the higher the TiB₂+Al₂O₃ content is, the more the regular shapes and homogeneous distributions of TiB₂ and Al₂O₃ will be present in the NiAl matrix.

Key words: (TiB₂+Al₂O₃)/NiAl composites; self-propagation high-temperature synthesis; microstructure; evolution mechanism

1 Introduction

Due to its low density (5.59 g/cm³), high melting point (1911 K), good thermal conductivity and good corrosion resistance [1], the NiAl intermetallic compounds have a wide application for components in high-temperature and corrosive environments, such as aero-engines, blades of gas turbine, thermal barrier coatings, and corrosion-resisting coatings. However, the low plasticity at room temperature and low strength at high temperature limit their applications in industry and production [2,3].

Up to now, many publications have involved in improving the room temperature plasticity and high temperature strength of Ni–Al intermetallic compounds. Adding different alloy elements such as Mo [4], Ti [5], Cr [6], and rare-earth metals [7] in NiAl is an efficient way to improve its properties. By adding or forming ceramics particles, such as Al₂O₃ [8,9], TiB₂ [10], NbB₂ [11] and TiC [12,13], the strength and creep properties of Ni–Al composite material can also be greatly improved. MICHALSKI et al [14] fabricated a

NiAl–Al₂O₃ composite with different amounts of Al₂O₃ in NiAl through spark plasma sintering and self-propagation high-temperature synthesis (SHS) of Ni and Al, and the results showed that the hardness of NiAl–Al₂O₃ increased and the fracture toughness of it was almost twice that of NiAl. The fabrication of NiAl matrix composites was carried out by a variety of novel processes such as combustion synthesis [11,14], mechanical alloying [13] and high pressure reaction sintering. It has been proved that combustion synthesis has significant advantages, such as low energy consumption, inexpensive fabrication equipment, simplicity of operation and in situ synthesis of composite components, compared with other conventional methods. Via combustion synthesis procedure, a series of NiAl matrix composites reinforced by TiB₂, NbB₂, TiC, Al₂O₃, ZrB₂ [15] and TiN [16] were obtained conveniently.

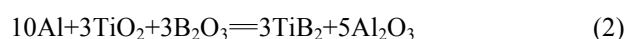
At present, many researches focus on TiB₂ and Al₂O₃ ceramics as a duplex reinforced phase in different matrices because of their similar thermal expansion coefficient, chemical and physical compatibility [17]. By adding TiB₂ into Al₂O₃ ceramics, the growth of Al₂O₃ grains and the propagation of cracks in the matrix can be

prevented. Consequently, the fine and well-distributed TiB_2 phase would help to enhance the strength and fracture toughness of the composite material. Thus, it would contribute to improving the abrasion resistance and fracture toughness in cutting tool materials [18,19].

The study on the previous work indicates that the fabrication of $\text{TiB}_2+\text{Al}_2\text{O}_3/\text{NiAl}$ composite materials through combustion synthesis has not been fully investigated. The fundamental objective of the present study is to investigate the microstructures and their evolution modes when different $\text{TiB}_2+\text{Al}_2\text{O}_3$ contents were formed in the NiAl matrix, using Ni, Al, B_2O_3 and TiO_2 as raw materials, via combustion synthesis. Also, the correlation between the $\text{TiB}_2+\text{Al}_2\text{O}_3$ content, microstructure and properties of the composites was investigated.

2 Experimental

The raw materials are commercial pure Ni powder (99.9%, 38–50 μm), Al powder (99.9%, 75 μm), B_2O_3 powder (98%, 75 μm), and TiO_2 powder (98%, 45 μm). The powder mixtures were considered as two parts: one was Ni+Al in which the mole ratio was fixed to $n(\text{Ni}):n(\text{Al})=1:1$, the other was $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$ in which the mole ratio was fixed to $n(\text{Al}):n(\text{TiO}_2):n(\text{B}_2\text{O}_3)=10:3:3$, and the latter was added into the former in mass fractions of 0, 5%, 10%, 15%, 20%, 25% and 30%, respectively. In order to produce a homogeneous mixture, all the powders were mixed in a three-dimension blender for about 6 h. Then, the mixtures were poured into metal molds with dimensions of $\phi 20 \text{ mm} \times 20 \text{ mm}$ and pressed at a pressure of 150 MPa into cylindrical compacts. Seven typical green compacts were fabricated with different contents of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$. In order to make the reactants react completely, the green compacts were preheated in an electric furnace at 300 $^\circ\text{C}$ for 30 min. After that, the compacts together with the molds were taken out and a small amount of Mg powders was poured onto the surface as the initiating combustion agent. At last the compacts were ignited by live tungsten wire immediately. The reactions between powders in raw materials were as follows:



Under the present condition, the reaction in green compact could self-sustain due to its high exothermic heat (SHS), the process is shown in Fig. 1.

The crystalline phases of the products were characterized by X-ray diffraction (XRD, Model D/Max 2500PC Rigaku, Japan). The morphologies of the fracture and microstructure were observed by scanning electron microscopy (SEM, Model KYKY2800B) and field emission scanning electron microscopy (FESEM,

NOVA NANOSEM 450, FEI). The compositions of samples were analyzed using electron probe microanalysis (EPMA, JXA-8230). The microhardness was measured by FM-700 with the load of 1 N and sustaining for 10 s. For each compound, five indentations were taken in the microhardness test and made an average of these points with a consideration of relative errors.

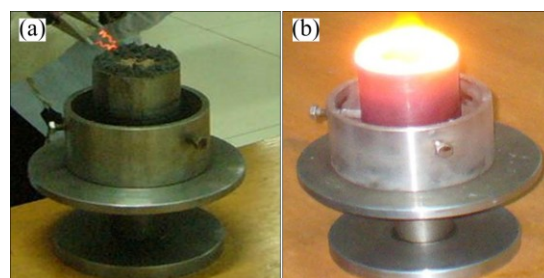


Fig. 1 Schematic diagrams of SHS reaction initiated by electrifying tungsten wire (a) and SHS reaction (b)

3 Results

In the sample without $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$, the Ni+Al reaction wave propagated steadily, and the speed was about 25 mm/s. When adding $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$ into Ni+Al, the igniting delay time prolonged. Once the reaction was initiated, the reaction wave spread rapidly. When the content of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$ increased to 15%, the spreading speed reached up to 40 mm/s. Thus, the whole reaction in the compacts almost took place at the same time, similar to thermal explosion. After the reactions finishing, the samples were slowly cooled down.

3.1 Phase analysis

Figure 2 shows the XRD pattern of the raw materials. It illustrates that the powder mixture is composed of Ni, Al, TiO_2 and B_2O_3 . Figure 3 shows the XRD patterns of products obtained by adding different contents of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$.

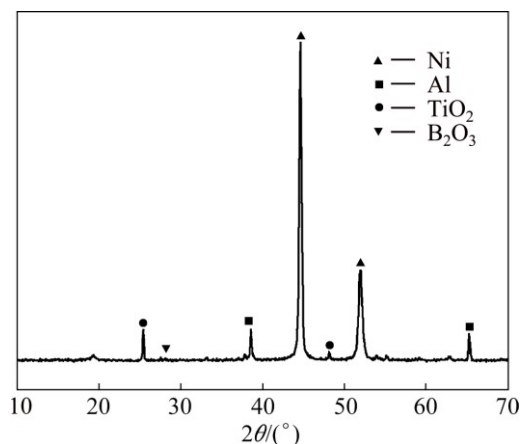


Fig. 2 XRD pattern of reactants

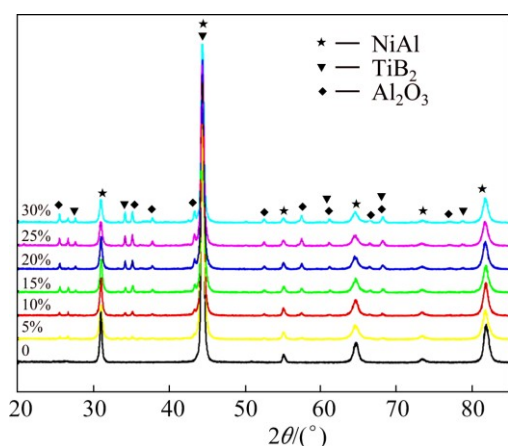


Fig. 3 XRD patterns of NiAl products with different contents of Al+TiO₂+B₂O₃

The reaction products simply consist of NiAl when the mixture contains only Ni and Al, which is in accordance with Reaction (1). After adding Al+TiO₂+B₂O₃, TiB₂ and Al₂O₃ ceramic phases appear besides

NiAl phase. The higher the reactant content of Al+TiO₂+B₂O₃ is, the more the TiB₂ and Al₂O₃ ceramic phases can be generated. Moreover, there are hardly any unreacted raw materials. Thus, it can be deduced that the reactions were completely carried out, and the phases which are expected according to Reaction (2) are obtained.

3.2 Microstructure and hardness

The microstructures on polished surface of samples with different contents of ceramics are shown in Fig. 4. In the samples only containing Ni+Al, the NiAl grains are coarse with a lot of needles and parallel plates, as shown in Figs. 4(a) and (b), and this is in accordance with Ref. [20]. After adding Al+TiO₂+B₂O₃, some white bars and plates (~5 μm) distributed along the boundary of the matrix appear, and the grains of NiAl matrix are refined remarkably, as shown in Figs. 4(c) and (d). Furthermore, some plates (10–15 μm) are alternatively distributed with a kind of grey phase, and their distributional patterns are very similar to herringbone

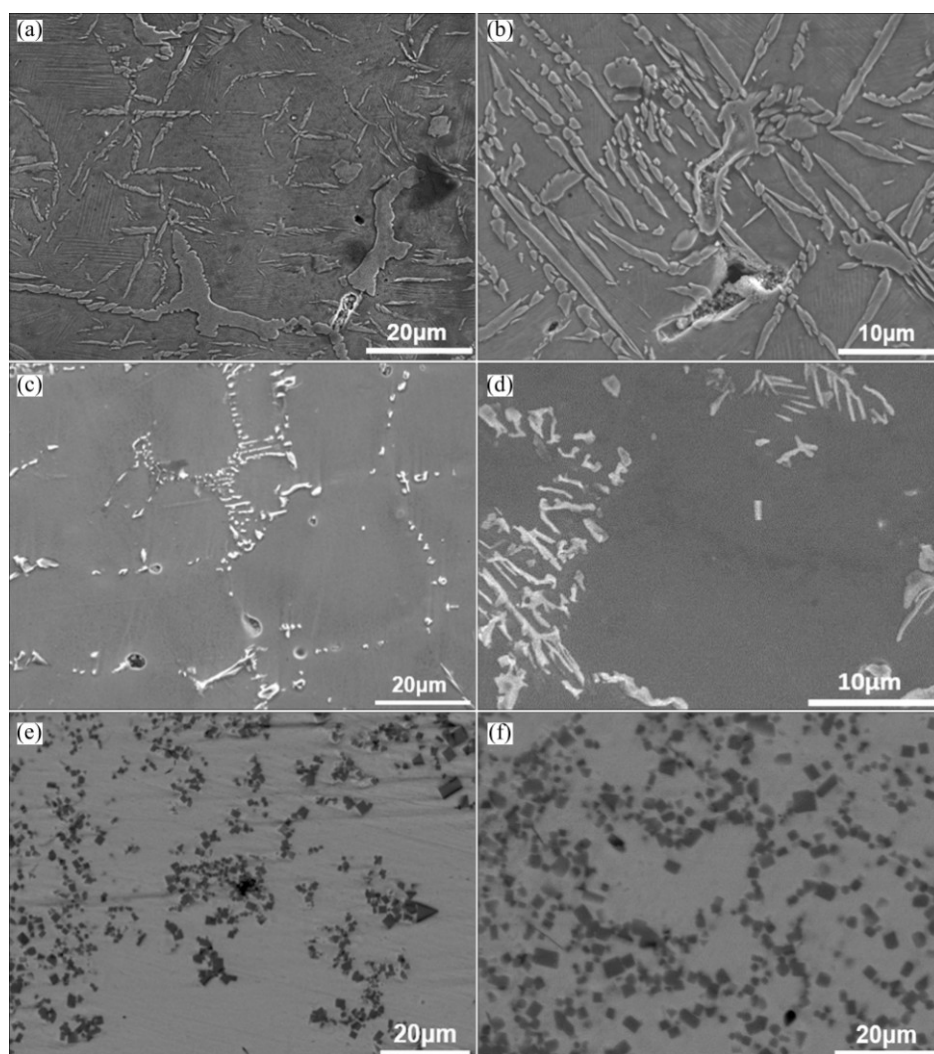


Fig. 4 SEM images of NiAl products with different contents of Al+TiO₂+B₂O₃: (a, b) 0%; (c, d) 10%; (e) 20%; (f) 30%

structure. When the content of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$ increases to 20%, the white phase turns to be fine regular particles (smaller than $5\ \mu\text{m}$), mainly in the shape of cubes or cuboids gathering together as clusters on an irregular dark grey conglomerations, as shown in Fig. 4(e). Also the aggregation ($60\text{--}70\ \mu\text{m}$) of white particle clusters on the irregular conglomerations and some white particles still distribute along the grains boundary of the matrix. The white particles seem to be inlaid in the dark grey conglomerations. With further increasing the content of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$, the amount of white particles increases significantly. But the size of aggregations of white particles, becomes smaller. In addition, some white particles disappear in the matrix, as shown in Fig. 4(f).

The microhardness test results are listed in Table 1. The average microhardnesses of the matrix in NiAl products without and with 10% ($\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$) are HV_{100} 398 and HV_{100} 501, respectively, which are close to the hardness of pure NiAl [20]. The microhardness of herringbone structure in the product with 10% ($\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$) is HV_{100} (1160–1730), which fluctuates remarkably. Compared with the product with 10% ($\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$), the hardness of white cube or cuboids gathering clusters in the products with 20% and 30% ($\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$) is higher, and the highest hardness is up to HV_{100} 2700. The hardness of dark grey irregular conglomerations which look like being pinned under the cube or cuboid particles is HV_{100} (1260–1480). The hardness of the products with 20% and 30% ($\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$) also varies from HV_{100} 570 to HV_{100} 1420, and the highest hardness is close to that of dark grey conglomerations.

Table 1 Microhardness and adiabatic temperature of samples with different contents of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$

Sample	Microhardness (HV)	Adiabatic temperature/K
Pure NiAl	398 ± 19	1911
NiAl–5%($\text{TiB}_2\text{--Al}_2\text{O}_3$)	410 ± 21	1938
NiAl–10%($\text{TiB}_2\text{--Al}_2\text{O}_3$)	501 ± 25	1965
NiAl–15%($\text{TiB}_2\text{--Al}_2\text{O}_3$)	567 ± 28	1992
NiAl–20%($\text{TiB}_2\text{--Al}_2\text{O}_3$)	595 ± 29	2018
NiAl–25%($\text{TiB}_2\text{--Al}_2\text{O}_3$)	608 ± 31	2045
NiAl–30%($\text{TiB}_2\text{--Al}_2\text{O}_3$)	596 ± 30	2072
Pure ($\text{TiB}_2\text{--Al}_2\text{O}_3$)	–	2448

3.3 Composition

The SEM images of typical samples and the results of EDS composition analysis are shown in Fig. 5 and Tables 2 and 3, respectively. Combining the results of XRD analysis, microhardness test and EDS composition analysis, we can deduce that the white bars and plates

which distribute along the boundary of the matrix or in herringbone structure at Spectrum 1 in Fig. 5(a) and the white particles in regular cube or cuboids shapes shown in Figs. 4(e) and (f) and Spectrum 3 in Fig. 5(b) are all TiB_2 grains. The gray matrix shown in Figs. 4(a) and (c) and Spectrum 2 in Figs. 5(a) and (b) is NiAl intermetallic compound. The irregular dark grey clusters which are around or under the cube or cuboids particles in Figs. 4(e) and (f) and Spectrum 3 in Figs. 5(a) and (b) are Al_2O_3 .

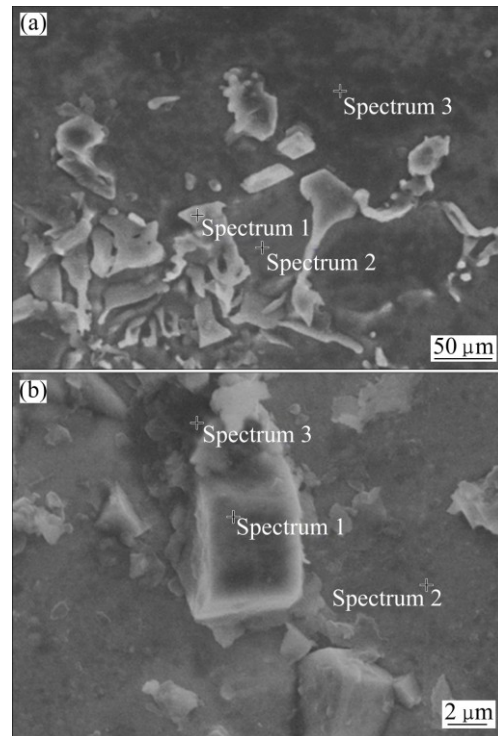


Fig. 5 SEM images of products with different contents of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$: (a) 5%; (b) 15%

Table 2 EDS compositions of composite with 5% ($\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$)

Element	x/%		
	Spectrum 1	Spectrum 2	Spectrum 3
Ni	0	46.27	6.47
Al	0	50.34	39.17
Ti	33.43	0	0
B	66.56	0	0
O	0	3.39	54.36

Table 3 EDS compositions of composite with 15% ($\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$)

Element	x/%		
	Spectrum 1	Spectrum 2	Spectrum 3
Ni	0	41.33	5.23
Al	0	52.36	37.27
Ti	30.75	0	0
B	69.24	0	0
O	0	6.31	57.50

4 Discussion

The mixture of reactants could be considered as two parts: one is Ni+Al which follows Reaction (1), the other is Al+TiO₂+B₂O₃ which follows Reaction (2). If the reaction systems are initiated at room temperature, the adiabatic temperature of Reaction (1) would be $T_{ad}=T_{m,NiAl}=1911$ K [21], while the adiabatic temperature of Reaction (2) would be $T_{ad}=2448$ K [22]. When preheating the samples at 300 °C, the adiabatic temperatures of Reactions (1) and (2) at 300 °C are much higher than their T_{ad} at room temperature. The T_{ad} of Ni+Al+TiO₂+B₂O₃ hybrid system can be calculated according to the thermodynamic data provided in Ref. [23]. With the increase of Al+TiO₂+B₂O₃ content, the T_{ad} of the mixtures rises to above 1911 K. The adiabatic temperatures of samples with different mass fractions of Al+TiO₂+B₂O₃ are listed in Table 1.

The reaction processes and microstructure evolution mechanisms of the system are shown in Fig. 6. As the melting points of Al and B₂O₃ are 660 and 445 °C, respectively, Al and B₂O₃ in the green powder (Fig. 6(a)) melt firstly during the reaction proces. The melted Al contacts with liquid B₂O₃ and infiltrates into the surface of TiO₂ particles, as shown in Fig. 6(b). Thus, B and Ti elements are displaced from B₂O₃ and TiO₂, respectively [24,25]. At the same time, Al₂O₃ and TiB₂ are produced rapidly through Reaction (2). Moreover, the surplus molten Al and solid Ni react to form liquid NiAl, as the melting point of NiAl is lower than the adiabatic temperature of reaction system, as shown in Fig. 6(c). The sequences of the whole reaction are as follows:

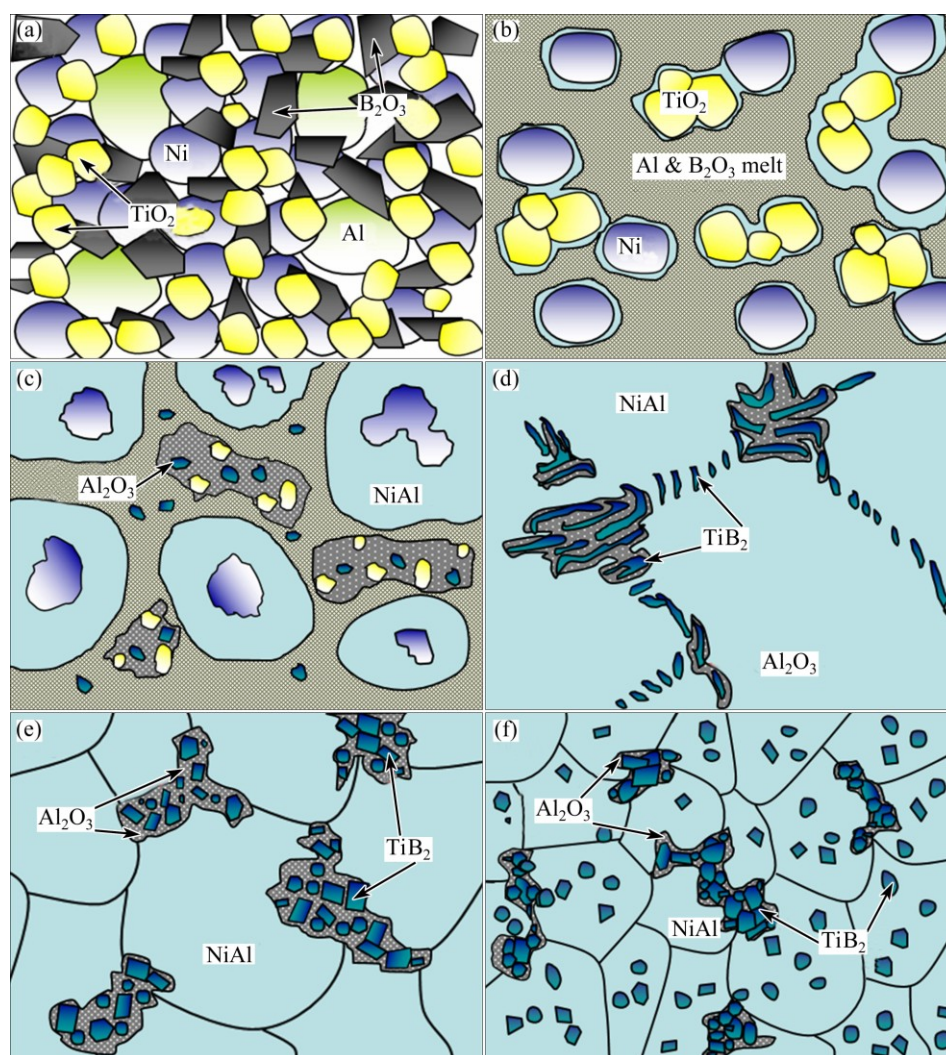
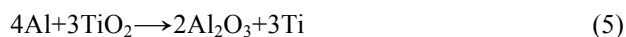


Fig. 6 Sketch of reaction processes and structure evolution modes: (a) Mixed initial powders; (b) Al and B₂O₃ begin to melt; (c) Grains of NiAl, TiB₂ and Al₂O₃ formatting; (d–f) Morphologies of products with 10%, 20% and 30% (Al+TiO₂+B₂O₃), respectively

When the content of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$ is less than 10%, both the amounts of TiB_2 and Al_2O_3 , and the temperature of the reaction system are not sufficient. Therefore, the TiB_2 grains grow as irregular bars and plates [26,27] (Figs. 4(d) and 6(d)). TiB_2 and Al_2O_3 grow into herringbone alternative structure through “self-assembly” mode. When the reaction system cools down below the NiAl melting point, NiAl grains begin to nucleate and grow to form the matrix of composite gradually.

With increasing the content of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$, the T_{ad} of reaction system increases and a great amount of TiB_2 and Al_2O_3 is produced. The abundant TiB_2 resources and higher temperature are better for the regularization of TiB_2 [26]. Thus, TiB_2 particles grow freely, develop fully and take shapes of cube or cuboids finally. As Al_2O_3 grains form around the TiB_2 grains almost simultaneously, it is possible for them to attach together to form large irregular conglomerations, as shown in Figs. 4(e) and (f) and 6(e) and (f). During the grow process of NiAl grains, the conglomerations are pushed to the corner of grains or distribute along the boundaries. Also, this kind of distribution impedes the boundary's movement and restricts the grain's growth of NiAl matrix.

When the $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$ content increases to 30%, much higher T_{ad} can be reached and more TiB_2 and Al_2O_3 are produced. All the above contribute a lot to TiB_2 grains to grow as regular cubes cuboids or hexagons, as shown in Figs. 4(e) and (f) and 7(a). In addition, a large amount of Al_2O_3 adheres to TiB_2 grains to nucleate, as

shown in Fig. 7(b), and grows around them. Because there are no plates to subdivide the spaces between TiB_2 grains, as shown in Fig. 4(b), the Al_2O_3 grains develop more freely until touching each other to form conglomerations, as shown in Figs. 4(c) and (d). In the meantime, there are also some single TiB_2 cubes or cuboids dispersed in the NiAl liquid and some Al_2O_3 grains adhere to the impurities in NiAl liquid to nucleate. Finally, when NiAl begins to solidify, almost all the above solid phases that form before NiAl will be pushed to the grain boundaries in the similar way shown in Figs. 4(b) and 6(e), which makes contribution to the formation of refine NiAl grains. What is more, the Al_2O_3 grains which nucleate on the impurities may remain in the NiAl matrix to help to improve the hardness. These Al_2O_3 grains may flock together and become big conglomerations because of the self-diffusion of Al_2O_3 . Therefore, the hardness of matrix in Fig. 4(c) varies greatly, and the highest hardness is close to that of pure conglomerations.

5 Conclusions

1) $(\text{TiB}_2+\text{Al}_2\text{O}_3)/\text{NiAl}$ composites were synthesized by combustion synthesis using Ni , Al , TiO_2 and B_2O_3 mixed powders. With increasing the content of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$, more TiB_2 and Al_2O_3 ceramic phases are generated in the NiAl matrix. When the reactant content of $\text{Al}+\text{TiO}_2+\text{B}_2\text{O}_3$ is less than 10%, TiB_2 and Al_2O_3 grow into herringbone alternative structure through self-assembly mode.

2) When more $\text{TiB}_2+\text{Al}_2\text{O}_3$ form, TiB_2 particles would grow freely and develop fully and take shapes of cube, cuboids or hexagons finally. At the same time, more Al_2O_3 which forms around TiB_2 grains tends to attach together to form large irregular conglomerations.

3) The microhardness of NiAl matrix composite increases by 50% from HV (398±19) to HV (608±31), due to grain refining with increasing the content of $\text{TiB}_2+\text{Al}_2\text{O}_3$ to 25%.

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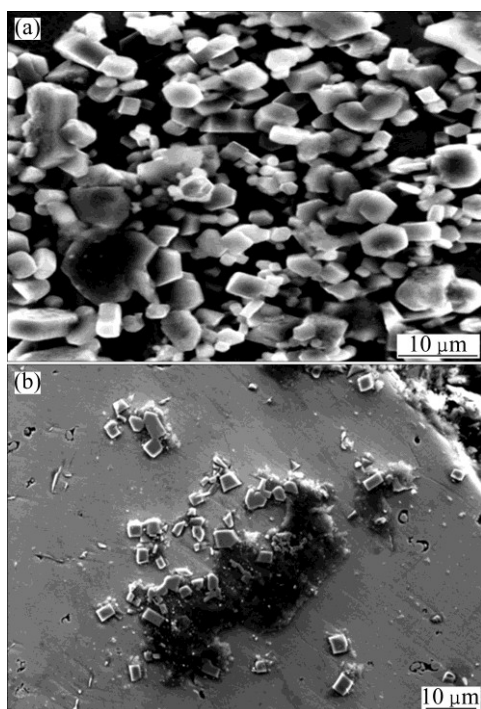


Fig. 7 SEM images of regular TiB_2 particles (a) and Al_2O_3 conglomerations (b)

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自蔓延高温合成($\text{TiB}_2+\text{Al}_2\text{O}_3$)/NiAl 复合材料的显微组织及演化机制

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摘 要: 采用自蔓延高温合成技术制备($\text{TiB}_2+\text{Al}_2\text{O}_3$)增强 NiAl 基复合材料, 并研究其相组成、显微组织和演化机制。结果表明: 复合材料的组织及陶瓷相的形貌随添加($\text{TiB}_2+\text{Al}_2\text{O}_3$)含量的不同而变化, TiB_2 在短时间内以自组装的模式形成长条状、片状、鱼骨状以及规则的四方体和六棱柱等不同形态。 TiB_2 的尺寸在 2–5 μm 之间, 与不规则的 Al_2O_3 伴生存在, 少量 TiB_2 分布于 NiAl 基体中。随着陶瓷相含量的增多, TiB_2 的形状趋于规则, 且在 NiAl 基体中的分布更加均匀。

关键词: ($\text{TiB}_2+\text{Al}_2\text{O}_3$)/NiAl 复合材料; 自蔓延高温合成; 显微组织; 演化机制

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