Laser melted TiC reinforced nickel aluminide matrix in situ composites

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Abstract

Titanium carbide reinforced nickel aluminide matrix in situ composites were produced using a newly patented laser melting furnace. Microstructure of the laser melted TiC/(Ni_3Al–NiAl) in situ composites was characterized by optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and transmission electron microscopy (TEM). Results showed that the constituent phases in the laser melted in situ composites are TiC, Ni_3Al and NiAl. Volume fraction of TiC and NiAl increase with increasing content of titanium and carbon. The growth morphology of the reinforcing TiC carbide has typically faceted features, indicating that the lateral growth mechanism is still predominant growth mode under rapid solidification conditions.
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1. Introduction

Nickel aluminides based on NiAl and Ni_3Al are of interest for structure applications at elevated temperature due to their high melting point, low density, high specific modulus, excellent thermal conductivity and good oxidation resistance [1–3]. Ni_3Al-based intermetallic alloys, in particular, possess an abnormal yield stress–temperature relationship up to ~1000 K, which is advantageous for structural applications at elevated temperatures. However, the brittleness at room temperature and inadequate yield strength at high temperatures prevent them from industrial application. Recently, a few studies have been carried out to improve the ambient temperature ductility by micro-alloying and macro-alloying [4–6]. As to their elevated temperature strength, one possible approach is to incorporate metallurgically stable, hard and fine ceramic particles in nickel aluminides [7–10]. It is well known that the TiC type MC carbide has the advantages of high hardness, high melting point, low density and excellent high temperature stability and, therefore is expected to be a suitable reinforcement in nickel aluminides. To the authors' knowledge, fabrication technology of Ni_3Al matrix composites has so far been focused on simultaneous combustion and densification synthesis [11], solid state reaction by mechanical alloying [12], self-propagating high-temperature synthesis [13–14], high temperature and high densification in situ synthesis [15] and so on. The advent of high-power lasers provides a new processing technique for the fabrication of bulk advanced materials. In this paper, nickel aluminide matrix in situ composites reinforced by TiC particles were fabricated by the Lasmelt[TM] process [16,17] using a newly invented chilled copper-mold laser melting furnace. Microstructures of laser melted/rapidly solidified TiC reinforced nickel aluminide in situ composites were characterized by optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and transmission electron microscopy (TEM), the growth morphologies of reinforcement were also investigated.

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Table 1
Chemical composition (at.%) of Ni–Al–Ti–C elemental powder blends for fabrication of the TiC reinforced nickel aluminide matrix in situ composites

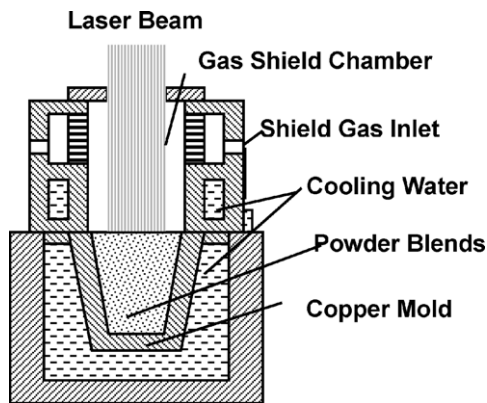| Sample no.	Ni	Al	Ti	C
C1	67.5	22.5	5	5
C2	60	20	10	10
C3	52.5	17.5	15	15

Fig. 1. Schematic illustration of the water-cooled copper-mold laser melting furnace.

2. Experimental procedures

Commercially pure nickel, aluminum, titanium and carbon powders were selected as starting precursor materials for fabricating TiC reinforced nickel aluminide matrix in situ composites by a laser melting technique. Particle size of the powders ranges from 4.5 to 7.5 μm. Three compositions of the powder blends, as listed in Table 1, were selected to produce the in situ composites having different volume fraction of the reinforcing phase TiC, which has an important influence on the mechanical properties of the composites. The powder blends were well mixed prior to laser processing. As schematically illustrated in Fig. 1, the Ni–Al–Ti–C elemental powder blends were preplaced in the water-cooled copper-mold in argon shielding atmosphere, and melted by a high power CO_2 laser beam. The laser melting was performed on a 5 kW CO_2 laser materials processing system equipped with a four-axis computer numerical controlled (CNC) work table. The laser melting processing parameters were: laser output power, 2.5 kW; laser beam diameter, 7 mm; laser irradiation time, 20–30 s.

Samples of laser melted/rapidly solidified in situ composites for microstructure observation were prepared by standard mechanical polishing and were etched in a solution of 25 vol% nitric acid, 50 vol% hydrofluoric acid and 25 vol% glycerin. The microstructure of the laser melted in situ composite was characterized under an optical microscope (ZEISS Axiotech 100HD), a scanning electron microscope (KYKY2800) with EDS attachment using the Noran Ventage DSI spectrometer, and a transmission electron microscope (H-800). The constituent phases of laser melted in situ composites were identified by X-ray diffraction method using a Rigaku D/max 2200 diffractometer with Cu Kα radiation operated at a voltage of 40 kV, a current of 40 mA and a scanning rate of 5°/min. Thin specimens for TEM observation were cut from laser melted ingots and were mechanically thinned to 40 μm before final thinning by argon ion bombardment. Microhardness of the laser melted in situ composite was measured using a MH-6 semi-automatic Vicker tester with a test load of 200 g and a dwell time of 15 s.

3. Results

Fig. 2 shows the XRD results of the laser melted/rapidly solidified in situ composites with Ni–17.5Al–15Ti–15C alloy powder. It indicates that the main constituent phases of the laser melted Ni–Al–Ti–C powder system are: TiC, Ni$_3$Al and NiAl. Typical microstructures of TiC/(Ni$_3$Al–NiAl) in situ composites laser melted with different elemental powder system are shown in Figs. 3–5, respectively. All three com-
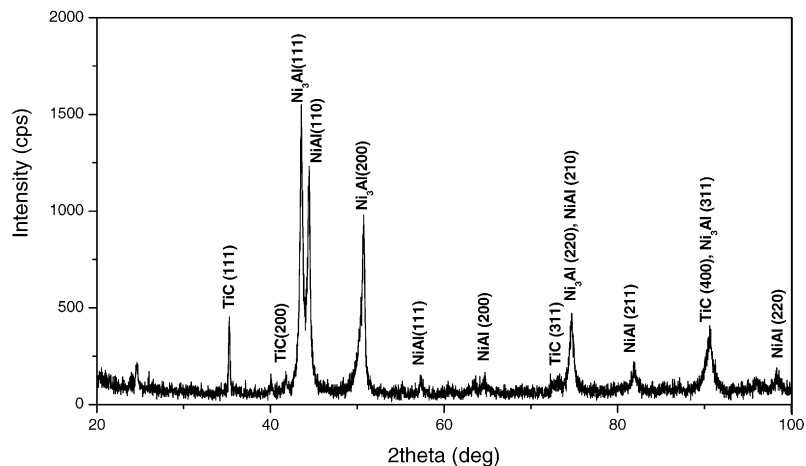

Fig. 2. Results of XRD analysis of a laser melted in situ composite with powder blends of Ni–17.5Al–15Ti–15C (at.%).
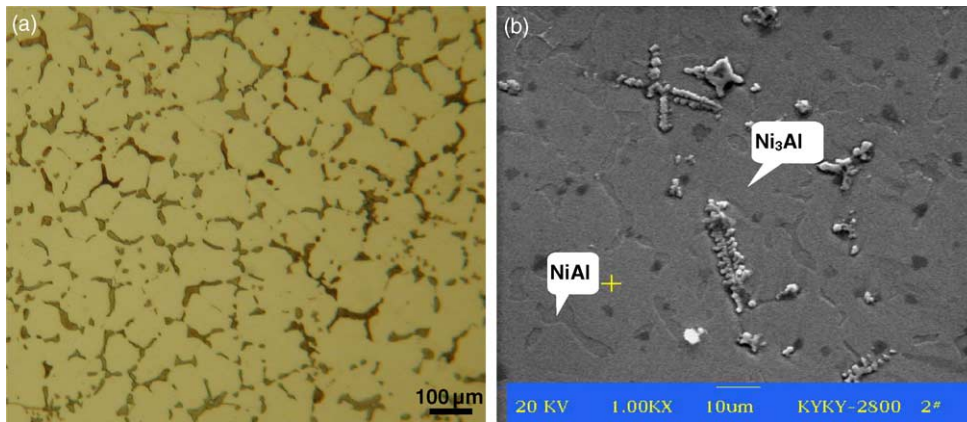
Fig. 3. OM micrograph (a) and SEM micrograph (b) showing the typical microstructure of laser melted in situ composite in composition (at.%) of Ni–22.5Al–5Ti–5C.
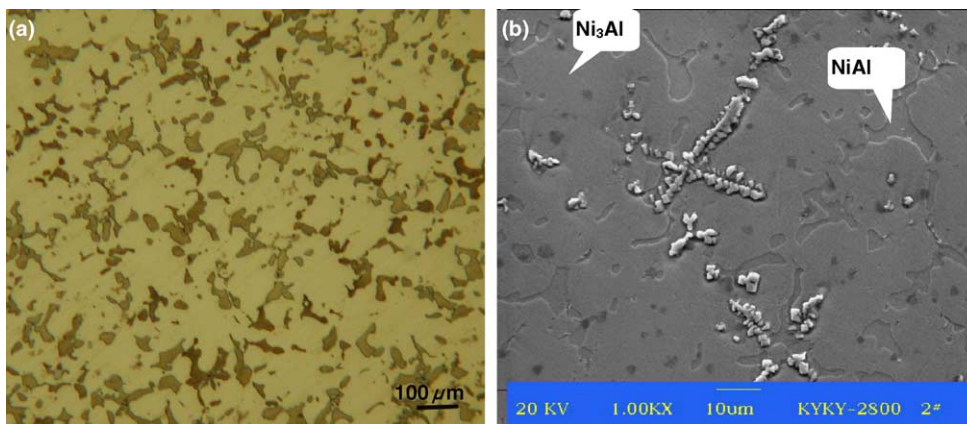

Fig. 4. OM micrograph (a) and SEM micrograph (b) showing the typical microstructure of laser melted in situ composite in composition (at.%) of Ni–20Al–10Ti–10C.

posites have rapidly solidified fine and dense microstructure due to the rapid heat conduction cooling of the water-chilled copper-mold. Volume fraction of the TiC reinforcement and NiAl increase with increasing the content of titanium (carbon), compared with Figs. 3–5.

The change in microhardness of the laser melted TiC/(Ni$_3$Al–NiAl) in situ composites as a function of the content of titanium and carbon is illustrated in Fig. 6. Clearly, all the three laser melted/rapidly solidified in situ composites have higher hardness. The microhardness of the laser melted
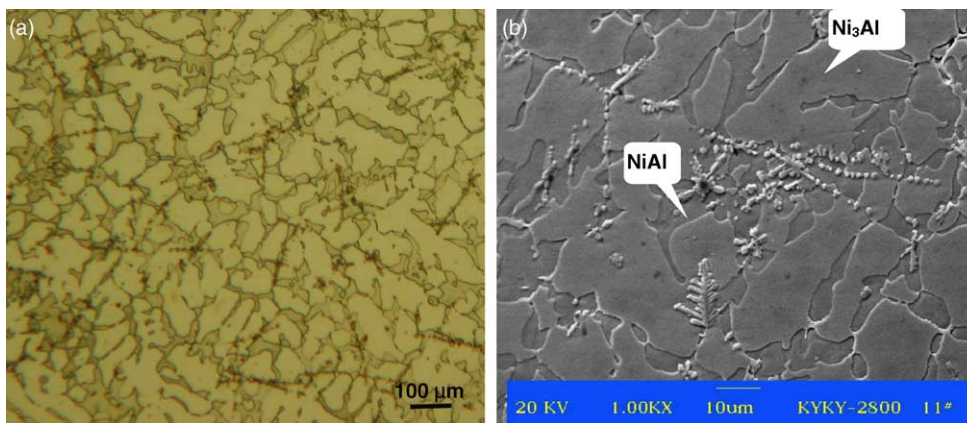

Fig. 5. OM micrograph (a) and SEM micrograph (b) showing the typical microstructure of laser melted in situ composite in composition (at.%) of Ni–17.5Al–15Ti–15C.
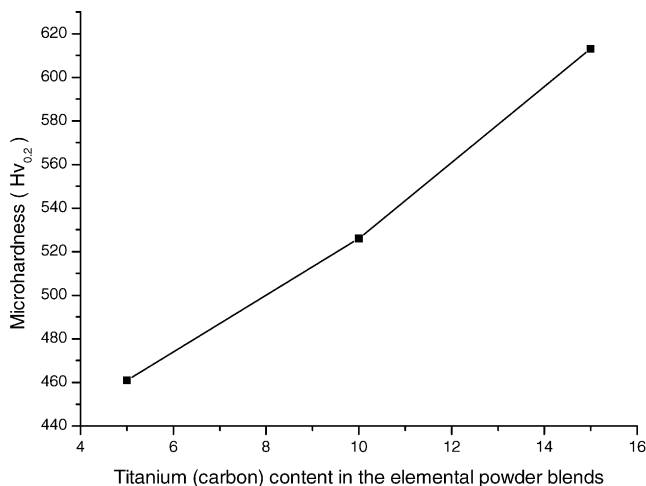
Fig. 6. Microhardness of the laser melted TiC/(Ni$_3$Al–NiAl) in situ composites as a function of titanium and carbon content.

in situ composite also increases with the increasing content of titanium and carbon because the volume fraction of primary TiC in composite increases with increasing content of titanium and carbon in elemental powder system.

As shown in Fig. 7(a), the advancing fronts of TiC in a laser melted in situ composite are faceted. The result of the selected area diffraction parameter in the [1 1 1] direction shows that the carbide has fcc crystal structure, as shown in Fig. 7(b). The calculated lattice parameter of carbide is approximately 0.4374 nm, which is higher than the standard lattice parameter, 0.4328 nm. It probably resulted from nickel and aluminum dissolved in TiC. The growth morphologies of TiC type MC carbide in laser melted/rapidly solidified TiC/(Ni$_3$Al–NiAl) in situ composites are shown in Fig. 8. The growth morphologies of reinforcements are in well-developed dendrites. It indicates that the composition of the precursor powder material has no influence on the growth morphology of TiC carbide.

4. Discussion

It is well known that the TiC type MC carbide has a high melting point (3200 °C) and large negative free energy of formation in the Ni–Al–Ti–C powder system. As a result, TiC carbide precipitates firstly from the melt as the primary phase during the rapid solidification process. Accompanying the precipitation of primary dendritic TiC, the residual melt is highly depleted in Ti and C, and is close to Ni–Al binary melt. Because nickel and aluminum can be dissolved in primary TiC dendrites, the nominal chemical composition of the residual melt after the formation of primary TiC dendrites might deviate from the zone of the formation of Ni$_3$Al intermetallic alloy. The authors think that the nominal chemical composition of residual liquid might lies at about the dashed line in Ni–Al binary phase diagram, as shown in Fig. 9 [18]. Hence, NiAl phase precipitates subsequently in the following rapid solidification process, then the peritectic reaction L + NiAl → Ni$_3$Al occurs. Because the nominal chemical composition of residual melt deviates from that of peritectic point, the peritectic reaction cannot proceed completely and the final peritectic products are NiAl and Ni$_3$Al. On the other hand, more content of nickel may be dissolved in TiC particles because the standard lattice parameter of nickel (0.35238 nm) is smaller than that of aluminum (0.404959 nm). As a result, the ratio of the nickel content to that of aluminum in the residual liquid decreases with increasing volume fraction of TiC, leading to an increase in volume fraction of NiAl phase.

TiC carbide is considered as a typical faceted crystal under slow cooling solidification conditions due to its high Jackson factor [19], and therefore its growth mechanism is well known to be the lateral growth [20,21]. As shown in Fig. 6, the growth morphologies of TiC in laser melted and rapidly solidified in situ composites are in well-developed dendrites. This phenomenon indicates that the element diffusion in TiC carbide required to form the equilibrium growth shape becomes harder to satisfy. As a result, carbide grows along its
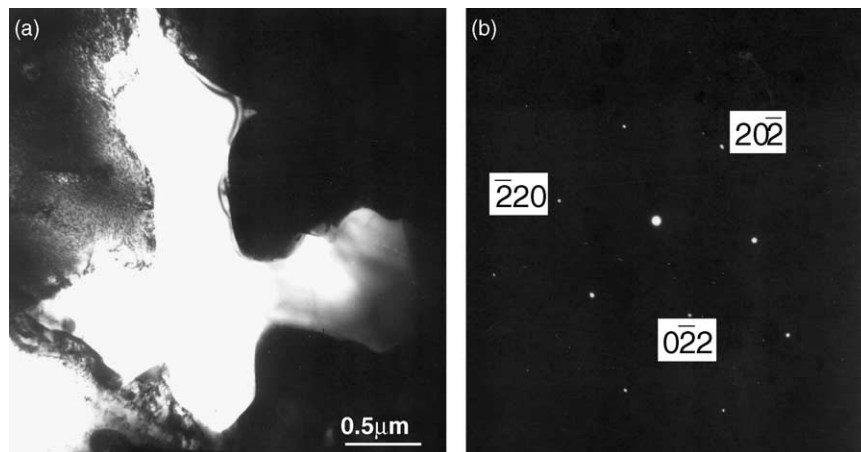

Fig. 7. Bright-field TEM images of (a) TiC and SAD patterns (b) TiC.
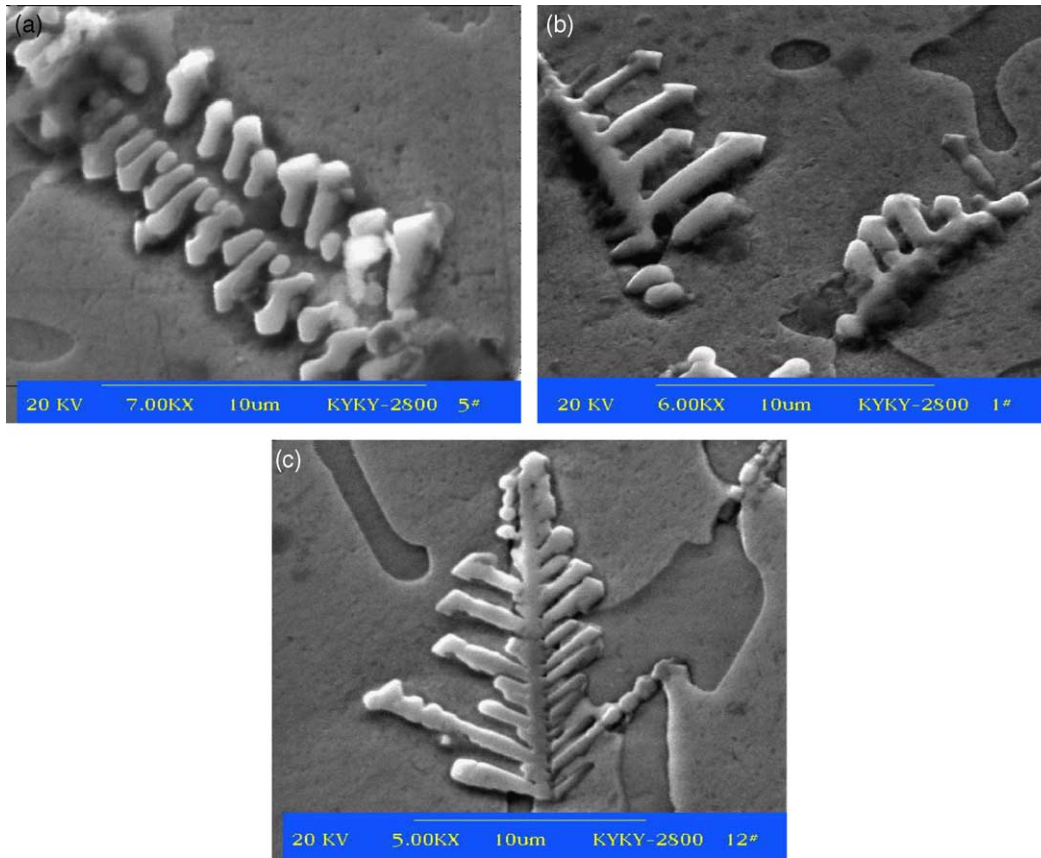
Fig. 8. Growth morphologies of TiC in laser melted/rapidly solidified in situ composites with power composition (at.%) of (a) Ni–22.5Al–5TiC–5C, (b) Ni–20Al–10Ti–10C and (c) Ni–17.5Al–15Ti–15C.
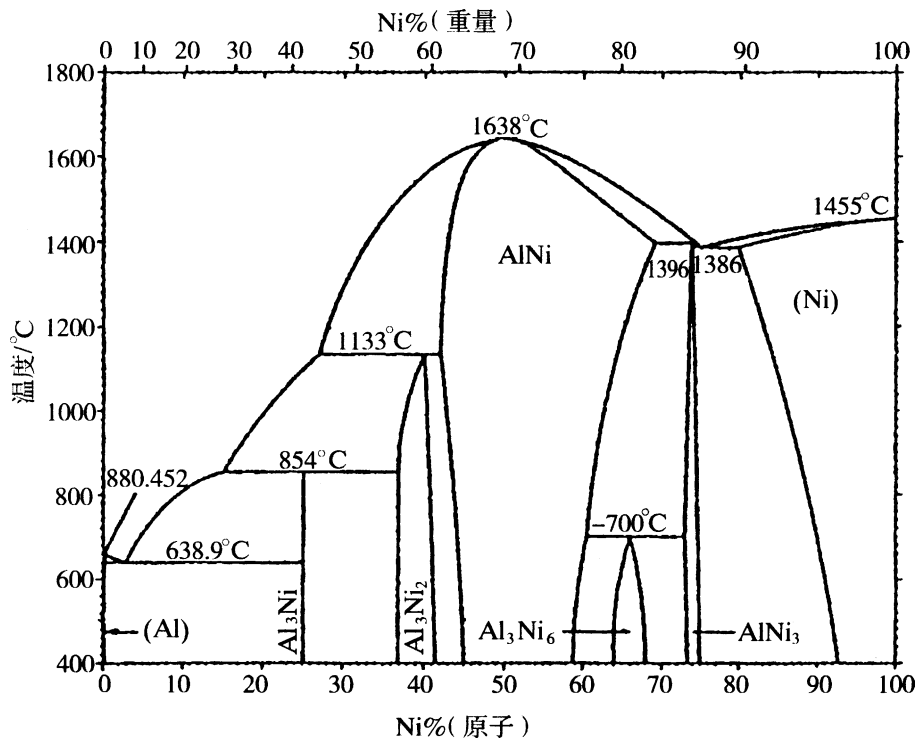

Fig. 9. Ni–Al binary phase diagram.

preferred growth direction, i.e. $\langle 0\,0\,1 \rangle$, and its growth morphology changes to be dendritic. It is worth noting that the growth surfaces of TiC are all faceted. However, all the tip surface of the secondary arms is strongly faceted, as shown in Fig. 8, which illustrated that TiC carbide still grows by the mechanism of lateral ledge growth.

According to above experimental results and analysis, it is clear that the Lasmelt process newly invented has the following advantages compared with conventional alloy-melting: (1) materials produced by Lasmelt process are free from ceramic inclusions from refractory crucible; (2) the Lasmelt process is a very rapid melting process; (3) materials produced by Lasmelt process have fine and dense microstructure due to the rapid heat conduction cooling of the water-chilled copper-mold.

5. Conclusions

Nickel aluminides matrix in situ composites reinforced by TiC were produced by the laser melting process using Ni–Al–Ti–C elemental powders. The laser melted TiC/(Ni$_3$Al–NiAl) in situ composites have high hardness and fine microstructure. The composition of elemental powder precursor materials has no influence on the constitution phases and carbide growth morphology in the in situ composite. However, the volume fraction of TiC and NiAl increases with increasing content of titanium and carbon. The growing surfaces of TiC reinforcements have typically faceted features, illustrating that TiC grows by a lateral growth mechanism under rapid solidification conditions.

Acknowledgement

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