JOURNAL OF MATERIALS SCIENCE **32** (1997) 6325–6329

# Preparation of TiAl/Mo and TiAl/NiAl composites by powder processing

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Processes for preparing TiAl/Mo and TiAl/NiAl composites are described and results of the investigation are reported. It was found that the hardness of the TiAl/NiAl composites was largely influenced by the amount of hard ternary phases formed through reactions between TiAl and NiAl. The possibility of ductilizing and toughening TiAl by introducing molybdenum as a ductile reinforcement has also been shown. For both composites, optimal process parameters still remain to be determined by further experiments to suppress the formation of hard ternary phases, which are believed to be detrimental to ductility and fracture toughness.

## 1. Introduction

Materials based on the intermetallic compound TiAl are of practical importance owing to their low density, high melting temperature, relatively good mechanical properties and environmental resistance at elevated temperatures. However, applications as lightweight high-temperature materials are largely limited by their poor ductility and fracture toughness at low or intermediate temperatures [1–3]. To tackle this problem, numerous studies have been conducted [1–15]. An improvement in ductility has been achieved by adding alloying elements such as chromium and manganese and grain refining or microstructural control via suitable thermal or thermomechanical treatment. As a result, a room-temperature tensile fracture strain of about 3%–4% has been measured for monolithic TiAl alloys [8–11]. In addition, incorporating metallic ductile phases into the brittle TiAl matrix has led to quite promising results: large improvements in both monotonic fracture toughness and cyclic fatigue threshold compared to monolithic TiAl are observed in niobium particle-reinforced TiAl [12, 13].

In a search for other measures by which to improve ductility and fracture toughness of TiAl-based materials, two TiAl matrix composites were prepared and investigated: TiAl/Mo and TiAl/NiAl. The results of fabrication processes, microstructures as well as hardnesses of the composites, are reported here. Other results, concerning phase formation and mechanical properties, have been published elsewhere [6, 15].

## 2. Experimental procedure

The two composites were prepared via different processes. One (TiAl/Mo) was made by hot pressing alternately stacked Ti–Al and molybdenum sheets, and the other (TiAl/NiAl) was processed using techniques of mechanical alloying. Details of the specimen preparations are given below.

### 2.1. Preparation of the TiAl/Mo specimen

Fig. 1 shows the manufacturing process used for preparation of the TiAl/Mo specimen. Elemental titanium and aluminium powders of sizes smaller than 100 μm, with purities of 99.8% and 99.9%, respectively, were mixed to the desired composition Ti–48 at % Al. The powder mixture was pre-compacted at room temperature using a 1000 kN load to a green compact of 50 mm diameter. This compact was further extruded to a 10 mm × 20 mm rectangular bar which was then rolled to a 0.5 mm thick sheet. The rolling direction was perpendicular to the extrusion direction. The as-rolled sheet had a width of about 100 mm. Cold-rolled Ti–Al sheets, as well as commercial molybdenum foils of 0.1 mm thickness (purity 99.9%) were slightly polished and cleaned with acetone to remove surface dirt. The Ti–Al sheets were sandwiched in alternate layers with molybdenum foil and hot pressed at 1250 °C for 2 h under a pressure of 20 MPa in an argon atmosphere. A heating rate of 10 K $\text{min}^{-1}$ was used. The cooling of the specimen was carried out in two steps: first cooling to about 500 °C at a cooling rate of 10 K $\text{min}^{-1}$ and then air-cooling to room temperature. During hot pressing, titanium and aluminium react to form intermetallic compounds TiAl and $Ti_3Al$ [4–6]. At the same time, diffusion bonding between the TiAl matrix and the molybdenum reinforcements occurs, so that a TiAl–Mo composite was obtained.

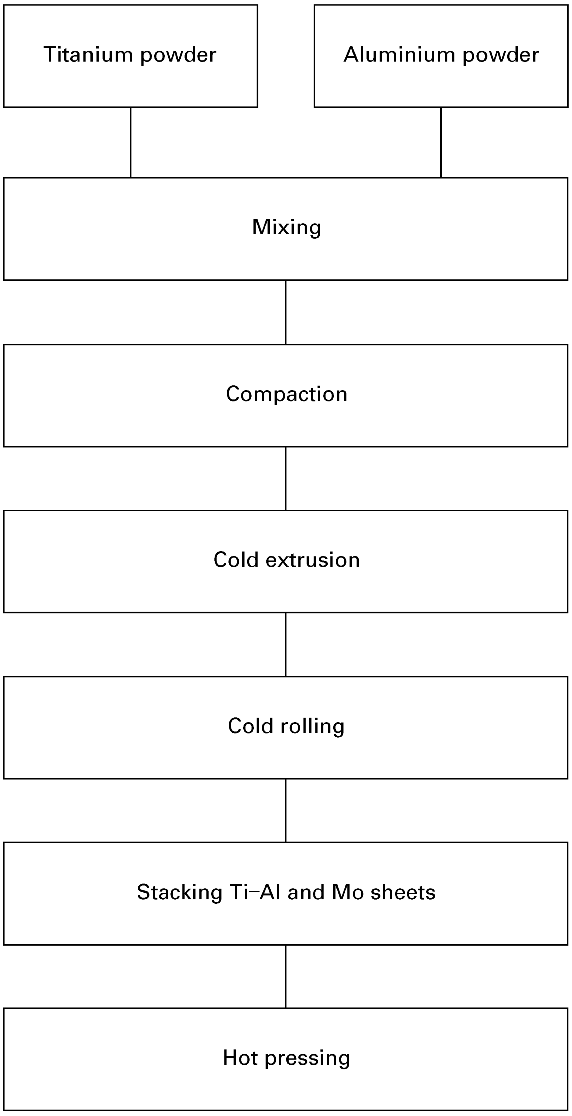

*Figure 1* Preparation process for the TiAl/Mo specimen.

## 2.2. Preparation of the TiAl/NiAl specimens

In Fig. 2 the process for preparation of the TiAl/NiAl specimens is schematically presented. Prealloyed $Ti_{50}Al_{50}$ and $Ni_{50}Al_{50}$ (at %) powders, both produced from high-purity elemental powders by reaction synthesis processes and having particle sizes of 20–60 μm and 40–125 μm, respectively, were mixed to different portions: 100% TiAl, 90% TiAl + 10% NiAl, 75% TiAl + 25% NiAl, 50% TiAl + 50% NiAl, and 100% NiAl. The pure TiAl and pure NiAl were chosen as reference samples. The powder mixtures were ball milled for 5 h at $-150\,^{\circ}C$, with 6.35 mm steel balls and a ball-to-powder weight ratio of 10:1 being used. The milling temperature of $-150\,^{\circ}C$ was realized through liquid nitrogen cooling of steel vials, which contain powders and balls during milling. Such a low milling temperature is believed to be beneficial for promoting particle fracture, reducing cold welding and suppressing new phase formation. All handling of the powder materials was carried out inside a specially constructed chamber filled with argon. The consolidation of milled powders was conducted via uniaxial pressing (pressure about 40 MPa) in a vacuum furnace (vacuum at least $10^{-2}$ torr $\simeq$ 1.33 Pa) at different temperatures. The holding time was typically 2 h; the total time at temperatures exceeding 600 °C was about 3 h. The resulting consolidated specimens were typically 10 mm diameter and 15 mm long.

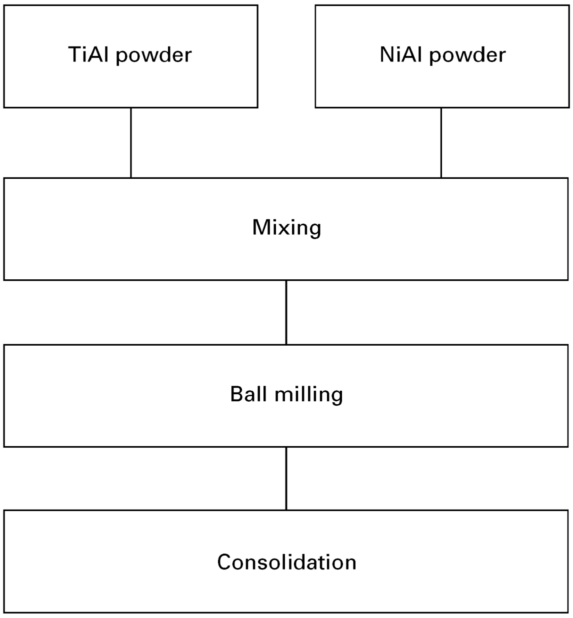

*Figure 2* Preparation process for the TiAl/NiAl specimens.

The prepared specimens were examined by optical and scanning electron microscopies, as well as X-ray diffraction analysis. Density determinations for consolidated specimens were carried out using the Archimedes' method described in ASTM C693. In addition, Vickers' hardness was measured to study individual constituents in the TiAl/Mo composite and to establish the relationships between hardness and consolidation temperature or composition for the TiAl/NiAl composites. At least ten hardness measurements were taken to obtain the mean value for one datum point.

## 3. Results and discussion

### 3.1. TiAl/Mo specimen

Fig. 3 shows the optical micrograph of the TiAl/Mo sandwich specimen. The TiAl matrix exhibits a Duplex structure consisting of lamellar TiAl/$Ti_3Al$ regions distributed in the TiAl phase. Between the TiAl and the molybdenum a reaction zone (RZ) is visible. As reported elsewhere [6], this reaction zone contains two diffusion layers: one is about 10 μm thick and consists of cubic $(Mo,Ti)_3Al$ phase, and the other is about 40 μm and consists of (Mo, Ti)Al phase with a body centred cubic (b c c) crystal structure. As shown in Fig. 4, the incorporated molybdenum exhibits a Vickers' hardness of about 60 HV1, much lower than that of the TiAl matrix (about 250 HV1). This implies that ductilizing and toughening of brittle intermetallic TiAl by introducing metallic molybdenum as ductile reinforcement are possible. However, the reaction zone containing both $(Mo,Ti)_3Al$ and (Mo, Ti)Al phases exhibits a hardness of about 330 HV1, much higher than the hardness of the matrix. This indicates

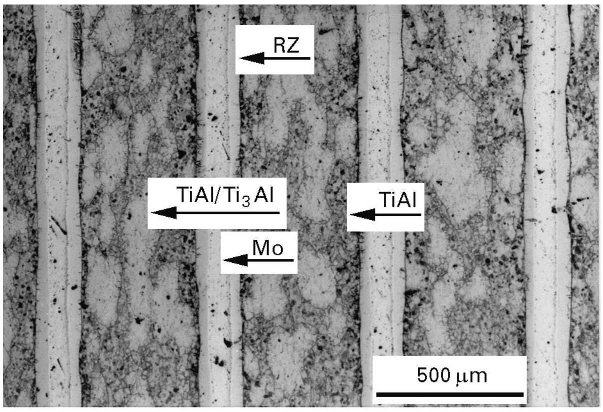

*Figure 3* Microstructure of the prepared TiAl/Mo specimen (RZ = reaction zone).

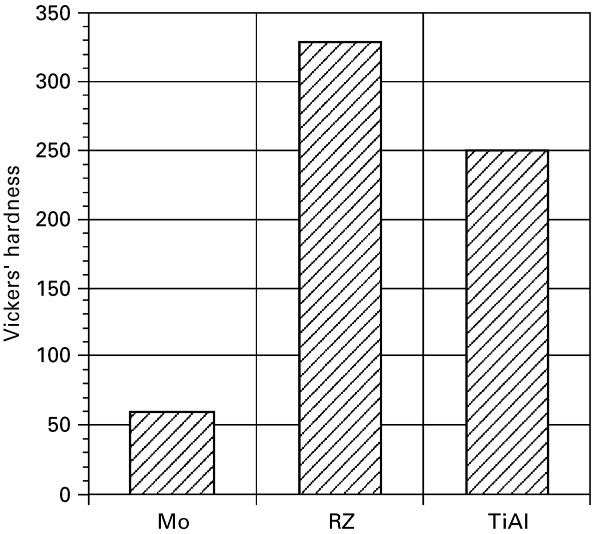

*Figure 4* Vickers' hardnesses of individual constituents in the TiAl/Mo composite (RZ = reaction zone).

that the reaction zone is more brittle than TiAl. Therefore, the growth of the reaction zone must be suppressed to achieve the desired ductilizing and toughening effect. In consideration of this, further investigations should be conducted to determine effective measures for suppression of the reaction zone growth.

## 3.2. TiAl/NiAl specimens

The particle size of powder mixtures after 5 h milling at −150 °C ranged from less than 1 μm up to 60 μm (Fig. 5). Relatively large particles were found upon closer inspection to be agglomerates of much smaller particles. No new phases were detected in milled powder mixtures by X-ray diffraction analysis. The crystallite sizes calculated from X-ray diffraction patterns using the Scherrer formula varied in the range 23–39 nm, larger than the critical size (about 21–22 nm for TiAl [15]) required for inverse Hall–Petch behaviour.

During consolidation at high temperatures, an apparent grain-growth process took place, resulting in crystallite sizes which are too large to be measured from X-ray diffraction patterns. As shown in Fig. 6, a typical microstructure of consolidated composite materials consists mainly of light grey TiAl grains and dark grey NiAl grains, with an apparent size ranging from less than 1 μm up to about 60 μm. This size is identical to the particle size of milled powders (see Fig. 5). Intergranular areas are mainly micropores and newly formed $NiTiAl_2$ or NiTiAl phases [14],

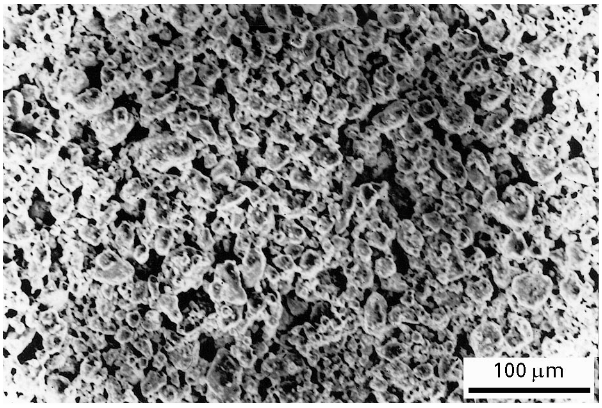

*Figure 5* Scanning electron micrograph of powder mixtures milled 5 h at −150 °C.

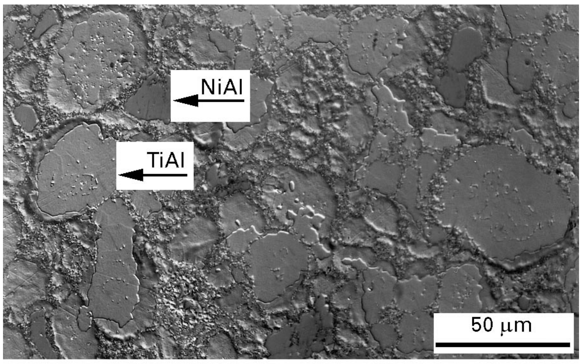

*Figure 6* Typical microstructure of consolidated TiAl/NiAl composites (50% TiAl + 50% NiAl, powders milled 5 h at −150 °C, consolidated at 1150 °C).

As shown in Fig. 7, the measured density of consolidated 50% TiAl + 50% NiAl specimens increases with increasing consolidation temperature. At 850 °C, the measured density was only about 81% of the theoretical density ($= 4.97\ \mathrm{g\,cm^{-3}}$ for 50% TiAl + 50% NiAl). This increased to about 92% at 1150 °C.

For a constant consolidation temperature of 1000 °C, the measured density increased with increasing NiAl amount (Fig. 8), simply due to the fact that the theoretical density of NiAl ($5.92\ \mathrm{g\,cm^{-3}}$) is higher than that of TiAl ($3.89\ \mathrm{g\,cm^{-3}}$). In fact, the consolidation of powder mixtures becomes more difficult with increasing amount of NiAl powder. This can be recognized by comparison of both curves in Fig. 8. The best consolidation is achieved for the powder mixture of 90% TiAl + 10% NiAl with a ratio of measured density to theoretical density of about 96%. This ratio becomes smaller with increasing NiAl amount, and is about 95% for 75% TiAl + 25% NiAl and about 88% for 50% TiAl + 50% NiAl.

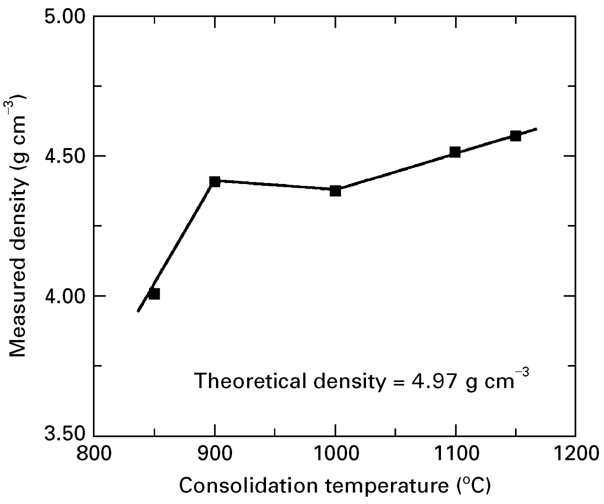

*Figure 7* Measured density of consolidated 50% TiAl + 50% NiAl as a function of consolidation temperature.

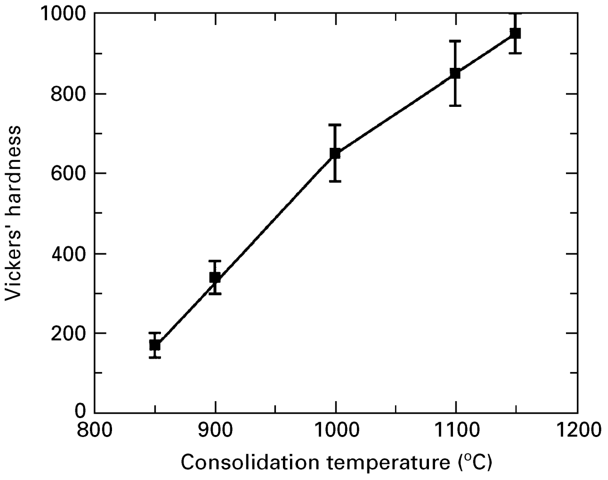

*Figure 9* Vickers' hardness of consolidated 50 TiAl + 50% NiAl as function of consolidation temperature.

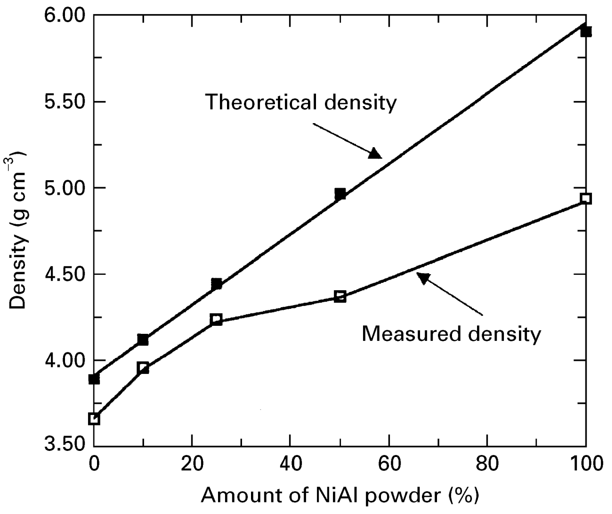

*Figure 8* Comparison of measured density and theoretical density of different TiAl/NiAl composites consolidated at 1000 °C.

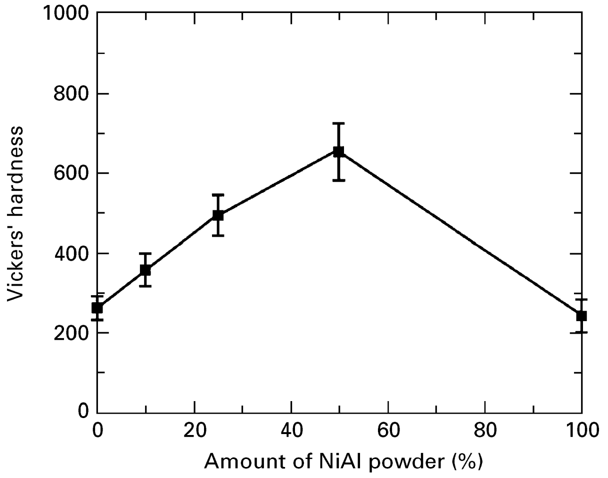

*Figure 10* Vickers' hardness of different TiAl/NiAl composites consolidated at 1000 °C.

The hardness of consolidated TiAl/NiAl materials is proved to be strongly dependent both on consolidation temperature and material composition. In Fig. 9, it can be seen that the hardness of consolidated 50% TiAl + 50% NiAl mixtures increases almost linearly with consolidation temperature. While a hardness of only 180 HV is measured after consolidation at 850 °C, the material consolidated at 1150 °C exhibits a hardness as high as 950 HV. For a constant consolidation temperature of 1000 °C, a clock-like curve of hardness versus the NiAl amount is established, with the maximum hardness of about 650 HV being achieved at 50% NiAl (Fig. 10).

By comparison of Fig. 9 with Fig. 7, one may jump to the conclusion that the increase in hardness is due to reduction of porosity, because both density and hardness increase with consolidation temperature. But this cannot account for the clock-like curve in Fig. 10, because the 50% TiAl + 50% NiAl composite with the maximum hardness only exhibits the lowest ratio of measured density to theoretical density. Therefore, we believe there is another more important factor controlling the hardness. This is the amount of newly formed ternary $NiTiAl_2$ or NiTiAl phases with high hardnesses. Because these phases form through reactions between TiAl and NiAl, their amount in consolidated materials must be enhanced not only by increase in the consolidation temperature but also by enlargement of the total contact area between TiAl and NiAl. Assuming the degree of mixing is the same for all three compositions, then the 50% TiAl + 50% NiAl powder mixture has the largest total contact area between TiAl and NiAl and thus the highest hardness for constant consolidation temperature (see Fig. 10).

The ternary phases with high hardnesses are surely beneficial for improvement of strength properties. However, in view of ductility and fracture toughness, these hard ternary phases are undesirable. In fact, the indentation fracture toughness of the TiAl/NiAl composites has been found to decline with increasing amount of ternary phases [14]. On this account, a low consolidation temperature should be used. The problem of porosity at low temperature can then be solved by applying a high pressure during consolidation.

## 4. Conclusions

TiAl/Mo and TiAl/NiAl composites have been prepared and investigated. The important results obtained in this study can be summarized as follows.

The lower hardness value measured for incorporated molybdenum implies that ductilizing and toughening of TiAl by introducing molybdenum ductile reinforcement are possible.

The hardness of TiAl/NiAl composites varies strongly with consolidation temperature and composition. Possible factors contributing to the hardness of the composites are the amount of the hard ternary phases and the material density, with the former being the major factor controlling the hardness.

Both composites contain hard ternary phases formed through reactions of TiAl with molybdenum and NiAl, respectively. The formation of these phases is detrimental for ductility and fracture toughness, and should therefore be suppressed through measures which are still to be determined by further experiments.

## Acknowledgements

The authors thank Mr F. Pirwitz, GKSS Research Center, Germany, Mr F.-Y. Hsu, Technical University Aachen, Germany, and Mr N. McMinn, University Calgary, Canada, for conducting part of the experimental work. The financial support of the Natural Science Foundation of Zhejiang Province, People's Republic of China, for one of us (G.-X. Wang) is also gratefully acknowledged.

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*Received 23 August 1995*
*and accepted 12 August 1996*