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Near-net shape processing of spherical high Nb-TiAl alloy powder by gelcasting

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Abstract: Spherical Ti-45Al-8.5Nb-(W,B,Y) alloy powder prepared by an argon plasma process was near-net shape by gelcasting. In the non-aqueous system, methaerylate-2-hydroxy ethyl, toluene, benzoyl peroxide, and *N*,*N*-dimethylaniline were used as the monomer, solvent, initiator, and catalyst, respectively. To improve sintering and forming behaviors, many additives were included in the suspension. The concentrated suspension with a solid loading of 70vol% was prepared. The high Nb-TiAl powder was analyzed by electron microscopy and X-ray diffraction. It was found that the green bodies had a smooth surface and homogeneous microstructure, exhibiting a bending strength as high as 50 MPa. After sintering at 1480°C for 2 h in vacuum, uniform complex-shaped high Nb-TiAl parts were successfully produced.

Keywords: titanium aluminum alloys; niobium alloys; powders; gelcasting; microstructure

1. Introduction

Strong, lightweight and high-temperature structural aerospace alloys have led to development of titanium-based alloys with a high concentration of aluminum. TiAl-based alloys have shown low density, relatively good oxidation resistance, high strength at room temperature, and good creep behavior at elevated temperatures, which make them promising for high-temperature applications [1-4]. It has been found that Nb is the essential and effective element to improve their mechanical properties, especially the high-temperature strength [5]; and the oxidation behavior of TiAl alloys is improved by Nb addition [6-7].

Compared with the microstructures of Y-free TiAl alloys after deformation, the grain sizes of Y-containing TiAl alloys were much finer, and the addition of 0.3at% Y decreased the flow stress and the deformation activation energy of TiAl alloys [8-9]. Boron is a fast diffuser through successive jumps between Ti- and Al-rich interstitial sites in γ-TiAl [10-12]. There are three traditional methods to shape TiAl alloys: forging, powder metallurgy (PM), and casting. It is difficult for TiAl alloys to take shape by forging due to thier inherent poor deformability and high material loss in machining processes [13].

Gelcasting is an attractive near-net-shape ceramic forming technique for making high-quality complex-shaped ceramic parts. Among various ceramic manufacturing techniques, gelcasting has been recognized as a very high-potential process: controllable casting, rapid forming cycle, and minimal molding defects. The process has been widely used in ceramics, and it is also significant to apply in the forming of metal products. In this process, high solid loading slurry is obtained by dispersing the powders in a premixed monomer solution and then cast in a mould of the desired shape [14]. After adding an initiator, a macromolecular network is created to hold metal particles very near each other and green bodies that have excellent and mechanical properties but contain a few percent of polymer can be obtained [15-16]. Remarkable advantages were clear in this process, for example, dried green bodies can be sintered directly because they contain less binder, and complex-shaped bodies can be fabricated at low cost. The gelcasting processed metal part is heated to burn out the polymer gel and is subsequently sintered for densification. The final product is a near-net-shape part that requires very little machining.

Therefore, the gelcasting technology was investigated in the manufacture of high Nb containing TiAl alloys with spherical Ti-Al-Nb alloy powders. Based on *in-situ* poly-

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merization of organic monomer binders, the macropolymeric network was formed by powder particles together. The effects of gelcasting parameters on the green body and mechanical properties of high Nb containing TiAl alloys were discussed in detail.

2. Experimental

High Nb containing TiAl alloy powder was used in this experiment, and its nominal composition (at%) is Ti-45Al-8.5Nb-(W,B,Y). The powder was prepared by an argon plasma process; in this way, the particle size was controlled, and there was a narrow range of particle size distribution, with free holes inside; and the alloy powder is spherical with high-tap density. In the present study, the average particle size of the alloy powder is 100 μm, the oxygen content is 3.8×10^9, and it also contains small amounts of nitrogen and carbon. The alloy powder consists mainly of α_2 phase and a small amount of β phase, showing a branched surface and segregating Al-rich phase within the network. The SEM micrograph of the high Nb containing TiAl alloy powder is shown in Fig. 1.

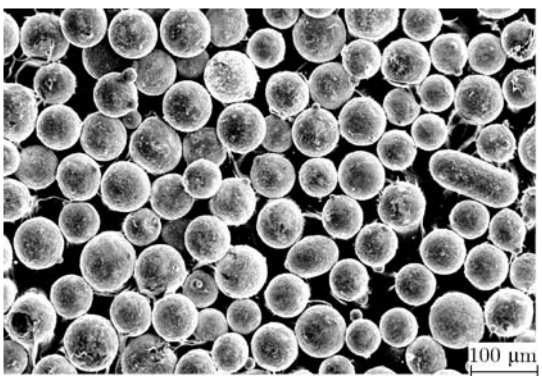

Fig. 1. SEM micrograph of the high Nb containing TiAl alloy powder.

The monomer in the non-aqueous system is methaerylate-2-hydroxyethyl (HEMA) and the solvent is toluene. The initiator and catalyst are benzoyl peroxide (BPO) and *N*, *N*-dimethylaniline, respectively. The dispersant is oleic acid.

Fig. 2 shows the gelcasting process in the nonaqueous system. First, HEMA and toluene were mixed with a certain ratio to make the gel former and a premixed solution, and then high Nb containing TiAl powder was added to make metal slurry. The slurry with good liquidity and high solid volume fraction was prepared. To improve solid loading, oleic acid as a dispersant was added to the slurry, and ammonia was added to adjust the pH values within 9-10. The slurry was then degassed in vacuum for 5 min after the initiator and catalyst were added. Afterwards, the slurry was poured into a mold, and the temperature was held at 30°C for 2 h to ensure that the gel reaction processed sufficiently, and the slurry was solidified in required shape and size by the mold. After demolding, a green body of the high Nb containing TiAl alloy was obtained; the wet green body was dried in a vacuum oven at 60°C for 24 h and then sintered at 1480°C for 2 h in vacuum.

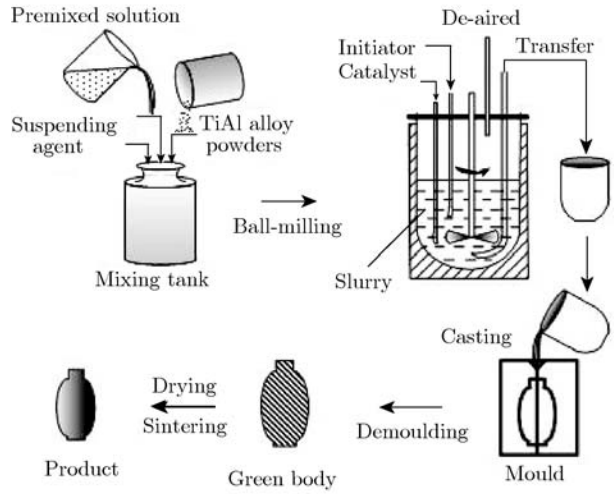

Fig. 2. Schematic representation of the gelcasting process.

By changing the parameters of solid loading, the monomer content of the premixed solution, and sintering conditions, the volume of the polymeric network among powder particles can be varied, resulting in more porous or dense structures. Moreover, the pores can be smooth and oriented by changing the sintering condition, leading to a three-dimensional interconnected porosity. Solid loading of 70vol% was chosen to create the desired porosity. The sintering temperature of 1480°C for 2 h in vacuum was used to fabricate uniform microstructures.

The apparent viscosity of TiAl slurries was measured with a NDJ-1 rotational viscometer at a shear rate of $60\ s^{-1}$. The bending strength of green bodies, which were cut to a size of 4 mm × 4 mm × 46 mm (span of 30 mm, GB/T 228—2002), was measured by the three-point bending test performed using an Instron material test system (CMT4305) at a loading rate of 0.5 mm/min. The sintering of the metal body was accomplished in a vacuum resistance furnace. Microstructures of TiAl powders and the cross-section morphology of the samples were observed by scanning electric microscopy (SEM, Cambridge S-250MK2). Constituent phases of the sample were identified using a D/MAX RB X-ray diffractometer with Cu K_α radiation. The density of sintered samples was determined using the Archimedes' principle. The compression test for sintered cylindrical TiAl specimens of ϕ10 mm × 20 mm (GB 7314—87) was also measured by the ignitron material test system (CMT4305). For SEM examination, the polished surfaces of the sintered solid samples were etched with the polishing solution (10vol% HNO_3+ 5vol% HF + 85vol% H_2O).

3. Results and discussion

3.1. Characterization of the high Nb-TiAl gelcasting process

The slurry is cast in a nonporous mould, wherein it is subsequently set by gel reaction for gelcasting. In general, the apparent viscosity of the slurry increases gradually following by increasing the solid loading. When the solid loading exceeds a critical value, the apparent viscosity takes a sharp rise.

Accordingly, the density of green bodies depends on solid loading. It is beneficial for obtaining high solid loading to control the final geometry of the part and to achieve a higher dimensional accuracy by minimizing the possible distortion during drying and sintering. On the other hand, shaping of intricate parts requires the slurry to pour the moulds easily. It means that the viscosity of the slurry has to be kept below some acceptable levels. The slurry with low viscosity, generally below 1 Pa·s at the shear rate of 60 s^{-1}, is required to ensure the mould filled well with the slurry without outside force for gelcasting [17-18].

Fig. 3 shows the evolution of the viscosity of a slurry (the volume ratio between HEMA and toluene is 1.0) with solid loading. The high Nb-TiAl powder was uniformly spherical with good liquidity. The solid loading can be as high as 70vol%. As shown in Fig. 3, the slurry viscosity takes the greatest change when the solid loading is between 65vol%-70vol%. The apparent viscosity comes to 1.05 Pa·s with the solid loading of 70vol%.

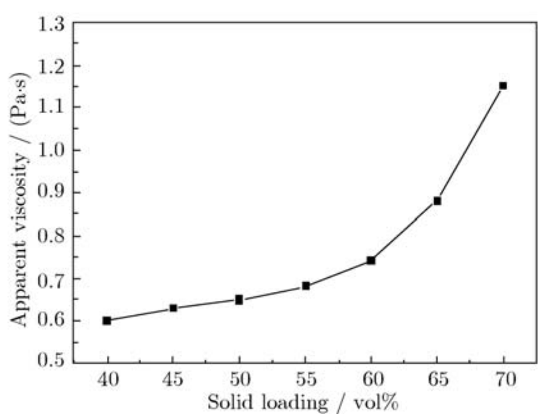

Fig. 3. Effect of solid loading on the apparent viscosity of high Nb-TiAl slurries.

Monomer concentration has great effect on the green body strength. Fig. 4 shows the effect of the volume ratio between HEMA and toluene in the slurry on the dry green strength. It is found that the bending strength of the dry green body increases with the monomer content, and when the content is 70vol%, the strength obtained is 50 MPa. Following with increasing the content of HEMA, the three-dimensional network of the green body was more closely connected. The three-dimensional network of the green body determines its strength, and the denser the network structure is, the less the powder is carried.

The relationship between monomer concentration and sintered body density at 1480°C for 2 h in vacuum is shown in Fig. 5. It shows that the density of sintered body at first increases and then decreases with the increasing volume ratio of HEMA to toluene. Therefore, the ratio should be right to meet the requirements of green body strength and to ensure good contact between the powders to get a better sintered body. The strength of the green body was about 50 MPa with the monomer concentration of 50%, and the density of the high Nb-TiAl alloy sintered at 1480°C for 2 h in vacuum was 4.08 g/cm^3 (the theoretical density of Ti-45Al-8.5Nb-(W,B,Y) (at%) is 4.11 g/cm^3).

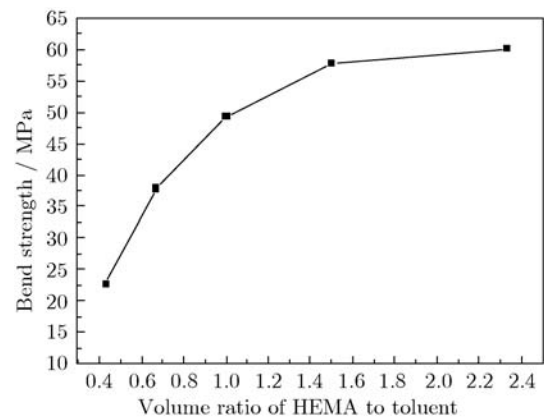

Fig. 4. Effect of the volume ratio between HEMA and toluene on the bending strength of high Nb-TiAl samples.

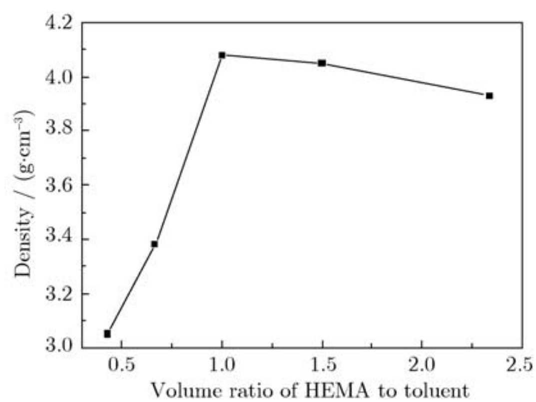

Fig. 5. Effect of the volume ratio between HEMA and toluene on the density of high Nb-TiAl samples.

3.2. Microstructure of the sintered high Nb-TiAl alloy

The XRD pattern shows the phase compositions of Ti-45Al-8.5Nb-(W,B,Y) alloys after sintering at 1480°C for 2 h in vacuum (as shown in Fig. 6). It mainly consists

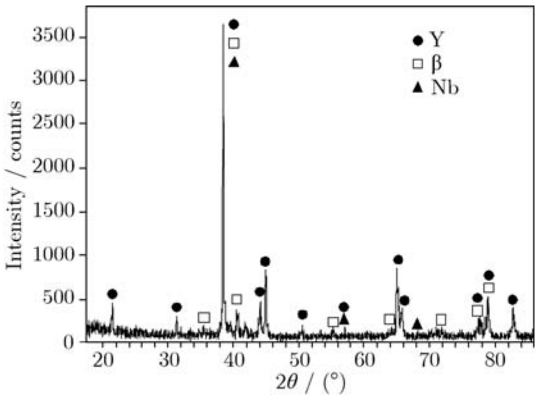

Fig. 6. XRD pattern of high Nb-TiAl samples by gelcasting (γ: TiAl; β: Ti_2Al).

of γ-TiAl phase, β-Ti_2Al phase, and residual Nb. It suggests that the reactive ability between Ti and Al elements is dominant in the Ti-Al-Nb ternary system due to their different diffusivities.

The micrographs of the high Nb-TiAl alloy by gelcasting are shown in Fig. 7. It is shown that the sample is almost fully densified and does not show any obvious pores in the microstructure. The equiaxed TiAl phase, Ti_2Al phase, and Nb-island are observed and the samples exhibit uniform microstructures. The white regions are residual Nb powders, the gray region located at the edge of the white regions is Nb dissolved by Ti_2Al phase, and the black region is TiAl phase.

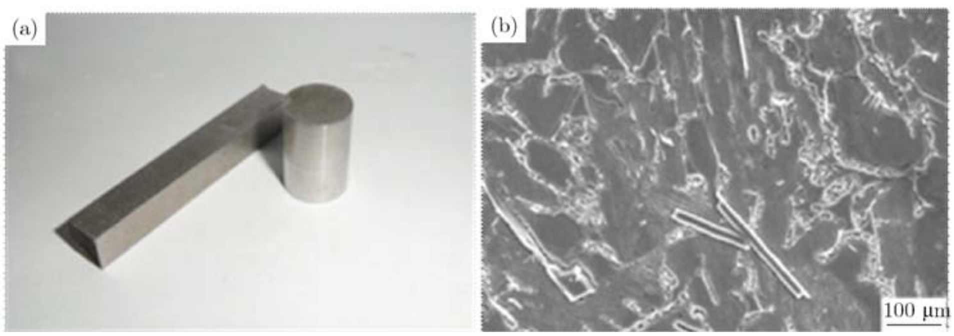

Fig. 7. Sample image (a) and SEM micrograph (b) of the high Nb-TiAl alloy by gelcasting.

4. Conclusion

The non-aqueous gelcasting system has been successfully applied to forming complex-shaped high Nb-TiAl parts. The maximum solid loading is 70vol% for the spherical powder. The volume ratio between HEMA and toluene has great effect on the properties of solidified colloid. The green bodies have been polymerized through HEMA and have a smooth surface and homogeneous microstructure, exhibiting a bending strength as high as 50 MPa. It should be emphasized that the combination of plasma spheroidization and gelcasting is effective to obtain high-performance near-net shape products. After sintering at 1480°C for 2 h in vacuum, uniform complex-shaped high Nb-TiAl parts with the density of 4.08 g/cm^3 were successfully obtained.

Acknowledgements

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