

Effect of Sn on Sintering Densification and Mechanical Properties of TiAl-Based Alloys (Post-print)

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Abstract

Using TiAl-8.5Nb pre-alloyed powder as the raw material and adding 1at.% Sn powder as a sintering strengthening agent, high-density high-niobium TiAl alloys were prepared by pressureless sintering technology to investigate the influence of Sn addition on the sintering densification process, microstructure, and mechanical properties of TiAl-based alloys. The study reveals that: the addition of 1at.% Sn element can reduce the sintering densification temperature of high-niobium TiAl alloy powder, increase the density and linear shrinkage rate of the sintered compact, thereby facilitating the reduction of alloy microstructure grain size and improvement of comprehensive properties. After Sn addition, when the alloy powder is sintered at 1500°C/2h, its density can reach 99.1% and the linear shrinkage rate reaches 9.3%; the alloy microstructure exhibits a uniform and fine α/β fully lamellar structure with a lamellar colony size of 40-60 nm; Sn element is mainly dissolved in the β phase, causing its axial ratio c/a value and unit cell volume to increase; the prepared alloy demonstrates a Rockwell hardness of 50.1 HRC, compressive strength of 2938 MPa, yield strength of 680 MPa, and compression rate of 29.1%, with all performance metrics exceeding those of the Sn-free high-niobium TiAl-based alloy.

Full Text

Effect of Sn Addition on Densification and Mechanical Properties of Sintered High Nb Containing TiAl Alloys

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Abstract

High Nb containing TiAl alloys (TiAl-Nb) are a new generation of materials for high temperature structural applications because of their low density, high strength and corrosion resistance at elevated temperatures. The alloys can be processed by powder metallurgy (PM) which have more advantages including low cost-effectiveness, near net forming for complex parts with fine grain size and uniform microstructure. However, the alloy powders are difficult to achieve full densification due to their lower sintering activity, which impairs the mechanical performance of sintered parts. The present work focuses on the densification performance of TiAl-Nb alloy powders with 1%Sn (atomic fraction) as sintering aids. The effects of Sn addition on the sintering densification process, microstructure and mechanical properties of sintered alloys were investigated. The results show that 1%Sn addition can significantly reduce the sintering densification temperature of alloy powders, and increase the relative density and linear shrinkage of sintered parts. This contributes to control microstructure grain size and improve the comprehensive properties. Sintered with 1500 °C for 2 h, 1% Sn containing TiAl-Nb based alloys show the best densification performance, with the relative density of 99.1% and linear shrinkage of 9.3%. The alloy samples exhibit fine and uniform full lamellar microstructure, and α_2 lamellar colonies are sized about 40-60 nm. Sn mainly dissolved into β phase, leading to the enhancement of axial ratio c/a and unit cell volume. The sintered TiAl-Nb-1Sn samples have been found to possess superior room-temperature mechanical properties, with a rockwell hardness of 50.1 HRC, a compressive strength of 2938 MPa, a yield strength of 680 MPa, and a compression ratio of 29.1%, which is obviously higher than those of TiAl-Nb alloys.

KEY WORDS TiAl alloys, powder metallurgy, Sn, sintering densification

Introduction

TiAl-based alloys are promising lightweight high-temperature structural materials for aerospace and automotive applications due to their low density, high strength, high elastic modulus, and excellent oxidation and creep resistance at elevated temperatures[1~3]. Building upon this foundation, high Nb-containing TiAl alloys with independent intellectual property rights developed in China have further increased the service temperature of conventional TiAl alloys and have become internationally recognized as a major development direction for high-temperature TiAl alloys[4~6]. However, low room-temperature ductility, difficult plastic processing and forming, and high manufacturing costs remain bottlenecks restricting large-scale industrial application of TiAl alloys, and the

addition of high-melting-point Nb further exacerbates these problems. In recent years, alloying and thermomechanical processing to control microstructure have effectively improved the room-temperature ductility of high Nb-containing TiAl alloys, but the forming difficulties remain unresolved[7~9].

Among various manufacturing methods, powder metallurgy (PM) technology offers unique advantages for near-net-shape fabrication of small complex parts, while also producing alloys with uniform composition, fine microstructure, and lower processing costs[10~12]. However, due to the low sintering activity and difficult densification of alloy powders, the mechanical properties of PM TiAl-based alloys cannot be fully realized[13~15]. Therefore, achieving pressureless sintering densification of TiAl-based alloy powders is a key issue in the development of PM TiAl-based alloys.

Enhanced sintering technology can be used to improve the sintering activity of high-melting-point powders. Its principle is to provide rapid diffusion channels between powder particle interfaces through a second phase to achieve sintering enhancement[16]. Currently, enhanced sintering technology has been widely applied in many material systems, such as W-Cu, Mo-Si-B, 8YSZ, TiB₂, etc.[17~20]. However, relevant research on its application to TiAl-based alloy powders is limited. Xia et al.[21~23] investigated the effects of Ni, Co, and Cu metal powders on the sintering performance of TiAl-based alloy powders, with preliminary results showing that micro-additions below 2% (atomic fraction, similarly hereinafter) could significantly promote sintering densification and also improve room-temperature mechanical properties to some extent. Soyama et al.[24] added 1%~5% Zr to TiAl-based alloy powders and found that with increasing Zr content, the porosity and densification temperature of samples decreased significantly, while hardness and creep resistance increased. These preliminary results confirm the feasibility of achieving enhanced sintering of TiAl-based alloys through the addition of certain micro-alloying elements, but extensive research is still needed on the selection of enhanced sintering elements and their influence mechanisms on the matrix. Among various micro-alloying elements, low-melting-point Sn is also a beneficial element for improving the comprehensive properties of TiAl alloy matrices. He Yuehui et al.[25] studied the effects of 0.15%~0.72% Sn (atomic fraction, similarly hereinafter) on the microstructure and properties of as-cast Ti-48Al alloys, finding that after adding 0.37% Sn, the alloy exhibited fine equiaxed grains with smooth and widened grain boundaries, and its room-temperature bending strength and ductility were significantly improved; when Sn content exceeded 0.60%, mechanical properties decreased due to the formation of a Sn-rich reticular third phase from non-uniform microstructure. Shida and Anada[26] studied the influence of Sn and other elements on the oxidation behavior of Ti-48.6Al binary alloys, confirming Sn as a neutral element with no adverse effect on high-temperature oxidation resistance. Wang et al.[27] reported on the microstructure and mechanical properties of cast Ti-(46-x)Al-xSn (x=0~8%, atomic fraction) alloys, with results showing that when Sn content was 1%, the as-cast alloy exhibited optimal comprehensive mechanical properties after homogenization heat treat-

ment. Currently, no relevant reports have been found on using Sn element as an enhanced sintering aid for alloy powders.

Experimental Procedures

In this work, using gas-atomized Ti-45Al-8.5Nb-0.2W-0.2B-0.02Y (atomic fraction, %, similarly hereinafter) pre-alloyed powder as raw material and adding 1% Sn powder as an enhanced sintering agent, we investigated the effects of Sn element on the sintering densification process of TiAl-based alloy powders, as well as on the matrix microstructure and mechanical properties.

Alloy ingots with a nominal composition of Ti-45Al-8.5Nb-0.2W-0.2B-0.02Y were prepared using a water-cooled copper crucible induction melting furnace, and high Nb-containing TiAl alloy powders were produced by crucible-free induction heating continuous Ar gas atomization. The alloy powders (37 μm) were uniformly mixed with Sn powder (purity 99.9%, 20 μm) at (0, 1%, atomic fraction), with a nominal composition of Ti-45Al-8.5Nb-0.2W-0.2B-0.02Y-xSn (abbreviated as TiAl-xSn, x=0, 1).

A forming agent of 1.5% (mass fraction) paraffin was added to the mixed powders, which were then die-pressed at a pressure of 1200 MPa. The green compacts were placed in a tube furnace and pressurelessly sintered under high-purity Ar protection. The temperature was first raised to 450 °C and held for 1 h for paraffin removal, then increased to the sintering temperature of 1440~1520 °C and held for 2 h, with a heating rate of 4 °C/min throughout. The sintered alloy components were obtained after furnace cooling.

The density of samples was measured using the Archimedes drainage method. Powder morphology and microstructure were observed using a BH200M optical microscope (OM) and a JSM-6480LV scanning electron microscope (SEM). The chemical composition of microstructures was analyzed using a Link-860 energy dispersive spectrometer (EDS). Phase analysis was performed using a Dmax-RB rotating anode X-ray diffractometer (XRD, CuK α , wavelength =0.15406 nm). Rockwell microhardness (HRC) of samples was measured using a benchtop HDI-1875 hardness tester. The alloy blocks were cut by electric spark wire cutting into compression test specimens with dimensions of diameter 3 mm and length 5 mm. Compression tests were conducted on an INSTRON universal material testing machine at a deformation rate of 0.002 s⁻¹. The compressive strength, yield strength, and compression ratio of specimens were determined from their engineering stress-strain curves.

2.1 Effect of Sn Addition on Alloy Density and Hardness

Figure 1 [Figure 1: see original paper] shows SEM images of the raw powders. The high Nb-containing TiAl alloy powder is spherical with an average particle size of 38 μm , while the Sn powder is irregularly shaped with an average particle size of 18 μm . The raw mixed powders have good fluidity but are difficult to form during compaction. Adding 1.5% paraffin as a forming agent ensures

successful compaction and increases the relative density of the green compacts to approximately 75%.

Figure 2 [Figure 2: see original paper] presents the influence of sintering temperature on the relative density and linear shrinkage of TiAl-xSn ($x=0, 1$) alloys. As shown in the figure, with increasing sintering temperature, the linear shrinkage and relative density of both alloy compositions continuously increase. At the same sintering temperature, the density and linear shrinkage of alloys with Sn addition are significantly higher than those without Sn. For the TiAl-0Sn alloy, when the sintering temperature is 1520 °C, the sample achieves a relative density of 97.9%. In contrast, the TiAl-1Sn alloy sintered at 1500 °C reaches a relative density of 99.1% with a linear shrinkage of 9.3%, demonstrating significantly higher densification than the TiAl-0Sn alloy prepared at 1520 °C. However, when the sintering temperature is further increased to 1520 °C, the TiAl-1Sn alloy samples experience slight melting. Evidently, adding 1% Sn reduces the sintering densification temperature and increases the density of alloy powders, indicating that Sn addition can promote the sintering densification process between alloy powder particles and exert its sintering strengthening effect.

Figure 3 [Figure 3: see original paper] shows the influence of sintering temperature on the Rockwell hardness of TiAl-xSn ($x=0, 1$) alloys. The variation of hardness with temperature follows a trend similar to that of sample density. The hardness of TiAl-0Sn alloy continuously increases with sintering temperature, stabilizing at approximately 46.8 HRC when the temperature reaches 1500–1520 °C. After adding 1% Sn, the hardness of TiAl-1Sn alloy prepared at the same temperature is generally higher than that of TiAl-0Sn alloy, peaking at approximately 50.1 HRC for the sample sintered at 1500 °C. Further increasing the sintering temperature leads to a decline in hardness.

2.2 Effect of Sn Addition on Microstructure

Figure 4 [Figure 4: see original paper] shows XRD spectra of TiAl-xSn ($x=0, 1$) alloys sintered at different temperatures. As seen in the figure, the phase compositions of TiAl-xSn ($x=0, 1$) alloys obtained at different sintering temperatures show basically no difference, consisting of γ -TiAl phase and β -Ti₃Al phase. No diffraction peaks of elemental Sn were detected in the TiAl-1Sn alloy either. Additionally, through micro-analysis comparing the XRD spectra of TiAl-xSn ($x=0, 1$) alloys, the β -phase diffraction peaks of TiAl-1Sn alloy were found to shift leftward compared with TiAl-0Sn alloy, while the diffraction peak angles of the β phase showed no obvious change, indicating that the added Sn element mainly dissolved in the β phase and caused lattice expansion of the β phase[25,28]. Further calculations from XRD analysis results show that for the TiAl-0Sn alloy sintered at 1500 °C, the β -phase unit cell parameters are $c=(0.40705\pm 0.00013)$ nm, $a=(0.40029\pm 0.00018)$ nm, $c/a=1.0169\pm 0.0008$, with a unit cell volume of $(6.518\pm 0.004)\times 10^{-23}$ nm³. After doping with 1% Sn, the β -phase unit cell parameters in the alloy become $c=(0.40827\pm 0.00008)$ nm, $a=(0.40081\pm 0.00012)$

nm, $c/a=1.0186\pm 0.0005$, with a unit cell volume of $(6.543\pm 0.006)\times 10^{-2}$ nm³. Evidently, Sn doping increases the lattice constant of the β phase, while also increasing the axial ratio c/a and unit cell volume.

Figure 5 [Figure 5: see original paper] shows SEM images of TiAl-xSn ($x=0, 1$) alloys sintered at different temperatures. As can be seen from the figures, at lower sintering temperatures (1440 °C), the TiAl-0Sn alloy still contains numerous original particle boundaries, with sintering necks forming between spherical powder particles during sintering and particle spacing shrinking to form a continuous pore network. In contrast, after adding 1% Sn, the alloy porosity decreases substantially, pores become smaller and tend toward near-spherical shapes, and a small amount of bright white regions exist at powder particle boundaries, which EDS analysis identifies as Sn-rich phases.

When the sintering temperature increases to 1500 °C, the prepared TiAl-0Sn alloy consists of 2/ full lamellar structure with lamellar colony sizes of 80~100 μ m, and still contains many small pores. The TiAl-1Sn alloy, however, is nearly fully dense, with significantly finer full lamellar colony sizes of approximately 40~60 μ m. When the sintering temperature further increases to 1520 °C, the microstructure of TiAl-0Sn alloy shows no obvious change, with only a slight reduction in pores, while the lamellar colonies of TiAl-1Sn alloy become coarser and the lamellar spacing increases noticeably.

The reactive diffusion process between Sn and the TiAl matrix can be analyzed in conjunction with the Ti-Al-Sn ternary phase diagram. In the early stage of sintering, the addition of low-melting-point Sn component can form a ternary eutectic liquid phase with the matrix at relatively low temperatures. The liquid phase spreads on the surface of alloy powder particles, promoting particle rearrangement and providing rapid diffusion channels for matrix material migration, thereby accelerating the matrix material transport process. When the sintering temperature is low (1440 °C), the diffusion process is insufficient, and Sn remains enriched at particle boundaries. When the sintering temperature is further increased to 1500 °C, diffusion becomes complete, resulting in uniform distribution of Sn element throughout the full lamellar alloy matrix.

2.3 Effect of Sn Addition on Mechanical Properties

Figure 6 [Figure 6: see original paper] illustrates the relationship between room-temperature compression properties of TiAl-xSn ($x=0, 1$) alloys and sintering temperature. As shown in the figure, with increasing sintering temperature, both the compressive strength and yield strength of the two alloy types show an upward trend. At the same sintering temperature, the strength properties of TiAl-1Sn alloy are generally higher than those of TiAl-0Sn alloy, with a slight improvement in compression ratio as well. For TiAl-0Sn alloy, the mechanical properties are optimal for the sample sintered at 1520 °C, slightly higher than that prepared at 1500 °C, with compressive strength and yield strength of 2870 MPa and 595 MPa, respectively, and a compression ratio of 28.3%. For TiAl-

1Sn alloy, the properties reach optimum for the sample sintered at 1500 °C, with values of 2938 MPa, 680 MPa, and 29.1%, respectively. The room-temperature compression engineering stress-strain curves of the two samples are shown in Figure 7 [Figure 7: see original paper]. It can be seen that the room-temperature compression properties of TiAl-1Sn alloy sintered at lower temperatures are significantly higher than those of TiAl-0Sn alloy.

After adding 1% Sn element, the mechanical properties of the alloy are comprehensively improved. Besides improving alloy densification, Sn doping can also provide certain strengthening and toughening effects on the TiAl alloy matrix. SEM and XRD results indicate that Sn element dissolves in the TiAl-based alloy, changing the lattice constants of the alloy's β phase and increasing the axial ratio c/a , which enhances the crystal structure anisotropy and thereby improves alloy strength and hardness values. Additionally, Sn addition refines grains to some extent, increasing the number of grain boundaries and hindering crack propagation, thus producing a fine-grain strengthening effect while improving both alloy strength and ductility. These research results are consistent with reports by Soyama et al.[24] and Wang et al.[27].

2.4 Effect of Sn Addition on Fracture Morphology

Figure 8 [Figure 8: see original paper] shows SEM images of fracture surfaces of TiAl-xSn ($x=0, 1$) alloys after compressive fracture. The results indicate that both TiAl-xSn ($x=0, 1$) alloys exhibit relatively rough fracture surfaces with no obvious essential difference in morphology, both showing typical transgranular cleavage fracture characteristics of full lamellar structures, including three modes: trans-lamellar fracture of lamellar colonies, cracking along lamellar interfaces, and stepwise fracture. During the fracture process, microcracks generally nucleate at β/α lamellar interfaces.

When cracks encounter grains with small angles between lamellar orientation and crack direction, they choose to propagate along lamellar interfaces with less resistance. When cracks encounter grains with large angles between lamellar orientation and crack direction, they first choose to propagate through lamellae along directions favorable to the stress state. When cracks encounter lamellar colonies at intermediate angles, stepwise propagation may occur[29]. Therefore, the fracture mechanism of the alloy microstructure is a mixed brittle cleavage fracture of the above three modes. When colony sizes are relatively coarse, crack propagation speed is faster, whereas TiAl alloys with fine lamellae have more β/α interfaces on the same stress cross-section, which hinders crack propagation. Consequently, the TiAl-1Sn alloy with finer grains exhibits slightly higher strength than the TiAl-0Sn alloy.

3 Conclusions

- (1) Adding 1% Sn can reduce the sintering densification temperature of TiAl-based alloy powders and increase the relative density and linear shrinkage

of sintered compacts, effectively promoting the sintering densification process of alloy powders. Specifically, the TiAl-1Sn alloy sintered at 1500 °C for 2 h achieves a relative density of 99.1% and a linear shrinkage of 9.3%.

- (2) Adding 1% Sn helps refine lamellar colony sizes. The TiAl-1Sn alloy prepared by sintering at 1500 °C exhibits a uniform full lamellar structure with an average lamellar colony size of 40~60 nm. The Sn element mainly dissolves in the β phase, increasing its axial ratio c/a and unit cell volume.
- (3) Adding 1% Sn helps improve the hardness, room-temperature strength, and plasticity of TiAl-based alloys. The TiAl-1Sn alloy sintered at 1500 °C demonstrates optimal mechanical properties with a Rockwell hardness of 50.1 HRC, compressive strength of 2938 MPa, yield strength of 680 MPa, and compression ratio of 29.1%, which are significantly superior to those of Sn-free high Nb-containing TiAl-based alloys.
- (4) Sn addition shows no obvious effect on the fracture mode of TiAl-based alloys. Both TiAl-xSn ($x=0, 1$) alloys exhibit three fracture modes: trans-lamellar fracture of lamellar colonies, cracking along lamellar interfaces, and stepwise fracture, which are typical of transgranular cleavage brittle fracture.

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