

Fabrication of Powder Metallurgy Ti-47Al-2Cr-2Nb-0.15B Alloy and Factors Influencing Its Mechanical Properties (Postprint)

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Date: 2023-03-18T00:00:00+00:00

Abstract

Pre-alloyed powder with a nominal composition of Ti-47Al-2Cr-2Nb-0.15B (at%) was prepared by crucible-free induction melting ultrasonic gas atomization and characterized. The room-temperature tensile ductility and high-temperature stress-rupture life of powder γ -TiAl alloy under two different pretreatment regimes were compared. The results demonstrate that vacuum degassing pretreatment reduces the size and quantity of pore defects in powder γ -TiAl alloy, thereby enhancing the stability of room-temperature ductility and high-temperature stress-rupture life. To investigate the effect of can materials on the microstructure and mechanical properties of powder γ -TiAl alloy, low-carbon steel and commercially pure titanium (CP-Ti) were selected as encapsulation materials. The results reveal that under hot isostatic pressing (HIP) at 1260°C, significant reactive diffusion occurs between the low-carbon steel can and the γ -TiAl alloy, with the resulting pore defects deteriorating the mechanical properties of the material. Under HIP at 1230°C, CP-Ti exhibits a lower pressure shielding effect on HIP pressure compared with low-carbon steel, the CP-Ti/ γ -TiAl compact undergoes more complete plastic deformation, and the powder γ -TiAl alloy exhibits higher strength. The powder γ -TiAl alloy is texture-free, fine-grained, and homogeneous, with tensile properties superior to cast γ -TiAl alloy.

Full Text

Preamble

Vol. 29 No. 2

CHINESE JOURNAL OF MATERIALS RESEARCH

February 2015

Preparation of Powder Metallurgy Ti-47Al-2Cr-2Nb-0.15B Alloy and Analysis of Factors Influencing Mechanical Properties

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Supported by National High Technology Research and Development Program of China No.2013AA031606.

Abstract

Pre-alloyed powders with a nominal composition of Ti-47Al-2Cr-2Nb-0.15B (at%) were prepared by electrode induction melting gas atomization and characterized comprehensively. The room-temperature tensile ductility and high-temperature rupture life of powder γ -TiAl alloys were compared under two different pretreatment conditions. Results demonstrate that vacuum degassing pretreatment reduces the size and quantity of porosity defects in powder γ -TiAl alloys, thereby improving the stability of room-temperature ductility and extending high-temperature rupture life.

To analyze the influence of container materials on the microstructure and mechanical properties of powder γ -TiAl alloys, low-carbon steel and commercially pure titanium (CP-Ti) were selected as container materials. At a hot isostatic pressing (HIP) temperature of 1260°C, significant reactive diffusion occurred between the low-carbon steel container and the γ -TiAl alloy, forming porosity defects that deteriorated mechanical properties. At 1230°C, CP-Ti exhibited less shielding effect on HIP pressure than low-carbon steel, enabling more complete plastic deformation of CP-Ti/ γ -TiAl compacts and resulting in higher strength of the powder γ -TiAl alloy. Powder γ -TiAl alloys exhibited no texture, fine grains, and uniform microstructure, with tensile properties superior to those of cast γ -TiAl alloys.

Keywords: metallic materials, powder metallurgy, γ -TiAl alloy, pretreatment, container material

1 Experimental Methods

Pre-alloyed powders with a nominal composition of Ti-47Al-2Cr-2Nb-0.15B (at%) were prepared by electrode induction melting gas atomization (EIGA-50/500) using 99.9996% pure argon gas at a pressure of 3 MPa. The particle size distribution was measured using a Mastersizer 2000 laser particle size analyzer, and powder morphology was observed with a Shimadzu SSX-550 scanning electron microscope. A TENSOR27 FTIR spectrometer was employed to analyze

the desorption process of adsorbed gases from the γ -TiAl pre-alloyed powders during heating.

Powder metallurgy (PM) alloys were fabricated by filling γ -TiAl pre-alloyed powders into low-carbon steel or CP-Ti containers in atmospheric environment, followed by powder vibration, vacuum degassing, argon arc welding, and densification via hot isostatic pressing (HIP) using a QIH-21 HIP furnace [7]. Tensile tests at room temperature and 650°C were conducted on a Shimadzu testing machine, while high-temperature rupture tests were performed on a SANS-GWT105 creep rupture testing machine. The specimen geometry and dimensions followed the standard Q/6S 977-2004 “Metal Mechanical Property Test Specimen Atlas” from the Beijing Institute of Aeronautical Materials. Microstructural observation was carried out using a ZEISS-AXIO optical microscope. Porosity defects in γ -TiAl powder compacts were characterized by VersaXRM-500 X-ray micro-computed tomography. The flow stress-temperature curve of low-carbon steel was measured using a Gleeble 3800 at a strain rate of 1 s⁻¹ and true strain of [value missing in original].

2.1 Particle Size Distribution and Morphology of Pre-alloyed Powders

Powder morphology and particle size distribution directly affect powder packing density and subsequent HIP densification behavior [7]. The particle size distribution of the pre-alloyed powders was therefore characterized. [Figure 1: see original paper] shows the differential size distribution of Ti-47Al-2Cr-2Nb-0.15B pre-alloyed powders. The particle size is predominantly below 250 nm, following a normal distribution, which is beneficial for powder mixing as smaller particles can fill the interstices between larger ones, increasing packing density and promoting complete densification during HIP.

[Figure 2: see original paper] presents the surface morphology of γ -TiAl pre-alloyed powders. The powders are primarily spherical with well-developed cellular structures on their surfaces, exhibiting grain sizes several orders of magnitude smaller than those of cast and wrought γ -TiAl alloys [8-9], typically ranging from 1-5 nm. Xu et al. [9] performed electron probe microanalysis (EPMA) line scans on γ -TiAl pre-alloyed powders of different particle sizes and found microsegregation in larger particles. Unlike the macrosegregation in cast γ -TiAl alloys, this microsegregation can be eliminated through subsequent HIP densification and heat treatment.

Due to the nature of powder metallurgy processes [7], γ -TiAl pre-alloyed powders are exposed to atmospheric conditions during handling and encapsulation. Practical limitations prevent complete vacuum environment during powder filling, vibration, degassing, and sealing. With an average particle size of approximately 100 nm, the high specific surface area of these fine powders leads to adsorption of atmospheric O₂ and H₂O [9]. To amplify this adsorption phenomenon (in actual operations, temperature is maintained at 18-25°C and hu-

midity at 25-35% to minimize gas adsorption), some γ -TiAl pre-alloyed powders were exposed to a humid environment (RH = 60%) for 3 hours. Ti-6Al-4V pre-alloyed powders were subjected to identical treatment for comparison. Infrared-mass spectrometry analysis of gas evolution during heating (25-800°C) of both powder types is shown in [Figure 3: see original paper]. Comparison with standard infrared spectra reveals distinct peak groups in both Figures 3a and 3b corresponding to gaseous H₂O (free water -OH at 3500-3950 cm⁻¹ and hydrogen-bonded -OH at 1300-2000 cm⁻¹), indicating that H₂O is the primary gas released during heating.

Compared with Ti-6Al-4V pre-alloyed powders, γ -TiAl powders more readily absorb atmospheric moisture, likely due to the higher activity of their surface oxide films (primarily Al₂O₃ and TiO₂) [5]. Y. T. Lee [10] reported that residual O₂ in container gaps and O₂ adsorbed on powder surfaces dissolve as interstitial elements in PM Ti-6Al-4V alloys without significantly affecting properties. However, few studies have investigated the effects of vacuum degassing pretreatment on PM γ -TiAl alloy mechanical properties. γ -TiAl pre-alloyed powders are more reactive than Ti-6Al-4V, and adsorbed gases alter powder surface states, potentially affecting subsequent HIP densification. Therefore, investigating pretreatment effects on PM γ -TiAl alloy mechanical properties is essential to provide experimental support for component service performance.

2.2 Effect of Pretreatment on Mechanical Properties of PM γ -TiAl Alloys

To analyze pretreatment effects on mechanical properties, PM γ -TiAl alloys prepared with and without vacuum degassing were tested for room-temperature tensile properties and 650°C/450 MPa rupture life. Sixty tensile specimens and ten rupture specimens were tested for each condition. The vacuum degassing schedule was: room temperature/2 h → 150°C/2 h → 400°C/3 h.

[Figure 4: see original paper] shows the frequency distribution of room-temperature tensile ductility under different pretreatment conditions. For vacuum-degassed samples, 56% exhibited room-temperature elongation exceeding 1.5%, compared to only 32% for non-degassed samples. Statistical analysis indicates that the proportion of samples with >1.5% elongation is 1.75 times higher for degassed samples, demonstrating that vacuum degassing pretreatment substantially improves the stability of room-temperature ductility in PM γ -TiAl alloys.

compares the 650°C/450 MPa rupture life under different pretreatment conditions. Degassed samples exhibit significantly longer rupture life than non-degassed samples. Studies [11] have shown that impurity elements such as oxygen significantly affect the strength and ductility of γ -TiAl alloys. In this work, a TCH 600 analyzer was used to measure H, O, and N contents in PM γ -TiAl alloys under different pretreatment conditions, revealing no significant differ-

ences. X-ray micro-computed tomography (Micro-CT) [12-13] was employed to characterize internal porosity distribution in powder compacts prepared under both conditions, with each analysis volume measuring $1145 \text{ m} \times 1517 \text{ m} \times 454 \text{ m}$.

[Figure 5: see original paper] shows the size and distribution of internal pores in PM γ -TiAl alloys under different pretreatment conditions. Vacuum degassing pretreatment significantly reduces both the size and quantity of porosity defects. This is attributed to the high chemical reactivity and alloying level of γ -TiAl pre-alloyed powders, which readily undergo oxidative contamination during atmospheric exposure, forming a hard α_2 layer on particle surfaces [9]. Without vacuum degassing, this α_2 layer may prevent interstitial element diffusion into the matrix, creating pores at particle boundaries that hinder densification. In summary, while vacuum degassing does not significantly alter O, N, and H contents, it markedly reduces internal porosity, which critically affects room-temperature ductility stability and high-temperature rupture life. Therefore, vacuum degassing pretreatment is essential.

2.3 Influence of Container Materials on Microstructure and Mechanical Properties of PM γ -TiAl Alloys

Containers are essential for HIP processing of powder alloys, serving two primary functions: (1) as hermetic vessels to prevent pressurizing gas from entering powder pores [14-15]; and (2) as dies to transfer external heat and gas pressure to powder particles, enabling complete densification during HIP. Container materials should possess adequate strength, good hermeticity and workability, excellent weldability, and easy removability after HIP.

Common HIP container materials for titanium alloys include low-carbon steel, stainless steel, CP-Ti, and Ti-6Al-4V [14]. Studies [5, 14-16] indicate that stainless steel containers have poor weldability, with weld tearing during HIP causing container expansion (severe leakage) and component failure, plus high manufacturing costs. Ti-6Al-4V containers can contaminate the titanium alloy matrix with V. Low-carbon steel containers offer easy machining, good weldability, and low cost. Therefore, this work focuses on comparing low-carbon steel and CP-Ti containers.

Xu et al. [9, 17] found that insufficient HIP temperature leaves distinct prior particle boundaries in powder compacts. Thus, HIP temperatures of 1230–1260°C were selected. Post-HIP density measured by Archimedes' method exceeded 99.5%, indicating full densification.

During HIP densification, significant reactive diffusion occurs between low-carbon steel containers and γ -TiAl alloys, contaminating the matrix. [Figure 6: see original paper] shows SEM-BSE images of reaction layers at container/alloy interfaces under two HIP temperatures. At 1230°C ([Figure 6a: see original

paper]), the low-carbon steel/ γ -TiAl interface is wavy with a ~ 100 nm thick reaction layer. Based on Ti-Fe and Fe-Al binary phase diagrams [18-19], intermetallic compounds such as Ti₃Fe and FeAl form at the interface, with Fe diffusing into the γ -TiAl matrix (confirmed by EDS). At 1260°C ([Figure 6b: see original paper]), reaction intensifies, producing a ~ 2 mm thick reaction layer with numerous pores at the interface and within the alloy. Micro-CT three-dimensional reconstruction ([Figure 6c: see original paper]) clearly reveals coarse dendritic Ti₃Fe and FeAl structures in the γ -TiAl matrix. In contrast, CP-Ti/ γ -TiAl at 1260°C shows interdiffusion with fewer pores ([Figure 6d: see original paper]). Consequently, the maximum HIP temperature for low-carbon steel containers should be below 1230°C, while CP-Ti containers can be used at higher temperatures.

Micro-CT analysis of pore distribution in compacts HIPed at 1230°C/140 MPa/3 h ([Figure 7: see original paper], analysis volume: 1145 nm \times 1517 nm \times 454 nm) reveals that low-carbon steel containers produce significantly more and larger pores than CP-Ti containers. This occurs because: (1) Al and Ti diffusion coefficients in Fe are higher than Fe diffusion coefficients in Ti and Al matrices [20], leaving pores in the γ -TiAl alloy; and (2) the diffusion flux through reaction-formed channels between Fe and γ -TiAl is much higher than that between Ti and γ -TiAl, increasing porosity in low-carbon steel canned compacts.

compares room-temperature tensile properties of PM γ -TiAl alloys canned with low-carbon steel and CP-Ti after HIP at 1230°C/140 MPa/3 h (12 specimens per group, sampled radially ~ 1 mm from the container inner wall). While ductility is similar, yield and tensile strengths differ by approximately 100 MPa.

[Figure 8: see original paper] shows flow stress-temperature curves for low-carbon steel, CP-Ti, and γ -TiAl alloy [21]. CP-Ti exhibits lower high-temperature flow stress than low-carbon steel across 600-1400°C. HIP is a dynamic compression process relying on container plastic deformation to transmit inert gas pressure [22]. However, container thickness and rigidity cause a pressure shielding effect, making transmitted pressure unequal to the gas pressure. Different materials exhibit varying shielding effects; CP-Ti's lower high-temperature flow stress (significantly below 1100°C) results in more complete densification and plastic deformation/recrystallization, further refining grain structure and improving properties [15]. Additionally, reactive diffusion and pore formation at low-carbon steel/ γ -TiAl interfaces degrade properties.

Although CP-Ti containers produce fewer pores, their large flow stress difference with γ -TiAl causes poor deformation coordination, leading to non-uniform component deformation during HIP [23-24]. CP-Ti also has inferior weldability compared to low-carbon steel, and low-carbon steel containers are more easily removed by chemical milling [15]. Therefore, applying a ceramic coating to low-carbon steel containers could inhibit interfacial reactions while maintaining uniform deformation and easy removal.

2.4 Microstructure and Tensile Properties of Typical PM γ -TiAl Alloys

summarizes average tensile properties at room temperature and 650°C for vacuum-degassed PM γ -TiAl alloys HIPed at 1230°C/140 MPa/3 h. For comparison, cast γ -TiAl alloys of identical composition (HIPed at 1260°C/150 MPa/3 h for defect healing) were also tested (12 specimens each). PM γ -TiAl alloys exhibit superior strength and ductility at both temperatures compared to cast alloys.

[Figure 9a: see original paper] shows the inverse pole figure (IPF) orientation map of the γ phase in cast γ -TiAl alloy, revealing coarse, non-uniform grains (100–600 μm). [Figure 9b: see original paper] presents the (111) and $\langle 110 \rangle$ pole figures, demonstrating distinct casting texture that persists after HIP and heat treatment, causing mechanical property anisotropy and scatter in actual components. [Figure 9c: see original paper] shows the IPF map for PM γ -TiAl alloy, exhibiting fine, uniform grains (5–10 μm) with some twinned grains. Gas atomization creates metastable microstructures with high chemical potential, which subsequent HIP and heat treatment can significantly modify, altering phase distribution and mechanical properties—a concept also discussed for PM Ti₂AlNb alloys [25]. [Figure 9d: see original paper] shows the (111) and $\langle 110 \rangle$ pole figures for PM γ -TiAl alloy, revealing no significant texture. The fine, uniform, texture-free microstructure of PM γ -TiAl alloys ensures consistent and stable overall performance in components.

3 Conclusions

1. Vacuum degassing pretreatment significantly reduces internal porosity in PM γ -TiAl alloys, improving room-temperature ductility stability and high-temperature rupture life.
2. Using low-carbon steel containers for PM γ -TiAl alloys causes reactive diffusion and pore formation; the HIP temperature should be lower than that for CP-Ti containers. At 1230°C, CP-Ti exhibits less pressure shielding than low-carbon steel, enabling more complete plastic deformation of CP-Ti/ γ -TiAl compacts and higher alloy strength.
3. Compared to low-carbon steel containers, CP-Ti containers have inferior machinability and weldability and are difficult to remove after HIP. Ceramic coatings on low-carbon steel containers could inhibit interfacial reactions while allowing higher HIP temperatures and easy removal.
4. PM γ -TiAl alloys exhibit fine, uniform grains without texture, with tensile properties superior to cast alloys. Powder metallurgy enables fabrication of γ -TiAl alloy components with consistent and stable overall performance.

References

1. C. Leyens, M. Peters, CHEN Zhenhua, et al, transl, *Titanium and Titanium Alloys* (Beijing, Chemical Industry Press, 2005) p.52
2. XU Lei, LI Juying, TIAN Xiao, CUI Yuyou, YANG Rui, Fabrication and characterization of TiAl based pre-alloyed powder produced by gas atomization, *Rare Metal Materials and Engineering*, 37(Suppl.3), 815(2008)
3. Alain Lasalmonie, Intermetallics: Why is it so difficult to introduce them in gas turbine engines? *Intermetallics*, 14(10-11), 1123(2006)
4. Fritz Appel, Jonathan D. H. Paul, Michael Oehring, *Gamma Titanium Aluminide Alloys: Science and Technology* (Germany, Wiley-VCH Verlag & Co. KGaA, 2011) p.479
5. WANG Gang, *An Investigation of the Fabrication and High Temperature Deformation Behavior of P/M TiAl Alloys*, PhD thesis, Institute of Metal Research, Chinese Academy of Sciences (2011)
6. ZHANG Ji, JING Yongjuan, FU Mingjie, GAO Fan, Microstructure optimization of ingot metallurgy TiAl, *Intermetallics*, 27, 21(2012)
7. CHENG Wenxiang, XU Lei, LEI Jiafeng, LIU Yuyin, YANG Rui, Effects of powder size segregation on tensile properties of Ti-5Al-2.5Sn ELI alloy powder, *The Chinese Journal of Nonferrous Metals*, 23(2), 363(2013)
8. HU Hanqi, *Fundamentals of Solidification* (Second Edition) (Beijing, Machinery Industry Press, 2000) p.255
9. L. Xu, C. G. Bai, D. Liu, W. Sun, D. J. Yu, Y. Y. Cui, R. Yang, Processing and properties of gamma TiAl sheet from atomized powder, in *Structural Aluminides for Elevated Temperature Applications*, edited by Y. W. Kim, D. G. Morris, R. Yang and C. Leyens (TMS, Warrendale, PA, 2008)
10. Y. T. Lee, H. Schurmann, K. J. Grundhoff, M. Peters, Effect of degassing treatment on microstructure and mechanical properties of P/M Ti-6Al-4V, *Powder Metallurgy International*, 22(1), (1990)
11. Shinichi Takagi, Chiaki Ouchi, Effect of oxygen content on microstructure and mechanical properties of cast and HIP' ed γ TiAl alloys, *Materials Transactions*, 38, (1997)
12. WANG Shaogang, WANG Sucheng, ZHANG Lei, Application of high resolution transmission X-ray tomography in material science, *Acta Metallurgica Sinica*, 49(8), 902(2013)
13. H. Jiang, K. Zhang, F. A. Garcia-Pastor, M. H. Loretto, D. Hu, P. J. Withers, M. Preuss, X. Wu, Microstructure and properties of hot isostati-

- cally pressed powder and extruded Ti₂₅V₁₅Cr₂Al_{0.2}C, *Materials Science and Technology*, 27(8), 1244(2011)
14. CHENG Wenxiang, *Investigation on Densification Behavior and Finite Element Modeling of Ti-5Al-2.5Sn ELI Pre-alloyed Powders*, Master thesis, Institute of Metal Research, Chinese Academy of Sciences (2013)
 15. WU Jun, *Study on Densification Behavior of Ti-5Al-2.5Sn ELI Pre-alloyed Powders under Hot Isostatic Pressing*, Master thesis, Institute of Metal Research, Chinese Academy of Sciences (2011)
 16. GUO Ruipeng, XU Lei, BAI Chunguang, WU Jie, WANG Qingjiang, YANG Rui, Effect of can design on tensile properties of typical powder metallurgy titanium alloys, *The Chinese Journal of Nonferrous Metals*, 24(8), 2051(2014)
 17. WANG Gang, ZHENG Zhuo, CHANG Litao, XU Lei, CUI Yuyou, YANG Rui, Characterization of TiAl pre-alloyed powder and its densification microstructure, *Acta Metallurgica Sinica*, 47(10), 1263(2011)
 18. K. C. Hari Kumar, P. Wollants, L. Delaey, Thermodynamic reassessment and calculation of Fe-Ti phase diagram, *Calphad*, 18(2), 223(1994)
 19. M. Palm, G. Inden, Experimental determination of the Fe-Al phase diagram in the temperature range 900–1400°C and reassessment of the Fe-Al system, *Intermetallics*, 3(6), 443(1995)
 20. M. K. Miller, D. T. Hoelzer, S. C. Deevi, E. A. Kenik, Atom probe tomography of FeAl alloys, *Intermetallics*, 13(12), 1287(2005)
 21. LIU Dong, *Hot Extrusion Process, Microstructure Control and Mechanical Properties of γ -TiAl Alloys*, PhD thesis, Institute of Metal Research, Chinese Academy of Sciences (2007)
 22. DONG Ping, An analysis of the shielding effect of container on isostatic pressing, *Metal Forming Technology*, 20(3), 12(2002)
 23. WANG Zhen, *Manufacturing, Microstructure Evolution and Mechanical Properties of γ -TiAl Alloy Sheet*, PhD thesis, Institute of Metal Research, Chinese Academy of Sciences (2014)
 24. W. B. Eisen, B. L. Ferguson, R. M. German, R. Iacocca, P. W. Lee, D. Madan, K. Moyer, H. Sanderow, and Y. Trudel Hardbound, *ASM Handbook Volume 07: Powder Metal Technologies and Applications* (American Society of Metals, 1998) p.1431
 25. WU Jie, XU Lei, LU Bin, CUI Yuyou, YANG Rui, Fabrication and rupture life of Ti₂AlNb alloy by powder metallurgy, *Chinese Journal of Materials Research*, 28(5), 391(2014)

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