Study on the Influence of the New Type of Powder Metallurgy Ti-Al-Nb Alloy Hot Press Sintering Process on the Microstructure

Shuang Fang^{*}, Mincong Zhang, Qiuying Yu and Huaping Xiong

Beijing Institute of Aeronautical Materials, Beijing, 100095, China.

Abstract. Based on the hot-pressing sintering process, the kinetics of hotpressing sintering crystal grain growth and the hot-pressing sintering densification kinetics, the mixing ratio of the raw material powder and the hot-pressing sintering system are designed. The pressure is maintained at 30 MPa from the temperature rise to the end of the sintering process, and wait until the temperature reaches the corresponding hot pressing sintering temperature (1200°C~1400°C), keep the corresponding time for 1.5h~4.5h, stop heating and depressurize, and the alloy billet is cooled to room temperature with the furnace. The main phases of Ti2AlNb powder and hot pressing sintering process are TiAl and Ti3Al.

1 Introduction

Ti2AlNb alloy is a Ti-Al intermetallic compound with orthogonal structure O phase as the matrix. Because it has good strength, plastic toughness and creep resistance at 650°C~700°C (typical room temperature performance data: tensile The strength is about 1100MPa, the elongation is 8-14%[1-2]), and the density is low, so it has good application potential in the aviation and aerospace fields, but it is generally believed that the long-term stable working temperature of this material is difficult to exceed 700°C.

The γ -TiAl intermetallic compound has a series of advantages such as low density, high specific strength, high specific rigidity and good high temperature performance. It is generally believed that its long-term use temperature can even reach 750°C~800°C (typical high temperature performance data: 800°C, tensile strength About 500MPa [3-5]). On the one hand, γ -TiAl intermetallic compounds can replace conventional Ti-based alloys, thereby increasing the service temperature of materials; on the other hand, they can replace Ni-based superalloys to achieve weight reduction and reduce The heavy effect is more obvious than that of Ti2AlNb alloy, which is very attractive for meeting the high-temperature service requirements and weight reduction requirements of high-performance aeroengines [6-9].

However, the biggest problem of γ -TiAl intermetallic compounds is poor plasticity at room temperature. For example, the room temperature elongation of such materials after forging and heat treatment is generally only about 1.0% to 3.0%, and it is difficult to exceed 4.0% [2, 4, 5], this results in poor hot and cold processing formability of the material, and

^{*} Corresponding author: fs107114@126.com

seriously affects the safety and reliability of the components, so that the design and application of γ -TiAl materials in many high-temperature parts of aircraft and engines are limited [10-12].

It should be said that there is currently a lack of a titanium-based superalloy with a long-term operating temperature of 700° C to 750° C (room temperature elongation of more than 5.0%) in the aviation field.

2 Experimental

The raw materials used in the experiment are pure Ti powder, pure Al powder and Ti2AlNb alloy powder. The powder is produced by Xi'an Ouzhong Material Technology Co., Ltd. The chemical composition of the raw material powder used in the test is listed in Table 1.

Element	Fe	С	0	Ν	Н	Ti	/	Ti
Content, wt%	0.023	0.023	0.1661	0.0066	0.0023	Bal	/	11
Element	Ti	0	Zn	Cu	Fe	Si	Al	A 1
Content, wt%	< 0.01	0.096	< 0.01	< 0.01	< 0.01	< 0.01	Bal	Al
Element	Al	Nb	С	Ν	0	Н	/	Ti2Al
Content, wt%	10.25	42.90	0.008	0.002	0.068	0.002	/	Nb

 Table 1. Chemical composition of pure Ti, Al and Ti2AlNb powder (wt%)

Through scanning electron microscopy secondary electron imaging, it is observed that the raw material powder used in the experiment is spherical particles of nearly equal diameter. As shown in Figure 1, The 400 mesh pure Ti powder has a relatively uniform particle size distribution. The 400 mesh pure Al powder has smaller spherical particles agglomerated near the 400 mesh powder particle size particles, and the 400 mesh Ti2AlNb alloy powder has smaller size spherical particles.

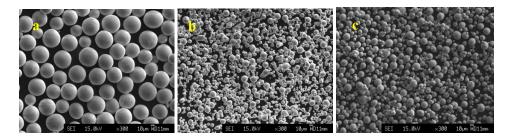


Fig. 1. SEM photo of the raw material powder (400 mesh)used in the experiment

(a)pure Ti powder;(b)pure Al powder;(c)Ti2AlNb alloy powder;

Based on the hot-pressing sintering process, the kinetics of hot-pressing sintering crystal grain growth and the hot-pressing sintering densification kinetics, the mixing ratio of the raw material powder and the hot-pressing sintering system are designed. The pressure is maintained at 30 MPa from the temperature rise to the end of the sintering process, and wait until the temperature reaches the corresponding hot pressing sintering temperature (1200°C~1400°C), keep the corresponding time for 1.5h~4.5h, stop heating and depressurize, and the alloy billet is cooled to room temperature with the furnace.

3 Results and discussion

The microstructure and morphology of the hot-pressed sintered body obtained under the conditions of 1200°C~1400°C, 30MPa, 1.5h~4.5h are shown in Fig. 2~4. It can be seen that there are no obvious pores in the hot-pressed sintered body under the conditions of different powder particle size, powder ratio and hot-pressed sintering system. According to the analysis of porosity by computer software, the samples are basically completely dense (above 99.5%). It shows that pure Ti powder, pure Al powder and Ti2AlNb alloy powder diffuse more completely.

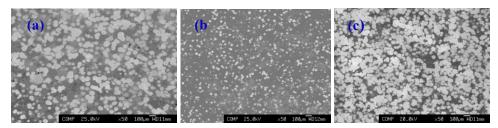


Fig. 2. Microstructure morphology of 400 mesh powder after hot pressing and sintering (a)VTi2AlNb=5%;(b)VTi2AlNb=10%;(c)VTi2AlNb=15%

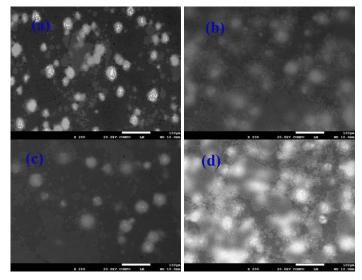


Fig. 3. Microstructure morphology of 400 mesh powder after hot pressing and sintering at different temperatures (a)1200C; (b)1250C; (c)1300C; (d)1400C

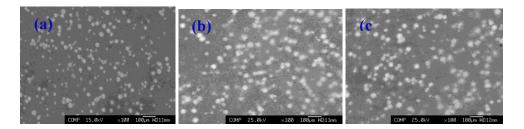


Fig. 4. Microstructure morphology of 400 mesh powder after hot pressing and sintering with different holding time (a)1.5h;(b)3h;(c)4.5h

The microstructure morphology of 400 mesh powder mixture (VTi2AlNb=10%) after hot pressing and sintering is shown in Figure 5. Under the backscatter detection conditions, the brighter the place, the higher the Nb content, the darker the place, the Al content Higher. In the microstructure, there are scattered tissues (phases) with high Nb content, with a size of about 20 μ m, and some agglomerated structures (phases) with high Nb content, with a size of about 40 μ m (see Figure 5(a)). From Figure 5(b), it can also be seen that due to the small particle size of the powder, there is basically no morphology close to the boundary of the powder particles in the microstructure.

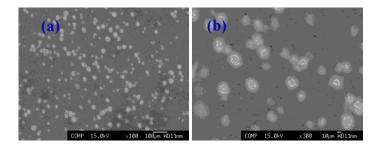


Fig. 5. Microstructure morphology of 400 mesh powder mixture (VTi2AlNb=10%) after hot pressing and sintering (a)100X;(b)300X

XRD was used to analyze the phase of the hot-pressed sintered body, and then the standard JCPDS card was used for calibration. As shown in Figure 6, it was found that the hot-pressed sintered body was mainly composed of TiAl, Ti2Al and Ti3Al.

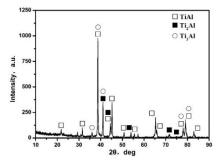


Fig. 6. XRD pattern of 400 mesh powder mixture (VTi2AlNb=10%) after hot pressing and sintering

The microstructure morphology of 400 mesh powder mixture (VTi2AlNb=5%) after hot pressing and sintering is shown in Figure 7. Compared with Figure 5(a), the size of the

microstructure (phase) with high Nb content in the microstructure is significantly increased. The size is about 50µm.

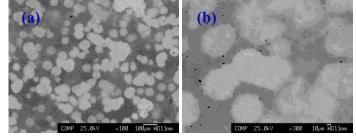


Fig. 7. Microstructure morphology of 400 mesh powder mixture (VTi2AlNb=5%) after hot pressing and sintering (a)100X;(b)300X

The phase of the hot-pressed sintered body was analyzed by XRD, and then the standard JCPDS card was used for calibration. As shown in Figure 8, it was found that the hot-pressed sintered body was mainly composed of Ti3Al and TiAl, mixed with 400 mesh powder (VTi2AlNb=10%) The microstructure composition after hot pressing and sintering is different and does not contain Ti2Al phase. This is because the increase in Ti content makes Ti2Al and Ti fully react.

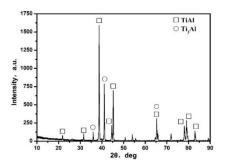


Fig. 8. XRD pattern of 400 mesh powder mixture (VTi2AlNb=5%) after hot pressing and sintering

When VTi2AlNb is increased to 15%, as shown in Figure 9, the microstructure with high Nb content (phase) increases significantly, and the morphology is not significantly different from VTi2AlNb=5%, but it is significantly different from VTi2AlNb=10%.

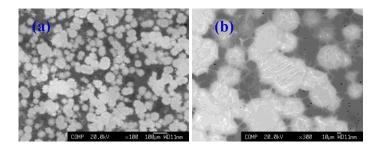


Fig. 9. Microstructure morphology of 400 mesh powder mixture (VTi2AlNb=15%) after hot pressing and sintering (a)100X;(b)300X

Using XRD to analyze the phase of the hot-pressed sintered body, and then use the standard JCPDS card for calibration, it is found that the hot-pressed sintered body is mainly composed of Ti2Al, TiAl, Ti and Ti2AlNb, and 400 mesh powder mixture (VTi2AlNb=5% and VTi2AlNb= 10%) There is no difference in microstructure composition after hot pressing and sintering.

Taking VTi2AlNb=10% as an example, using 400 mesh powder, the effect of hot pressing sintering temperature on the microstructure was studied. As shown in Figure 10, it is a 400 mesh powder mixture (VTi2AlNb=10%) hot pressed at 1300°C The sintered microstructure morphology, it can be seen that not only a small amount of high Nb content structure (phase) is retained in the microstructure, but also a lath-like structure exists.

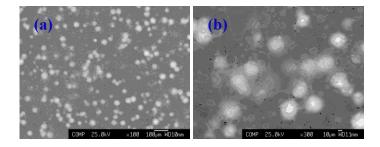


Fig. 10. Microstructure morphology of 400 mesh powder mixture (VTi2AlNb=10%) after hot pressing and sintering at 1300°C (a)100X;(b)300X

XRD was used to analyze the phase of the hot-pressed sintered body, and then a standard JCPDS card was used for calibration. As shown in Figure 11, it was found that the hot-pressed sintered body was mainly composed of Ti3Al and TiAl.

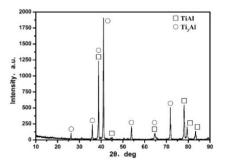


Fig. 11. XRD pattern of 400 mesh powder mixture (VTi2AlNb=10%) after hot pressing and sintering at 1300°C

Continue to increase the hot-pressing sintering temperature to 1400°C, the structure (phase) with high Nb content in the microstructure gradually decreases, the lath structure still exists, and the gap between the laths gradually increases. Using XRD to analyze the phase of the hot-pressed sintered body, and then use the standard JCPDS card for calibration, it is found that the hot-pressed sintered body is mainly composed of Ti3Al and TiAl.

Taking VTi2AlNb=10% as an example, using 400 mesh powder, the effect of hot pressing and sintering time on the microstructure was studied. As shown in Figure 12, it is a 400 mesh powder mixture (VTi2AlNb=10%) after hot pressing and sintering for 3 hours. It can be seen that not only a small amount of high Nb content structure (phase) is retained in the microstructure, but part of the structure has obviously grown up.

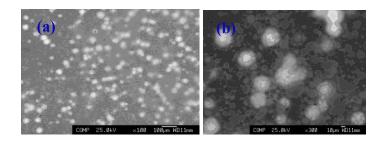


Fig. 12. Microstructure morphology of 400 mesh powder mixture (VTi2AlNb=10%) after 3h hot pressing and sintering (a)100X;(b)300X

XRD was used to analyze the phase of the hot-pressed sintered body, and then the standard JCPDS card was used for calibration. As shown in Figure 13, it was found that the hot-pressed sintered body was mainly composed of Ti3Al and TiAl.

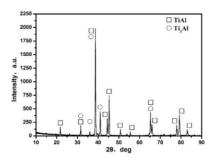


Fig. 13. XRD pattern of 400 mesh powder mixture (VTi2AlNb=10%) after hot pressing and sintering for 3 hours

Continue to increase the hot-pressing sintering time to 4.5h, not only a small amount of high Nb content structure (phase) still remains in the microstructure, but some of the structure obviously grows up, which is not significantly different from the structure after 3h of hot-pressing sintering. Using XRD to analyze the phase of the hot-pressed sintered body, and then use the standard JCPDS card for calibration, it is found that the hot-pressed sintered body is mainly composed of Ti3Al and TiAl.

In summary, the phase composition and phase content of the sample with the addition of Ti2AlNb powder and the hot pressing sintering process are listed in Table 2.

Numbering	Nb%	system	Mesh	AlTi	Ti3Al	Ti2Al	Ti2AlNb
Ti-4	5	1200°C		82	18	/	/
Ti-5	10	30MPa		82	16	2	/
Ti-6	15	1.5h		72	16	11	1
Ti-11	5	1250°C		89	11	/	/
Ti-12	10	30MPa		78	22	/	/
Ti-13	15	1.5h		77	23	/	/
Ti-14	5	1300°C		77	23	/	/
Ti-8	10	30MPa		81	19	/	/
Ti-15	15	1.5h	400	83	17	/	/
Ti-7	10	1400°C 30MPa 1.5h	+00	87	13	/	/
Ti-9	10	1200°C 30MPa 3h		78	22	/	/
Ti-10	10	1200°C 30MPa 4.5h		77	23	/	/

 Table 2. Different Ti2AlNb powder addition amount and phase composition and phase content of samples after hot pressing sintering process

4 Conclusions

Based on the hot-pressing sintering process, the kinetics of hot-pressing sintering crystal grain growth and the hot-pressing sintering densification kinetics, the mixing ratio of the raw material powder and the hot-pressing sintering system are designed. The pressure is maintained at 30 MPa from the temperature rise to the end of the sintering process, and wait until the temperature reaches the corresponding hot pressing sintering temperature ($1200^{\circ}C^{-1400^{\circ}C}$), keep the corresponding time for $1.5h^{-4.5}$ h, stop heating and depressurize, and the alloy billet is cooled to room temperature with the furnace. The addition amount of Ti2AlNb powder and the hot pressing sintering process are mainly TiAl and Ti3Al for the phase of the sample.

References

- [1] Mao Yong, Li Shiqiong, Zhang Jianwei, Peng Jihua, Zou Dunxu, Zhong Zengyong. Acta Metall Sinica, 2000, 36(2): 135-140.
- [2] Zhang Yonggang, Han Yafang, Chen Guoliang, Guo Jianting, Wan Xiaojing, Editorin-Chief Feng Di, Intermetallic Structural Materials, National Defense Industry Press, January 2001
- [3] C.H. Koo and T.H. Yu. Pack cementation coatings on Ti3Al-Nb alloys to modify the high-temperature oxidation properties. Surface and Coatings Technology, 126, 2000, p171-180.
- [4] Y. W. Kim, D. M. Dimiduk. Progress in the understanding of gamma titanium aluminides, JOM, 43(8), 1991, p40-47.
- [5] C. Koepee, A. Bartels, J. Seeger and H. Mecking. General aspects of the thermomechanical treatment of two-phase intermetallic TiAl compounds. Metallurgical and Materials Transaction A, 24(8), 1993, p1795-1806.

- [6] W. Gao, Z. Li, D. Zhang. A new high-temperature, oxidation-resistant Ti-based materials. Oxidation of Metals, 57(1-2), 2002, p99-114.
- [7] S. L. Semiatin. Wrought processing of ingot-metallurgy gamma titanium aluminide alloys [A]. Kim Y W, Wagner R, Yamaguchi M, ed. Gamma Titanium Aluminides [C]. TMS, Wrarrendale, PA, 1995: p509-524.
- [8] Y.W. Kim. Intermetallic alloys based on gamma titanium aluminum [J]. JOM. 1989, 41(7): 24.
- [9] C. Scheu, E. Stergar, M. Schober, L. Cha, H. Clemens, A. Bartels, F.P. Schimansky, A. Cerezo. High carbon solubility in a γ -TiAl-based Ti-45Al-5Nb-0.5C alloy and its effect on hardening [J]. Acta Materialia. 2009, 57: 1504–1511.
- [10] Y.Y. Chen, H.Z. Niu, F.T. Kong, S.L. Xiao. Microstructure and fracture toughness of a β phase containing TiAl alloy [J]. Intermetallics. 2011,19: 1405–1410.
- [11] D. Vujic. Effect of rapid solidification and alloying addition on lattice distortion and atomic ordering in L10 TiAl alloys and their temporary alloys [J]. Metall Trans A. 1988, 19(10): 2445.
- [12] T. Kawabata, T. Abumiya, T. Kami, et al. Mechanical properties and dislocation structures of TiAl single crystals deformed at 4.2-293K [J]. Acta Metall Mater. 1990, 38(8): 1381-1393.