Hot Deformation Mechanism and Ring Rolling Behavior of Powder Metallurgy Ti₂AlNb Intermetallics

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Abstract Powder metallurgy (PM) Ti–22Al–24Nb–0.5Mo (at.%) alloys were prepared by hot isostatic pressing. In order to study the feasibility of PM + ring rolling combined process for preparing Ti₂AlNb rings, thermal mechanical simulation tests of PM Ti₂AlNb alloys were conducted and two rectangular PM rings (150 mm in height, 75 mm in thickness, 350 mm in external diameter) were rolled as a validation experiment. Experimental results show that the flow stress of Ti₂AlNb alloys exhibited a significant drop at the very beginning of the deformation (true strain <0.1), and became stable with the increase in strain. Stress instability phenomenon of PM Ti₂AlNb alloys was more obvious than that of wrought alloy. Flow stress fluctuation at the initial stage of deformation is related to phase transition of Ti₂AlNb alloys which strongly depends on heat treatment and thermal mechanical deformation process. Processing windows during initial stage of ring rolling process is very crucial. A sound PM Ti₂AlNb rectangular ring blank (height = 150 mm, thickness = 30 mm, external diameter = 750 mm) was successfully rolled in two passes by using the improved heat preservation method and optimized rolling parameters. Tensile properties of PM Ti₂AlNb alloy were improved, and the porosity was reduced after ring rolling.

KEY WORDS: Ti₂AlNb alloy; Powder metallurgy; Hot deformation; Ring rolling

1 Introduction

Ti₂AlNb alloy is a potential intermetallic compound which can be used in gas turbine engine components such as compressor [1–3] due to its low density, high strength and excellent comprehensive performance at elevated

temperature [4, 5]. Compressor is one of the core components of the aeroengine, and the external dimension of engine compressor is usually very large (diameter > 900 mm). Ring rolling is one of the most important and necessary deformation processes to prepare large-size components with ring shape. Figure 1a shows the conventional route to prepare Ti₂AlNb ring. However, it is difficult to find a suitable way to prepare sound Ti₂AlNb ring components due to its initial brittleness and other economic factors [6–8]. Previous studies have reported that the casting defects such as center-line porosity, chemical inhomogeneity, regions of varying density would cause crack during rolling process [9, 10]. Therefore, to reduce the risk of cracking, about 10 times upsetting steps are needed to prepare wrought ring preforms and these repeated thermal mechanical steps would increase the preparation period.

Powder metallurgy (PM) through hot isostatic pressing (HIP) is considered as a near-net-shape process with small

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Fig. 1 Preparation route of Ti₂AlNb ring blanks: **a** conventional method, **b** PM + rolling method

amounts of machining, and the mechanical properties of PM alloy are more stable and microstructure is more uniform than that of casting or wrought alloys [11–14]. Compared with as-cast preforms, PM Ti₂AlNb preforms have two advantages: the first is the higher ratio of materials usage, and the second is the better chemical and microstructure homogeneity due to mixing of pre-alloyed powder [15, 16]. Therefore, PM + ring rolling combined process (as shown in Fig. 1b) is considered as a more reasonable and economical way.

Recently, many researchers have studied hot deformation behavior of Ti₂AlNb alloys [17–20]. However, there are a few reports on hot workability of PM Ti₂AlNb alloys prepared by HIPing [21, 22]. The aim of this article is to study the feasibility of the PM + rolling combined process for preparing Ti₂AlNb ring components. High-temperature deformation behavior of PM Ti₂AlNb alloy was conducted by thermal mechanical simulation tests. Two rectangular ring preforms (150 mm in height, 75 mm in thickness, and 350 mm in external diameter) were prepared by PM method and taken into actual rolling process as a validation study. Microstructure evolution and mechanical properties of PM Ti₂AlNb alloys during ring rolling and post-heat treatment were also investigated.

2 Experimental

Pre-alloyed Ti₂AlNb powder was prepared by electrode induction melting gas atomization (EIGA). The powder with a normal size distribution from 5 to 250 μm was measured by using a Mastersizer 2000 laser size analyzer. The powder was canned in mild steel containers and degassed at elevated temperature, and then, capsules were sealed and gas tight [23, 24]. HIPing was conducted at 1040 °C with applied pressure of 120–150 MPa for 3 h. Table 1 shows the chemical composition of pre-alloyed powder and as-HIPed compacts, and impurities content of either powder or compacts is at a low level.

Thermal mechanical simulation tests were conducted on a Gleeble-3800 simulator for studying the hot deformation behavior of PM Ti₂AlNb alloys. Heat treatment (HT) after thermo-mechanical treatment (TMT) is a common process to obtain a stable and homogeneous microstructure or properties for Ti₂AlNb alloy [24]. To make a comparison study of hot deformation behavior after HT, heat treatment for as-HIPed Ti₂AlNb alloy was conducted at 980 °C/2 h/air cooling and 900 °C/24 h/air cooling. The microstructures of as-HIPed and as-HIPed + HT alloys are shown in Fig. 2. Cylindrical samples with 8 mm in diameter and 10 mm in height were cut from as-HIPed and HT Ti₂AlNb alloys for compressing tests. Thermocouples were welded to the specimens for testing the temperature change, and graphite was applied to both sides of the specimens for reducing friction effect. Compressing temperatures were ranging from 930 to 1050 °C, and the constant strain rates were from 0.001 to 10 s^{−1}. The samples subjected to a heating rate at 5 °C/s were preserved for 3 min at elevated temperature and then compressed to a final true strain of 0.69. Both temperature and strain were controlled by the connected computer to ensure that the actual testing results were stable and reliable.

Ti₂AlNb ring preforms for ring rolling were prepared by typical PM route, and the initial size of the work piece was height (H) = 150 mm, thickness (T) = 75 mm, external diameter (ED) = 350 mm. Table 2 shows the parameters of ring rolling process. According to the hot compression results of PM Ti₂AlNb alloy and conventional rolling experience of wrought alloy, the rolling parameters and heat preservation method were optimized. The first rolling pass continued about 30 s, both PM Ti₂AlNb rectangular billets were rolled to 30% reduction in thickness. After the first rolling pass, one of them was subjected to second rolling pass (60% total reduction). It is shown in Fig. 3 that the hot workability of PM Ti₂AlNb preforms during ring rolling is very well, because no cracks or folds were observed.

PM + rolled ring (~30% reduction) was cut to prepare testing samples, and experiments for studying microstructure of PM Ti₂AlNb preforms during rolling

Table 1 Chemical composition and gas contents of Ti₂AlNb powder and as-HIPed compacts (wt%)

Samples	O	N	H	Ar	Al	Nb	Mo	Ti
Powder	0.069	0.0080	0.0050	<0.0001	10.4	41.0	0.90	Bal.
Compacts	0.068	0.014	0.0025	<0.0001	10.6	41.3	0.90	Bal.

Fig. 2 Microstructures of compressed samples: **a** as-HIPed Ti₂AlNb alloy, **b** as-HIPed + HT Ti₂AlNb alloy**Table 2** Parameters of ring rolling for PM Ti₂AlNb ring preforms

Rolling steps	Parameters
As-HIPed preforms	EIGA powder + HIPing, capsules removed Size: $H_0 = 150$ mm, $T_0 = 75$ mm, $ED_0 = 350$ mm
Heat preservation	Ceramic coatings + asbestos package
Pre-heating	23–1025 °C (5 °C/s), holding for 2 h
The first pass rolling	30 s
Re-heating	1025 °C, holding for 2 h
The second pass rolling	30 s
Post-heating	Furnace cooling, HT (solution + aging treatment)
Machining	Size: $H_1 = 150$ mm, $T_1 = 55$ mm, $ED_1 = 450$ mm (30% deformation in T) Size: $H_2 = 150$ mm, $T_2 = 30$ mm, $ED_2 = 750$ mm (60% deformation in T)

process were conducted. Samples were ground, polished, chemically etched, and then observed by scanning electron microscopy (SEM). Specimens for electron backscatter diffraction (EBSD) were prepared by electro-chemical polishing with a solution of 6% perchloric acid, 34% butanol and 60% carbinol. Tensile tests at room temperature were conducted on Zwick/Roell-Z050, and tests at 650 °C were conducted on MTS E45.105. Porosity is an important issue for PM alloys and would sometimes cause performance deterioration especially at elevated temperature, so X-ray micro-

computed tomography (Micro-CT) was used to assess the porosity in PM Ti₂AlNb alloy [25, 26].

3 Results and Discussion

3.1 Optimization of Process Parameters

Compression test on a thermomechanical simulator is a convenient way to identify the optimal processing parameters for ring rolling. To study the workability of PM

Fig. 3 **a** Horizontal ring rolling equipment, **b** final PM + rolled Ti₂AlNb blank (~60% reduction)

Ti₂AlNb alloys more clearly, the deformation results of PM alloys were compared to that of wrought Ti₂AlNb alloys prepared by casting ingots [27]. Flow behaviors under various compression conditions of as-HIPed Ti₂AlNb alloys and wrought Ti₂AlNb alloys are shown in Fig. 4. It can be seen that the stress increases rapidly at the beginning of deformation and reaches a peak value within 0.1 strain. Then stress decreases gradually and becomes stable after the true strain is 0.1. The initial stage of deformation is very important; thus, special attention should be paid to the change of flow stress within 0.1 strain. When the strain rate is at a low level ($\dot{\varepsilon} = 0.001 \text{ s}^{-1}$), the flow stress decreases slowly from the peak value for both Ti₂AlNb alloys. For as-HIPed alloys, when the strain rate $\geq 0.01 \text{ s}^{-1}$, there is a quick drop of flow stress after reaching the peak value, this stress fluctuation phenomenon was also observed in wrought Ti₂AlNb alloys only when the strain rate was 10 s^{-1} . In other words, the phenomenon of work hardening is more obvious for PM alloys especially at the initial stage of deformation.

To make compare this stress fluctuation phenomenon of both alloys more clearly, a response surface method (RSM) of the stress decline is shown in Fig. 5. True stress at $\varepsilon = 0.1$ ($S_{0.1}$) was defined as a critical value for a starting of stable state of deformation. Then stress declined as different values between peak stress (S_p) and $S_{0.1}$ was calculated. The stress fluctuation phenomenon comparison of PM and wrought Ti₂AlNb alloys in Fig. 5 indicated that stress fluctuation of wrought alloys (Fig. 5b) was affected by both temperature and strain rate. When the deformation temperature is lower than $1000 \text{ }^\circ\text{C}$ or the strain rate is higher than 1 s^{-1} , there is a sudden rise of stress decline for wrought Ti₂AlNb alloys. In Fig. 5a, the stress fluctuation of PM Ti₂AlNb alloys was mainly controlled by strain rate rather than temperature of present work, because the stress fluctuation showed a quick growth at low strain rate. Any sudden change in stress can affect the workability of the material; thus comparison with wrought alloys, the initial

stress fluctuation phenomenon of PM Ti₂AlNb alloys is more obvious, so the process parameter optimization would be more difficult for ring rolling of PM Ti₂AlNb alloys.

To study the stress fluctuation mechanism of PM Ti₂AlNb alloys, the peak stress comparison of Ti₂AlNb alloys at $\dot{\varepsilon} = 0.1 \text{ s}^{-1}$ is shown in Fig. 6. The deformation resistance is an important value to evaluate the workability and identify the process parameters. The results show that heat treatment has no obvious influence on the flow behaviors or peak stress of as-HIPed Ti₂AlNb alloys. The peak stress of wrought alloys decreases with increasing temperature which is mainly related to the dislocation glide. But for PM Ti₂AlNb alloys, a maximum (at $\sim 1000 \text{ }^\circ\text{C}$) and a minimum (at $\sim 1030 \text{ }^\circ\text{C}$) peak stresses are observed in Fig. 6a.

It is shown in Figs. 5 and 6 that the stress fluctuation behavior of PM Ti₂AlNb alloys is very obvious in the initial stage and the peak stress shows a decrease–increase–decrease trend with temperature change, while the deformation behavior of wrought Ti₂AlNb alloys is more stable. The deformation behavior is controlled by many factors such as loading method, temperature and microstructure evolution. The temperature range of phase fields for as-HIPed Ti₂AlNb alloys ($T_{B2} > 1055 \text{ }^\circ\text{C} > T_{B2+\alpha2} > 1000 \text{ }^\circ\text{C} > T_{B2+\alpha2+O}$ [15]) and wrought alloys ($T_{B2} > 1060 \text{ }^\circ\text{C} > T_{B2+\alpha2} > 1000 \text{ }^\circ\text{C} > T_{B2+\alpha2+O}$ [28]) is similar. But the structural states of them are different. The preparation route of wrought alloys is that Ti–22Al–25Nb (at.%) ingot was homogenized at $1150 \text{ }^\circ\text{C}$ for 4 h and then forged into rods at a temperature above the beta transus temperature [27]. Before hot compression tests were conducted, wrought alloys had already been deformed at $1160 \text{ }^\circ\text{C}$, and this step is also called primary deformation or blooming process which is helpful to obtain good deformed preforms. While for PM alloys, pre-alloyed powder was prepared by gas atomization through rapid cooling, and the metastable phase composition of powder cannot be easily changed to stable phase after HIPing as we have discussed before [15]. During the thermal mechanical deformation

Fig. 4 Flow behaviors of as-HIPed Ti_2AlNb alloy and wrought Ti_2AlNb alloy [27] at various conditions

process, the residual metastable phase transitions would influence the deformation behavior, especially in the initial stage.

Workability is defined as the degree of deformation that can be achieved in a particular metal working process without creating an undesirable condition such as cracking or fracture. In general, workability depends on the local condition of stress, strain, strain rate and temperature in combination with material characteristics. The strain rate sensitivity value m in the dynamic material model (DMM) approach is based on changes in σ with $\dot{\epsilon}$ using different flow curves at a given microstructure. The m value is considered to evaluate the workability of specific material in some cases [29].

$$m = \partial \ln \sigma / \partial \ln \dot{\epsilon}. \quad (1)$$

Figure 7 shows the m value counter maps of PM Ti_2AlNb alloys and wrought Ti_2AlNb alloys. It can be seen that the

average m value of PM alloys is larger than that of wrought alloys. The maximum m value of PM alloys only occurs at the low strain rate. For wrought alloys, when the deformation temperature is in $\text{B2} + \alpha_2$ region, the m value distribution is more uniform. This variation of m value in counter maps is similar to the stress fluctuation phenomenon mentioned in former section which indicates that the workability of PM Ti_2AlNb alloys is more sensitive to strain rate. Therefore, processing windows during initial stage of PM Ti_2AlNb alloys is very narrow and accurate control of the process is crucial.

3.2 Ring Rolling of PM Ti_2AlNb Preforms

Thermal mechanical simulation results show that the workability of PM Ti_2AlNb alloys is comparable to that of wrought Ti_2AlNb alloys, but the deformation behavior of PM Ti_2AlNb alloys in the initial stage is more unstable.

Fig. 5 Stress decline comparison of **a** PM Ti₂AlNb alloys, **b** wrought Ti₂AlNb alloys

Fig. 6 Peak stress comparison of PM **a**, wrought **b** Ti₂AlNb alloys at various temperatures ($\dot{\epsilon} = 0.1 \text{ s}^{-1}$)

Deformation conditions in practical environment are much more complicated than that of simulation tests (compression experiment is an adiabatic process). In this case, two PM ring preforms were rolled as a validation experiment to further study the feasibility of PM + rolling combined

process. Due to heat loss, heat preservation and setting of rolling parameters should be properly arranged as shown in Table 2. In order to find out the effect of rolling process on the properties of PM Ti₂AlNb alloys, microstructures and tensile properties of PM and PM + rolled Ti₂AlNb alloys were studied.

As shown in Fig. 8a, the as-HIPed Ti₂AlNb alloy microstructure generally consists of three phases, equiaxed α_2 phase, and lamellar O + B2 phases inside grain. B2 phase is the basic phase with a bcc structure showing a light gray color under secondary electron imaging (SEM). The black area along the grain boundary is α_2 phase, and the ductility of α_2 is poor because of its hexagonal structure, and therefore, crack can be easily formed between α_2 and B2 matrix [30]. The gray area with a lath shape inside grains is O phase which has the good combination property at elevated temperature. Figure 8c shows the microstructure of PM + rolled Ti₂AlNb alloy. The average grain size of as-HIPed Ti₂AlNb alloy is about 15–30 μm and shows obvious change after ring rolling with 30% deformation in thickness. Figure 8b is a magnified image of Fig. 8a, and Fig. 8d is a magnified image of Fig. 8c, the lamellar O + B2 microstructure of as-HIPed alloy inside the grains can be seen more clearly, and a homogeneous distribution of needle-shaped O phase between basic B2 phases after rolling was obtained. For as-HIPed Ti₂AlNb alloy, the O phase lath size is about 1–5 μm in length and 1 μm in thickness. After rolling, the previous lamellar O + B2 structure disappeared and changed into needle-shaped structure. For as-HIPed + rolled Ti₂AlNb alloy, the length of O phase laths is within 1 μm and the thickness is much smaller than that of as-HIPed alloy.

Fig. 7 Counter maps of strain rate sensitivity value m : **a** as-HIPed Ti_2AlNb alloys, **b** wrought Ti_2AlNb alloys

Fig. 8 Microstructures of PM Ti_2AlNb alloys: **a, b** as-HIPed, **c, d** as-HIPed + rolled (30% deformation)

Figure 9 shows different phase distributions of PM Ti_2AlNb alloys characterized by EBSD. The green area is O phase, blue area is α_2 phase, and yellow area is B2 phase. The phase distribution and grain orientation of as-HIPed Ti_2AlNb alloys are homogeneous. According to HKL-Channel 5 software analysis, the volume fractions of each phase are shown in Table 3. After 30% deformation, the volume fraction of O phase shows a significant decrease and the volume fraction of B2 phase increased.

Mechanical properties of PM Ti_2AlNb alloys are very sensitive to microstructure and heat response. To study the

influence of thermal mechanical history on tensile properties of PM Ti_2AlNb alloys, tensile tests at room temperature and 650 °C were conducted and the results are shown in Table 4. It indicated that the tensile strength of PM + rolled Ti_2AlNb alloys is higher than that of as-HIPed alloys, but the ductility shows a little decrease. The yield strength at both testing temperatures of PM + rolled Ti_2AlNb alloys is about 50% higher than that of as-HIPed alloys. After rolling process, finer microstructure consisting of O + B2 phase was obtained. This tensile strength improvement is likely due to the phase composition change of Ti_2AlNb .

Fig. 9 Microstructures of as-HIPed (a), as-HIPed + rolled (b) Ti_2AlNb alloy by EBSD analysis (blue color shows α_2 phase, yellow color shows B2 phase, and green color shows O phase)

Table 3 Phase distribution of PM Ti_2AlNb alloy observed by EBSD analysis

	V_{α_2} (%)	V_O (%)	V_{B2} (%)
As-HIPed	7.3	58.1	34.6
30% rolled	5.6	22.6	71.8

Table 4 Tensile properties of as-HIPed and as-HIPed + rolled Ti_2AlNb alloys

	T (°C)	0.2% YS (MPa)	UTS (MPa)	El (%)
HIPed	23	833	1034	8.0
	650	545	715	15.5
30% rolled	23	1240	1365	5.0
	650	835	1090	5.0

0.2% YS, UTS, El are abbreviations of yield strength, ultimate tensile strength and elongation, respectively

Porosity is one of the most important issues for the application of PM alloys. Though no pores or defects were found in as-HIPed Ti_2AlNb alloys tested by conventional fluorescence detection or ultrasonic flaw detection, but micro-pores also exist. In this case, Micro-CT was used to assess the porosity in PM Ti_2AlNb alloys. The results are shown in Fig. 10; after rolling process, both the counts and the size of micro-pores of as-HIPed Ti_2AlNb alloys decreased. To ensure good mechanical properties of PM Ti_2AlNb alloys, gas pores inherent in the gas atomized powders must be minimized and the PM preparation route should be under good control. Micro-CT results show that rolling can improve the porosity of as-HIPed Ti_2AlNb alloys, and would improve the performance stability from the point of view of defect healing.

As discussed above, ring rolling can improve the tensile strength as well as the porosity of as-HIPed Ti_2AlNb alloy, but the ductility decreased. To obtain good property

Fig. 10 Porosity distributions of as-HIPed and 30% rolled Ti_2AlNb alloys by Micro-CT analysis

combination of PM Ti_2AlNb alloys, HT is a common and necessary method to adjust materials structure and is especially useful to promote phase transition for PM alloys. Heat treatment consisting of solution and aging process is thought to be helpful to obtain better mechanical performance. To improve the ductility of PM + rolled Ti_2AlNb alloys, heat treatment was conducted at 980 °C/2 h/air cooling and 900 °C/24 h/air cooling. Figure 11 shows a

Fig. 11 Comparisons of yield strength and elongation of PM Ti_2AlNb alloys after HT

comparison of yield strength and elongation of as-HIPed and rolled Ti₂AlNb alloys. After heat treatment, the tensile strength of rolled Ti₂AlNb alloys decreased a little and the elongation was improved significantly. Compared with as-HIPed Ti₂AlNb alloys, both the tensile strength and elongation of PM Ti₂AlNb alloys were improved after ring rolling by using the same post-HT. Ring rolling is helpful to improve the tensile properties of PM Ti₂AlNb alloys.

4 Conclusions

- (1) The workability of PM Ti₂AlNb alloys is comparable to that of wrought Ti₂AlNb alloys, but the stress fluctuation of PM alloys is obvious due to the metastable phase transition. Processing windows for PM Ti₂AlNb alloys' ring rolling process in the initial stage is narrow.
- (2) According to the hot compression results of PM Ti₂AlNb alloy, the rolling parameters and heat preservation method were optimized. A sound Ti₂AlNb rectangular ring blank has been successfully prepared which indicates that PM + rolling combined process has good potential in preparing large-size Ti₂AlNb ring blanks.
- (3) Ring rolling can improve the tensile strength as well as the porosity of as-HIPed Ti₂AlNb alloy due to the finer O + B2 microstructure inside the grains. After post-heat treatment, PM + rolled Ti₂AlNb alloys perform a good tensile property combination of strength and ductility.

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