

# Microstructure and Mechanical Properties of TiNbZr Alloy during Cold Drawing

Wang Liqiang<sup>1</sup>, Yang Guanjun<sup>2</sup>, Yang Huabin<sup>3</sup>, Cao Jimin<sup>3</sup>,  
Lü Weijie<sup>1</sup>, Zhang Di<sup>1</sup>

<sup>1</sup> State Key Laboratory of Metal Matrix Composites, Shanghai Jiaotong University, Shanghai 200240, China

<sup>2</sup> Northwest Institute for Nonferrous Metal Research, Xi'an 710016, China

<sup>3</sup> Xi'an SMA Material Co., Ltd, Xi'an 710016, China

**Abstract:** The cold workability of solution-treated TiNbZr biomedical  $\beta$  titanium alloy was investigated. TiNbZr alloy was fabricated by vacuum consumable arc melting furnace. Cold drawing was carried out for further deformation of the studied alloy. During cold drawing, the alloy exhibited excellent workability. Deformation twins appeared when the reduction of cold deformation was around 20%. Dislocations slipping contributed much to plastic deformation in further drawing. The ultimate tensile strength will go up to 1170 MPa and the elongation is larger than 10% when the reduction reaches 80%. Small grains ranging from 20 nm to 50 nm can be obtained when the reduction is 80%.

**Key words:**  $\beta$  titanium alloys; cold drawing; microstructure; mechanical properties

$\beta$  titanium alloys, composed of non-toxic  $\beta$ -stabilizing alloying elements such as Nb, Ta, Zr, Mo and Sn show good biocompatibility and low toxicity to human being<sup>[1–3]</sup>. Compared with  $\alpha$  and  $\alpha+\beta$  titanium alloys,  $\beta$  titanium alloys possess excellent properties such as higher strength and better plasticity<sup>[4–8]</sup>. The deformation mechanisms of  $\beta$  titanium alloys include dislocation slip, twinning and stress-induced martensitic phase transformation. Hanada et al reported that the mechanisms of deformation depend much on phase stability<sup>[9,10]</sup>. Twinning is replaced by dislocation slipping with higher  $\beta$  phase stability. Recently, a new mechanism of the plastic deformation of crystalline metals without dislocations has been studied by researchers<sup>[11,12]</sup>. Takashi Saito's group developed a new kind of titanium alloys<sup>[13,14]</sup>. In these cold-worked alloys, dislocation-free plastic deformation mechanism appeared, which contributed most to the plastic deformation of the alloys.

Cold drawing, as a major mode of cold deformation, plays an important role in the application of  $\beta$  titanium alloys. Nowadays, cold rolling has been utilized as an ordinary mode

to study the deformation behavior of  $\beta$  titanium alloys. Few researches have focused on the microstructure and the mechanical properties of newly developed TiNbZr  $\beta$  titanium alloy deformed by cold drawing

In this paper, the characteristics of TiNbZr  $\beta$  titanium alloy treated by different reductions of cold drawing were studied. The mechanical properties of this alloy were also tested for its further applications.

## 1 Experimental Procedure

The alloy was melted in a consumable vacuum arc remelting furnace twice to ensure compositional homogeneity. The specimen of  $\Phi 7$  mm was obtained through forging, hot rolling and solid solution treatment at 780 °C for 1 h. The specimens of  $\Phi 6.3$  mm,  $\Phi 5.4$  mm,  $\Phi 4.4$  mm and  $\Phi 3.3$  mm were obtained by cold drawing of 20%, 40%, 60% and 80% reduction, respectively.

Microstructures were observed by optical microscopy (OM) and transmission electron microscopy (TEM). Samples for optical microscopy (OM) were prepared using conventional

Received date: April 19, 2008

Biography: Wang Liqiang, Candidate for Ph. D., State Key Laboratory of Metal Matrix Composites, Shanghai Jiaotong University, Shanghai 200240, P. R. China, Tel: 0086-21-54749117, E-mail: Wng-liqiang@163.com

Copyright © 2009, Northwest Institute for Nonferrous Metal Research. Published by Elsevier BV. All rights reserved.

techniques of grinding and mechanical polishing. The composition of etched solution was hydrofluoric acid, nitric acid and water in proportion of 1:3:6 (volume ratio). Samples for TEM were treated by electrolytic apparatus with the electrolyte of 95% carbinol and the perchloric acid solution was used in the final stage of thinning. The phase analysis was carried out by X-ray diffraction (XRD) and tensile test was operated on Instron1195 tensile testing machine.

## 2 Results and Discussion

### 2.1 OM analysis

Fig.1 shows the transverse (T) and longitudinal (L) microstructure of the samples deformed by the reductions ( $\varepsilon$ ) of 20%, 40%, 60% and 80%, respectively. With the increase of the reduction of cold deformation, the microstructure of the samples changed gradually. When the alloy was deformed by  $\varepsilon=20\%$ , the microstructure presented uniform equiaxed grains along transverse direction and the longitudinal grains were drawn much longer, as shown in Fig.1a, 1b. The grains along transverse direction became smaller gradually when the reduction was higher than 20%. As observed in Fig.1c and Fig.1e, smaller grains were obtained when the alloy was deformed by  $\varepsilon=40\%$  and 60%. The longitudinal grains became much longer. When the reduction increased to 80%, grain boundaries along transverse direction broke up, as shown in Fig.1g. Fibriform structure appeared along longitudinal direction, shown in Fig.1h.

### 2.2 XRD phase analysis

X-ray diffraction (XRD) investigations reveal that only  $\beta$  phase was observed in both the samples treated by solid solution and cold drawing by 20% reduction, as shown in Fig.2a, 2b. No stress-induced  $\alpha''$  martensite phase was detected by XRD. It is known that  $M_s$ (the start temperature of martensite transformation) and cooling rate have great effect on  $\alpha''$  martensite phase transformation. As reported in many papers, in  $\beta$  titanium alloys, zirconium should work as the  $\beta$ -stabilizers<sup>[13,15,16]</sup>, which decreases  $M_s$ . A large amount of Nb and Zr will suppress the phase transformation from  $\beta$  to  $\alpha''$  martensite in solid solution or cold drawing. Employing a molecular orbital method, electronic structures were calculated for bcc. Ti alloyed with a variety of elements, and two alloying parameters were determined theoretically. One is the bond order ( $Bo$ ) which is a measurement of the covalent bond strength between Ti and an alloying element. The other is the metal d-orbital energy level ( $Md$ ) which correlates with the electronegativity and the atomic radius of elements. In addition, the number of average valence electrons is considered as  $e/a$ . As for the studied TiNbZr alloy, the three parameters of the studied alloy were calculated as  $e/a=4.244$ ,  $Bo=2.894$  and  $Md=2.527$ . These major parameters affect the mechanism of cold deformation obviously. Owing to the higher  $Bo$  and lower  $Md$ , which influences stabilization of  $\beta$  phase much, only  $\beta$  phase without any  $\alpha''$  martensite was obtained during solid

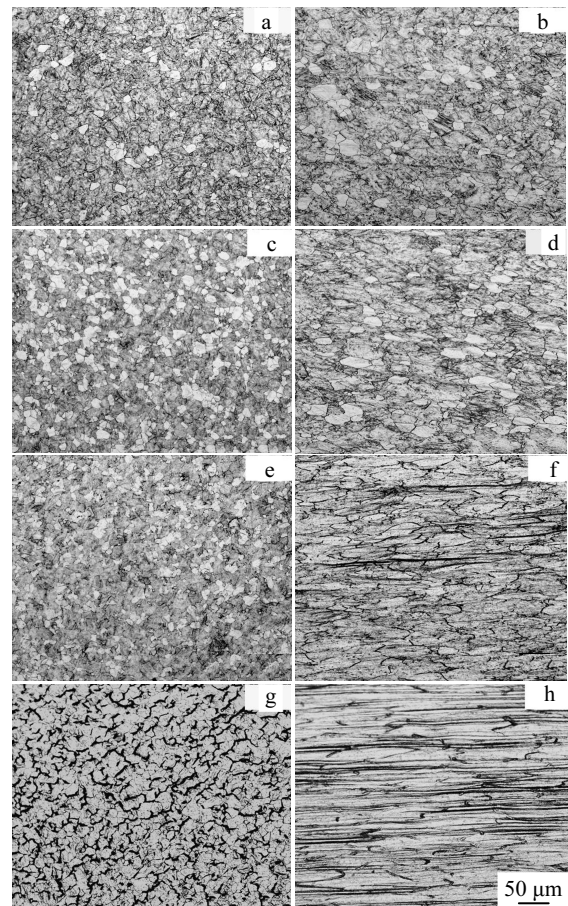


Fig.1 OM microstructure of the specimens after cold deformation: (a), (b)  $\varepsilon=20\%$ ; (c), (d)  $\varepsilon=40\%$ ; (e), (f)  $\varepsilon=60\%$ ; (g), (h)  $\varepsilon=80\%$ ; (a), (c), (e), (g) transverse (T); (b), (d), (f), (h) longitudinal (L)

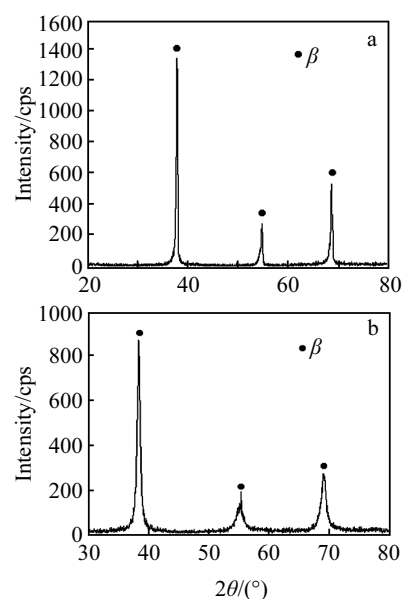


Fig.2 XRD patterns of TiNbZr specimens: (a) solution-treated and (b) cold-drawn by 20% reduction

solution and cold drawing by  $\varepsilon=20\%$ . The transforming driving force from  $\beta$  phase to  $\alpha''$  martensite was not so sufficient to complete phase transformation.

### 2.3 Deformation behavior

Fig.3 shows TEM micrographs of cold drawing sample deformed by 20% reduction. Much platelet-shape deformation twins was presented in the  $\beta$ -Ti matrix, as shown in Fig.3a. Besides this deformation twins, dislocation tangle was investigated in the sample deformed by 20% reduction, as the black arrows showed in Fig.3b. Macroscopic shear bands (SB) around dislocations were also observed. Dislocation slipping affects the plastic deformation greatly. As studied above in Fig.2b, during this deformation, no stress-induced  $\alpha''$  martensite was detected by transmission electron microscopy. During the cold drawing, the resistance for slipping caused by dislocation cells and dislocation pileup induced the deformation twins, as shown in Fig.3a. As it was reported, the mechanism of deformation depended much on the stabilization of  $\beta$  phase<sup>[10,16]</sup>. Hanada et al reported that for a higher stabilization of  $\beta$  phase,  $\{332\}\langle 113\rangle$  twinning was replaced by  $\{111\}$  slipping and the mechanism of deformation of  $\beta$  titanium alloys changed from twinning to slipping. The image of dislocation structure of  $\beta$ -Ti matrix deformed by 60% reduction is shown in Fig.4a. As seen in the image, accompanying with shear bands, dislocation structure tended to form cell structure. The SAD patterns from the areas noted by arrow show that the grains obtained in this condition were much smaller than that of 20% reduction. When the alloy was drawn by 80% reduction, the equiaxed grains with the size of 20 nm-50 nm were obtained, as shown in Fig.4b. Comparing with the samples deformed by the reductions of 20%, 60% and 80%, respectively, grains could be divided into nano-size by shear bands gradually. Dislocations slipping, as the main mechanism of deformation, contributed much to the deformation.

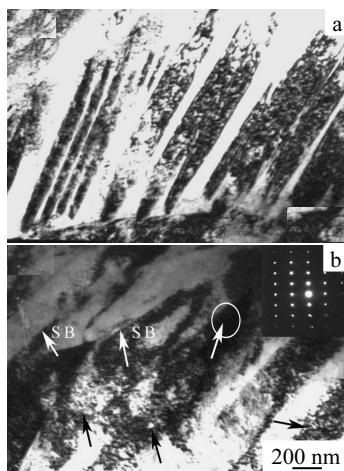


Fig.3 TEM micrographs of cold drawing samples deformed by 20% reduction: (a) the bright field image of deformation twins and (b) dislocation structure and narrow shear bands. Insert present SAD patterns from the areas noted by arrow

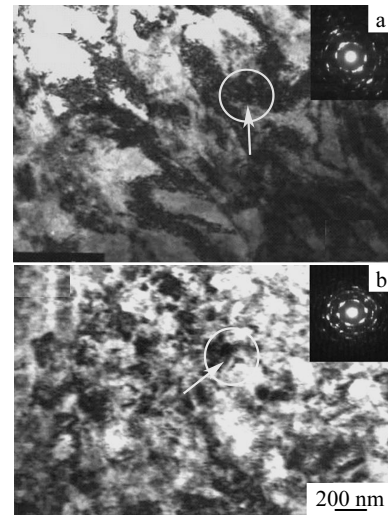


Fig.4 TEM images of deformed samples: (a)  $\varepsilon=60\%$  and (b)  $\varepsilon=80\%$ . Insert present SAD patterns from the areas noted by arrow

### 2.4 Effect of reduction of deformation on mechanical properties

Fig.5 shows the influence of cold deformation on mechanical properties of the specimens. Comparing with the solution-treated sample, when the reduction was low ( $\varepsilon<20\%$ ), the strength and plasticity changed quickly. The ultimate tensile strength reached 857 MPa and the elongation decreased to 9.5% in the condition of 20% reduction. As studied above, deformation twins occurred in the 20% reduction sample. These platelet-shape twins strengthened the alloy at a large extent. With the increase of the reduction, the strength increased gradually and the plasticity decreased slowly. When the reduction reaches 80%, the ultimate tensile strength went up to 1170 MPa while the elongation still kept around 10%. According to Hall-Patch equation:  $\sigma_s = \sigma_0 + Kd^{-1/2}$ , where  $\sigma_s$  is yield strength,  $\sigma_0$  is initial strength,  $K$  is constant, and  $d$  is grain size. The grains size ranging from 20 nm to 50 nm caused by cold drawing contributed much to the strength of the alloy.

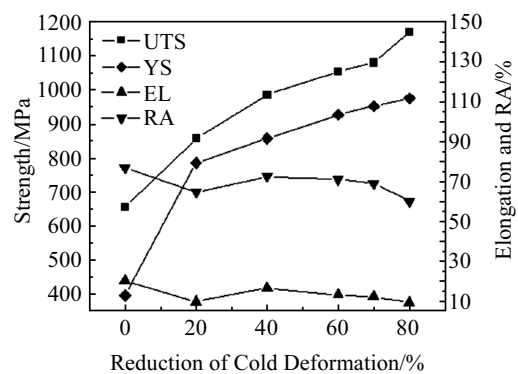


Fig.5 Influence of cold deformation on mechanical properties of TiNbZr specimens

### 3 Conclusions

1) No  $\alpha''$  martensite phase is detected for the alloy treated by solid solution and cold drawing.

2) Deformation twins appear when the reduction of cold deformation is around 20%, which strengthens the alloy and induces further plastic deformation drastically.

3) When the reduction is over 20%, the strength increases gradually and the plasticity nearly keeps the same. When the reduction of cold deformation increases to 80%, the ultimate tensile strength goes up to 1170 MPa and the elongation is above 10%. Both excellent strength and plasticity are obtained during cold drawing.

4) Cold drawing contributes much to grain refinement. Small grains ranging from 20 nm to 50 nm are obtained when the reduction is 80%.

### References

- Long M, Rack H J. *Biomaterials*[J], 1998, 19: 1621
- Nagai Y, Toyama T, Tang Z et al. *Scripta Mater*[J], 2006, 54: 1751
- Kim H Y, Sasaki T, Okutsu K et al. *Acta Mater*[J], 2006, 54: 423
- Kuroda D, Niinomi M, Morinaga M et al. *Mater Sci Eng A*[J], 1998, 243: 244
- Miyazaki S, Kim H Y, Hosoda H. *Mater Sci Eng A*[J], 2006, 438-440: 18
- Yang G J, Zhang T. *J Alloys Compd*[J], 2005, 392: 291
- Ho W F, Ju C P, Chern Lin J H. *Biomaterials*[J], 1999, 20: 2115
- Kim J I, Kim H Y, Inamura T et al. *Mater Sci Eng A*[J], 2005, 403: 334
- Hanada S, Yoshio T, Nishimura T et al. *Sixth World Conference on Titanium*[C]. Paris: Societe Francaise de Metallurgie, 1988: 105
- Hanada S, Izumi O. *Metall Trans*[J], 1987, 2: 265
- Kiritani M. *Mater Sci Eng A*[J], 2003, 350: 1
- Yasunaga K, Iseki M, Kiritani M. *Mater Sci Eng A*[J], 2003, 350: 76
- Saito T, Furuta T, Hwang J H et al. *Science*[J], 2003, 300: 464
- Gutkin M Y, Ishizaki T, Kuramoto S et al. *Acta Mater*[J], 2006, 54: 2489
- Yang Y, Li G P, Cheng G M et al. *Scripta Mater*[J], 2008, 58: 9
- Abdel-Hady M, Hinoshita K, Morinaga M. *Scripta Mater*[J], 2006, 55: 477

## 冷拉过程中 TiNbZr 合金的微观组织和力学性能

王立强<sup>1</sup>, 杨冠军<sup>2</sup>, 杨华斌<sup>3</sup>, 曹继敏<sup>3</sup>, 吕维洁<sup>1</sup>, 张 荻<sup>1</sup>

(1. 上海交通大学 金属基复合材料国家重点实验室, 上海 200240)

(2. 西北有色金属研究院, 陕西 西安 710016)

(3. 西安赛特金属材料开发有限公司, 陕西 西安 710016)

**摘要:** 研究了 TiNbZr 生物  $\beta$  钛合金的冷加工性能。TiNbZr 合金由真空自耗电弧炉熔炼, 实验过程中采用冷拉变形方式。在冷拉过程中, 合金表现出良好的冷加工性能。当冷变形率在 20% 左右时, 出现变形孪晶, 使得合金强度有大幅度提高。在随后的冷变形过程中, 位错滑移为主要的塑性变形方式。当冷变形率为 80% 时, 抗拉强度达到 1170 MPa, 延伸率也大于 10%。在该冷变形率下, 晶粒得到显著细化, 晶粒尺寸在 20 nm 到 50 nm 之间。

**关键词:**  $\beta$  钛合金; 冷拉; 微观组织; 力学性能

---

作者简介: 王立强, 男, 1980 年生, 博士生, 上海交通大学 金属基复合材料国家重点实验室, 上海 200240, 电话: 021-54749117, E-mail: wang\_liqiang@163.com