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Tensile properties and plastic deformation modes of β Zr–Nb alloys

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Tensile properties and plastic deformation modes of β zirconium–niobium alloys were investigated at 290 and 77 K in the wide composition range from metastable to stable β phase. Three types of plastic deformation modes, $\{332\}\langle 113\rangle$ twinning, $\{112\}\langle 111\rangle$ twinning and slip, were observed depending on alloy composition and temperature. $\{332\}\langle 113\rangle$ twinning, which occurs in metastable β zirconium alloys, is related to the stability of β phase to ω decomposition and leads to low yield stress and large elongation. On the other hand, $\{112\}\langle 111\rangle$ twinning, which appears in stable β zirconium alloys, results from high critical stress for slip due to solution hardening and high Peierls stress and does not affect tensile properties significantly. The results obtained for zirconium–alloys are similar to those for β titanium alloys, strongly suggesting that $\{332\}\langle 113\rangle$ twinning is an important plastic deformation mode which is common to β phase alloys containing athermal ω phase.

1. Introduction

It has been revealed that the deformation behaviour of metastable β titanium alloys depends significantly on the alloying content [1–4]. This was caused mainly by the change of plastic deformation mode from $\{332\}\langle 113\rangle$ twinning to slip with increasing alloying content. It has also been suggested that twinning occurs in metastable β titanium alloys where the shuffling of atoms required for the twinning is favourable [5]. Zirconium is the IVa element identical to titanium, and it is known that the structure and decomposition of β phase titanium and zirconium alloys are quite similar [6]. Therefore, if the above-described criterion for the occurrence of twinning is realistic, $\{332\}\langle 113\rangle$ twinning should occur in metastable β phase zirconium alloys and affect the mechanical properties. Very little information is, however, available on plastic deformation modes in β zirconium alloys.

On the other hand, twinning has recently been found to occur in stable β titanium alloys having high critical stress for slip due to solution hardening and high Peierls stress [7]. Thus, $\{112\}\langle 111\rangle$ twinning is also expected to occur in stable β zirconium alloys.

The purpose of the present work is to examine plastic deformation modes and to report the interrelationship among plastic deformation modes, stability of β phase and mechanical properties in β zirconium alloys with the wide composition range from metastable to stable β phase.

2. Experimental details

Zirconium–niobium alloys containing 13.5 to 80 mass % niobium were arc melted under an argon atmosphere. In this paper niobium-rich alloys are also called β zirconium alloys for convenience. The arc melted buttons with a weight of approximately 100 g were hot rolled to 3 mm thick plates, scalped, and then cold rolled to 1.5 mm thick plates. Tensile specimens with dimensions of 3 mm × 1.5 mm × 16 mm were spark-machined from the plates and sealed in a quartz tube under a vacuum of 2×10^{-3} Pa. They were homogenized at various temperatures shown in Table I to obtain a constant grain size of 100 to 120 μm and quenched into iced water by breaking the quartz tube immediately after quenching, followed by mechanical and chemical polishing. Tensile tests at 290 and 77 K were performed on an Instron type testing machine at an initial strain rate of 5.2×10^{-4} sec^{-1}.

3. Results and discussion

Plastic deformation modes of slightly deformed zirconium–niobium alloys were determined by optical and transmission electron microscopy. On deformation at 290 K, optical microscopy revealed that there were three types of microstructures, as shown in Fig. 1. Figure 1a shows broad bands observed in Zr–13.5% Nb. Similar broad bands were also observed in Zr–15 and 17% Nb, although the density of the bands decreased with increasing niobium content. In addition to the broad bands, a fine distribution of Widmanstätten plates is seen in Fig. 1a. The amount of the Widmanstätten plates was a function of niobium content, i.e. it was suppressed as niobium content increased, and no plate was observed in Zr–Nb alloys containing more than 50% niobium. Crystal structure of the Widmanstätten plates was not examined in this work, because it is unlikely that the plates affect plastic deformation modes or tensile properties

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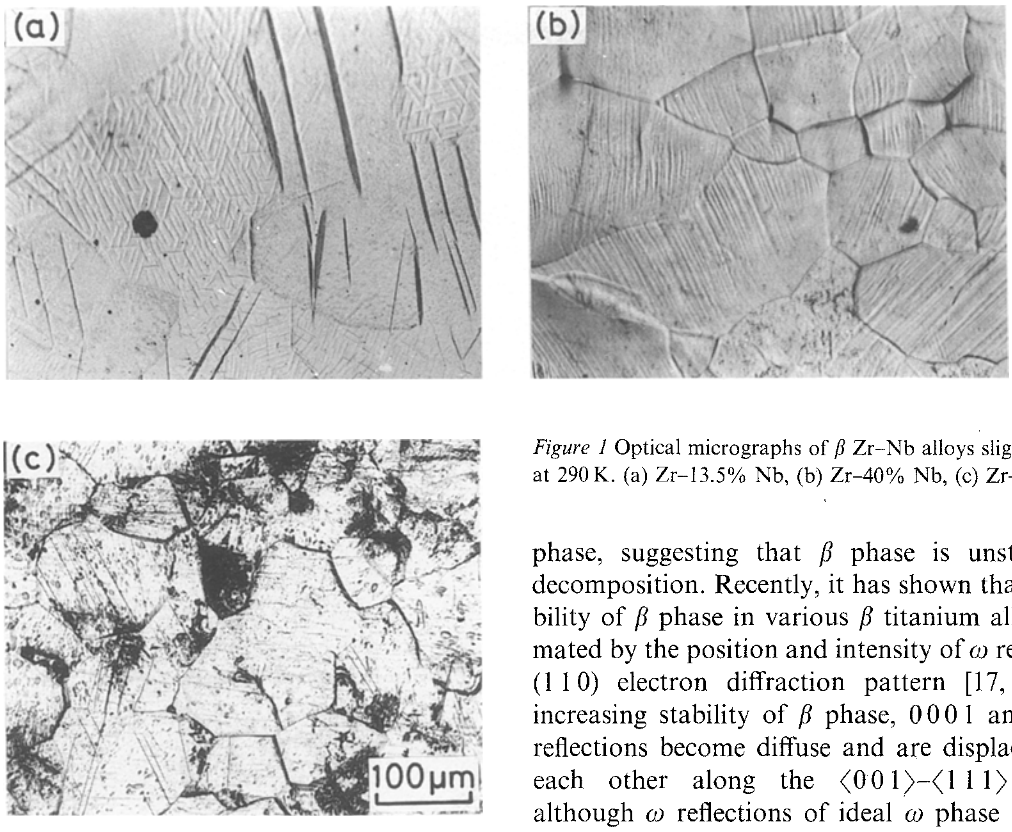

Figure 1 Optical micrographs of β Zr–Nb alloys slightly deformed at 290 K. (a) Zr–13.5% Nb, (b) Zr–40% Nb, (c) Zr–50% Nb.

significantly as described later. Fig. 1b shows fine wavy traces observed in Zr–40% Nb. Such wavy traces appeared in Zr–19, 22, 30 and 80% Nb. In Zr–50 and 60% Nb straight narrow bands formed (Fig. 1c).

Fig. 2a shows a transmission electron micrograph of a broad band. One can see that a high density of dislocations is introduced in the band. Electron diffraction patterns in Figs 2b, c and d were taken from the boundary region, matrix and band, respectively. Fig. 2e illustrates a key diagram of Fig. 2b, indicating that the broad band is a $(2\,3\,3)[\bar{3}\,1\,1]$ twin. $\{3\,3\,2\}\langle 1\,1\,3\rangle$ twinning has been widely observed in metastable β phase titanium alloys, such as Ti–V [1, 2, 5, 8], Ti–Mo [9–13], Ti–Nb [14], Ti–Fe [15] and Ti–Cr [16] alloys. The occurrence of $\{3\,3\,2\}\langle 1\,1\,3\rangle$ twinning, not $\{1\,1\,2\}\langle 1\,1\,1\rangle$ which is common in b c c alloys, has been explained in terms of instability of β phase to ω decomposition. That is, the explanation is based on an idea that shuffling of atoms required for $\{3\,3\,2\}\langle 1\,1\,3\rangle$ twinning is easier in metastable β phase than in stable β. Fig. 2c shows clearly the existence of athermal ω phase, suggesting that β phase is unstable to ω decomposition. Recently, it has shown that the instability of β phase in various β titanium alloys is estimated by the position and intensity of ω reflections in $(1\,1\,0)$ electron diffraction pattern [17, 18]. With increasing stability of β phase, $0\,0\,0\,1$ and $0\,0\,0\,2$ ω reflections become diffuse and are displaced toward each other along the $\langle 0\,0\,1\rangle$–$\langle 1\,1\,1\rangle$ direction, although ω reflections of ideal ω phase lie on lines joining b c c reflections. Fig. 3 shows composition dependence of d^*_{0002}/d^*_{222} in Zr–Nb alloys. The obtained result is in good agreement with that of Sass [6]. It should be noted that $\{3\,3\,2\}\langle 1\,1\,3\rangle$ twinning was observed predominantly in Zr–13.5, 15 and 17% Nb having d^*_{0002}/d^*_{222} of larger than 0.66, since similar observations have been made for Ti–V and Ti–Mo alloys [17]. It is concluded from these results on β Zr and Ti alloys that $\{3\,3\,2\}\langle 1\,1\,3\rangle$ twinning is operative as a common mode of plastic deformation when β phase is unstable to ω phase decomposition and contains athermal ω particles which raise critical stress for slip.

Only dislocations were observed in Zr–19, 22, 30, 40 and 80% Nb. Therefore, it is evident that wavy traces in Fig. 1b indicate slip markings. On the other hand, narrow bands as well as dislocations were observed in Zr–50 and 60% Nb. A transmission electron micrograph of the narrow band is shown in Fig. 4. A diffraction pattern taken from a boundary region of the band (Fig. 4b) and a key diagram of the pattern (Fig. 4c) indicate that the narrow band is a $(\bar{1}\,\bar{1}\,2)$ $[1\,1\,1]$ twin.

Tensile properties at 290 K are summarized in Fig. 5, where the composition dependence is divided

TABLE I Heat treatments and content of interstitial elements (mass %)

Alloys	Heat treatments	Oxygen	Nitrogen	Hydrogen
Zr–13.5% Nb	1123 K, 7.2 k sec	0.071	0.0016	0.0025
Zr–15% Nb	1123 K, 7.2 k sec	0.064	0.0004	0.0028
Zr–17% Nb	1123 K, 7.2 k sec	0.063	0.0005	0.0042
Zr–19% Nb	1123 K, 7.2 k sec	0.063	0.0009	0.0051
Zr–22% Nb	1123 K, 7.2 k sec	0.062	0.0013	0.0035
Zr–30% Nb	1273 K, 7.2 k sec	0.066	0.0014	0.0061
Zr–40% Nb	1273 K, 7.2 k sec	0.063	0.0019	0.0053
Zr–50% Nb	1323 K, 36 k sec	0.075	0.0021	0.0057
Zr–60% Nb	1323 K, 36 k sec	0.060	0.0067	0.0074
Zr–80% Nb	1473 K, 10.8 k sec	0.045	0.0024	0.0106

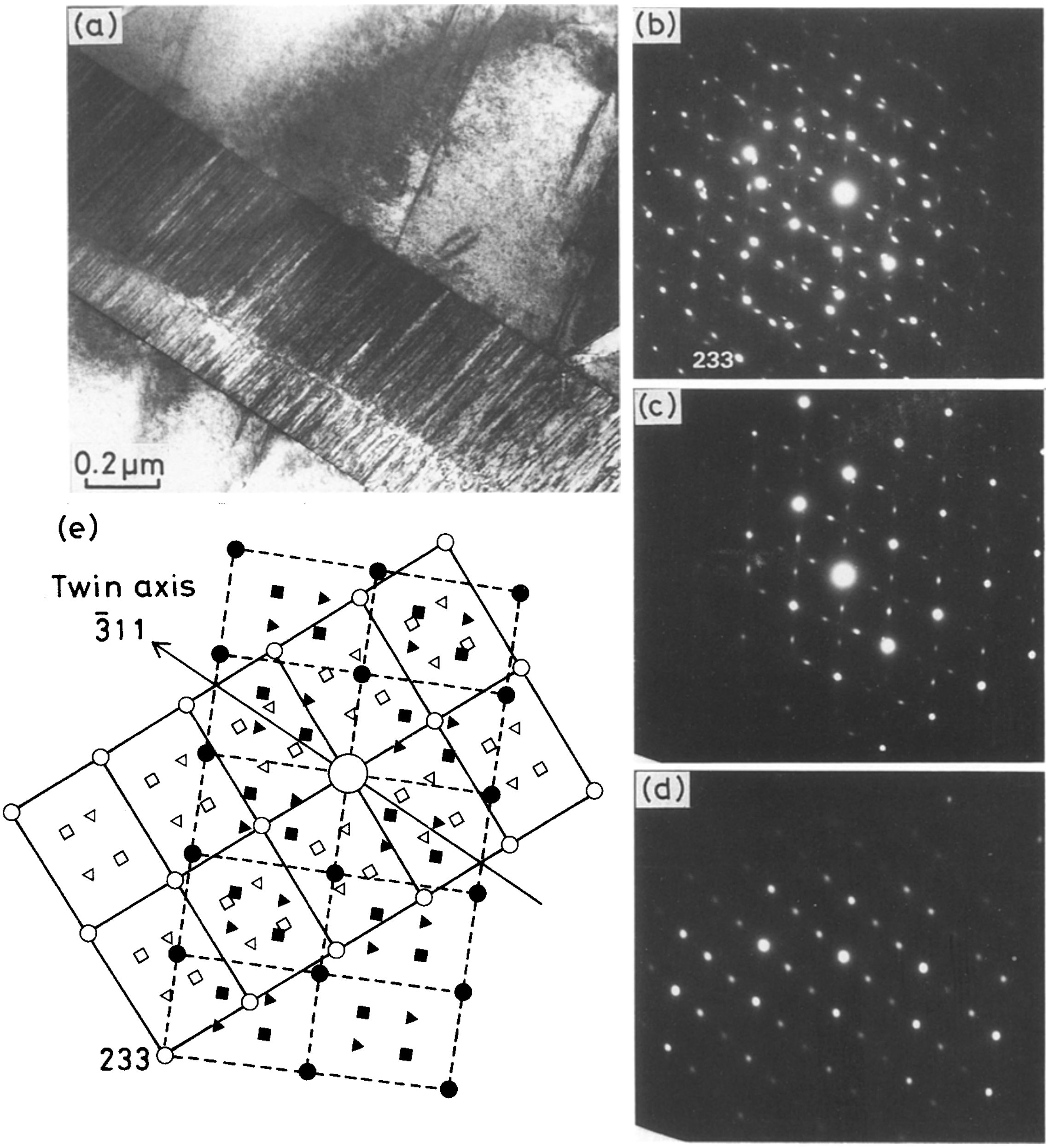

Figure 2 (a) Transmission electron micrograph of a broad band in Zr–15% Nb. Diffraction patterns taken from boundary region (b), matrix (c) and band (d). (e) key diagram of (b). (Matrix: ○ bcc, □ ω_1, △ ω_2; twin: ● bcc, ■ ω_1, ▲ ω_2.)

into four regions I to IV depending upon the plastic deformation mode. Yield stress ($\sigma_{0.2}$) in region I, where the deformation mode is mainly $\{332\}\langle 113\rangle$ twinning, is low, while in region II, where only slip operates, yield stress increases with increasing niobium content. Since the number of Widmanstätten plates decreases in this region as niobium content increases, the plates are not ascribed to the increase of yield stress. Moreover, the intensity of ω reflections also decreases with increasing niobium content, indicating that ω particles are not responsible for the increase of yield stress. In addition, as shown in Table I, oxygen content is almost constant in this composition range. On the other hand, it has been reported that niobium is remarkably solution-hardened by zirconium additions [19]. Therefore, the increase of yield stress is most likely caused by solid solution hardening accompanied with the increase of niobium content. Thus, $\{112\}\langle 111\rangle$ twinning seems to occur at a 50 at % solute content alloy which usually exhibits maximum solution strengthening.

Fracture stress (σ_B) decreases in region I with increasing niobium content. As shown in Fig. 2d, single variant ω phase transformation is enhanced in a stress-induced $\{332\}\langle 113\rangle$ twin. This tendency was pronounced as β phase became unstable; i.e. as niobium content decreases. High fracture stress in a low niobium content alloy can be explained by large work hardening due to the stress-induced ω phase transformation in $\{332\}\langle 113\rangle$ twins and a high density of $\{332\}\langle 113\rangle$ twins. Fracture stress in region II is close to yield stress at the corresponding composition. These samples exhibited low work hardening in a similar manner to many stable β phase titanium alloys [2]. Occurrence of $\{112\}\langle 111\rangle$ twins in region III leads to considerable work hardening and the resultant maximum fracture stress.

Elongation to fracture and reduction of area are

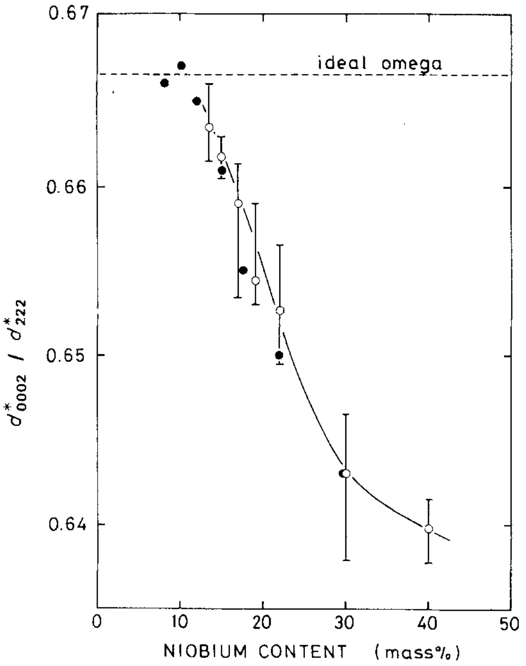

Figure 3 Composition dependence of reciprocal distance ratio d^*_{0002}/d^*_{222} in Zr–Nb alloy. (○ this work, ● Sass).

low in the most unstable β phase alloy (Zr–13.5% Nb), which is related to stress-induced ω phase transformation in {3 3 2}⟨1 1 3⟩ twins. Maximum elongation is obtained when {3 3 2}⟨1 1 3⟩ twinning occurs and ω phase transformation is not induced significantly in twins (Zr–15 and 17% Nb). In higher niobium content alloys elongation seems to be independent of composition. However, reduction of area has a maximum in a 40% niobium alloy in which β phase is stable and deformation mode is slip.

On deformation at 77 K, three types of plastic deformation modes were also observed. Typical examples of optical micrographs are shown in Fig. 6. Transmission electron microscopy revealed that broad bands in Fig. 6a and narrow bands in Fig. 6c correspond to {3 3 2}⟨1 1 3⟩ and {1 1 2}⟨1 1 1⟩ twins, respectively. In addition, wavy traces in Fig. 6b were found to be mainly slip markings and in this sample few twins of {1 1 2}⟨1 1 1⟩ were observed.

Tensile properties at 77 K are summarized in Fig. 7, where the composition dependence is divided into three regions I to III depending upon plastic deformation mode. Yield stress in region I, where deformation mode is mainly {3 3 2}⟨1 1 3⟩ twinning, is low. In region II, where only slip operates, yield stress increases, as niobium content increases. These characteristics in regions I and II are quite similar to those at 290 K. In region II, however, yield stresses at 77 K are much higher than those at 290 K, which can be interpreted in terms of temperature dependence of Peierls stress in bcc structure. Yield stress in region III, where {1 1 2}⟨1 1 1⟩ twinning occurs with slip, is lower than that of Zr–30% Nb deformed only by slip. As seen in Fig. 5, yield stress of the alloys containing between 40 and 80% niobium are higher than that of Zr–30% Nb. Therefore, {1 1 2}⟨1 1 1⟩ twinning seems to occur in alloys having high yield stress for slip due to solution hardening and high Peierls stress at low temperature.

Fracture stress in region I is relatively high, which is again related to stress-induced {3 3 2}⟨1 1 3⟩ twinning accompanied with ω phase transformation. Fracture stress of the alloys in regions II and III increases as the composition approaches 50% Nb. Two sharp peaks are seen in composition dependence of elongation. It is likely that the large elongation in Zr–15% Nb is due to occurrence of {3 3 2}⟨1 1 3⟩ twinning, while small elongation in Zr–13.5% Nb is related to the brittleness caused by the large amount of ω phase formed during {3 3 2}⟨1 1 3⟩ mechanical

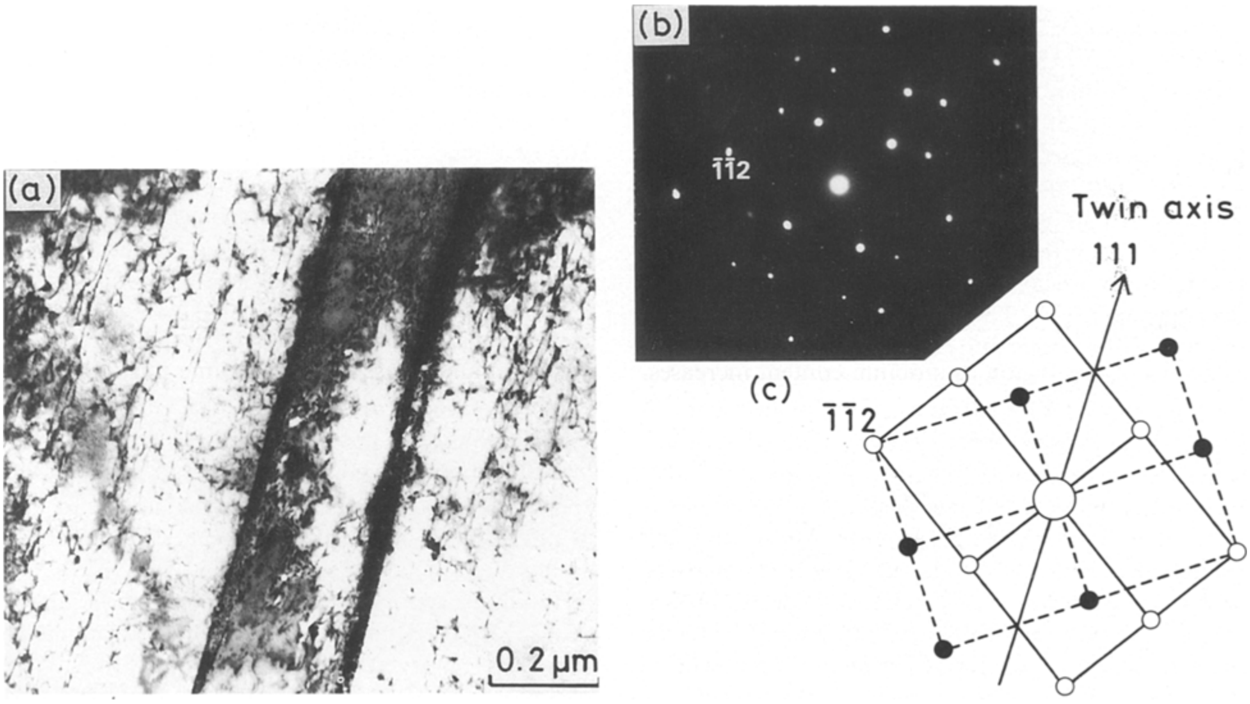

Figure 4 (a) Transmission electron micrograph of a narrow band in Zr–60% Nb deformed at 290 K, (b) Diffraction pattern taken from a boundary region, (c) key diagram of (b). (matrix; ○ bcc; twin: ● bcc).

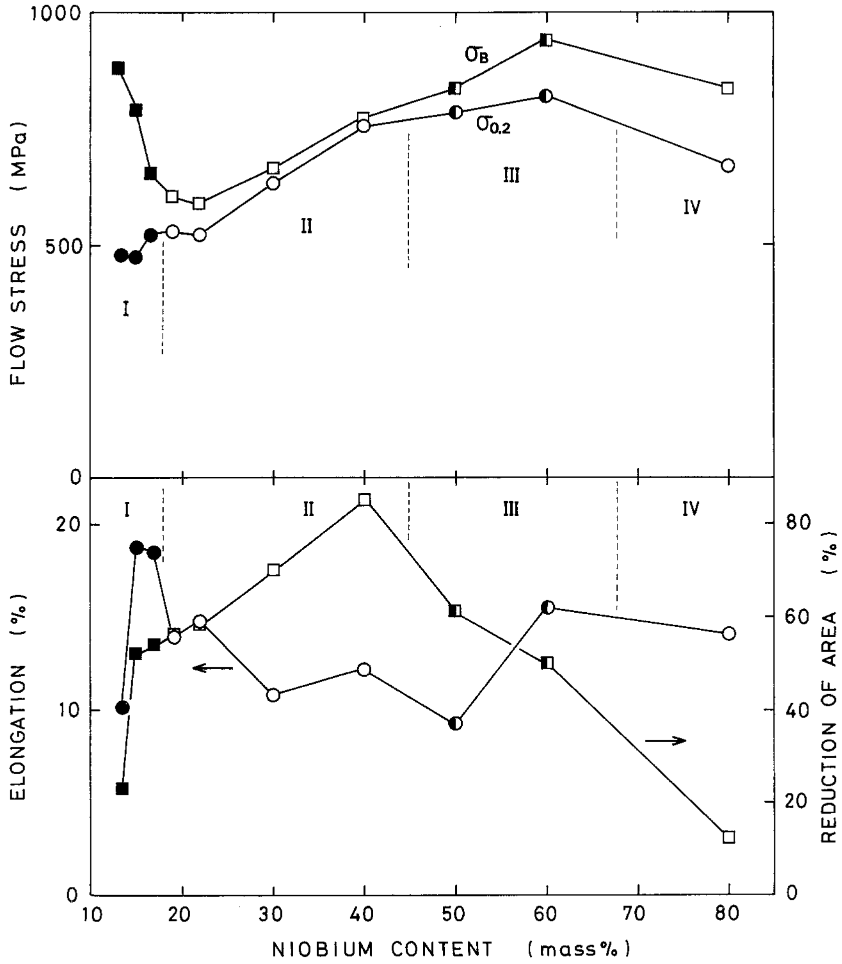

Figure 5 Composition dependence of tensile properties in Zr–Nb alloys deformed at 290 K. (■● $\{332\}\langle 113\rangle$ twinning, □○ slip, ◧◐ $\{112\}\langle 111\rangle$ twinning and slip)

twinning. On the other hand, large elongation in stable Zr–50% Nb may be explained by appropriate coexistence of $\{112\}\langle 111\rangle$ twinning and slip, because stable β titanium alloys which deform only by slip usually exhibit small work hardening, large reduction of area and the resultant small elongation. This explanation is consistent with the fact that large elongation in metastable β titanium alloys is obtained with large work hardening rate which results from formation of $\{332\}\langle 113\rangle$ twins, even if ω phase transformation is not induced in the twins. Elongation and reduction of area in Zr–80% Nb are extremely low. Scanning electron microscopy on fracture surface revealed that although all the alloys other than Zr–80% Nb showed dimple type transgranular fracture, Zr–80% Nb did coexistence of intergranular and transgranular cleavage fracture in agreement with previous work [20]. This means that Zr–80% Nb has ductile–brittle transition temperature between 290 and 77 K, which is characteristic of deformation in bcc metals and alloys. The cause of the brittleness is not clear at present, but it may be related to crack initiation accompanied with twin formation.

Another marked change between 290 and 77 K was found in Zr–17 and 19% Nb. Elongation and reduction of area at 77 K is very low in comparison with those at 290 K. Nevertheless, dimple-type transgranular fracture was observed at 290 and 77 K. To investigate the cause, X-ray diffraction and electron diffraction analysis were carried out at low temperatures. Intensity of 0002 ω reflection was found to increase in Zr–17 and 19% Nb with decreasing temperature. No apparent change, however, was observed in other alloys. This result is in agreement with previous work [21, 22]. Therefore, remarkable embrittlement at 77 K in Zr–17 and 19% Nb can be explained by taking account of athermal ω phase transformation during cooling. As shown in Figs 5 and 7, deformation mode in Zr–17% Nb changes from $\{332\}\langle 113\rangle$ twinning at 290 K to slip at 77 K, suggesting that considerable amounts of ω phase formed during cooling suppresses twinning. Furthermore, reduction of area in Zr–13.5, 15, 17, and 19% Nb is comparably low. This implies that large amount of ω phase is formed during deformation and/or cooling. It is known that formation of athermal or isothermal ω phase lowers elongation and reduction of area and makes fracture surface dimple type transgranular. Thus, low ductility at 77 K in Zr–17 and 19% Nb is considered to result from ω embrittlement.

4. Conclusions

Tensile properties and plastic deformation modes of β Zr–Nb alloys were investigated at 290 and 77 K over the wide composition range from metastable to stable β phase. The obtained results are summarized as follows:

1. Three types of plastic deformation modes, $\{332\}\langle 113\rangle$ twinning, $\{112\}\langle 111\rangle$ twinning and slip, were observed in the sequence of $\{332\}\langle 113\rangle$ twinning → slip → $\{112\}\langle 111\rangle$ twinning (→slip) with increasing niobium content.
2. The transition of $\{332\}\langle 113\rangle$ twinning → slip

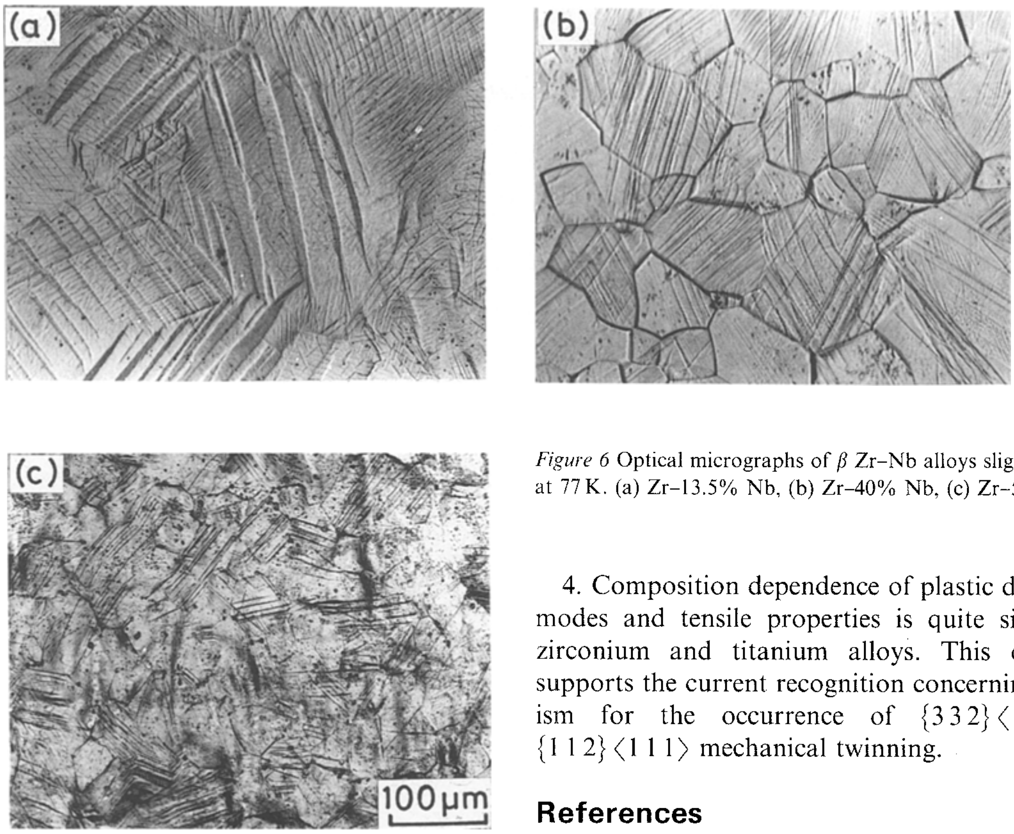

Figure 6 Optical micrographs of β Zr–Nb alloys slightly deformed at 77 K. (a) Zr–13.5% Nb, (b) Zr–40% Nb, (c) Zr–50% Nb.

was related to the stability of β phase to ω decomposition. $\{3\,3\,2\}\langle 1\,1\,3\rangle$ twinning leads to low yield stress and large elongation except in the most unstable Zr–13.5% Nb where formation of large amounts of quenched ω reduces elongation.

3. $\{1\,1\,2\}\langle 1\,1\,1\rangle$ twinning occurs in stable β Zr–Nb alloys. Occurrence of $\{1\,1\,2\}\langle 1\,1\,1\rangle$ twinning can be explained in terms of high critical stress of slip which results from solution hardening.

4. Composition dependence of plastic deformation modes and tensile properties is quite similar in β zirconium and titanium alloys. This observation supports the current recognition concerning mechanism for the occurrence of $\{3\,3\,2\}\langle 1\,1\,3\rangle$ and $\{1\,1\,2\}\langle 1\,1\,1\rangle$ mechanical twinning.

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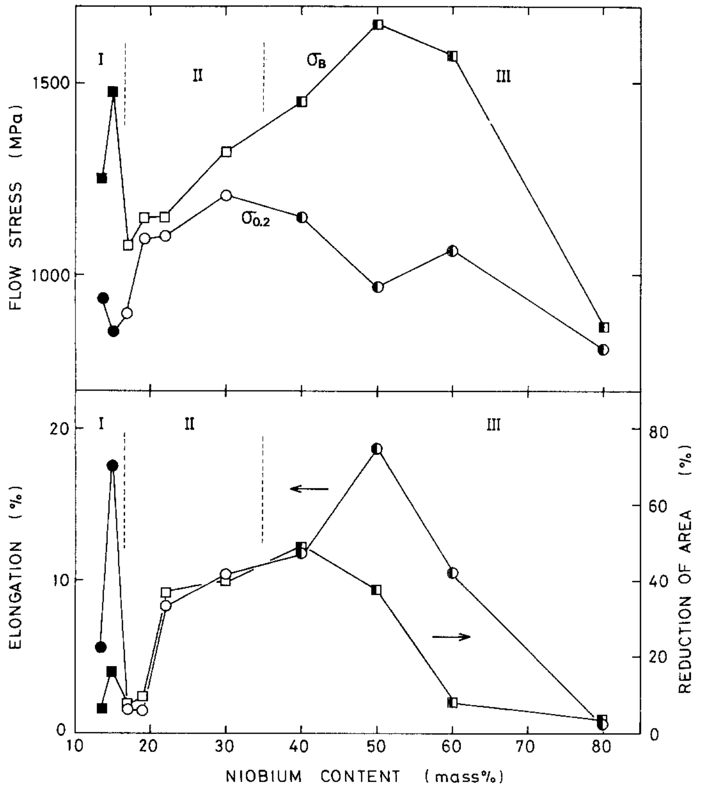

Figure 7 Composition dependence of tensile properties in Zr–Nb alloys deformed at 77 K. (■● $\{3\,3\,2\}\langle 1\,1\,3\rangle$ twinning, □○ slip, ◧◐ $\{1\,1\,2\}\langle 1\,1\,1\rangle$ twinning and slip)

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