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Fabrication of Beta-Ti-Type Ti-Nb-Ta-Zr (TNTZ) Wire with High-Ductility by Arc-Melt-Type Melt-Extraction Method

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Beta-Ti-type Ti-30Nb-10Ta-5Zr (TNTZ) wires with high-ductility were developed using the arc-melt-type melt-extraction method. The continuous melt-extracted wire with very smooth surface and small fluctuations in the diameter realizes the high tensile strength and superior bending ductility of TNTZ alloy. The melt-extracted Beta-Ti wire shows the possibility for the use in biomedical applications. The arc-melt-type melt-extraction method is very effective for developing new Beta-Ti-based biomaterials. [doi:10.2320/matertrans.M2009163]

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1. Introduction

Among metallic biomaterials such as stainless steels, Co-Cr alloys, bulk metallic glasses (BMGs), pure Ti, and Ti alloys, Beta-Ti-type Ti-Nb-Ta-Zr (TNTZ) quaternary alloys with a bcc structure are most suitable for biomaterials. This is because of some of their attributes such as low Young's modulus, low cyto-toxicity, high specific strength, and superior corrosion resistance.¹⁻⁴ If TNTZ alloys are to be used in biomedical applications, we must first ensure whether the biomaterials having different shapes can be prepared using these alloys. Recently, we succeeded firstly in fabricating the continuous, rapidly quenched Ti-rich Ti-Zr wires containing no bio-toxic elements by arc-melt-type melt-extraction method.⁵ These continuous Ti-Zr alloy wires exhibit a good white luster, very smooth surface, high tensile strength, and good bending ductility. In addition, their diameter is almost constant. The arc-melt-type melt-extraction method can be effectively used for fabricating new Ti-based metallic crystalline wires for biomaterials. Binary Ti-Zr alloys have a good wire-forming ability. However, these wires do not exhibit a Beta-phase with a bcc structure; instead, they exhibit an Alpha-phase with an hcp structure. Hitherto, the fabrication of a melt-extracted Beta-Ti metallic wire has not been reported. In this study, we have attempted to fabricate melt-extracted TNTZ alloy wires with a Beta-phase by the arc-melt-type melt-extraction method.

2. Experimental Procedure

Ti₄₅Nb₃₀Ta₁₀Zr₅ (Ti-30Nb-10Ta-5Zr) alloy, which has a bcc structure was used in the experiment. We also fabricated Alpha-Ti former type Ti_{67.7}Zr_{32.3} (Ti-32.3Zr, Ti-Zr) alloy wires for the purpose of comparison.⁵ The compositions are expressed in nominal weight per cent. A master ingot was prepared by arc melting a mixture of pure Ti, Nb, Ta, and Zr (purity > 99.9%) in a highly purified Ar gas atmosphere. Rapidly solidified wires were produced by the arc-melt-type

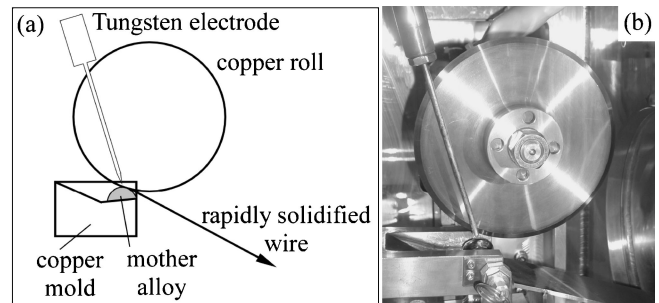


Fig. 1 Schematic illustration of arc-melt-type melt-extraction method used in this study. (a) Schematic illustration of the method and (b) photograph of the apparatus.

melt-extraction method.⁵⁻⁷ Figure 1 shows (a) a schematic illustration and (b) the corresponding image of the apparatus (NISSIN-GIKEN, NEV-AT3). The diameter of the Cu-roll used in this method was 200 mm, and the angle of the edge was fixed at 60°. The rotation speed was maintained at 1000 rotation per minutes (rpm) or 2000 rpm, and the circumferential velocity of the Cu roll was 21 ms⁻¹ in the case of 2000 rpm. Mother alloys were arc melted from the master ingots under an Ar gas atmosphere on a water-cooled Cu mold. This apparatus allowed us to control the position of the Cu roll. The extraction of the molten mother alloy was performed by moving down the rotating Cu roll. The above conditions are the similar as those used in the fabrication of the Ti-Zr alloys⁵ and Zr-based metallic glasses.⁶ The constituent phases of the wire were confirmed by X-ray diffraction (XRD) using Cu-K α radiation, differential scanning calorimetry (DSC) at a heating rate of 0.67 Ks⁻¹, and optical microscopy (OM). The surface morphology of the wires was examined by OM and scanning electron microscopy (SEM). The tensile strength of the wire specimens was measured by an Instron-type testing machine at a strain rate of 4.2 \times 10⁻⁴ s⁻¹ in air. The length of wire for mechanical tensile strength measurement is 10 mm. The wire elongation was measured by the position of the crosshead and not by

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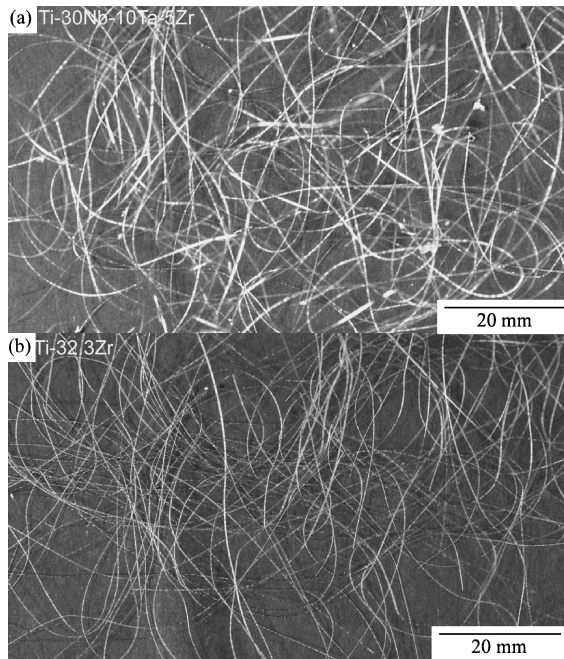


Fig. 2 Outer appearance of melt-extracted Ti-based alloy wires prepared at 2000 rpm; (a) Ti-30Nb-10Ta-5Zr and (b) Ti-32.3Zr.

using a strain gage. The ductility of the wires was evaluated by means of a simple bending test. A ductile wire is one that can be bent through 180° without fracture.

3. Results and Discussions

Figure 2 shows the outer appearance of melt-extracted Ti-based alloy wires fabricated at 2000 rpm. The TNTZ alloy (a) exhibits a good wire-forming ability and therefore continuous wires can be produced from it. The length and fluctuations in diameter of the TNTZ wire are similar to those of the Ti-Zr wire (b). A corresponding decrease in the diameter of the TNTZ wire is observed with an increase in the rotation speed of Cu; however, the length of the wire is not sensitive to the rotation speed. The constituent phases of melt-extracted TNTZ and Ti-Zr wires prepared at a rotation speed of 2000 rpm were investigated using XRD analysis and DSC measurement. Figure 3 shows the XRD patterns of melt-extracted alloy wires. Sharp diffraction peaks in the XRD pattern of the Ti-Zr wire were identified as those belonging to the Alpha-(Ti, Zr) phase. In contrast, the TNTZ wire showed sharp diffraction peaks corresponding to the Beta-phase with a bcc structure. The formation of the Alpha-phase in TNTZ wires cannot be detected by XRD analysis. Figure 4 shows the DSC curves of a melt-extracted wire at a heating rate of 0.67 K s^{-1} . The Ti-Zr wire shows properties similar to that of an endothermic reaction. This is due to the phase transition of the Ti-Zr wire from the Alpha-phase to the Beta-phase.⁵⁾ The TNTZ wire does not exhibit any sharp endothermic or exothermic peaks. The XRD analysis and DSC measurement confirm the formation of a Beta-single phase in the TNTZ wire.

Figure 5 shows typical examples of the outer surface appearance of the melt-extracted TNTZ wire ((a) and (b)) and the Ti-Zr wire ((c) and (d)). The OM images of the TNTZ

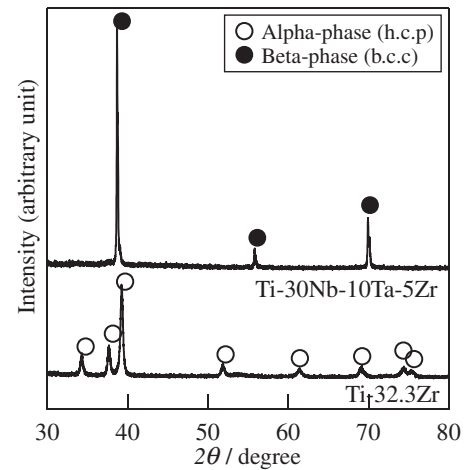


Fig. 3 X-ray diffraction patterns of melt-extracted Ti-30Nb-10Ta-5Zr (TNTZ) and Ti-32.3Zr (Ti-Zr) wires.

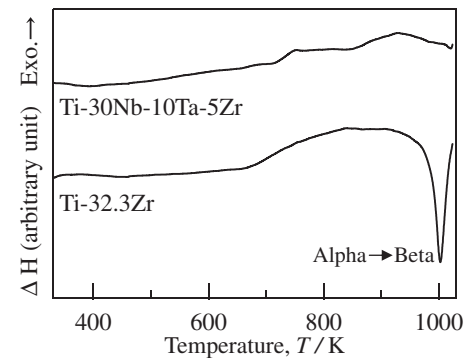


Fig. 4 DSC curves of melt-extracted Ti-30Nb-10Ta-5Zr (TNTZ) and Ti-32.3Zr (Ti-Zr) wires obtained at a heating rate of 0.67 K s^{-1} .

wire (a) show no fluctuation in diameter, but a small amount of fluctuation, indicated by white arrows, can be observed in the images of the Ti-Zr alloy wire (c). From Figs. 5(b) and (d), we can observe that the TNTZ and the Ti-Zr wires have a rough surface and not a mirror surface that is characteristic of melt-extracted amorphous wire.⁶⁾ In order to investigate the surface morphology of the melt-extracted wires in detail, we carried out SEM observations. The SEM images of the melt-extracted wires along with the SEM images of a conventional steel code are shown in Fig. 6. The SEM image of the TNTZ wire (a) shows a fine dendrite structure, while that of the Ti-Zr alloy wire (b) shows a facet structure. The facet structure was formed in the Alpha-Ti-type Ti-Zr wire with a wide composition range in the $\text{Ti}_{100-x}\text{Zr}_x$ ($x = 17.5\text{--}88.4$) alloys.⁶⁾ The surface morphology of the Beta-Ti-type TNTZ alloy wire was observed to be different from the surface morphologies of the Alpha-Ti-type Ti-Zr alloys. It should be noted that the degree of surface roughness of the melt-extracted TNTZ wire (a) and the Ti-Zr wire (b) is smaller than that of the conventional steel code prepared by wire-drawing in a mass production system (c). Rapid solidification of the extracted wire in the thermal-melt process during its free flight in an Ar gas atmosphere results in the formation of a fine crystalline precipitates formation, leading to a very smooth surface. The XRD and DSC measurements shown in Figs. 3 and 4 also confirm the formation of the crystalline

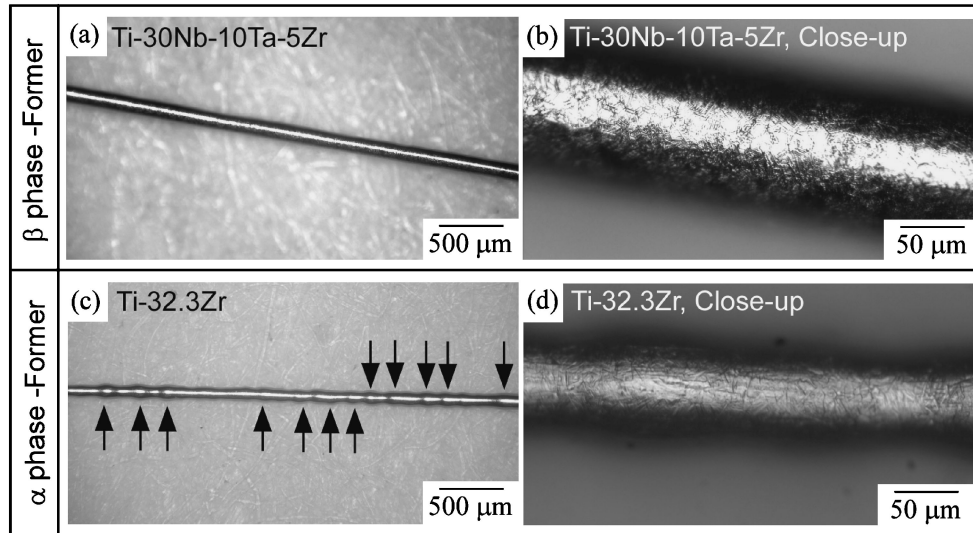


Fig. 5 Outer surface appearance of Beta-Ti-type melt-extracted Ti-30Nb-10Ta-5Zr (TNTZ) wires and Alpha-Ti-type Ti-32.3Zr (Ti-Zr) wires; (a) and (b) Ti-30Nb-10Ta-5Zr, and (c) and (d) Ti-32.3Zr.

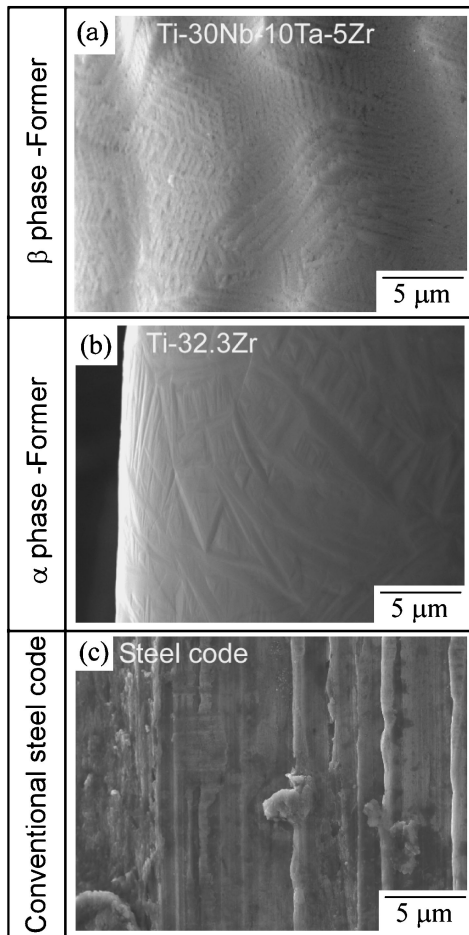


Fig. 6 SEM images of melt-extracted in Beta-Ti-type Ti-30Nb-10Ta-5Zr (TNTZ) and Alpha-Ti-type Ti-32.3Zr (Ti-Zr) wires; (a) Ti-30Nb-10Ta-5Zr, (b) Ti-32.3Zr, and (c) Conventional steel code.

precipitates on the wires. The difference in the morphological properties of the melt-extracted wires between the TNTZ and Ti-Zr alloys may be attributed to either the crystallization behavior of the melt or phase transition, or both during the free flight of the TNTZ and Ti-Zr wires in the Ar gas

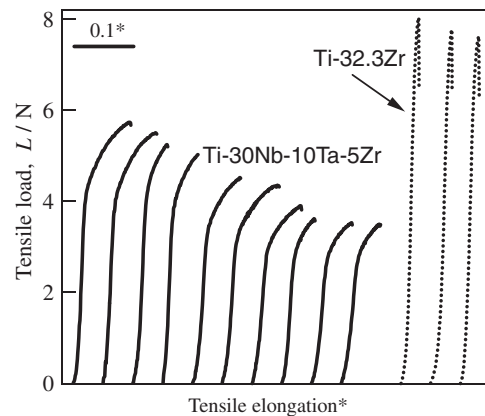


Fig. 7 Tensile load-elongation curves of melt-extracted wires in Beta-Ti-type Ti-30Nb-10Ta-5Zr (TNTZ) and Alpha-Ti-type Ti-32.3Zr (Ti-Zr) wires. (*) The length of wire for mechanical tensile strength measurement is 10 mm. The tensile elongation value of 0.1 means 1.0 mm elongation. The elongation of the wires was measured by determining the displacement of a crosshead and not by using a strain gage. The absolute values of the tensile stress and elongation could not be determined because of the fluctuations in the diameter of the wires.

atmosphere. The extremely fine dendrite formation in the TNTZ alloy leads to the formation of the continuous Beta-Ti wire with small fluctuation in diameter and significantly smooth surface roughness.

The mechanical properties of melt-extracted TNTZ crystalline wires were investigated by the tensile test in air. Figure 7 shows the tensile load-elongation curves of 10 samples and 3 examples of Ti-Zr wire.⁵⁾ In the present study, the tensile stress was not evaluated because of the fluctuations in diameter of the wire. The difference observed in the tensile load-elongation curve in each sample is because of the experimental difficulties in holding a wire of approximately 100 μm diameter in a chuck. The length of wire for mechanical tensile strength measurement is 10 mm. The tensile elongation value of 0.1 means 1.0 mm elongation. The wire elongation was measured by the position of the crosshead and not by using a strain gage. Thus, the absolute

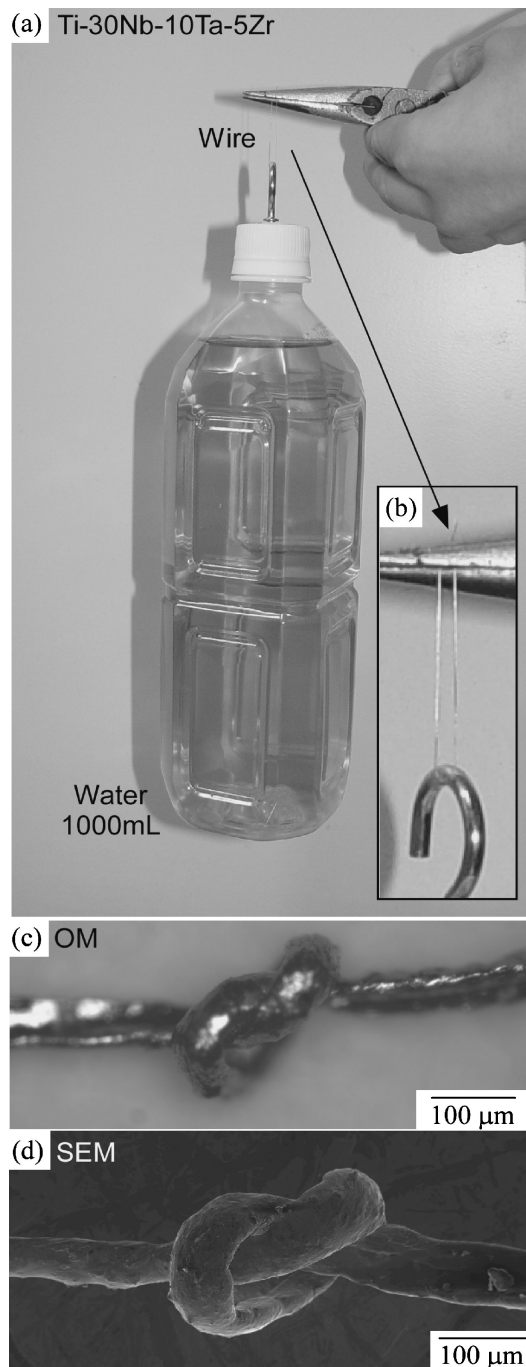


Fig. 8 Demonstration of high strength, full ductility and easy handling of melt-extracted Beta-Ti-type Ti-30Nb-10Ta-5Zr (TNTZ) wire. (a) A typical example of lifting a heavy load, (b) close up of (a), (c) OM image of the knot, and (d) SEM image of the knot.

values of tensile stress and tensile elongation cannot be discussed here. However, it can be observed that as compared to the Ti-Zr wire, the melt-extracted TNTZ wire exhibits superior mechanical properties for biomaterials, such as lower Young's modulus and a larger plastic deformation region. The melt-extracted TNTZ wire was bent through 180° without fracture. Thus, ductile Ti alloy wires with superior mechanical properties can be obtained using the TNTZ alloy. Figure 8 is a demonstration of the high tensile strength, ductility, and easy handling of one of the TNTZ melt-extracted wires. The TNTZ wire did not get fractured

when it was used to lift a heavy load of approximately 1 L water (a). The TNTZ wire can be bent through 180° without fracture in spite of the heavy load as shown in the close-up image (b). The wire was not damaged even when it was held tightly by a pair of needle-nose pliers. Furthermore, the OM and SEM images ((c) and (d), respectively) of the knot formed by the TNTZ wire highlight the full ductility of the wire. Thus, owing to their high tensile strength, low Young's modulus, large plastic deformation region, and superior ductility, the melt-extracted Beta-Ti-type TNTZ metallic wires can be used in biomedical applications. It is well known that Beta-Ti type TNTZ alloy shows high plasticity and ductility because of the b.c.c.-structure.¹⁻⁴⁾ The melt-extracted wire in the present study also exhibits the superior bending ductility, indicating that the very smooth surface without any crack initiation sites and significantly small fluctuation in the diameter are effective for removing the loss of the superior mechanical properties of TNTZ alloy.

4. Conclusion

In this study, new Beta-Ti-type (TNTZ) metallic wires with full ductility were developed using the arc-melt-type melt-extraction method. The results were summarized, and the following conclusions were drawn:

- (1) A melt-extracted wire with the Beta-Ti single phase can be obtained from Ti-30Nb-10Ta-5Zr alloy by the arc-melt-type melt-extraction method. A fine dendrite structure was observed on the surface of the Beta-Ti wire. Because of this dendrite structure, the fluctuation in the diameter and surface roughness of the wire was negligible.
- (2) The continuous melt-extracted TNTZ wire exhibits high tensile strength and full ductility. Thus, the arc-melt-type melt-extraction method is very effective for fabricating Beta-Ti metallic wires for biomaterials.

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