Available online at www.sciencedirect.com
ScienceDirect
Acta Materialia 80 (2014) 149–158
Acta MATERIALIA
www.elsevier.com/locate/actamat

High strength and superconductivity in nanostructured niobium–titanium alloy by high-pressure torsion and annealing: Significance of elemental decomposition and supersaturation

Kaveh Edalati [a,b,*], Takeshi Daio [c], Seungwon Lee [a,b], Zenji Horita [a,b], Terukazu Nishizaki [d,*], Tadahiro Akune [d], Tsutomu Nojima [e], Takahiko Sasaki [e]

[a] *WPI, International Institute for Carbon-Neutral Energy Research (WPI-I2CNER), Kyushu University, Fukuoka 819-0395, Japan*
[b] *Department of Materials Science and Engineering, Faculty of Engineering, Kyushu University, Fukuoka 819-0395, Japan*
[c] *International Research Center for Hydrogen Energy, Kyushu University, Fukuoka 819-0395, Japan*
[d] *Department of Electrical Engineering and Information Technology, Kyushu Sangyo University, Fukuoka 813-8503, Japan*
[e] *Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan*

Received 23 June 2014; received in revised form 12 July 2014; accepted 29 July 2014

Abstract

A powder mixture of Nb–47 wt.% Ti (a well-known composition for superconducting magnets) was subjected to severe plastic deformation using high-pressure torsion (HPT) and subsequently annealed at 573 K. Ti gradually dissolved in Nb with increasing shear strain, with a fast kinetics comparable to lattice diffusion at 700–1200 K. At large strains, a complete transition to a nanostructured β phase occurred at room temperature, which is far below the equilibrium temperature of 690 K. Nanoclusters of Ti with a body-centered cubic structure were also detected at large strains. Subsequent annealing led to elemental decomposition, formation of a nanoscale lamellar structure and segregation of Nb at grain boundaries. Superconductivity occurred at temperatures below 9 K, while the transition temperature decreased with increasing shear strain because of supersaturation of Ti in Nb and increased with annealing because of elemental decomposition. The Nb–Ti alloy after HPT exhibited hardness/strength peaks followed by softening at large strains, while hardening occurred after annealing. The maximum hardness, tensile and bending strengths were 4, 1.7 and 2.7 GPa, respectively.

Keywords: Severe plastic deformation (SPD); Ultrafine-grained (UFG) materials; Electrical properties; Magnetic properties; Critical temperature of superconductivity

1. Introduction

High-pressure torsion (HPT) [1,2], equal-channel angular pressing [3,4] and accumulative roll-bending [5,6] are well-known severe plastic deformation (SPD) methods in which ultrafine-grained microstructures with high densities of lattice defects are achieved. Among the different SPD processes, HPT is especially effective at introducing extremely large shear strains up to steady states [7–10]. In addition to grain refinement and the resultant strengthening, the HPT method is capable of controlling several other structural features, such as enhanced atomic diffusion [11–14], allotropic phase transformations in pure elements such as Ti [15–17], Zr [18–20], Co [21,22], Si [23] and C [24], elemental decomposition in supersaturated alloys such as Al–Zn [25], Al–Mg [26], Al–Zn–Mg [26] and Co–Cu [27],

* Corresponding authors. Addresses: Department of Materials Science and Engineering, Faculty of Engineering, Kyushu University, Fukuoka 819-0395, Japan (K. Edalati); Department of Electrical Engineering and Information Technology, Kyushu Sangyo University, Fukuoka 813-8503, Japan (T. Nishizaki). Tel.: +81 92 802 2992 (K. Edalati). Tel.: +81 92 673 5636 (T. Nishizaki).
E-mail addresses: kaveh.edalati@zaiko6.zaiko.kyushu-u.ac.jp (K. Edalati), terukazu@ip.kyusan-u.ac.jp (T. Nishizaki).

http://dx.doi.org/10.1016/j.actamat.2014.07.065
1359-6454/

and solid solution supersaturation in alloys such as Al–Cu [14], Al–Fe [28], Cu–Ag [29], Cu–Ni [30], Cu–Cr [31], Ag–Ni [32], Ni–Al–Cr [33] and Fe–Cu [34].

Despite the publication of numerous papers regarding the microstructural refinement and mechanical property improvement of SPD-processed materials [1–34], there have been very few reports on the evolution of mechanical properties [6,35–40] and superconducting properties [41–43] of superconductors after processing by SPD. It was reported that the strength of Nb (a well-known superconducting metal) significantly increases after HPT processing because of significant grain refinement to the submicrometer level [38–40]. It was also reported that the critical temperature for superconductivity increases in Nb after the grain refinement by HPT because of the quantum confinement effect and size-dependent superconductivity, while the critical current density increases after HPT because of the vortex pinning effect by lattice defects such as dislocations and grain boundaries [43]. These reports suggest that the application of SPD through the HPT method can be an effective approach to achieve both high strength and enhanced superconductivity in superconducting materials.

It is of particular scientific and industrial interest to examine whether the strength and the critical temperature T_c for superconductivity can be increased in other superconducting materials such as Nb–Ti alloys after grain refinement by HPT processing. In Nb–Ti alloys, which are widely used in superconducting magnets, the critical current density in a magnetic field is enhanced due to the presence of non-superconducting hexagonal close-packed (hcp) Ti particles [44–48]. Since the Ti particles pin vortices and enhance the superconductivity, the amount and size of the Ti phase are important in controlling the superconducting properties in the Nb–Ti alloys [44–48]. The Nb–Ti alloys are usually processed by conventional plastic deformation techniques such as wire drawing, extrusion or rolling followed by thermal annealing [44–48]. However, the grain refinement after SPD processing is expected to be much more significant than that after the conventional plastic deformation methods [7,8].

In this study, we thus investigate the evolution of microstructure, mechanical properties and superconductivity along with HPT processing, as well as after subsequent annealing, in an Nb–47 wt.% Ti alloy, and show the importance of elemental decomposition and supersaturation. Since earlier papers reported that the application of HPT to powder mixtures results in a more significant grain refinement when compared to bulk alloys [17,30,31], powder mixtures were used as starting materials in this study.

2. Experimental procedures

Nb powders with a purity level of 99.9% and particle sizes less than 75 μm were manually mixed with 47 wt.% (63 at.%) of Ti powders with a purity level of 99.9% and particle sizes less than 75 μm. The powder mixtures were further homogenized by mechanical mixing in acetone with sufficient agitation in a glass container even after the acetone dried out. It should be noted that these high-purity powders were fresh, with minor oxidation, and energy-dispersive X-ray spectrometry (EDS) showed that no contamination occurs during mechanical mixing or after the HPT processing.

HPT was conducted at room temperature to consolidate the powder mixtures into discs of 10 mm diameter and 0.8 mm thickness under a pressure of $P = 4$ GPa using a pair of HPT anvils, as described in detail in Ref. [49]. Shear strain ($\gamma = 2\pi rN/h$, where γ is the shear strain, r is the distance from the disc center, N is the number of turns and h is the disc thickness [7,8]) was introduced through rotations for either N = 0, 1, 2, 5, 10, 20, 50 or 100 turns, with a rotation speed of $\omega = 1$ rpm. The samples processed through N = 50 were subsequently annealed at 573 K for either 1, 24 or 240 h.

Disc samples were first polished to a mirror-like surface on both sides and the Vickers microhardness was measured on the upper surfaces of discs at 3.5 mm away from the disc center with an applied load of 200 g for 15 s at several radial directions.

Second, X-ray diffraction (XRD) analysis was performed on samples using Cu K_α radiation at 40 kV and 40 mA in a scanning step of 0.01° and a scanning speed of 1° min^{-1}. LaB_6 standard powders were used concurrently with the samples during XRD analysis to offset the XRD analyzer and find identical absolute peak positions.

Third, scanning electron microscopy (SEM) with EDS was performed at 15 kV to examine the microscopic distribution of Nb and Ti in the disc samples.

Fourth, for transmission electron microscopy (TEM) and scanning-transmission electron microscopy (STEM), thin foils were prepared from the disc samples at 3.5–4 mm away from the disc center with a focused ion beam system using gallium ions under an accelerating voltage of 30 kV. TEM and STEM were performed at 200 and 300 kV for microstructural observation in bright-field and dark-field modes, respectively, to record selected-area electron diffraction (SAED) patterns, EDS mappings and elemental analyses. It should be noted that the microstructural observations using TEM and STEM were conducted on the cross-section of the discs.

Fifth, for bending tests, miniature rods with a square cross-section of 0.5 × 0.5 mm^2 and a length of 9 mm were cut from the discs at 2.5 mm away from the disc center. Three-point bending tests were carried out at room temperature to measure the bending load and displacement in the direction perpendicular to the disc surface at a crosshead speed of 0.5 mm min^{-1}. The bending stress was calculated through the Euler–Bernoulli beam theory using the equation given in Ref. [17]. It should be noted that the bending properties are very important for Nb–Ti alloys, especially when they are used in the form of superconducting wires.

Sixth, miniature tensile specimens having 1.5 mm gauge length, 0.7 mm width and 0.6 mm thickness were cut from discs at a position 2 mm away from the disc center, as