Acta Materialia 135 (2017) 400–410

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Partial recrystallization of gum metal to achieve enhanced strength and ductility

J.-L. Zhang [a, b], C.C. Tasan [b, *], M.J. Lai [a], D. Yan [a], D. Raabe [a]

[a] Max-Planck-Institut für Eisenforschung, Max-Planck-Straße 1, 40237, Düsseldorf, Germany
[b] Department of Materials Science and Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA, 02139, USA

ARTICLE INFO

Article history:
Received 6 February 2017
Received in revised form
22 June 2017
Accepted 24 June 2017
Available online 26 June 2017

Keywords:
Gum metal
Partial recrystallization
Microstructure heterogeneity
ω-Phase
Dislocation channeling

ABSTRACT

Here we present a microstructure design approach which leads to partial recrystallization and nano-precipitation within the same single-step heat treatment. This produces a dual-constituent microstructure in Ti-Nb based gum metal, which consists of nano-ω-particle-rich ultrafine recrystallized grain chains embedded in ω-lean subgrain-containing recovered zones. This partially recrystallized microstructure exhibits an improved strength-ductility combination that surpasses the inverse strength-ductility relationship exhibited by materials with similar composition. The strengthening effects due to precipitates and grain refinement were studied by nanoindentation. The deformation mechanisms of the partially recrystallized material were investigated by in-situ scanning electron microscope tensile tests, micro-strain mapping and post-mortem microstructure characterization. The improved mechanical properties are attributed to the high yield strength of the recrystallized grains and the sequential activation of dislocation slip and dislocation channeling.

© 2017 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

1. Introduction

Partial recrystallization has been widely used as a tool to improve either the ductility or the strength of metallic materials. Typical examples of the former effect are low temperature annealing of nanostructured materials [1–3] and texture modification of Mg-alloys containing rare earth elements [4,5]. Regarding the latter effect, recrystallization is known to drastically decrease the strength of pre-strained materials [6,7]. Thus, various methods based on partial recrystallization have been studied to preserve the strength, such as post-recrystallization aging to form precipitates [8,9] and stacking fault energy reduction to retain nano-twins in the non-recrystallized zones [10,11]. Here, we propose an approach to achieve an enhanced strength-ductility combination by incorporating nano-precipitation in partial recrystallization with a single-step heat treatment. After the heat treatment of a strongly cold worked microstructure, the recrystallized and recovered constituents in the obtained microstructure are expected to have different strengthening and deformation mechanisms. This is due to the spatially differing recrystallization kinetics leading to variations in grain size and nano-phase content. This approach is demonstrated on gum metal [12], which is a metastable β (bcc)-structured Ti-Nb based alloy that forms nano-sized martensitic α"- or ω-phase depending on the thermomechanical treatments. Gum metal provides the possibilities of (i) tailoring the starting microstructure guided by thorough studies on its cold worked microstructures [13,14], and (ii) introducing multiple nano-structured features (α"- and ω-phase) [15–18]. Although gum metal is a biomedical candidate alloy due to its biocompatibility and low Young's modulus (40 GPa) [12,19,20], its yield strength and its fracture resistance must be improved for biomedical applications [20–22]. These two requirements are addressed with the proposed approach.

2. Experimental methodology

2.1. Recrystallization treatment design

Gum metal was 80% cold rolled leading to deformation-induced microstructural features including elongated grains and shear bands, fiber-type crystallographic texture, and in-grain dislocation substructures. This pre-straining level with a true strain[1] of ~1.6

* Corresponding author.
E-mail address: tasan@mit.edu (C.C. Tasan).

[1] This is calculated from $\ln(t_0/t_f)$, where t is the rolled metal sheet thickness.

http://dx.doi.org/10.1016/j.actamat.2017.06.051
1359-6454/© 2017 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

(medium deformation) provides sufficient driving force for recrystallization, and it conserves the freedom to control the recrystallization volume fraction as well. Partial recrystallization was realized at 800 °C (above the β-transus), based on the results of previous recrystallization studies [23–25] to avoid concurrent recrystallization and α-phase formation. The presence of α-phase can lead to undesired changes in the Young's modulus and work hardening rate of gum metal [12]. 50 K s^{-1} was used as the heating rate to ensure temperature homogeneity and to suppress recovery by narrowing its time window before recrystallization.[2] A moderate cooling rate of −50 K s^{-1} was selected to favor nano-ω precipitation, which is beneficial due to the associated precipitation hardening effect and dislocation channeling mechanism [26], by avoiding both α-phase and martensitic α"-phase transformation. The former is a diffusional transformation enhanced by slow cooling rates [25,27], whereas the latter was reported to form instead of ω during fast cooling [28]. Fast cooling leaves a very limited time for ω-phase transformation before the temperature reaches the α"-phase transformation start temperature (~room temperature (RT)) [28,29]. Even though the oxygen in the gum metal chemical composition could help retard the α"-phase transformation [16,30], we intended to eliminate the possibility of forming α"-phase by using a moderate cooling rate.

2.2. Experimental details

Gum metal with a composition of Ti-35.7Nb-1.9Ta-2.8Zr-0.4O (wt.%) was produced via arc-melting under argon atmosphere and cast into a rectangular copper mold. The ingot was subsequently annealed for 4 h at 1200 °C for homogenization and then furnace cooled. The average initial grain size (d) of the as-solution treated (ST) material was ~60 μm. The rectangular block was cold rolled at RT to ~80% thickness reduction to obtain the described starting material.

All heat treatments were carried out on rectangular samples ($6 \times 4 \times 1$ mm^3) in a DIL805A/D dilatometer (Bähr Thermoanalyse GmbH), which enables an accurate control of temperature, heating/cooling rate, and atmosphere. Argon and helium were used respectively as the protecting atmosphere during holding and as the cooling agent. A newly upgraded multi-probe microstructure tracking method [31] was used to study the primary recrystallization behavior. Among the partially recrystallized states, the 20% (800°C-60 s) and 70% (800°C-300 s) recrystallized materials were selected for mechanical property investigations. Some partially recrystallized materials were additionally aged at 350 °C for 2 h to study the ω-phase distribution.

All samples probed by scanning electron microscope (SEM) were wet-ground and polished. Final polishing was carried out using a solution of silica particle suspension with 25% H_2O_2. The microstructures in all states were characterized in a Zeiss-Crossbeam XB 1540 FIB-SEM (Carl Zeiss SMT AG). A EDAX/TSL system (AMETEK GmbH) equipped with a Hikari camera was used for electron backscatter diffraction (EBSD) measurements. Secondary electron (SE) and backscattered electron (BSE) imaging were conducted using an accelerating voltage of 15 or 30 kV. The recrystallization fraction (f_{Rex}) was estimated using two methods: (i) large field (~200 × 500 μm^2 in each state) EBSD grain average misorientation maps to differentiate the recrystallized and recovered[3] zones; (ii) color orientation contrast images of an integrated area of ~1 × 1.5 mm^2 in each state, taken in a Zeiss Merlin equipped with a Bruker EBSD system (Bruker Nano GmbH), to yield adequate statistics. Color orientation contrast images, captured and integrated by three forward-mounted backscatter detectors positioned below the EBSD detector, assign different colors to pixels with respect to their orientation, as the electron beam is raster scanned across the specimen surface [32].

Transmission electron microscopy (TEM) specimens were lifted out following a site-specific method [33] in a dual-beam focused ion beam (FIB) Helios Nanolab 600i (FEI). TEM observations were performed in a JEOL JEM-2200 FS (JEOL GmbH) at an operating voltage of 200 kV, through which bright field (BF), dark field (DF) images and selected area diffraction patterns (SADP) were recorded by a Gatan CCD camera (Gatan, Inc.). Scanning transmission electron microscopy (STEM) images were captured using a scanning transmission electron imaging (STEI)-BF detector with a 100 cm camera length.

Dog-bone-shaped tensile samples (gauge geometry: $4 \times 2 \times 1$ mm^3) were cut by electrical discharge machining (EDM) with the gauge length parallel to the rolling direction (RD). Tensile tests were carried out using a 5 kN Kammrath & Weiss tensile stage coupled with in-situ imaging of speckled patterns on tensile sample surfaces using a high speed camera. The data were used for digital image correlation (macro-DIC: spatial resolution ≈ 120 μm) analyses performed by employing the ARAMIS software (GOM GmbH). To study the deformation mechanisms, in-situ tensile tests were conducted using a SEM tensile stage designed in house, applying a recently developed correlative microstructure and DIC micro-strain mapping method [34]. In each deformation stage, micro-strain was mapped with 0.25 μm spatial resolution, and the microstructure was imaged by BSE (also by EBSD in selected stages). Note that due to the difference of the spatial resolutions between the macro- and micro-DIC strain measurements, percentage and rational numbers are used here respectively for clarity.

The heights of the deformation-induced slip steps that appeared on the pre-polished tensile sample surfaces were measured with a NI/Veeco dimension 3100 atomic force microscope (AFM) operated in tapping mode. The lateral resolution of a scan map was set as 512 points along a distance of 30 μm. A scan rate of 0.25 Hz and a tip velocity of 25 μm s^{-1} were used for the measurements. The data analysis was performed with the help of the Gwyddion software following a three-point plane correction of the zero level. The heights extracted as line profiles were averaged within a 1 μm width along the drawn line.

Nanoindentation tests were carried out using a Hysitron TriboScope 950 nanoindentation system with a Berkovich shaped indenter in a load-controlled mode and with a maximum load of 2500 μN. The hardness of a matrix of 10 × 10 indents with a spacing of 15 μm were mapped.

3. Results

3.1. Recrystallization kinetics and microstructure evolution

Recrystallization kinetics curves at 800 °C (Fig. 1a) obtained from EBSD maps (solid line) and color orientation contrast images (dashed line) reveal consistent trends that follow the general recrystallization kinetics curve of single phase materials [7]. 20% and 70% recrystallized materials were selected for further characterization, since they show representative microstructures of the nucleation stage and of the grain impingement stage. In all microstructures shown in Fig. 1, the top and bottom parts of the EBSD maps are taken from the center layer and the roll contact surface, respectively.[4] The image quality (IQ) overlaid inverse pole figure

[2] This is following the assumption that recovery generally has a lower activation energy than recrystallization at high temperatures [7].

[3] Recovered describes a microstructure state that is in recovery.

[4] In the roller contact surface, ~100 μm thick zone was excluded due to low EBSD indexing (confidence index< 0.1).

Download English Version:

https://daneshyari.com/en/article/5435927

Download Persian Version:

https://daneshyari.com/article/5435927

Daneshyari.com