Materials Science & Engineering A 645 (2015) 328–332

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Grain refinement via formation and subdivision of microbands and thin laths structures in cold-rolled hafnium

Henglv Zhao [a], Song Ni [a,*], Min Song [a], Xiang Xiong [a], Xiaopeng Liang [a,b], Huizhong Li [b]

[a] State Key Laboratory of Powder Metallurgy, Central South University, Changsha 410083, China
[b] School of Materials Science and Engineering, Central South University, Changsha 410083, China

ARTICLE INFO

Article history:
Received 16 June 2015
Received in revised form
2 August 2015
Accepted 5 August 2015
Available online 13 August 2015

Keywords:
Electron microscopy
Grain refinement
Dislocations
Shear bands
Hardness measurement

ABSTRACT

The microstructural evolution and mechanical properties of pure hafnium subjected to cold rolling were investigated in this paper. Structural characterization indicates that slip dominated the plastic deformation and the microstructural evolution follows the sequence: original coarse grains – formation of microbands – formation of thin laths in microbands and formation of elongated grains in locally formed shear bands – the longitudinal splitting and transverse breakdown of laths to elongated segments – formation of nano-grains through further transverse breakdown of elongated segments. Hardness results indicate that strain hardening occurred during the whole rolling process, which can be attributed to dislocation activities and grain refinement effect.

1. Introduction

The hexagonal close-packed (HCP) metal pure hafnium (Hf) possesses many interesting properties such as high density and strength, large cross-section for neutron capture, and excellent corrosion resistance. Because of these superior properties, it has obtained lots of attentions for various applications ranging from biomedical materials, control rods in nuclear reactors to solid solution strengthening element in many super alloys used in aircraft engines [1–6].

In recent years, extensive investigations have been carried out on HCP metals, such as magnesium [7,8], titanium [9,10] and zirconium [11,12], to elucidate their dominant deformation mechanisms under different deformation methods. It is known that HCP metal lacks the critical number of independent slip system which is needed to obtain homogenous deformation, thus twining is also regarded as an important deformation mode to accommodate strain in deformation and the extent of twining is associated with strain, strain rate, deformation temperature and texture [2,11]. In previous studies on Ti and Zr deformed at room temperature [9–14], besides contribution of twinning and dislocation activities, deformation induced grain refinement is mainly attributed to dynamic recrystallization. Yang et al. [9] reported that in shear bands where deformation is highly localized, grain refinement follows the process from original grains to microbands, dislocation cells, subgrains by dislocation activities, and finally to equiaxed nanograins by lattice rotation.

There are also many investigations done on Hf and its alloys [1–6]. Yadav et al. [1] reported a strong strain rate sensitivity for Hf, and found that twinning was suppressed under the condition of high temperature and low strain rate. Clarissa et al. [2] investigated the effect of dislocation slip and twinning on work-hardening behaviors of Hf, and found that dislocation motion can be inhibited by twins, which led to a high flow stress. Cerreta et al. [3] investigated the influence of grain size and texture on the mechanical response of pure Hf and calculated the Hall-Petch constant for Hf. Muller et al. [6] investigated the preferred orientation of cold-rolled hafnium and the change in texture with the progress of recrystallization. In this study, pure Hf was subjected to cold rolling. The microstructural and mechanical property evolutions were investigated to elucidate the deformation mechanism and structure–mechanical property relationship of Hf.

2. Experimental

The material used in present study was commercially pure Hf (99.99%). X-ray diffraction analysis indicated a pure HCP phase and no obvious texture in the material. Bar materials with dimension of 30 mm × 10 mm × 3 mm (L × W × H) were rolled at room temperature with a reduction of 0.3 mm per pass in thickness, to

* Corresponding author.
E-mail address: song.ni@csu.edu.cn (S. Ni).

http://dx.doi.org/10.1016/j.msea.2015.08.019
0921-5093/© 2015 Published by Elsevier B.V.

obtain deformed samples with reductions of 30%, 40%, 50%, and 60% respectively. Mechanical properties of both raw material and the rolled samples were measured by Vickers microhardness. Hardness test was performed with a load of 200 g for 15 s. At least 10 indent locations were made on each sample.

The microstructure of the samples before and after deformation was investigated by optical microscopy (OM) and transmission electron microscopy (TEM). Samples for OM investigations were mechanically ground and polished to mirror-like surface, and then etched with an acidic solution consisting of 10% HF, 45% HNO_3 and 45% H_2O. Those samples were observed at 1000 × magnification under polarized light. The thin foils for TEM investigation were prepared as follows: the rolled samples were first cut perpendicular to TD plane with a thickness of 0.5 mm and further cut into shorter strips with length of 3 mm (it is worth mentioning that hardness measurements, OM and TEM investigation were all conducted on TD plane), and then were mechanically thinned down into 30 μm thick. Those thin strips were further glued onto copper rings with a diameter of 3 mm and finally subjected to low- energy ion milling (30 μA, 5 kV Ar) in Gatan PIPS system to perforation.

3. Results and discussion

3.1. Optical microstructures

Fig. 1 shows the microstructure observed under optical microscope. Fig. 1(a) shows the OM image of the un-deformed specimen, in which equiaxed grains with an average grain size of 20 μm were observed. When the specimen was rolled with a thickness reduction of 40%, the equiaxed grains became elongated, as shown in Fig.1(b). As shown in Fig.1(c) and (d), when the reduction reached 60%, grains were further elongated and several localized shear bands were clearly distinguished from the neighboring deformed matrix, with the width of several micrometers and inclined to the rolling direction at an angel of 35–40°, as indicated by red arrows.

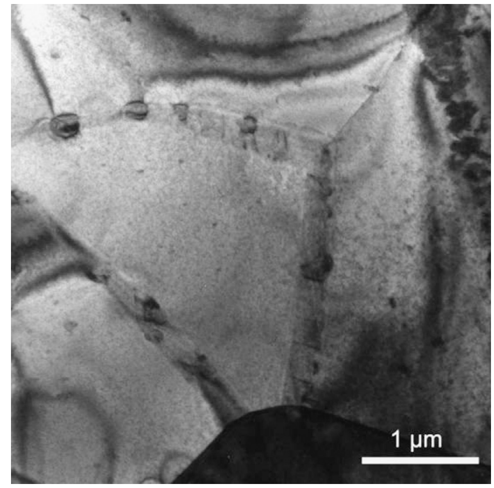

Fig. 2. A TEM image of the un-deformed raw material.

3.2. TEM observations

Fig. 2 shows a typical TEM image of the un-deformed raw material. No twins and very few dislocations were observed inside the equiaxed grains. Fig. 3 shows TEM images of the rolled specimen with a thickness reduction of 40%. In Fig.3(a), high density of dislocations tangled with each other are observed within the parallel microbands. The microband boundaries (MB boundary) were indicated by red arrows. The width of the microband ranges from ~600 nm to 5 μm. No twins were observed. It worth mentioning that, the microbands tend to split into thin laths. Part of the thin lath boundaries (TL boundary) are indicated by dashed

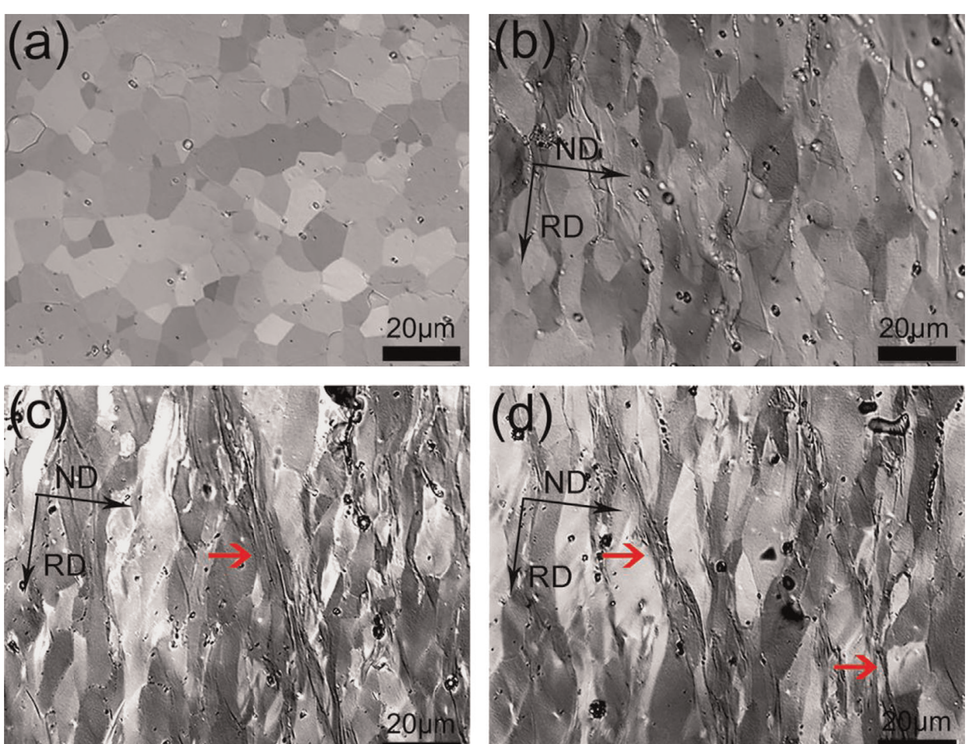

Fig. 1. Optical micrographs of (a) the un-deformed specimen; (b) specimen with thickness reduction of 40%; (c) and (d) specimen with reduction of 60%. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)