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Atomic-scale understanding of stress-induced phase transformation in cold-rolled Hf

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ABSTRACT

Identifying the character and the source of partial dislocations associated phase transformation from a hexagonal close-packed (HCP) structure to a face-centered cubic (FCC) structure is essential for understanding phase transformation mechanisms, but was rarely done using microscopy. Here, we report a stress-induced HCP to FCC phase transformation in pure hafnium during cold rolling. Detailed transmission electron microscopy investigations revealed that transformation-related partials stemmed from the dissociation of <a>-type dislocations. Successive gliding of partials on every other basal plane resulted in the orientation relationship between the two phases of $<11\bar{2}0>_{hcp}//<110>_{fcc}$, $<10\bar{1}0>_{hcp}//<\bar{1}12>_{fcc}$ and $[0001]_{hcp}//<111>_{fcc}$.Besides, a new way to form a twin relationship in the FCC structure was discovered.

1. Introduction

Solid-solid phase transformation in bulk materials has been one fundamental and long-standing research area, as the mechanical and physical properties of materials could change abruptly with structural transition [1–5]. Among different kinds of phase transformation, the transformation from a hexagonal close-packed (HCP) structure to a face-centered cubic (FCC) structure has been widely observed in metals and alloys, including Co [6,7], Co-32%Ni alloy [8,9], Ti [10,11], Ti-based alloys [12–14], stainless steel [1,2] and InAs nanowires [15]. Theoretically, the HCP to FCC phase transformation can be achieved through two approaches: Shockley partials glide on every other basal plane [1,2,6–9,12–15]; or shear-shuffle mechanisms through gliding two-layer disconnections or pure shuffle mechanisms through gliding four-layer disconnections on every other prism plane [11]. In the first approach, the transformation-related partials at the fronts of the FCC phase have Burgers vectors $\frac{a}{3}<1\bar{1}00>$ and their slip plane is the basal plane. The displays of these transformation-related partials have typically two scenarios: all the partials have the same Burgers vector; or they can have any one of the three Burgers vectors in the group of $\frac{a}{3}<1\bar{1}00>$ and hence are randomly arrayed. However, no consensus has been reached on which scenario is accounted for the HCP to FCC phase transformation, for there is still a lack of detailed and accurate atomic level identification for these transformation-related partials. Besides, it has not been clear on the sources of these partials.

Hafnium (Hf) has recently attracted increasing research interests due to its unique properties and significant applications in modern society [16–19]. For example, due to its large cross-section for thermal neutron capture and high corrosion resistance, Hf has been utilized as control rods in different types of nuclear reactors since late 1950s [19]. Pure Hf is of an HCP crystal structure (the α phase) at room temperature and a body-centered cubic (BCC) crystal structure (the β phase) at a temperature above 2016 K [16]. A phase transformation from the α phase to a simple hexagonal crystal structure (the ω phase) occurs under an applied pressure of 22 GPa–38 GPa [20–22]. High-energy ball milling on Hf powder leads to a transformation from the HCP phase to an FCC phase when the crystal size reaches several nanometers [23]. However, the HCP to FCC phase transformation has never been reported in bulk Hf. Moreover, the mechanism for HCP to FCC phase transformation is still unclear.

In the present study, we report the HCP to FCC phase

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transformation in cold-rolled bulk Hf processed at room temperature. Through comprehensive high-resolution transmission electron microscopy (TEM) investigation of FCC Hf lamellas in bulk Hf, the display of transformation-related partials was visualized successfully at the atomic level, and the source of these partials was deduced. Besides, we also discovered a new way for the formation of FCC twins in Hf.

2. Experimental procedure

The material used in this study was commercially pure Hf (99.99 wt%), purchased from Beijing General Research Institute of Nonferrous Metals. The as-received Hf plate exhibited equiaxed grains with an average size of ~20 μm. Small bars with dimensions of 30 mm × 10 mm × 3 mm were cut from the plate by spark machining. These small bars were annealed at the temperature of 1000 °C for 1 h and then were rolled at room temperature multiple times with a thickness reduction of 0.3 mm per pass to obtain deformed samples with a total thickness reduction of 60%. TEM specimens were cut from the transverse direction, mechanically ground to 50 μm, dimpled and then Ar^+ ion-milled using a Gatan 691 PIPS system with a voltage of 3 kV. For the initial and final milling, the ion incidence angle was set at 7° and 3°, respectively. TEM and high-resolution TEM investigations were performed using a FEI Titan G^2 60-300 Cs-corrected microscope operated at 300 kV.

3. Experimental results

3.1. HCP to FCC phase transformation in Hf

TEM and high-resolution TEM were used to identify the phase transformation in Hf. Fig. 1(a) shows a bright-field TEM image of the un-deformed hafnium. No lamella and few dislocations were seen in the un-deformed material. Fig. 1(b) shows a typical TEM image of cold-rolled samples with a thickness reduction of 60%.Lamellas with widths of up to tens of nanometers and lengths of several hundred nanometers formed in the cold-rolled material, as indicated by yellow arrows in Fig. 1(b). Fig. 1(c) presents a selected area electron diffraction (SAED) pattern obtained from the area in Fig. 1(b) that contains both the lamella structure and the matrix. The SAED pattern suggests that an HCP and an FCC structures co-exist in the selected area and the zone axis of the HCP and FCC structures presented in the pattern is $[1\bar{2}10]$ and [110], respectively. Dark-field imaging using FCC and HCP diffraction spots indicated that the lamellae are of the FCC structure while the matrix appears as the single HCP phase. The orientation relationship between the HCP and FCC phases is: $<11\bar{2}0>_{hcp}//<110>_{fcc}$ and $(0001)_{hcp}//\{111\}_{fcc}$. Fig. 1(d) presents a $[1\bar{2}10]_{hcp}$ and $[110]_{fcc}$ high-resolution TEM image showing the interface between an FCC lamella and the HCP matrix, further confirming the orientation relationship. Based on the orientation relationship and the crystallography of the two structures, it is expected to have $<10\bar{1}0>_{hcp}//<\bar{1}12>_{fcc}$(see the Supplementary Material Fig. S1). Interestingly, a high density of dislocations exists in the FCC lamellas. Some dislocation cores are indicated using white "T" in Fig. 1(d). The Burgers vectors of most dislocations in the FCC phase have a directional component parallel to the c-axis ([0001]) of the HCP matrix, implying that these dislocations accommodate the c-axis strain and thereby the introduction of the FCC lamellas may enhance the deformability of the material.

Based on the orientation relationship of the two phases, Fig. 2 depicts a possible mechanism of the phase transformation, in which the lattice parameters were measured from high-resolution TEM images: Shockley partial dislocations with Burgers vectors of the $\frac{a}{3}<10\bar{1}0>$ type were generated and glided on every other (0001) plane during rolling deformation. The dashed line in Fig. 2(a) presents a partial dislocation gliding on a(0001) habit plane. The following discussion assumes 5 Shockley partials emitted on atomic layers 1, 2, 3, 4, 5, respectively. The original atomic stacking sequence in the HCP matrix is … ABABABABAB …. Slip of the partial on layer 1 (a B stacking plane) results in a new stacking sequence of … ACBCBCBCBC … (the column "Step 1"). As the remaining steps go on, the final stacking sequence of … ACBACBACBA … (the column "Step 6") forms and a ten-layer FCC structure is created. This transformation process indicates that coordinated activation of $\frac{a}{3}<10\bar{1}0>$ partials gliding on every other(0001) plane gradually converts the HCP structure to the FCC structure. The model described in Fig. 2 leads to the orientation relationship obtained from the SAED and high-resolution TEM data shown in Fig. 1(c) and (d). Fig. 2(b) shows the lattice parameters and the orientation relationship of the two phases. The lattice parameters with error bars are measured in high-resolution TEM images. It shows that when the HCP structure transformed to the FCC structure, the lattice expansion amounted to +1.81%, and +6.06% along the$[10\bar{1}0]$, and[0001] directions, respectively.

Note that three different $\frac{a}{3}<10\bar{1}0>$ partial dislocations gliding on the basal plane with the angle 120° between each other would produce exactly the same stacking fault despite of the difference in the gliding directions. The atomic illustration is shown in the left bottom of Fig. 3. The solid circles in the figure represent the atoms of HCP phase, while the hollow circles indicate the atom positions after Shockley partial dislocations gliding on every other (0001) plane, converting the HCP stacking sequence to FCC stacking sequence. Though a coordinated sliding on every other (0001) plane is required for the HCP to FCC transformation, there is no restriction to the activation of any of the three $\frac{a}{3}<10\bar{1}0>$ dislocations on each (0001) plane. Fig. 3 shows there exists two scenarios for the HCP to FCC transformation—scenario A: all Shockley partial dislocations that contribute to the transformation have the same Burgers vector or gliding direction. This collectively produces a net macroscopic strain [24–26]; and scenario B: Burgers vectors are randomly selected, which leads to much smaller or even zero net macroscopic strain [27,28]. While both scenarios result in the HCP to FCC transformation, the net macroscopic strains introduced by the two types of transformation are different.

Fig. 4 shows two typical high-resolution TEM images of the HCP/FCC interfacial regions. The stacking sequence of the HCP phase is … ABABAB …, while the stacking sequence of the FCC phase is … ABCABC …, as shown in Fig. 2. In the transitional zone, several adjacent Burgers circuits based on the HCP lattice were drawn. Both 30°and 90°Shockley partial dislocations were observed (the identification of partial dislocations is shown in the Supplementary Material Fig. S2), and this is also the case for many other interfacial regions, indicating that scenario B is favored in this study.

3.2. The sources of phase transformation—related partials

Hf belongs to the IVb group in the periodic table, having a c/a ratio of 1.581 that is smaller than the ideal value of 1.633. During a deformation process of Hf, strain can be accommodated through slips and twinning [29,30]. Typically, Shockley partial dislocations can form in two ways: nucleation from special positions, which include the boundaries of nanocrystalline grains [7,27,31], free surface [15,31]and crack tips [32,33], and dissociation of full dislocations [34,35]. As the grain sizes of the Hf were much larger than the critical grain size needed for partial dislocation emission from grain boundaries [31,36], no grain boundary source was available. For most grains in the bulk coarse-grained Hf, there was also no free surface or crack tip. Therefore, the phase transformation—related partials formed only via the dissociation of full dislocations.