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Nanotwin's formation and growth in an AlCoCuFeNi high-entropy alloy

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ABSTRACT

Nanotwins were formed in the AlCoCuFeNi high-entropy alloy produced by melt-spinning. The Cu-rich nanotwins have a face-centered-cubic (FCC) structure diffused in the ordered body-centered-cubic (B2) matrix. The nanotwins decompose from the matrix with the K–S crystallographic relationship: $\{111\}_{FCC} \parallel \{110\}_{B2}$ and $<110>_{FCC} \parallel <111>_{B2}$. After annealing, the spherical nanotwins grow into rodlike grains. The growth direction is parallel to the {111} twin planes of the FCC phase. The hardness and elastic modulus change with the twin size, which is tested by nanoindentation. This study deepens the fundamental understanding of the phase transition in HEAs.

The high-entropy alloys (HEAs) have been widely studied since firstly proposed by Yeh et al. in 2004 [1–8]. Till today, the HEAs with the face-centered-cubic (FCC), body-centered-cubic (BCC) or hexagonal-closed-packed (HCP) structure have been reported [2,4,5,9]. The different processing conditions influence the structure of HEAs, especially the cooling rate [3]. High cooling rates could suppress the decomposition of HEAs efficiently [3]. A rapid solidification rate greatly favors the formation of the solid-solution phase vs. undesired precipitated compounds by exploiting the incubation times of nucleation for various competing phases in the HEAs [10,11]. Meanwhile, the number of the phases formed, and their structures depend on the alloying elements in HEAs synthesized under identical processing conditions [12]. According to the alloying effect of elements on microstructural characteristics in stainless steels, elements can be classified into two types: one is the FCC stabilizer, such as Ni, Mn, Cu, C and N, and the other is the BCC stabilizer, such as Al, Cr, Mo, Si, and Nb [9]. It is a good case in noting that the phase of $Al_xCoCrFeNi$ changing from the single FCC, FCC + BCC to complete BCC phases with increasing the Al content [1,9,13]. The reports in the literature indicate that the Al addition promotes B2 ordering in HEAs (e.g., AlCrCuFeNi [9], AlCoCrCuFeNi [14], and $Al_{1.3}CoCrCuFeNi$ [15]). The Cu–Cu always forms a preferred nearest-neighbor pair in the alloy melt [13,15], and the Cu segregation occurs on different length scales, such as the interdendritic regions and the nanoscaled Cu-rich precipitates [1,12,15]. The nanoscaled phase separation by the Cu partitioning and local $L1_2$ ordering was observed in the as-spun $CoCrFeCuNiAl_{0.5}$ HEA [16].

In some HEAs with the precipitates, the different morphology with spinodal structures, such as the plate-like precipitates, the rhombohedron-shaped precipitates, and the nanoprecipitates decomposing from the BCC phase exist at low temperatures [3, 15]. These precipitates are not randomly distributed, but keeping some orientational relationships with the BCC matrix [3]. For example, the Cu-rich plate-like precipitates are coherent with the BCC matrix in the AlCoCrCuFeNi HEA, and the edges of the rhombohedron-shaped precipitates are aligned along the <111 > directions [3]. However, there are no reports of the nanotwin's formation, growth, and their orientational relationship with the BCC matrix. In fact, there are the nanoscaled twins at the earliest reference of HEA [1], named nanoprecipitates. However, they are not realized to be nanotwins. Their formation and growth processes have not been studied. In the present work, we investigate the nanotwin's formation, growth process, and their property variation of the AlCoCuFeNi HEA fabricated by melt-spinning, followed by annealing.

The target alloy used in the present work has the nominal composition of AlCoCuFeNi (in atomic proportion). The alloys were melted five times by arc-melting in a Ti-gettered high-purity argon atmosphere. The specimen was then crushed into an appropriate size for single-roller melt-spinning with a wheel tangential speed of 15 m/s. The width and thickness of the ribbons are about 1–1.5 mm and 15 μm, respectively. The ribbons were annealed at 873 K for 60 min. The nature of phase transitions was examined by the x-ray diffraction (XRD) using the Cu Kα radiation from 20° to 100° of 2θ with 2°/min. The working voltage and current are 40 kV and 40 mA, respectively. We grind the as-spun ribbons into powders to conduct the XRD experiment. The

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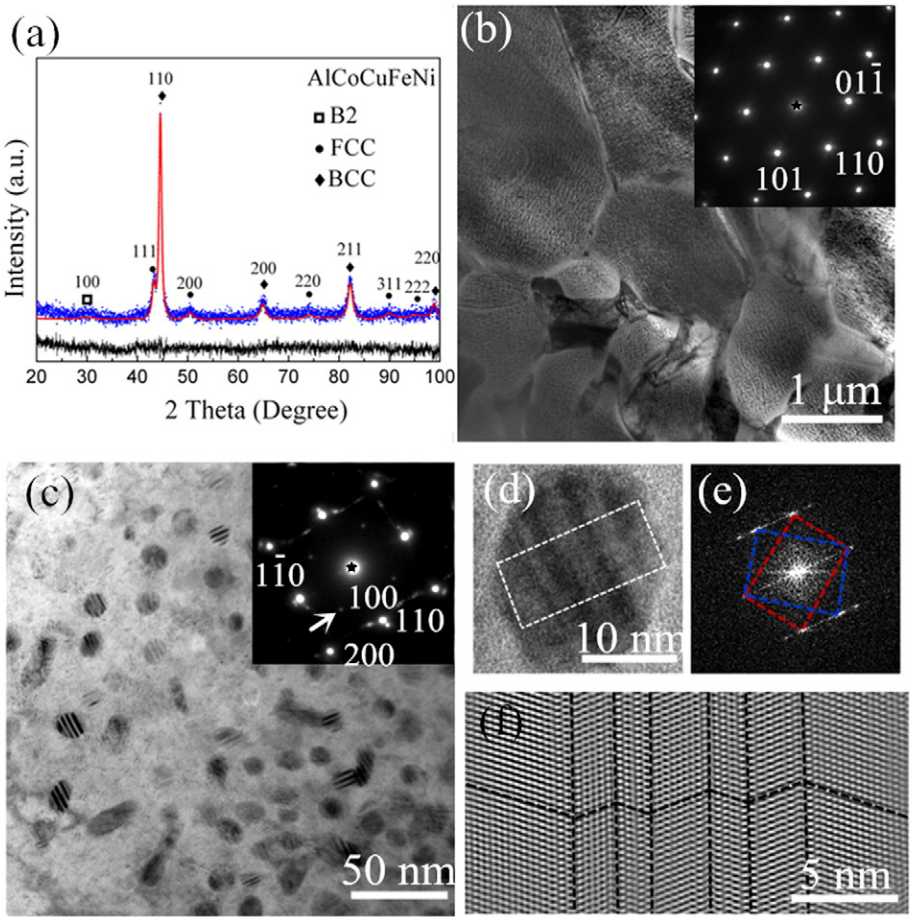

Fig. 1. The structure of the melt-spun equiatomic AlCoCuFeNi HEA. (a) The XRD pattern indicates the ordering B2 and FCC phase; (b) TEM bright-field image showing a polycrystalline structure with nanoscaled precipitates, and the inset is the $[\bar{1}11]$ zone-axis SAED; (c) high-magnification bright-field image of nano-precipitates, and the inset is the [001] zone-axis SAED; (d) HRTEM of a nanotwin in (c); (e) The corresponding FFT pattern of (d), in which the twinning relation is labeled; (f) the enlarged Fourier-filtered HRTEM image of (d), in which the twin boundaries and twinning relations are marked with dashed black lines.

transmission-electron-microscopy (TEM) observations were performed at 200 kV in a JEM-2010 microscope equipped with an energy-dispersive X-ray (EDX) spectrometer. Thin specimens for TEM observations were prepared by dimpling and ion-beam milling with a low energy ion beam (0.5 keV) to avoid damage at the latter milling stage. The nanoindentation test employed the Hysitron Triboindenter (TI-900) with the load increased up to the maximum load, $P_{\max}$, of 7000 μN at a loading and unloading rates of 2000 μN/s. The data were averaged in groups of five.

Fig. 1 displays the structure of the melt-spun equiatomic AlCoCuFeNi HEA, including the XRD pattern, TEM images, and their corresponding selected-area-electron-diffraction (SAED) patterns. Fig. 1a shows the XRD pattern of the melt-spun equiatomic AlCoCuFeNi HEA. The blue dots display the original data, and the red line is the fitting curve with

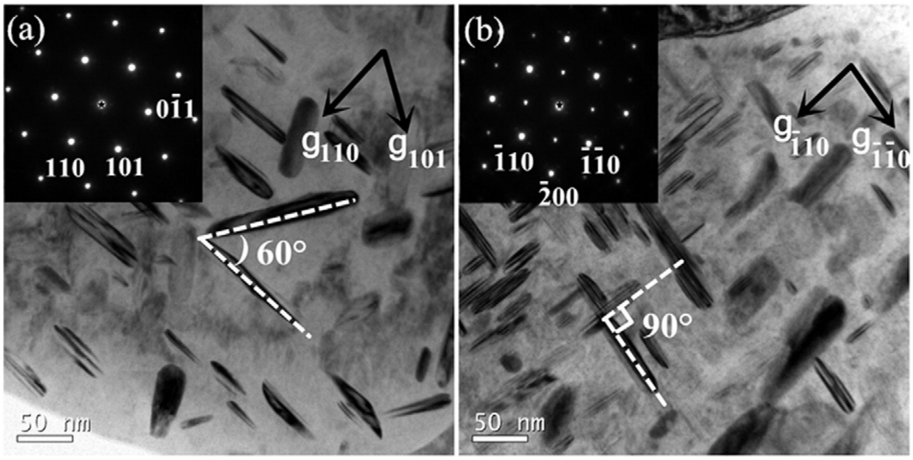

Fig. 2. TEM images of the melt-spun equiatomic AlCoCuFeNi HEA after annealing: (a) the rodlike grains with the intersection angle of two {111} planes is 60°, and the inset is the $[1\bar{1}1]$ zone-axis SAED of the B2 phase; (b) the rodlike grains with the intersection angle of two {111} planes is 90°, and the inset is the [001] zone-axis SAED of the B2 phase.