

In situ observations of crack propagation in as-cast Cu-1.5Fe-0.5Co (wt%) alloy

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ABSTRACT

As-cast Cu-1.5Fe-0.5Co (wt%) alloy displays both high tensile strength of 307 MPa and elongation of 33%. In situ transmission electron microscopy was used to investigate crack propagation in the alloy, to analyze the origin of the good properties. At different deformation stages in thin Cu foils, the interactions of a propagating crack with iron-rich nanoparticles and growth twins are investigated. Crack-bridging processes via near-tip twinned bridges were identified. The multiple deformation mechanisms act synergistically to contribute to high strength and high ductility in the alloy.

1. Introduction

Iron-rich nanoparticle reinforced Cu has recently drawn the attention of researchers because it exhibits extraordinary strength while maintaining high elongation [1–6]. These Cu samples were reported to exhibit strengthening and improved ductility due to the formation of refined grains ($> 1 \mu\text{m}$) and iron-rich nanoparticles in the grain interior [1,5]. Transmission electron microscopy (TEM) analysis revealed a coherent interface between the nanoparticle and the Cu matrix [1–7]. These coherent nanoparticles were proposed to modify the dislocation motion state during deformation, and to improve the tensile strength without sacrificing the ductility, similar to the strengthening effect of nanoscale twin boundaries (TBs) in pure Cu [5,8]. On fracture surface of the nanoparticle reinforced Cu there were fine dimples, contrary to the coarse microvoids on the fractographs of nanoparticle-free Cu [1–5]. The former ex situ studies mentioned above, however, cannot accurately identify the complex deformation processes that prevail during the interaction between a crack and the microstructure [9]. Crack propagation is an essential clue for unveiling the fracture mechanism, involving crack tip stress distribution, microstructure-strength relationship, etc. In situ SEM (scanning electron microscope) tensile test have been performed to study the crack propagation process in iron-rich nanoparticle reinforced Cu [1], which, however, was limited to observations at the surface of the bulk specimen and in micron scale [10,11]. Thus far, the detailed mechanisms and crack tip processes responsible for high strength and high ductility of the nanoparticle

reinforced Cu have remained unclear, particularly at the sub-micron and nano scale. In this regard, in situ TEM straining can be used to explicitly reveal the deformation mechanisms, an indispensable method for observing deformation processes in real time [12].

Here, pure Cu and Cu-1.5Fe-0.5Co (wt%) alloy were prepared by casting. Tensile tests were carried out on as-cast pure Cu and Cu-1.5Fe-0.5Co (wt%) alloy. By using the in situ TEM straining experimentation, the deformation and fracture behaviors of Cu-1.5Fe-0.5Co (wt%) alloy under uniaxial tension were investigated. Particular attention was paid to dislocation activities, and interactions between the crack-tip and the microstructures of the alloy.

2. Experimental

Pure Cu and Cu-1.5Fe-0.5Co (wt%) alloy were prepared in a vacuum chamber by melting pure Cu, Fe and Co (with a purity of 99.99%, 99.50%, 99.95%, respectively) in a medium frequency electrical furnace and solidifying in a graphite mold. The furnace temperature was increased from room temperature to 1300 °C and maintained for 20 min. After homogenizing, the furnace temperature was reduced to 1150–1200 °C and the melt was poured into the graphite mold. Using this technique, pure Cu and Cu-1.5Fe-0.5Co (wt%) alloy were cast into 50 mm $\times$ 65 mm $\times$ 195 mm cuboid samples.

Tensile tests were carried out using a model CMT4105 TMS equipment at an initial strain rate of $1.3 \times 10^{-3} \text{ s}^{-1}$ until failure. The tensile dog-bone specimens had a 30 mm gauge length, 12.5 mm width, and

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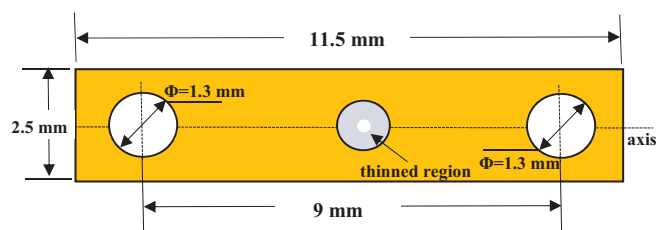


Fig. 1. The geometrical morphology and size of in situ tension specimen.

2 mm thickness. The fractured surface was investigated using a LEO1450 SEM. Specimens for in situ TEM tensile test with the geometry as shown in Fig. 1 were prepared by using electric discharge machining and thinned to a thickness of 80 μm by paper grinding (3000 grit). The central part of the tensile tests was further thinned by using a twin-jet apparatus with a solution of 25% HNO_3 + 75% CH_3OH at -30°C using a current/voltage of 50–60 mA/8–10 V. In situ straining tests were accomplished under uniaxial loading on a 200 kV JEM-2100F TEM by using a Gatan Model 654 single-tilt straining holder with the strain range of 2.0 mm and minimum step of 1 μm . The microstructures of the alloy, before and after in situ tensile test, were characterized in a 200 kV JEM-2010 TEM with a double-tilt holder.

3. Results and discussion

Tensile tests exhibit a substantial tensile strength elevation from 160 to 307 MPa, together with increment in ductility of 5% in Cu-1.5Fe-0.5Co (wt%) alloy, compared with the tensile properties of pure Cu (Fig. 2). The fractured surfaces of as-cast samples following tensile testing show two distinct topographies. The fractograph of pure Cu presents microvoid growth (Fig. 2a), whereas, the fractograph of Cu-1.5Fe-0.5Co (wt%) alloy is mainly covered by uniform and fine sub-micron dimples (Fig. 2b). During the tensile deformation, iron-rich nanoparticles decohere from Cu matrix arising from large stress concentration around the nanoparticle/Cu interface, resulting in pore forming and further leading to dimple fracture [13,14].

Fracture occurs in copper thin films in a different way from bulk fracture, originated from the discrepancies in the microstructures and stress states of the samples [15]. With the alloy foil elongated inside the TEM, initial crack formed from the circular edges roughly perpendicular to the loading direction in the thinned region. As the strain increased, initial deformation in the nanoparticle-free zone occurred at and ahead of the crack tip, as shown in Fig. 3a–d. Dislocations were emitted from the crack tip (Fig. 3a). Then, nanovoids nucleated in the

zone marked by a black circle in Fig. 3b and further coalesced with the main crack producing a sharp-tip crack (Fig. 3c). Afterwards, the crack tip blunted through the emission of dislocations (Fig. 3d) [16]. Thus in the nanoparticle-free zone, crack propagate in the manner of nanovoid initiation, growth and coalescence with the main crack. At the same time, dislocations were observed. Similar nanovoid coalescence process was observed during in situ TEM tensile test of nanocrystal Au and Ag thin films [17–20]. In the ~ 10 nm grained Au and Ag films, however, nanovoids grew and coalesced mainly through nanograin boundary grooving or sliding, in the absence of any dislocation activity within the grain. Deformation and fracture of such Au and Ag films are accompanied by the diffusion-controlled or shear-induced grain boundary (GB) rotation near the tip of a blunt microcrack, as well as nanovoid nucleating at the triple GB junction initiated by triple junction dislocations, and then joining the growing main crack [20]. It is noteworthy that the interface energy of grain boundary (γ_{GB}) was introduced to describe the strengthening effects of GBs [21–23], which aids in the understanding of the strength of nanomaterials. But in coarse-grained Cu-1.5Fe-0.5Co (wt%) alloy (the average grain size is ca. 87 μm), generation of nanovoids under load is induced by the superposition of the external stress and stresses created by lattice dislocation pile-ups stopped at either obstacles (e.g. the nanoparticle in Fig. 3e, f) or other positions in the grain interior (Fig. 3a, b). This is distinct from the process in nanocrystal Au and Ag films. It seems that if the contribution of GB sliding to the plastic strain is large in nanocrystals, the local stress may not exceed the stress required to generate mobile dislocations inside the grains [18]. Overall, the deformation in Cu-1.5Fe-0.5Co (wt%) alloy is governed by dislocation activities, differing to the deformation of nano-grained materials which is attributed to GB migration, GB sliding, or grain rotation [17–20,24].

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When the crack tip came near to an iron-rich nanoparticle, dislocations emitted from the crack tip piled-up around the nanoparticle (Fig. 3e–h), which can reduce the crack propagation rate [25]. Noticeably, some dislocations slipped through the nanoparticle along the interface and the others entered into the nanoparticle (Fig. 3e). This phenomenon is consistent with the dislocation motion state reported in [5], when the slipping dislocations encounter nanosized coherent interfaces, they slip along the coherent interface and enter into the nanoparticles. Some dislocations stay inside the nanoparticles and at the phase boundaries. Others pass through the coherent interface and slip to the adjacent nanoparticles. As such, iron-rich nanoparticles effectively impede crack propagation from blockage of dislocations impinging on the coherent nanoparticle/Cu interface which simultaneously offers pathways for dislocations glide along and through. This means that iron-rich nanoparticles in Cu-1.5Fe-0.5Co (wt%) alloy present substantial barriers for dislocation motion, which contributes to its high strength and significant strain hardening. Meanwhile, the nanoparticles provide coherent interfaces for the easy motion of dislocations, which permits the simultaneous generation of significant plastic deformation. Still, further extension of the crack accumulated more dislocations around the nanoparticle and eventually generated interface debonding (Fig. 3f, g). Interestingly, the main crack propagated by-passed the debonded interface (Fig. 3h), distinct from the normal case where the debonded interface can grow and coalesce with the main crack [26]. The stress concentration at the crack tip under loading was alleviated by dislocation generation and emission [16]. This crack tip stress can be transferred to the nanoparticle zone (the nanoparticle/matrix interface and its surrounding area), where emitted dislocations piled up, and further produced strain hardening at the zone. As a result, the main crack deviated from this hardened nanoparticle zone and propagated along other softer area, simultaneously bypassing the debonded interface. So the interface debonding in Fig. 3f, g cannot facilitate the crack initiation and propagation. Overall, crack interaction with iron-rich nanoparticles in Cu matrix is beneficial for the

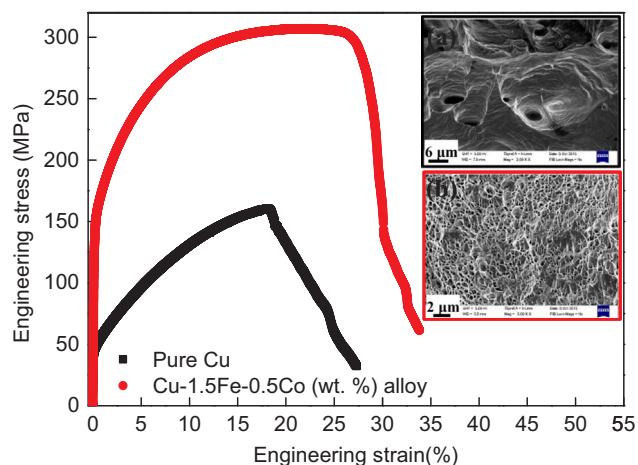


Fig. 2. Tensile engineering stress–strain curves for pure Cu and Cu-1.5Fe-0.5Co (wt%) alloy; inserted SEM images are related to the fracture surfaces of (a) pure Cu and (b) the alloy, respectively.

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