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In-situ TEM investigation of fracture process in an Al-Cu-Mg alloy



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ABSTRACT

The deformation and fracture process in Al–Cu–Mg alloy were investigated by using the in situ straining transmission electron microscopy (TEM) method. Some major aspects of the fracture process, including dislocation emission and migration, thinning of dislocation free zone (DFZ), crack propagation (both in continuous and discontinuous manners) and slipping/twinning deformation, can be observed. The rod-like T dispersoids, which may increase the microcrack initiation sensitivity, also can effectively prevent the fast and continuous propagation of the crack. DFZ ahead of crack tip can be thinned in a mixed mode characterized by tearing and shear deformation, while nanovoids, which are typical characteristics in DFZ during discontinuous crack propagation, may originate from the enrichment of defects such as dislocations and vacancies. Deformation twinning at crack tip can slow down crack propagation and change crack propagation path, thus may be beneficial to the fracture toughness of the alloy.

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1. Introduction

The deformation and fracture behaviors of structural materials are always the main aspects for its performance. Preliminary research to reveal the deformation and fracture mechanism generally accomplished via a comprehensive understanding of macroscopical deformation features, fractographs and some static mechanical properties such as tensile strength and fracture toughness [1–5]. With the development of materials science and electron microscopy, a real-time and high resolution observation of the deformation and fracture process in materials, especially for them within micro-nano scales, becomes available and increasingly receives widespread attentions [6]. In the past few decades, by means of in situ straining transmission electron microscopy (TEM) methods and related computer simulations, many researchers focused on the deformation and fracture behaviors of pure metals [7-14], metallic alloys [7,15-17] and nanomaterials (including nanocrystalline [18,19], nanotubes [20], nanowires [21] and micro- or nanopillars [22,23]) to reveal characteristics and mechanisms of plastic deformation, dislocation activities, crack propagation as well as the crack-tip substructures. In situ straining TEM methods, which directly, continuously and microscopically show the dynamical processes during deformation and fracture, can effectively avoid the probable misunderstandings induced by

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static observation and theoretical speculation [6], thus are of great importance to fully understand the deformation and fracture behaviors of various materials at micro-nano or atomic levels.

As a typical representative of commonly-used metallic alloys, the age-hardenable Al–Cu–Mg alloys are widely used in aerospace and automobile industries due to their excellent properties of strength, fracture toughness and fatigue and corrosion resistance [24,25]. In the common heat treatment conditions, Al–Cu–Mg alloys are typically composed of multiphases and polycrystallines, which may make their deformation and fracture behaviors quite different with those of either pure metals and single crystals, or composites and nanomaterials. To date, too much concern has been paid to the fracture process, fractographic features and mechanical properties of aluminum alloys [1–5], and three typical crack propagation modes, namely opening or tensile mode (Mode I), sliding mode (Mode II) and tearing mode (Mode III), were systematically revealed. However, no relevant in situ TEM investigations were carried out to unravel the deformation and fracture behaviors of Al–Cu–Mg alloy in detail.

In this study, by using the in situ straining TEM method, the deformation and fracture behaviors of Al–Cu–Mg alloy under uniaxial tension were investigated. Particular attention was paid to dislocation activities, crack-tip substructure evolution as well as interactions between crack tip and intermetallic particles.

2. Experimental

2024 Alloy with an nominal composition of Al-4.2Cu-1.5Mg- 0.6Mn-0.5Fe-0.5Si (wt%) was cast and homogenized at 460 $^\circ C$

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for 16 h before being hot rolled to a 2 mm thin sheet. The alloy was subsequently solution treated at 495 °C for 45 min and water quenched to room temperature, followed by natural ageing at 25 °C for more than 200 h (T42 state). Specimens for in situ TEM tensile experiment with the geometry as shown in Fig. 1 were prepared by using electric discharge machining (EDM) cutting and mechanical grinding to 200 µm thick thin foil. The central parts of the specimens were further thinned by using a twin-jet electropolisher with solution of 30% nitric acid and 70% methanol below -25 °C at 15 V. In situ straining experiments were accomplished on the 300 kV FEG-TEM Tecnai F30 G² by using a Gatan Model 654 single-tilt straining holder with the strain range of 2.0 mm and minimum step of 1 um. The whole specimen was subjected to uniaxial loading whereas the loading modes in local regions at the crack tip may be much more variable and complicated with the changes of crack propagation path. Series of Bright field (BF) TEM, high resolution TEM (HRTEM) and high angle angular dark field scanning transmission electron microscopy (HAADF-STEM) images were recorded to reveal the deformation and fracture behaviors from multiple scales and perspectives.

3. Results

For T42 2024 alloy, many rod-like T (Al₂₀Cu₂Mn₃) dispersoids and various morphologies of dislocations, which generally formed during water quenching of the supersaturated solid solution, collectively constitutes the main microstructural aspects of the alloy. Detailed introduction can be found in our previous contributions [26,27]. Under uniaxial tensile loading, crack initiates from the circular edges of the thinned region which are apt to cause stress



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

concentration. With the strain increasing, crack gradually propagates and plastic deformation tends to occur at and ahead of the crack tip, which are characterized by dislocation emission, fast migration and final pile-up far away from the crack tip. The typical morphology of the crack and its adjacent regions is shown in Fig. 2, in which two distinct regions between A and B, B and C can be distinguished, namely dislocation free zone (DFZ) and inverse pileup dislocation group (Dp), respectively. The range of Dp is much larger than that of DFZ, and the dislocation density in Dp decreases with the distance to crack tip. Furthermore, strong interactions can also be observed between emitted dislocations and T dispersoids (site D) as well as the intrinsic dislocations (such as helical dislocation at site E), which play important roles in hindering the emission and migration of dislocations from the crack tip.



Fig. 3. HAADF-STEM image showing the microstructure ahead of crack tip. Double arrow indicates the tensile loading direction.



Fig. 2. A montage of TEM images showing the dislocation emission, migration and pile-up at and ahead of crack tip, where A indicates the crack tip, regions between A and B, B and C are dislocation free zone (DFZ) and inverse pile-up dislocation group (Dp), respectively. Single and double arrows indicate the crack propagation direction and tensile loading direction, respectively.

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