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Direct measurement of precipitate induced strain in an Al-Zn–Mg-Cu alloy with aberration corrected transmission electron microscopy

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ABSTRACT

Precipitates and their associated strain fields significantly influence mechanical properties and, consequently, the industrial performance of aluminum alloys. In this work, we present a direct measurement of strains induced by η′ and η precipitates in an Al-Zn-Mg-Cu alloy using aberration-corrected high-resolution transmission electron microscopy and quantitative strain analysis. The results demonstrate that the strain induced by precipitates in the Al-Zn-Mg-Cu alloy shows significant tensile strains perpendicular to the longitudinal direction of the precipitate discs on the side of the discs and along the longitudinal direction at both ends of the η′ and η precipitates. This strain field can be described by an equivalent dislocation model, in which the lattice mismatch between the precipitate and the matrix is equivalent to a series of dislocation pairs along the precipitate/matrix interfaces.

1. Introduction

Precipitates and their related strain fields are instrumental for achieving desired mechanical properties in metallic materials. For example, for typical Al-Zn-Mg(Cu) alloys, the internal strain induced by nanometre-scaled precipitates serves as a well-known solution for enhancing the alloys' strength because the precipitates can impede the motion of dislocations, thus resisting deformation (Polmear, 2005). The usual precipitating process of these alloys can be summarized as follows (Berg et al., 2001; Liu et al., 2011; Liu et al., 2015):

$$\text{Solidsolution} \rightarrow \text{GPzone} \rightarrow \text{Metastable}\eta'\text{phase} \rightarrow \text{Stable}\eta\text{phase}.$$

Among the precipitates, the η′ precipitate is generally regarded as the hardening phase (Wang et al., 2002). Therefore, to control the properties of precipitation-hardened materials, a quantitative description is desired to estimate the strain field produced by the inclusion of precipitate particles. Early research presented that the strain of heterophase interfaces could be simulated by a continuous set of dislocations with infinitesimal "Burgers vector" located at the interface between the matrix and the precipitate (Sutton and Balluffi, 2006). Nevertheless, this model is mainly employed to analyze relatively straight single hetero-interfaces. Using the equivalent inclusion method, a theoretical model has been proposed to describe the strain outside of a disc-shaped particle (Mura, 1987), although the model was only successful for single ellipsoidal inclusion under very specific conditions. Recently, Douin et al. (Douin et al., 2010) presented an elastic model for needle-shaped particles embedded in an Al matrix, which used a dipole of edge dislocations at the longest dimension to replace the lattice mismatch. These works mainly focused on the strain field induced by a unidirectional lattice mismatch, which can be simplified as a series of edge dislocations for thin films (Sutton and Balluffi, 2006) or an edge dislocation dipole for needle-shaped particles (Douin et al., 2010) that are perpendicular to the lattice mismatch direction. However, for general precipitates, particularly those that do not have a needle shape with different heterophase interfaces along different crystallographic planes, strains are introduced at these heterophase interfaces. Accordingly, a more universal dislocation model is desired to simulate the strain fields induced by coherent or semi-coherent precipitates, including precipitates with complex or unknown structures, at their different heterophase interfaces with the matrix.

Recently, quantitative high-resolution transmission electron microscopy (HRTEM) has been developed as a powerful means to measure strains in materials at the atomic scale (Johnson et al., 2007; Hÿtch et al., 2008). Meanwhile, the aberration-corrected HRTEM technique could effectively eliminate the delocalization

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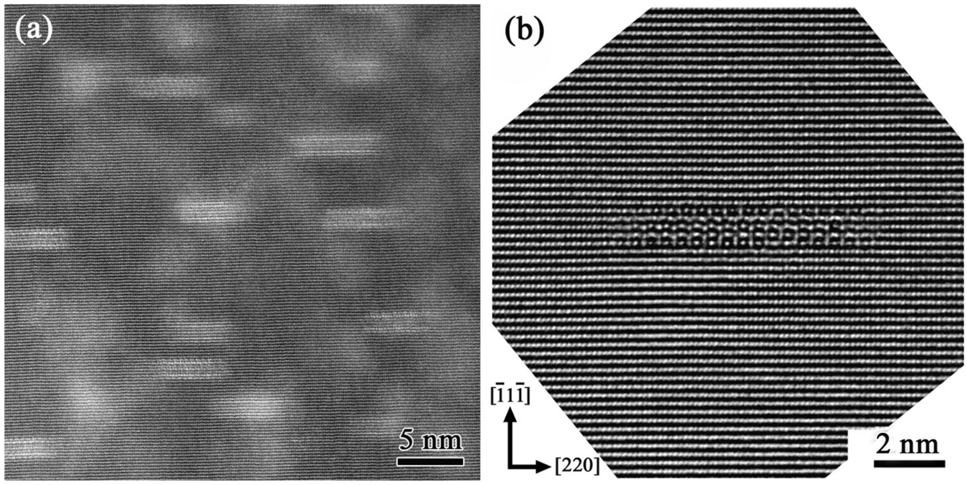

Fig. 1. (a) Aberration corrected HAADF-STEM image of η′ phases viewed along the $[\bar{1}12]_{Al}$ zone-axis in an Al-Zn-Mg-Cu alloy. (b) Aberration corrected HRTEM image of a η′ phase.

effect of HRTEM imaging on quantitative strain analysis, thus improving the reliability and accuracy of the quantitative analysis (Du and Phillipp, 2006; Du et al., 2010). Under favourable conditions, quantitative HRTEM analyses, such as LADIA (lattice-distortion analysis) (Du et al., 2002; Zhang et al., 2011; Wang et al., 2014; Lu et al., 2015) and GPA (geometric phase analysis) (Hÿtch et al., 1998; Hÿtch et al., 2008; Zhao et al., 2008), are able to derive the local lattice strain directly from the HRTEM images for objects with coherent or semi-coherent structures. In the present work, an Al-Zn-Mg-Cu alloy was chosen for the investigation. Precipitates including metastable η′ precipitates and stable η precipitates were observed through aberration corrected HRTEM, and the induced strains were measured by the LADIA quantitative analysis. Based on the experimental measurement and the theoretical calculation, an equivalent dislocation model associated with the lattice mismatch is presented, which can accurately simulate the strain field around complex-structured precipitates at their heterophase interfaces with the matrix in different crystallographic planes.

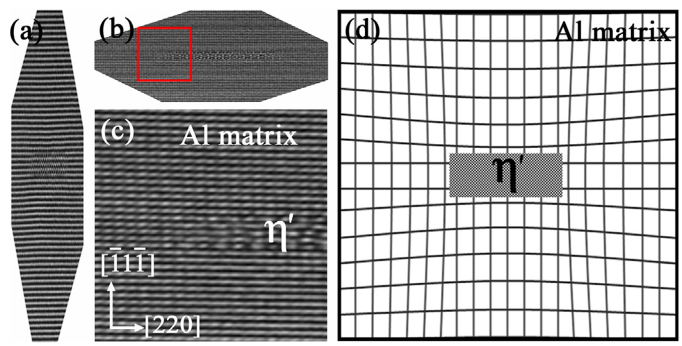

Fig. 2. (a) and (b) Shrunken HRTEM images of Fig. 1b along the [220] (horizontal) and [$\bar{1}1\bar{1}$] (vertical) directions to exemplify the distortion of the matrix parallel to the vertical and horizontal axes, respectively. (c) Enlarged HRTEM image of the red-boxed area in (b). (d) Schematic illustration of the lattice distortion around the η′ phase deduced from a–c.(For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

2. Experimental

The nominal chemical composition of the studied aluminum alloy was Al–7.8%Zn–1.6%Mg–1.8%Cu–0.13%Zr (wt.%). The amounts of Fe and Si were maintained below 0.15%. Samples were solution-treated at 748 K for 4 h, quenched in cold water at 293 K and then aged at 393 K for 24 h or 433 K for 4 h to introduce metastable η′ or stable η precipitates. TEM specimens were then prepared by conventional cutting, grinding and ion-milling, and a Gatan precision ion polishing system with a liquid nitrogen cooling stage was used. HRTEM images and high-angle annular-dark-field scanning transmission electron microscopy (HAADF-STEM) images were obtained using an aberration-corrected transmission electron microscope (Titan^3 G2 60–300). By adjusting the spherical aberration coefficient (*Cs*) to a small value that was close to zero at Scherzer defocus (Haider et al., 1998), we can effectively minimize the delocalization effect from the HRTEM imaging. Quantitative strain analysis was performed with LADIA software (Du et al., 2002; Du and Phillipp, 2006). Via a cross-correlation calculation, the effects from the shot noise and intensity variation of the background were eliminated from the quantitative measurement. A two-dimensional spline fitting of the intensity peaks provided additional sub-pixel precision for the determination of the image peak positions. To eliminate the effects of the variations of the local focus value and specimen thickness, the HRTEM images with a uniform image contrast and pattern were employed for the strain analysis (Du and Phillipp, 2006).

3. Results

After aging at 393 K for 24 h, η′ precipitates with an average size of ~6 nm × 1.4 nm are observed with a homogenous distribution in the Al matrix (Fig. 1a). Fig. 1b shows a typical HRTEM image of a η′ precipitate in the Al-Zn-Mg-Cu alloy obtained by aberration-corrected TEM. The η′ precipitate was viewed along the $[\bar{1}12]_{Al}$ direction.

To reveal the lattice distortion in the matrix induced by the η′ precipitate, the HRTEM image was shrunk along the [220] (horizontal, Fig. 2a) and [$\bar{1}1\bar{1}$] (vertical, Fig. 2b and c) directions. Slight lattice expansions are observed in the Al matrix around the η′ precipitate (Fig. 2a and c), which are sketched in Fig. 2d remarkably. This result is likely attributed to the lattice mismatch between the precipitate and the matrix. The strain fields around the η′ precipitate were measured using the LADIA method. Normal strains ε_{xx} (Fig. 3a) and ε_{yy} (Fig. 3c) show mirror symmetry along the y and x axes of the disc-shaped precipitate, respectively. In contrast, the shear strain, ε_{xy}, shows a diagonal symmetry around the precipitate. For the ε_{xx} strain, a strongly concentrated expansive strain is observed at each side of the precipitate in the horizontal direction. For the ε_{yy} strain, the concentration is slightly weaker compared with ε_{xx}, while the