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Study of coherence strain of GP II zones in an aged aluminum composite

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ABSTRACT

Strain mapping using the geometric phase analysis (GPA) technique was applied to Al–GP II (Guinier–Preston) nanoscale precipitates, using both high resolution transmission electron microscopy (HRTEM) micrographs as well as the exit wave function (EWF) obtained by focal series reconstruction. The experimental strain results were compared with strain maps obtained from an atomic model which consisted of an Al supercell containing a GP II precipitate. It was built as a reference from literature data. The experimental results demonstrate a complex strain distribution and larger fluctuations than the reference strain maps. These differences were found to be partly a consequence of image artifacts produced by the technique as well as complex microstructural events which were present at the development stage studied.

1. Introduction

It is well known that nanoscale GP II zones formed during aging of Al alloys cause an important increase in mechanical properties such as flow and ultimate tensile strength. Its ability for altering dislocation propagation in the matrix relies on distinct hardening mechanisms such as chemical hardening, modulus, as well as coherency and order strengthening. The morphology of these zones has a very important role in achieving the optimum mechanical properties. Strain energy and interfacial energy are the most important parameters that dictate the precipitate's morphology through a complex interaction that has been described elsewhere [1]. The strain energy depends on the lattice mismatch between precipitate and matrix lattice and is maximized when the 2 lattices are coherent. The completely coherent GP II phase produces a higher elastic strain in the matrix than the θ' phase which is semicoherent and causes hardness values to decrease when present. As a consequence, there have been some attempts to quantify the elastic strain in the Al matrix [2–4]. However, so far this aspect is still a matter of debate since different values have been found. Also the exact GP II chemical composition has not been determined accurately. It is important to mention that the most accepted model is the one that involves three Al atomic planes separated by 2 Cu atomic planes. However, it has been demonstrated that even inside of these planes there are some Al and Cu concentration gradients. The same complex situation regarding the exact chemical composition occurs with GPB precipitates (Guinier–Preston–Bagaryatsky) that commonly appear in these alloys and which includes Mg in addition to Al and Cu.

Amongst the different techniques available for mapping strain, those based on electron scattering offer the highest spatial resolution. The classical technique for strain mapping in the TEM is convergent beam electron diffraction (CBED), however as already noted in the original paper [5] the presence of strain gradients in the illuminated area may introduce high order Laue zone (HOLZ) lines splitting and broadening and thus severely hamper the interpretation of CBED patterns in the close vicinity of defects. The CBED technique also requires the specimen to be quite thick (usually thicker than 100 nm), making it practically inapplicable for samples containing a high density of small defects (such as the less than 10 nm wide GP II zones studied in this work) because of the very low chance of sampling the projection of an individual defect.

Dark-field inline or off-axis electron holography is a very promising technique for evaluating strain fields across a large field of view while maintaining a high spatial resolution of 1 nm for the inline configuration [6] and 4 nm for the off-axis configuration [7]. Note that, apart from the potentially higher spatial resolution,

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Table 1
Proportions of elemental powders used in this work (wt%).

Element	wt%
Al	92.66
Cu	4.26
Mg	1.49
Mn	0.59
Si	0.50
Ti	0.15
Zn	0.25
Cr	0.10

and the much simpler experimental setup, the inline configuration potentially also offers better signal to noise properties compared to its off-axis counterpart [6].

Because of their small size, studying the strain field around individual GP II zones does not require a very large field of view which is why we have decided to apply the geometric phase analysis (GPA) technique developed by Hytch et al. [8] which is based on processing HRTEM micrographs. Despite its limitation of a relatively small field of view, the method has been applied successfully on quantifying strain on antiphase domain boundaries [8], strain fields around dislocations [9], Ge nanowires [10], and more recently on Al–Pb interfaces [11]. However until now, it has not been any attempt to quantify the elastic strains by applying this technique on aluminum precipitates, so that is why the main objective of the present work is to estimate these values in GP II nanoscale precipitates.

2. Materials and methods

Al-2024 alloy with composition adjusted to the range of commercial materials was fabricated by powder metallurgy techniques using elemental powders and graphite particles as a reinforcement. The exact composition is shown in Table 1. More details about the fabrication procedure can be found in reference [12]. After that, samples were solution treated at 530 °C and artificially aged at 140 °C for different times. Microhardness measurements were done with the intention to determine how this property behaved as a function of aging time. The maximum hardness was found at 24 h in the reinforced material and near to 48 h in none reinforced material. Consequently, a time of 12 h of aging (i.e. before the maximum hardness has been reached) was selected in the reinforced samples with the purpose to determine the elastic strain fields around GP II zones. Samples for electron microscopy were prepared using ultrasonic cutting, mechanical grinding, jet electropolishing and ion milling. Microstructure observations and energy dispersive X-ray (EDX) chemical analysis were carried out in the Sub-Electron-Volt Sub-Angstrom Microscope (SESAM) (Carl-Zeiss NTS) operated at 200 kV while HRTEM work was performed on a JEOL 4000FX microscope operated at 400 kV. In order to avoid commonly observed artifacts in strain maps caused by aberrations in HRTEM images we applied the GPA technique to the complex-valued exit face wave function (EWF) [11] reconstructed from a series of HRTEM micrographs recorded at different values of the defocus [13] with the sample oriented in the [0 0 1] zone axis of the matrix material. By using GPA, the variation in local lattice constant in the HRTEM micrographs was calculated by taking a strain free area as a reference. This is achieved by extracting the geometric phase maps for 2 non-colinear reflections, each obtained by first multiplying the Fourier transform of the image with a mask around those reflections and performing the inverse Fourier transform again [8]. Since, in the absence of changes in the specimen thickness, orientation, crystal structure, and electrostatic or magnetic fields the geometric phase is directly proportional to the local lattice displacement, it may be used for computing strain maps. We applied a modified version of the original GPA algorithm [8] which allows extracting the geometric phase from the EWF [11]. A cosine mask of 0.4 nm^{-1} in size was applied to obtain the (2 0 0) and (0 2 0) geometric phase maps, perpendicular (*x*-axis) and parallel (*y*-axis) to the GP II zones respectively.

In addition, a reference atomic model consisting of a GP II precipitate embedded in an Al supercell was created to make a comparison with the experimental results using the software QSTEM [14]. It was considered that this precipitate existed throughout the whole supercell thickness (12 nm). The GP II precipitate model consisted of a tetragonal lattice with two Cu planes separated by three Al planes with lattice parameters of 0.4049 nm × 0.4049 nm × 0.768 nm [15]. Additionally, a uniform strain of 2% was introduced in the first five (2 0 0) adjacent planes according to the lattice mismatch between the GP II structure and the Al matrix which was considered to have a face centered cubic structure and a lattice parameter of 0.4049 nm. The EWF of this model structure was simulated using the software QSTEM [14]. This software is based in the multislice method proposed by Cowley and Moodie [16] by which the specimen is divided into a number of thin slices perpendicular to the incident-beam direction. In each slice, the effects of Fresnel diffraction (propagation) in the slice and the effects of the specimen potential in the slice (transmission) are treated separately. The slice thickness was kept constant and it was determined to be close to 1/2 of the Al lattice parameter in order to be approximated as a weak phase object according to literature [17]. By aligning the atomic columns with each slice, the multislice errors are minimal.

Finally, since Al has an anisotropy factor close to 1, linear isotropic elasticity theory considering plain strain conditions was used to calculate the stress state acting around GP II zones using the following expression [18]:

$$\begin{bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{xx} \\ \tau_{xy} \end{bmatrix} = \begin{bmatrix} \lambda+2\mu & \lambda & \lambda & 0 \\ \lambda & \lambda+2\mu & \lambda & 0 \\ \lambda & \lambda & \lambda+2\mu & 0 \\ 0 & 0 & 0 & \mu \end{bmatrix} \begin{bmatrix} \varepsilon_{xx} \\ \varepsilon_{yy} \\ 0 \\ 2\gamma_{xy} \end{bmatrix} \quad (1)$$

3. Results and discussion

3.1. Nature and morphology of GP zones

Fig. 1(A) shows a TEM micrograph of the GP II zones obtained in the material after aging for 12 h. Two mutually perpendicular variants were detected when the specimen was oriented along the [0 0 1] matrix zone axis. The graph in Fig. 1(B) shows EDX data from a line scan acquired across a GP II precipitate.

These compositional profiles helped us to decide whether the early precipitates were GPB or GP zones, or in other words, whether the newly formed phase contained Mg, or not. The EDX line scan shows in a qualitative way that there was no Mg in the GP II zones. Mg was detected in other big Al dispersoids that were observed in the microstructure but were not presented in this work. The EDX line scan shown in Fig. 1(B) exhibits only enrichment in Cu since the counts for this element were significantly higher in reference to the matrix. The increase in Cu counts that is observed on the left hand side was caused by the close proximity of another GP II zone. It should be pointed out that the observed width of the Cu-rich layers is in good agreement with the average GP II width of approximately 1.5 nm, taking into account the limited resolution of the EDX acquisition.

3.2. Strain mapping of GP zones

Fig. 2(A) and (B) presents the phases of the EWFs that were reconstructed from an experimental focal series and simulated from the reference Al supercell, respectively. As can be seen, there is some contrast variation across the image in the experimental case, however as has been explained elsewhere [19], for centrosymmetric structures like the present sample, slightly varying amplitude changes in HRTEM images do not influence markedly strain mapping results.

It is important to point out that in this micrograph the periodicity of the lattice fringes is well defined – a prerequisite for applying GPA [19]. The exception is the left side of the GP II zone on which it seems that the reconstruction has revealed some alterations in the lattice fringe contrast. As we will show, these changes in the lattice contrast are related to the presence of an edge dislocation at the far left edge of the field of view. However, in order to obtain more representable strain information, our analysis will be focused in the right hand side of the GP II zone. The EWF in Fig. 2B has been used to generate the reference strain maps that will be compared with the experimental ones.

It is important to point out that dynamical scattering phenomena are assumed to be present in both EWFs. Also it should be mentioned that in principle, the EWF reconstructed from a series of HRTEM micrographs did not contain objective lens aberrations since this reconstruction was made using several defocus values. However (as would be explained in later sections) changes in the local lattice orientation, specimen thickness and chemical gradients along the sample thickness are parameters which are difficult