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Atom Probe Study of Shear-Induced Dissolution of GP Zones in Al-3Cu Alloy

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A model Al-3Cu-(0.05Sn) (wt.%) alloy containing a bimodal distribution of relatively shear-resistant θ' precipitates and shearable GP zones is considered in this study. It has recently been shown that the addition of the GP zones to such microstructures can lead to significant increases in strength without a decrease in the uniform elongation. In this study, atom probe tomography (APT) has been used to quantitatively characterise the evolution of the GP zones and the solute distribution in the bimodal microstructure as a function of applied plastic strain. Recent nuclear magnetic resonance (NMR) analysis has clearly shown strain-induced dissolution of the GP zones, which is supported by the current APT data with additional spatial information. There is significant repartitioning of Cu from the GP zones into the solid solution during deformation. The evolution of the sizes and shapes of the Cu containing features in the solid solution, as a function of applied plastic strain, are discussed.

Keywords: *GP zones, dissolution, atom probe, precipitation hardening.*

1. Introduction

It is well known that during the fatigue loading of engineering alloys dislocations can arrange themselves into channels known as persistent slip bands (PSB's) where extensive strain accommodation occurs [1]. In alloys containing shearable precipitates, dislocation motion within the PSB's may lead to shear-induced dissolution of the precipitates creating a composite structure: a soft solid solution within the PSB and a precipitate hardened structure outside of the PSB, e.g. [2]. Shear induced dissolution of precipitates can also occur during monotonic loading, and in the case of Al alloys, within the regimes of strains relevant for conventional forming processes [3]. The process is a means to dynamically repartition solute from the precipitates to the matrix during deformation and can strongly influence the resulting mechanical properties. Indeed, it has recently been suggested that under certain circumstances this is a means to enhance the work hardening of precipitate-containing Al alloys at large strains [4,5] with beneficial effects on the elongation and fracture properties exhibited.

Many researchers have established that precipitate shearing occurs in some systems during straining. Perhaps not surprisingly, there have been limited studies of the kinetics of the shear-induced dissolution process or its dependence on the nature of the precipitates and processing variables. The experimental technique of choice for characterisation of precipitates in alloys has traditionally been transmission electron microscopy (TEM). The problem is that it is very difficult to resolve nanometer size precipitates in alloys using TEM after deformation because of the large dislocation densities. The first measurements of the kinetics of shear-induced dissolution of precipitates in Al alloys have recently been reported using NMR [3]. These investigators showed that in an Al-3Cu alloy aged to give a bimodal distribution of shear-resistant Al_2Cu precipitates (θ' phase) and shearable GP zones, approximately 50% of the Cu in the GP zones was repartitioned to the solid solution during straining at room temperature. NMR has the advantage that it is a bulk technique and provides average quantities. The disadvantage is that it provides no spatial information about the dissolution process. This spatially resolved information is necessary for the development and verification of model descriptions of the dissolution process. This is required if the dissolution process is to be exploited as an alloy design tool.

In this work we use APT to examine the effect of applied plastic strain on the distribution of GP zones in the same Al-3Cu alloy previously studied by NMR to provide atomistic information about the dissolution process.

2. Experimental procedure

A model Al-3Cu-(0.05Sn) (wt.%) alloy containing a bimodal distribution of relatively shear-resistant θ' precipitates and shearable GP zones was used for this study. The material was solution treated for 1 h at 520 °C followed by water quenching, aged for 1 h at 200 ºC (θ' production) with an additional heat treatment at 65 ºC for 600 h (GP zone production). Such a microstructure is suitable for a study of the strain-induced dissolution of GP zones using APT because the relatively shear-resistant θ' precipitates have the effect of spreading, or homogenizing, the deformation during straining. In the absence of the shear-resistant precipitates a heterogeneous deformation structure with the plasticity heavily localized in shear bands would be expected. Transmission electron microscopy observations of the microstructures of deformed Al-3Cu containing θ' precipitates of the same size considered in this study illustrate that the plasticity is relatively uniform throughout the microstructure [6]. This provides some confidence that the observations made using APT are likely to be reasonably representative of the bulk behaviour. Samples for APT study were prepared from the gauge length of tensile samples deformed using an Instron tensile testing machine to plastic strains of 3% and 9%. The plastic strain was controlled by the use of an extensometer. A sample without strain was also examined for comparison purposes.

The APT work was performed on sharp needles with tip radii ~50-100 nm prepared by standard two-stage electropolishing techniques using a Local Electrode Atom Probe (LEAP) instrument at ~20 K with a pulse fraction (the ratio of the pulse voltage to current standing voltage) of 25% and a pulse repetition rate of 200 kHz, under a vacuum of below 10^{-10} Torr.

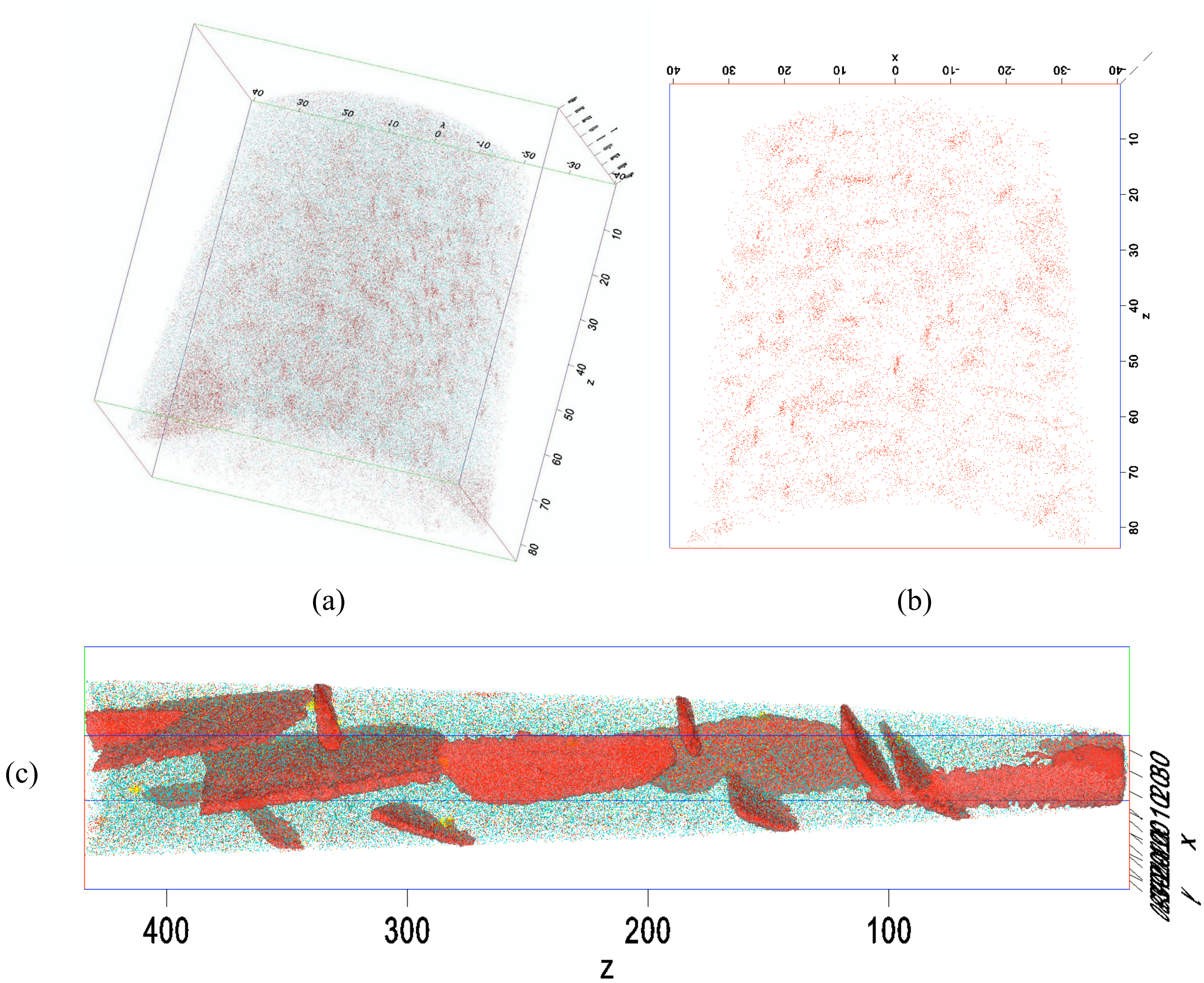
3. Results

3.1 APT Maps

Fig. 1 shows three-dimensional reconstructions of the atom probe data of the Al-3Cu alloy after tensile deformation. At 0% strain, Fig. 1a shows a volume containing two θ' plates, cut-off at the bottom edges, with GP zones homogenously distributed between them. Fig. 1b is a 5 nm slice of the data in Fig. 1a and clearly shows that the platelet GP zones are ~5 nm in length, which is consistent with TEM observations of the same material [3]. After tensile deformation, the θ' plates remain but the GP zones cannot be observed, as shown in Figs. 1c and 1d for 3% and 9% applied plastic strains, respectively. It is interesting to note that in the larger volume of the 3% strain sample (Fig. 1c), the angles between θ' plates are no longer consistently 90º, as they are in undeformed samples. This is expected and is due to the accumulation of geometrically necessary dislocations (GNDs) at the plate-matrix interface [7]. In order to discern atomic-scale information about the solute arrangements from the reconstructed APT data, a data-mining approach has been carried out by application of cluster-finding algorithms; the method of which is described in the next section.

3.2 Evolution of Solute Distribution

Quantitative APT analysis of nano-scale solute atom arrangements within a microstructure is a non-trivial exercise. This is made even more complicated when they co-exist with larger features such as precipitates. Characterisation of multi-scale microstructures using ‘cluster-finding’ algorithms is made complex because the optimal parameters to reveal the smaller solute atom aggregations are not the same as those required to highlight the larger precipitate features.



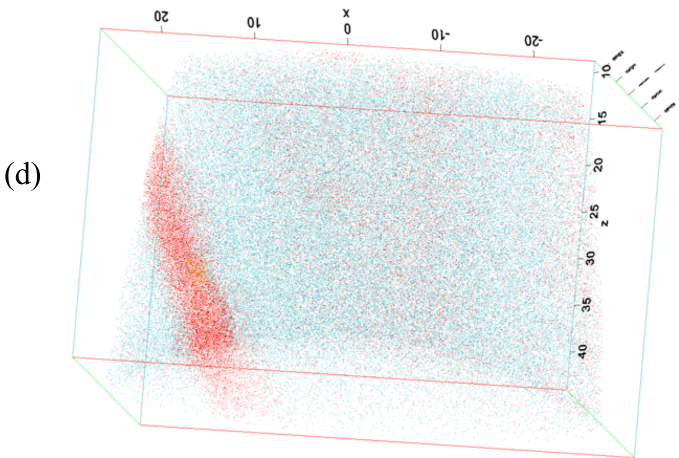


Fig. 1: Three-dimensional atom maps of the Al-3Cu(-0.05Sn) (wt.%) alloy after tensile deformation. (a) 0% strain; (b) 5 nm volumetric slice of the data presented in (a); (c) 3% plastic strain; (d) 9% plastic strain. Red (dark) represents Cu atoms and green (light) represents Al atoms. All scale bars are in nm.

Parameter selection for the data-mining algorithms used in this work is only briefly outlined and the details are reserved for an upcoming publication. The θ' plates were highlighted by using a technique based on the maximum separation method [8] with a d_{max} value determined by comparison of frequency histograms of 5th nearest neighbour (5NN) Cu solute atom distances from both experimental and random data (Fig. 2a); and a large N_{min} parameter chosen to isolate these obviously large features. Cluster finding was carried out on the remaining Cu solute atoms using the Core-Linkage algorithm [9] where d_{max} was determined by the same method (see Fig. 2b), $d_{link} = d_{max}/2$, and $N_{min} = 2$.

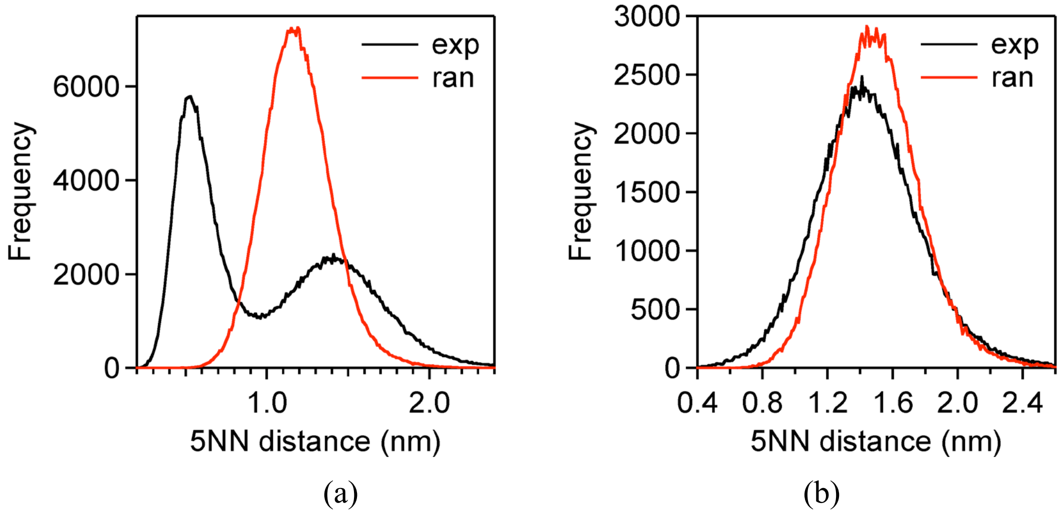


Fig. 2: Examples of fifth nearest neighbour (5NN) frequency histograms of the distance between Cu solute atoms in the Al-3Cu(-0.05Sn) (wt.%) alloy with 0% strain. All the data is represented in (a), whereas in (b) the θ' plates have been removed.

Results from application of the cluster-finding algorithms to the data in Fig. 1 are shown in Fig. 3a as histograms of the number density per cm^3 of Cu-Cu features as a function of size (number of Cu atoms). It can be seen that there are significantly less Cu-Cu features sized 2-100 atoms in the case of 3% strain compared to 0% deformation, and even fewer in the sample strained plastically to 9%. The 5nm GP zones are expected to contain 50-150 Cu atoms each, taking into account APT detector efficiency. For singleton Cu atoms on the other hand, Fig. 3b shows a correspondingly significant increase for the 3% strain case as compared to 0% deformation. This is direct evidence of the shear-induced dissolution of the GP zones, re-partitioning the Cu solute atoms back into the solid solution.

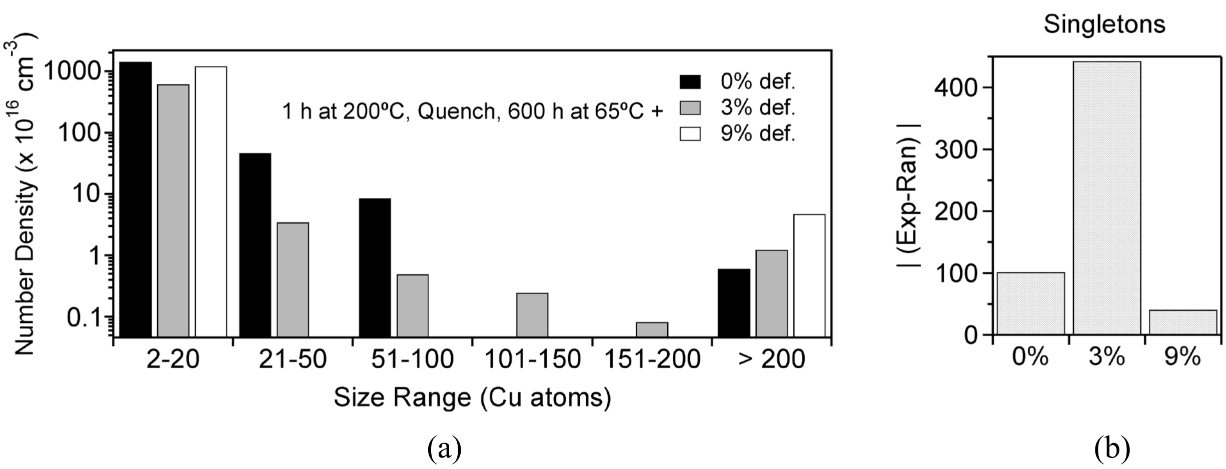
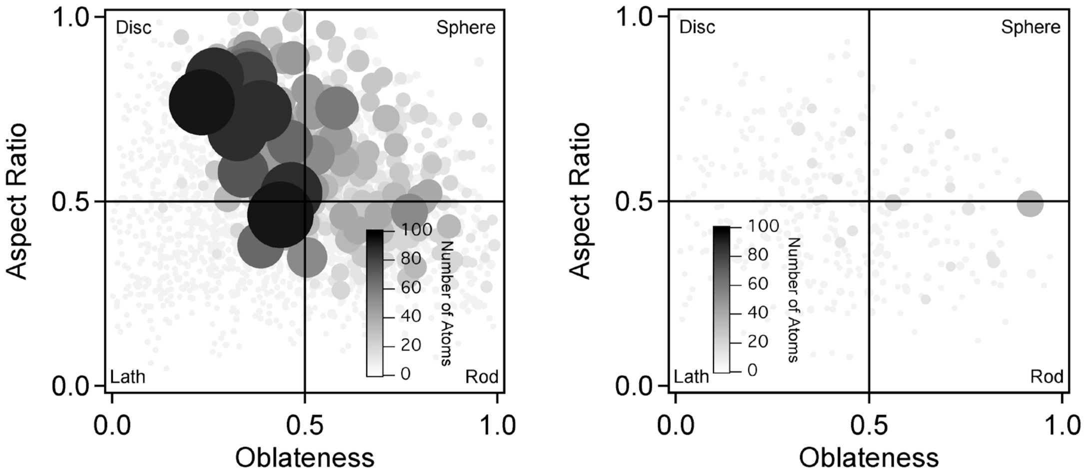


Fig. 3: (a) Experimental-minus-random number densities of Cu-Cu features versus size range (in Cu atoms) for the Al-3Cu(-0.05Sn) (wt.%) alloy after 0%, 3% and 9% plastic strain. (b) Absolute value of experimental-minus-random number density of Cu singleton atoms (since random > exp.).

The analysis shown in Fig. 3 provides information only about the sizes and number densities of the Cu containing solute aggregations. APT can also provide information about the shapes of the Cu-Cu features. The principal dimensions (s_1, s_2, s_3; where $s_1 \geq s_2 \geq s_3$) [10] describing the shape of each of the Cu-Cu features is plotted in Figs 4a and 4b in terms of aspect ratio (s_2/s_1) and oblateness (s_3/s_2) to generate a feature morphology map (rod, sphere, lath or disc) for the 0% and 9% plastic strain samples, respectively. The sizes of the features represented in Figs. 4a and b are indicated by the relative size of the data point markers and the greyscale legend. Clearly, the disc-shaped features that populate the upper left quadrant of Fig. 4a correspond to the GP zones observed in Fig. 1a and 1b (0% plastic strain), and these have been dissolved as seen by their absence in the same quadrant of Fig. 4b (9% plastic strain).



(a) (b)

Fig. 4: Morphology maps (aspect ratio vs. oblateness) of Cu-Cu features found in the Al-3Cu(-0.05Sn) (wt.%) alloy by the Core-Linkage algorithm after (a) 0% and (b) 9% plastic strain. These results were obtained from the data after the large θ' plates were removed (refer to text).

4. Discussion and Conclusions

APT has revealed spatially resolved (Fig. 1) and quantitative (Fig. 3) measures of the shear-induced dissolution of GP zones and the repartitioning of Cu from the zones into the solid solution in a model Al-Cu alloy initially having a bimodal microstructure containing small GP zones and large θ' plates. The results have also provided quantitative measures of the evolution in GP zone shape during deformation (Fig. 4).

This study is motivated by the need for a better understanding of the kinetics of the shear-induced precipitate dissolution in alloys subjected to straining. In the alloy considered in this study, the GP zone dissolution process has been correlated with an enhanced work hardening at large strains [3,4]. This is beneficial and may be responsible for the enhanced combinations of strength and elongation observed in alloys of this type containing such bimodal precipitate microstructures [4]. As reported elsewhere in these proceedings by da Costa Teixeira *et al.* [5], the initial work hardening rate of single-phase Al-Cu alloys is raised significantly by increasing the Cu concentration in solid solution. This has been interpreted in terms of a dependence of the dislocation junction strength (α in Taylor's equation) on the concentration of Cu in solid solution. The shear-induced dissolution of GP zones reported here gives rise to a concentration of Cu in solid solution that is strain-dependent. A key aspect that is not yet understood is the dependence of the plastic behaviour of the matrix on the heterogeneity of the Cu distribution in solid solution. In no case is the Cu solute distributed perfectly randomly, but there are different degrees of heterogeneity (both in terms of spatial and compositional fluctuations), and from the point of view of the effect on work hardening, it is not clear at what point the beneficial effect of Cu in solution on the work hardening rate is influenced by heterogeneity in the solute distribution. It is clear from the results presented in Fig. 3a that a significant difference exists between the number density of small Cu clusters (2 to 20 atoms) in the samples subjected to 3% and 9% plastic strain. There is a much larger number density of these Cu clusters in the latter case and a correspondingly smaller number density of singelton Cu atoms in the solid solution (Fig. 3b). The effect on plasticity, as a result of the solute heterogeneity at this scale is a question that APT has a role in helping to answer.

Acknowledgements

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