Laser-Induced Reversion of δ' precipitates in an Al-Li Alloy

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Atom probe tomography (APT) technique has been improved significantly, making it a well-established nano-analysis tool in the field of material science. It has been extensively applied to the investigation of different types of materials due to its ability to map the distribution of single atoms in a material in real space on a nearly atomic scale [1]. In this paper, we investigate the details of the laser pulse mode APT analyses, in particular the laser induced specimen-heating effect using an interface reaction in an Al-Li alloy as a model system. This alloy is known to have a low-temperature, metastable, miscibility gap [2]. It has been shown that under classical conditions of ageing of this alloy, including solution treatment, fast quench to room temperature and thermal ageing at intermediate temperatures (e.g., 100–2008C), the precipitation behavior was dominated by the presence of the metastable $\delta'(Al_3Li)$ phase with an L12 structure [3]. The influence of the laser power on the morphology, the composition, and the diffusion of $\delta'(Al_3Li)$ precipitates in the aluminum-lithium-based alloy is identified. A simple model is used to explain the observed experimental behavior and to estimate the corresponding tip-apex temperature for various laser energies. APT analyses were performed with both a CAMECA laser assisted wide angle tomographic atom probe (LAWATAP) for the field ion microscopy (FIM) mode and the CAMECA local electrode atom probe (LEAP 4000X HR). Data were acquired utilizing either the voltage- or the laser pulse mode. A diode-pumped (Nd: YAG) solid-state laser operating in the frequency tripled ultraviolet region with a wavelength of 355 nm, a pulse duration of approximately 12 ps and a repetition rate of 200 kHz was used. The laser pulse energy was systematically varied through the following values: 10, 30, 40, 50, 60, 80, and 100 pJ. The reconstruction algorithm used was the standard evolution algorithm [4].

Figure 1 shows APT analyses by using laser pulse mode with various laser pulse energies. These analyses were performed to monitor the effect of thermal processes induced by laser on the morphology and composition of the $\delta'(Al_3Li)$. The figure shows a series of reconstructed volumes of the tips analyzed by laser pulses at the following laser energies: 10, 30, 40, 50, 60, 80, and 100 pJ. From Figures 1a–1e, spherical precipitates in the microstructure are clearly visible in the range of laser energies from 10 to 60 pJ. Conversely, Figure 1f shows that the precipitates begin to lose their distinctive shapes at 80 pJ. At an even higher energy of 100 pJ, precipitates are no longer detected as individual particles (Fig. 1g). Moreover, some enriched Li regions can be seen in the top view of the reconstructed volume in Figure 1h. It is clear from this figure that the precipitates are no longer detected with their spherical morphology. Using the simple law of mass conservation and an Arrhenius-type relationship for the diffusion [5] together with the experimental APT measurements allowed us to obtain different numerical values for different parameters such as: the average diameter d of the precipitates, their number density Nv, their volume fraction f, their average Li compositions within the precipitates c_p, the Li composition of the matrix c_t , the diffusion lengths L , the effective diffusion coefficients D for the Li atoms and the corresponding temperatures T at each laser pulse energy. Comparing the proposed values of c_p, c_t, and T with the metastable miscibility gap of $\alpha + \delta'$ in the Al-Li system that was previously summarized by

[2] is shown in Figure 2. The measured data points are located nearly within the miscibility gap of the diagram. It is also clear that the average Li concentrations in the α and δ' precipitates follow the gap closure of the miscibility gap with the temperature as the laser pulse energy is increased up to 80 pJ; this behavior is supported by considering the rise in T due to the laser irradiation at the sample tip. The data at 10–80 pJ coincide with the miscibility gap, which is well below the critical temperature T_c. This experimental behavior indicates that the increase in temperature as the laser energy increases is sufficiently rapid to destabilize all of the precipitates present in the initial microstructure, leading to a supersaturated c_{Li} in the matrix. The results at a laser pulse energy of 100 pJ corresponds to full reversion, where no clear evidence of precipitation was detected (Figure 1). The corresponding temperature at 100 pJ was suggested to be in the range of T= 5533–540 K. This range of T is in agreement with the reported range of the solvus temperature of δ' in the Al-2.0 wt.% Li alloy.

In this study, atom probe tomography presented a series of snapshots during in-situ reversion of δ'(Al_3Li) initiated by laser irradiation, using different laser energies. In addition, the attempt shown in this study might provide a method to investigate real sample temperatures during laser-APT analyses using an interface reaction itself as a probe.

References:

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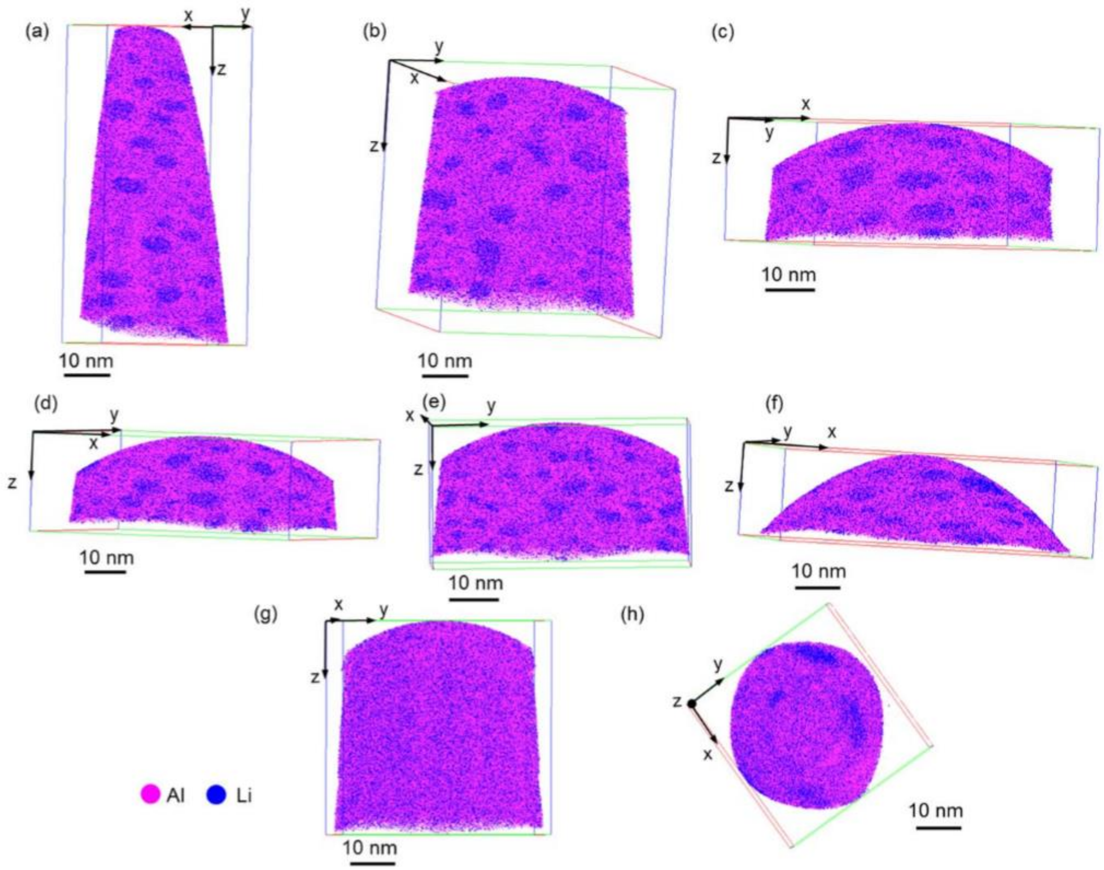

Figure 1. Comparison of the morphology of the δ' precipitates under illumination of the laser pulses at various values of energy E measured for different samples: (a) 10, (b) 30, (c) 40, (d) 50, (e) 60, (f) 80 and (g, h) 100 pJ.

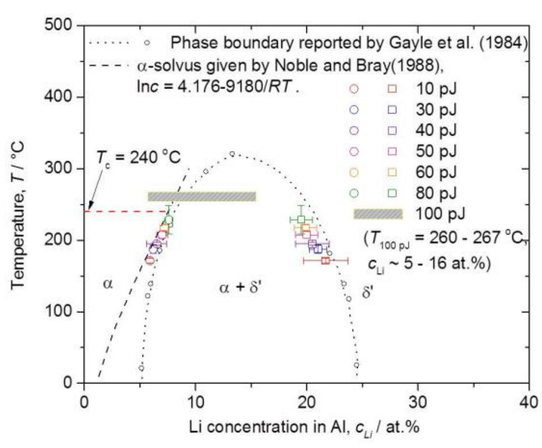

Figure 2. Comparison of the experimental Li concentrations and the estimated temperatures with an Al-Li phase diagram adapted from Ref [2]. The data obtained at E = 10 – 80 pJ agree with the miscibility gap, at well below the T_c. At 100 pJ, the tip temperature is estimated to be in the range of solvus temperature of δ' precipitates.