



New insights into plastic instability in precipitation strengthened Al–Li alloys

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Abstract—A mechanistic model that describes the microscopic mechanisms underlying plastic instability in precipitation strengthened Al–Li based alloy systems is proposed in this work. The model is based on experimental observations from high resolution nanoindentation tests and transmission electron microscopy (TEM) based methods, including *in situ* TEM tensile straining. These experiments show that dynamic strain aging (DSA), which is widely accepted as the underlying mechanism for plastic instability, cannot sufficiently account for the occurrence of plastic instability in Al–Li based alloy systems. It is proposed that an altogether different mechanism controls plastic instability, namely a diffusion-controlled pseudo-locking mechanism that accompanies order hardening. This mechanism does not require the concurrent operation of DSA by Li, which may be a nonviable mechanism given the low binding energy of Li to dislocation cores, for plastic instability to occur.
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1. Introduction

A number of technologically important alloys, including steels, Mg, Cu and Al-based alloys are known to exhibit discontinuous yielding during plastic deformation at critically low strain rates and over a range of temperatures [1–9]. This phenomenon, known as plastic instability or the Portevin–Le-Chatelier (PLC) effect, has been extensively researched in order to understand the underlying microscopic mechanisms that govern it. Such understanding is critical to the design of alloys and process routes that mitigate undesirable effects such as reduction in ductility and formation of surface striations associated with the phenomenon [1,9].

Several models, both phenomenological and theoretical, have been proposed to explain the origin of plastic instability in solution strengthened alloys [10–16]. Most of these models associate PLC-type plastic instability with dynamic strain aging (DSA), which refers to the solute aging of mobile dislocations temporarily trapped at localized obstacles such as forest dislocations. The increase in strength arising from this DSA process is claimed to govern negative strain rate sensitivity (nSRS), which is an anomalous behavior that is manifested macroscopically as serrations in stress–strain curves [17]. Yet recent modeling efforts have shown that this DSA cannot independently lead to nSRS

[18–20]. In view of this, Soare and Curtin [20] proposed a rate-dependent constitutive model for DSA in solution strengthened Al–Mg alloys that is not only predictive but also captures the physical mechanism associated with DSA. They show that solute aging of mobile and forest dislocations through cross-core diffusion must take place concurrently for nSRS to occur. Their model however suggests that since forest hardening is insignificant in precipitation strengthened alloys, plastic instability is not expected to occur in such materials unless a solute aging effect on the precipitate strengthening mechanism exists.

These theoretical models [18–20] question the widely accepted view that DSA by Li atoms alone governs plastic instability in precipitation strengthened Al–Li based alloys [21–25]. The plausibility of DSA by Li to activate nSRS in Al–Li alloys is further undermined by the rather weak binding energy of Li atoms to dislocation cores [26]. Others attribute the presence of instabilities in these alloys to the shearing of δ' precipitates [27–30]. This second view is largely predicated on the fact that these precipitates are shearable and are often present in Al–Li based alloys that exhibit plastic instabilities. However, there is still no convincing model based on precipitate shearing that gives a clear mechanistic description of the origin of plastic instability in these alloys.

In this work, a number of techniques, including nanoindentation, micro-tensile testing, TEM and *in situ* TEM straining have been employed to critically examine the role of precipitates in mitigating or enhancing plastic instability

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and, more specifically, to identify the microscopic mechanism(s) that govern the phenomenon in Al–Li based alloys. The alloy used in this study, is AA2198, a new generation Al–Cu–Li–Mg–Zr alloy with desirable combination of specific strength and damage tolerance, making it suitable for aerospace structural applications. The attractive properties of this alloy are due to the presence of several metastable strengthening precipitates such as GP zones, δ' (Al_3Li), T_1 (Al_2CuLi), θ' (Al_2Cu), Ω (Al_2Cu) and S' (Al_2CuMg). These metastable phases are known to transform to equilibrium phases such as θ (Al_2Cu), T_2 (Al_5CuLi_3) and T_B ($\text{Al}_7\text{Cu}_4\text{Li}$) after prolonged aging at elevated temperatures [31–35], leading to a decrease in strength.

2. Experimental procedure

The AA2198 alloy used for this investigation was received in the T351 state, i.e. stretched to 2% elongation followed by natural aging. This temper state hereafter referred to as NA temper, was used as a starting material for subsequent heat treatment. Two different artificial aging treatments, 155 °C for 14 h and 370 °C for 10 h which produced peak (PA) and overaged (OA) tempers respectively were carried. The composition range of the AA2198 alloy in at.% is given in Table 1.

Dog-bone shaped samples for microtensile testing with dimensions of $27 \times 3 \times 0.6$ mm and gauge length of 11 mm were produced by wire-cut electro-discharge machining (W-EDM) from the short-transverse (S-T) direction. The microtensile tests were conducted at a strain rate of 5×10^{-5} /s in a Zwick universal testing station equipped with a non-contact multi-zone laser extensometer. The laser extensometer has a resolution of 1 μm and thus facilitates the probing of the local strain distribution during straining in addition to the global strain response. The local and global strains were calculated from the displacements between markers placed 1 mm and 10 mm apart, respectively. Nanoindentation tests using two different loading profiles – constant strain rate (5×10^{-3} /s) and strain rate jump protocols, were also carried out on these tempers using a Nanoindenter XP (Agilent, GmbH) equipped with a Berkovich indenter. The strain rate jump tests were performed at three different indentation strain rates ranging from 0.01 to 0.0005 s^{-1} using a base strain rate of 0.05 s^{-1} . Details of the method used to conduct the nanoindentation strain rate jump test are given elsewhere [36].

3 mm diameter disks for TEM analysis and rectangular shaped foils for the *in situ* TEM tensile straining tests prepared from the samples were electropolished in a twin-jet device with a solution of 67% methanol and 33% nitric acid at a temperature of -20 °C and current of 12 V. A number of TEM lamellae were also produced by FIB milling in a Nova-200 dual-beam scanning electron microscope (SEM). The thickness of the FIB-milled lamellae was measured in the SEM prior to examination in the TEM. The TEM samples were examined in a JEOL 3010 microscope operated at 300 kV and a Philips CM200 microscope operated at 200 kV. The volume fraction of the δ' phase in the NA and OA tempers was estimated with the relationship prescribed by Cahn and Nutting [37]. The reported average volume fraction is the average of at least three different micrographs taken from different areas of the sample. The mean particle size of the δ' phase in each temper is

the average radius of over 180 particles. *In situ* TEM tensile straining was also carried out inside the JEOL 3010 microscope using a displacement controlled single tilt, straining specimen holder. The microscope is equipped with a Gatan Orius CCD camera that allowed the recording of the experiments at a video rate of 33 frames per second.

3. Microtensile and nanoindentation results

Fig. 1(a) shows the true stress, σ_t , vs. true strain, ϵ_t , response of the PA, NA and OA tempers from the microtensile experiments. Serrated flow indicative of plastic instability was observed in the OA temper but not in the PA and NA tempers. A small region of the plot is magnified in the insert in Fig. 1(a) to more clearly reveal the serrations. An important observation is that the NA temper sustained a higher plastic strain than the OA temper even though the yield strength of the former is more than a factor of 2 higher than the latter. This underscores the detrimental effect of plastic instability on ductility.

Fig. 1(b) shows a plot of local and global strains vs. measurement time for the OA temper, the former being taken from the mid-section of the sample with a gage length of 1 mm. The true stress response is also superimposed on the plot. Plastic instability, as captured by the stress and global strain response, was not very obvious in the early stage of plasticity. However, the local strain response, characterized by large stepwise increases in strain, reveals that the onset of the instability occurs shortly after yielding. This observation highlights the localized and microscopic nature of plastic instability.

In order to more carefully probe the microstructural influences on the serrated mechanical response and strain rate sensitivities, nanoindentation experiments were carried out; the high-resolution load–displacement capabilities of nanoindentation make it an effective tool for probing nanoscale perturbations such as plastic instability or strain localization [39]. Fig. 2(a) shows the nanoindentation response of the different tempers. The response observed in the micro-tensile test, specifically the absence of plastic instability in all but the OA temper, was also observed in the load vs. displacement curve. As clearly shown in the magnified insert in the figure, the instability however manifested as characteristic steps with large bursts of displacement; the inherently load controlled nature of the nanoindentation tests precludes the formation of load drops during deformation [17,40]. Fig. 2(b) presents a section of the nanoindentation curve obtained from the strain rate jump test. It shows the load vs. displacement response accompanying the change in nanoindentation strain rate from a very low strain rate, 0.0005/s, to the base strain rate, 0.05/s. In the case of the PA temper, the change from the low to high strain rate regime was accompanied by an upward deviation from the loading path. In contrast, a downward deviation from the loading path was observed in the OA temper upon changing the strain rate from the low to the high regime. On the other hand, the loading path was continuous in the NA temper, except for the transient response that usually accompanies a sudden change in strain rate during deformation. These trends are more clearly shown in Fig. 2(c), where the corresponding hardness response for the PA and OA tempers is shown. A significant increase in hardness accompanied the increase of the strain rate in the case of the PA

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