



# Temperature dependence of plastic instability in Al alloys: A nanoindentation study



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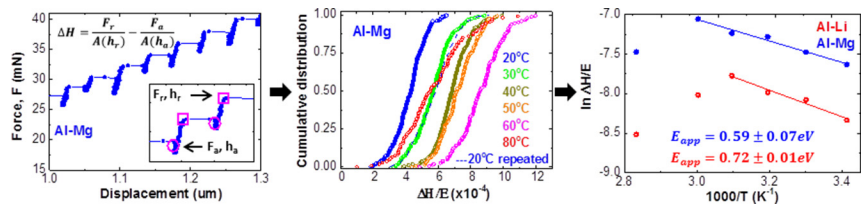
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## HIGHLIGHTS

- Plastic instability manifests as characteristic displacement bursts in nanoindentation load–displacement curves.
- The difference in hardness ( $\Delta H$ ) between consecutive displacement burst events is material and temperature dependent.
- The activation energy ( $\Delta E$ ) for plastic instability can be estimated from plots of  $\ln \Delta H$  vs.  $1/T$ .
- The  $\Delta E$  determined with the nanoindentation method are consistent with those derived from uniaxial tensile tests.

## GRAPHICAL ABSTRACT



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## ABSTRACT

An elevated temperature nanoindentation based method for characterizing the thermal dependence of plastic instability and assessing the activation energies associated with the phenomenon in Al alloys is presented in this work. The method exploits the nanoscale force–displacement resolution capabilities of the Nanoindenter, precludes the ambiguities inherent in the uniaxial testing based methods and offers increased reliability because of the statistical significance of the data achieved. The activation energies estimated for an Al–Mg and an Al–Li alloy with the proposed method were found to be  $0.59 \pm 0.07$  eV and  $0.72 \pm 0.01$  eV, respectively, and are consistent with values derived with other methods. The rate controlling mechanisms associated with these activation energies are described in terms of existing models for plastic instability in these alloy systems.

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## 1. Introduction

The term plastic instability, as used in this work, refers to repetitive yielding that occurs in certain alloys during plastic deformation at critically low strain rates and over a range of temperatures, typically above room temperature. This phenomenon, otherwise known as Portevin-Le Chatelier (PLC) effect represents a material instability that results in severe strain localization, reduction in ductility and formation of striations on the surfaces of sheet metals during forming processes. The

combination of these deleterious effects with the environmentally induced embrittlement that may occur during service further accelerates the failure of the materials susceptible to PLC-type plastic instability [1]. This has implications for metal sheet forming applications, particularly in the automotive and aerospace industries where Al-based alloys, driven by their excellent strength-to-weight ratios, are becoming increasingly important.

Various aspects of PLC-type plastic instability, particularly the influence of strain rate [2,3], temperature [4,5] and precipitation [6–9] have been investigated. Such investigations form the basis for the development of mechanistic, theoretical and numerical models to explain the underlying microscopic mechanisms that govern plastic instability in

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solution strengthened and precipitation strengthened Al alloys. It is widely accepted that dynamic strain aging (DSA), i.e., the aging of both temporarily trapped mobile dislocations and forest dislocations by solutes with sufficient mobility, governs plastic instability in solution strengthened alloys [10]. It is argued that the negative strain rate sensitivity (nSRS) observed in alloy systems that exhibit plastic instability arises because the stress required to liberate the strain-aged mobile dislocations from the solute-decorated forest dislocation junctions is higher than that required to keep the mobile dislocations in motion upon their release [11,12]. The amount of solute that diffuses to the trapped mobile dislocations and the associated 'break-free' stress are functions of the waiting time at the junctions between the trapped mobile dislocations and the dislocation forest. This in turn depends on the applied strain rate, temperature and the average dislocation density. The lower the strain rate, the longer the waiting time and the higher the 'break-free' stress. Conversely, higher temperature increases the solute diffusion rate and in turn leads to a higher 'break-free' stress [4,13]. While DSA effectively describes the origin of plastic instability in solution strengthened Al–Mg base alloys, it has been shown that it cannot sufficiently account for the phenomenon in precipitation strengthened Al–Li based alloy systems [6,14,15]. A diffusion-controlled locking mechanism arising from the relaxation of the antiphase boundary of the ordered  $\delta'$  ( $\text{Al}_3\text{Li}$ ) phase in Al–Li alloy systems during deformation at low strain rates was recently proposed as the mechanism governing PLC type plastic instability in Al–Li based alloys [6]. However, a quantitative treatment of the energetics required for such a mechanism has yet to be validated.

In order to gain further insights into the different rate controlling mechanisms in these two alloy systems, it is useful to probe the thermal dependence of the plastic instabilities. Such investigations provide insights into the local deformation mechanisms and facilitate the estimation of thermal activation parameters, such as activation volume and activation energy, associated with plastic instability. Different approaches [5,16–18] based on data from uniaxial tensile tests have been employed by various groups to arrive at these parameters. However, these approaches, as will be elaborated upon later, either have limited applicability, potential sources of error or are based on data lacking statistical significance. More so, the activation energies derived from these methods are usually less than those derived from theoretical and atomistic studies [11,13]. Yet it is not clear where the discrepancy lies.

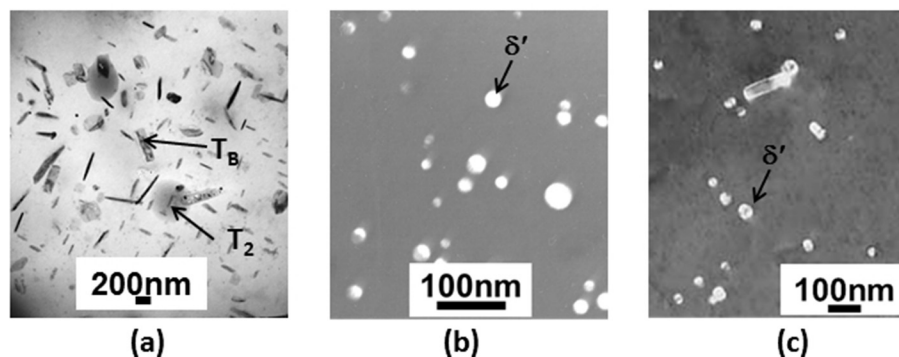
In this work, we present an elevated temperature nanoindentation test method for characterizing the thermal dependence of plastic instability and assessing the activation energy associated with the phenomenon in Al–Mg and Al–Li based alloys. Nanoindentation is particularly well suited for these kinds of investigations because of its high-resolution force–displacement capabilities, which makes it an effective tool for probing nanoscale perturbations such as plastic instability.

This work demonstrates the plausibility of the method, highlights the differences in plastic instability in these two alloy systems and extends upon the current state of mechanistic models for plastic instability more generally.

## 2. Materials and methods

Two aluminium alloys, a solid solution strengthened Al–Mg alloy, AA5182 and a precipitation strengthened Al–Li based alloy, AA2198, were used for this investigation. The Al–Mg based alloy had a nominal composition in wt% of 4.5% Mg, 0.2–0.5% Mn, 0.35% Fe, 0.25% Zn, 0.15% Cu and 0.15% Cr. It was received and used in the annealed and rolled state. The Al–Li based alloy had a nominal composition in wt% of 2.9–3.5% Cu, 0.8–1.1% Li, 0.25–0.8% Mg, 0.1–0.5% Ag, 0.04–0.18% Zr and 0.08% Si. This alloy was received in the naturally aged and stretched state and was then artificially aged at 370 °C for 10 h; a treatment which leads to an overaged temper state. A micrograph showing the key microstructural features obtained after this heat treatment is shown in Fig. 1(a). The microstructure mainly consist of the metastable  $\delta'$  ( $\text{Al}_3\text{Li}$ ) precipitates, which has an  $\text{L1}_2$  structure and is the main strengthening phase in this temper state, and equilibrium precipitates such as the  $\text{T}_2$  ( $\text{Al}_5\text{CuLi}_3$ ) and  $\text{T}_B$  ( $\text{Al}_7\text{Cu}_4\text{Li}$ ) phases. Further details of the heat treatment protocol and the resulting microstructure are published elsewhere [19]. Samples from both alloys were prepared for the nanoindentation tests from the short-transverse direction of the rolled sheets and were then mechanically ground and polished prior to testing.

A ZHN Universal Nanomechanical hardness tester (Zwick/ASMEC) was used to conduct the nanoindentation experiments. Unlike conventional nanoindentation systems, the force generation and force measurement systems in the ZHN nanoindenter are decoupled. The ability to record the force response in the material, in addition to the high force resolution (20nN) of this system, makes it possible to easily access the nanoscale force perturbations arising from the plastic instabilities. Further advantages of the decoupled force generation and measurement system will be highlighted later. The ZHN nanoindenter is integrated with a laser heating system (Surface, GmbH) that can simultaneously heat up both the sample and indenter tip up to 500 °C. The laser heating system significantly minimises thermal drift by confining the heat to the sample and tip. The system additionally incorporates a water-cooled and temperature controlled system to ensure mechanical and thermal stability. The indentations made with the ZHN system were performed in a quasi-static, displacement controlled mode at a nominally constant strain rate of 0.003/s and at temperatures between 20 and 80 °C. The samples were held at each temperature for 30 min to ensure thermal stability and to prevent thermal gradients prior to testing. At least 15 indents were conducted for each test temperature, and all were made on a single sample of each alloy with a sapphire Berkovich indenter to a depth of 3  $\mu\text{m}$ . The samples were re-



**Fig. 1.** (a) Bright field and (b) Dark field images, showing the precipitates in the artificially aged Al–Li (AA2198) alloy prior to testing at elevated temperatures. Several precipitates including  $\text{T}_2$  ( $\text{Al}_5\text{CuLi}_3$ ),  $\text{T}_B$  ( $\text{Al}_7\text{Cu}_4\text{Li}$ ) and the main strengthening phase,  $\delta'$  ( $\text{Al}_3\text{Li}$ ), are present in the grain interior of this temper state. (c) Dark field image of the alloy showing  $\delta'$  phase after all tests at elevated temperatures. Fig. 1(a) and (b) are taken from [19] with permission.

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