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Dynamic strain aging studied at the atomic scale

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Abstract—Dynamic strain aging arises from the interaction between solute atoms and matrix dislocations in strained metallic alloy. It initiates jerky dislocation motion and abrupt softening, causing negative strain rate sensitivity. This effect leads to instable flow phenomena at the macroscopic scale, appearing as a serrated stress–strain response and deformation banding. These macroscopic features are referred to as the Portevin–Le Chatelier effect (PLC). Here we study the atomistic origin of dynamic strain aging in an Al-4.8 at.% Mg alloy using atom probe tomography (APT) and transmission electron microscopy (TEM). Samples were prepared from as-cold rolled (90% thickness reduction), stabilized (120 °C, 20 h) and recrystallized sheets (400 °C, 10 min), respectively. In the stabilized state, Mg was found to decorate <1 1 0> aligned dislocations with up to ~12.5 at.%. Tensile tests in combination with thermographic and laser speckle observations were used to map the deformation bands for the site-specific extraction of APT samples from regions inside the PLC bands. We observed an asymmetrical Mg distribution along some of the dislocations, matching model predictions for high dislocation speeds at peak drag stress by Zhang and Curtin. In this case, the Mg distribution is characterized by depletion in the compressive regime above the dislocation slip plane and enrichment in the dilatation region below the slip plane. Mg also depletes in a tail-like form behind fast-moving dislocations, further promoting slip localization.

Keywords: Portevin–Le Chatelier effect; Atom probe tomography; Dynamic strain aging

1. Introduction

5xxx series Al–Mg alloys are widely used for automotive sheet-forming applications, where Mg is the main alloying element [1,2]. These alloys are mostly strengthened by solid-solution hardening since the Al_3Mg_2 (β phase) precipitates, formed above 5 wt.% Mg content, have no significant particle strengthening effect [1]. Upon deformation at room temperature, 5xxx Al alloys exhibit undesirable deformation traces on the as-formed sheet surface, which reduce the ductility of the material, create undesired optical effects and limit sheet-forming applications. The surface traces result from unstable plastic flow, entailing strain localization effects. Such phenomena become apparent upon straining as localized bands on the surface of tensile specimens and as serrations in stress–strain curves, known as the Portevin–Le Chatelier (PLC) effect [2–7].

The PLC effect is the macroscopic flow pattern that is associated with the phenomenon of dynamic strain aging (DSA), which occurs as a flow instability in a deforming alloy under specific conditions, such as the presence of solutes, medium strain rate and elevated temperatures, depending on the activation barriers for reorganization and the interaction between the solutes and the dislocations [6,7]. Several models have been suggested to explain DSA; however, the underlying atomistic interaction mechanisms between solutes and dislocations are still under debate [2–12]. All models agree on attributing the jerky dislocation motion and the resulting serrated flow pattern to a dynamical solute atom–dislocation interaction (SADI). In this perception, dislocation motion is assumed to be hindered by solute atoms that segregate to them. The dislocations are then assumed to be collectively released at a slightly higher resolved shear stress, resulting in negative stress/strain sensitivity, leading to flow instability, i.e. the PLC effect [5,13]. The understanding of DSA phenomena has improved over the years due to the introduction of advanced models that are based on considering SADI mechanisms at a more atomistic scale, also enabling the interpretation of temperature and strain rate effects [8,11]. Associated models dealing with the solute-drag effect have been developed [14–18], which also consider the spatial distribution of the solute atoms around mobile dislocations during deformation as a function of the dislocation velocity. Most of these models include specific assumptions about the distribution of the solutes around dislocations, and this motivated us to conduct a corresponding experimental analysis at the atomic scale. Among the various characterization techniques, atom probe tomography (APT) is one of the most suitable tools with which to analyze solute atom distributions in three dimensions with

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near-atomic resolution [19–28]. In some cases, the resolution of APT is even high enough to resolve lattice planes in crystalline samples, enabling identification of single dislocations under certain circumstances [21–25,29–31]. This method is referred to as atom probe crystallography [29–31]. Alternatively, APT coupled with transmission electron microscopy (TEM) can help to retrieve both structural and chemical information at comparable resolutions [32–35].

In the case of atom probe crystallography, the first task required for identifying dislocation vectors in atom probe data sets lies in revealing the sample's crystallography in the 3-D reconstruction [29–31]. Several methods have been suggested for the extraction of crystallographic information from atom probe data sets [22–24]. The centers of crystallographic poles are generally observed as low atomic density zones in the detector histogram and in the associated APT reconstruction. The crystallographic orientations can be determined by identifying at least two crystallographic poles in the reconstruction. The indices of each pole are then obtained by measuring their angular distances and lattice spacings [21]. For the current work, we implemented the method suggested by Moody et al. [25], which optimizes the use of the spatial distribution map technique suggested by Geiser et al. [23]. This method enables a full crystallographic analysis through the identification of multiple crystallographic directions within a single APT reconstruction.

Dislocations can be then detected in the so crystallographically characterized APT data set via two approaches, namely either by solute segregation, as discussed by Miller [26], or by the disturbance of lattice planes due to atomic displacements in the vicinity of the dislocation core, as presented by Blavette et al. [27].

Here, we study SADI in an Al–Mg alloy using the APT technique in conjunction with crystallographic reconstruction. Site-specific sample preparation is carried out from regions inside the PLC bands formed during the tensile tests. Atom probe analyses of deformed and undeformed samples are carried out and compared with each other. TEM is also used to study the underlying dislocation configurations.

2. Experimental procedure

An aluminum alloy with 4.8 at.% Mg content was cast using vacuum induction melting. The alloy was heated at 400 °C for 3 h to ensure homogenization, then cold-rolled to 90% engineering reduction, i.e. 1 mm thickness. The sheet was cut into three samples. The first one was heat treated at 120 °C for 20 h (stabilization annealing, referred to as H36 condition [1]). During this heat treatment dislocations align into low energy configurations forming a dislocation cell structure. The second sample was fully recrystallized at 400 °C for 10 min. The third sample was left in the as-cold-rolled condition, where dislocations form entangled networks. Tensile tests were conducted on all three types of samples (Fig. 1).

The tensile specimens had 50 mm gauge lengths and 5 mm × 1 mm cross-sections. They were exposed to a uniaxial tensile load at an initial strain rate of $1.67 \times 10^{-5}\,s^{-1}$. Two methods were used for imaging the PLC bands. The first method is a laser speckle technique (LST) [36], which uses a laser beam to illuminate the sample surface, imaging local strains via changes in the speckle pattern. A high-speed camera (FASTCAM 1024PCI) captured

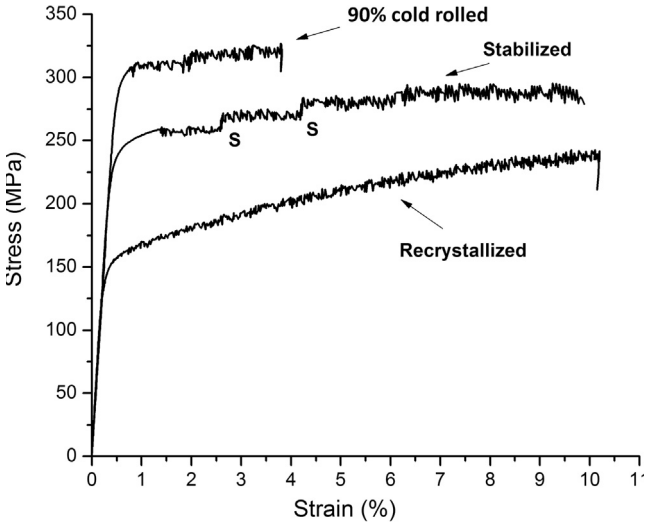

Fig. 1. Stress–strain curves of an Al-4.8 at.% Mg alloy (90% thickness reduction, cold rolled) in three conditions, i.e. recrystallized (400 °C, 10 min), stabilized (120 °C, 20 h) and as-cold rolled (reference state). Sudden stress shifts in the stabilized material are marked by S.

500 frames s^{-1} at a resolution of 128 × 512 pixels. The second method is imaging the bands in terms of temperature fields [37–39], where the dissipative deformation heat is mapped. The temperature variations range from a few hundredths to almost 0.5 °C. An infrared thermographic camera (VarioCAM HR inspect) with a sensitivity of 0.03 K and 50 frames s^{-1} at 640 × 480 pixels spatial resolution was chosen. Both methods were applied simultaneously during straining at room temperature (~23 °C).

The investigation of the Al–Mg alloy by APT was divided into two steps. The first one comprised the investigation of samples in the two heat-treated conditions. The second step consisted in probing regions directly inside the PLC bands. For this measurement, the tensile test was interrupted upon observation of a band. Site-specific atom probe and TEM specimens were then extracted from this PLC band by using a focused ion beam (FIB).

Atom probe samples from the stabilized condition were prepared by electropolishing [21]. For both the recrystallized state and the PLC band investigation, samples were prepared by a dual-beam FIB (FEI Helios NanoLab 600™). Site-specific sample preparation was conducted according to the lift-out protocol described in Ref. [40]. A 100 nm thick protective layer of platinum was deposited by FIB on the regions of interest (ROIs) before lift-out. FIB shaping of the specimens was performed at 30 kV ion beam voltage with a final milling step at 2 kV in order to control Ga implantation [40]. APT experiments were carried out using an Imago LEAP™ 3000X HR metrology system in voltage pulsed mode at 200 kHz repetition rate with a specimen temperature of about 30 K.

TEM investigations of the Al–Mg alloy were divided into two steps. The first set of experiments were carried out on a region inside a PLC band which had formed during a tensile test conducted on the recrystallized specimen. In a second set of experiments, we used the FIB-implanted Ga ions for decoration and, hence, identification of dislocation configurations. Since the Ga might affect defect configurations, we studied and compared dislocation structures before and after Ga implantation. This investigation was