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In situ evaluation of dynamic precipitation during plastic straining of an Al–Zn–Mg–Cu alloy

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Abstract

The coupling between precipitation and plasticity has been systematically investigated in an Al–Zn–Mg–Cu alloy using in situ small-angle X-ray scattering measurements during thermomechanical tests. Material pre-aged to two different initial precipitate conditions has been examined. Each pre-aged condition has been strained at 160 °C and we show that the plasticity induces an accelerated coarsening kinetics, which we characterize in terms of the evolution of the precipitate size. This acceleration is correlated with the degree of plastic strain, but does not depend markedly on strain rate. The experimental data strongly suggests that the accelerated kinetics is mainly linked with the accumulation of a supersaturation of vacancies during plastic flow that increases the effective diffusion constant.

Keywords: Aluminium alloys; Precipitation; Plasticity; Small-angle X-ray scattering

1. Introduction

Precipitation hardening is one of the most potent ways to increase the strength of aluminium alloys and is used extensively in transportation applications. Obtaining an optimized nanostructure of precipitates (i.e. a fine and dense distribution of shear-resistant phases) requires steps of phase nucleation and diffusion-controlled growth. These stages are both known to interact strongly with the presence of an applied stress and/or an applied strain through a wide variety of mechanisms.

The sequential influence of strain on precipitation is a well-charted area [1–10]. When a supersaturated solid solution is deformed and subsequently aged to form precipitates, the presence of dislocations due to the plastic straining promotes nucleation of more stable phases (as compared to an undeformed material that may nucleate more metastable phases) and tends to accelerate precipitate growth via pipe diffusion. In alloys where the homogeneous nucleation of the hardening-efficient phases is difficult, prior plastic strain can greatly enhance the hardening response. This is the case in the Al–Cu–Li or the Al–Cu–Mg systems [8,11]. However, when the phases that nucleate on dislocations are not the most efficient at hardening, prior plastic straining is usually observed to slightly decrease the hardening response or maintain it at a stable level, such as in the Al–Zn–Mg–Cu (7000 series) system [1].

When straining and precipitation occur together, the interaction is dynamic and thus more complicated. Elastic stresses can have an effect on precipitate nucleation (e.g. variant selection) for precipitates that have significant elastic misfit stresses with the matrix such as in the Al–Cu system [12]. The effect of plastic straining on precipitation involves a complicated balance between several possible mechanisms [13]: acceleration of nucleation due to the presence of dislocations [14,15]; acceleration of growth or coarsening due to pipe diffusion and solute transport by dislocation sweeping (solute collector) [16]; acceleration of nucleation, growth or coarsening due to a non-equilibrium vacancy concentration related to the plastic

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deformation [17]; and partial or complete dissolution of precipitates due to shearing by dislocations [18]. Given the variety of possible effects, it is not surprising that many different effects have been observed experimentally, such as accelerated nucleation [15,19], accelerated growth and coarsening [20,21], or precipitate dissolution [18]. It can be expected that the relative importance of the different interactions between precipitation and plastic strain depend on the conditions used for the precipitation process (supersaturation, temperature, precipitate morphology) as well as the characteristics of the imposed strain (strain, strain rate, temperature).

From an application point of view, many processes exist that imply such an interaction. One can find such interactions in processes that imply large strains that profoundly modify the whole microstructure such as friction stir welding [11] or severe plastic deformation [22,23]. More moderate strains applied during a precipitation process that can have a significant influence on the resulting microstructure are found in age-forming or creep-forming, [20,24–28] where the combination of strain and precipitation is used to minimize the level of internal stresses and to achieve a complicated part shape. Interactions between precipitation and strain are also important during a material's service lifetime such as during creep or fatigue.

Characterizing accurately the precipitation processes coupled with plastic straining is not straightforward, in part due to the presence of the dislocations in the microstructure that complicate electron microscopy observations. Small-angle X-ray scattering (SAXS), when performed on a system where a high contrast in electronic density exists between the precipitates and matrix, is generally sensitive to the precipitation state, since the influence of dislocations on the measured scattering is very weak. It is therefore a useful tool for microstructure quantification, and can be performed in situ during straining and heating, thereby making it possible to directly access the coupling between precipitation and plastic strain.

The present study is concerned with the AA7449 alloy, which is a classic alloy of the Al–Zn–Mg–Cu family for aerospace applications. This alloy can be subjected to the age-forming process in order to reduce the level of quench-induced internal stresses. In this process, plastic strain is applied at the temperature used for artificial ageing (120–160 °C). We have shown in a previous contribution [20] that under creep conditions of an initially fully aged material (T7651 temper), the plastic strain induced a significant acceleration of the precipitation kinetics. The present contribution will involve partially heat-treated materials (i.e. a residual supersaturation is still present), and more controlled straining conditions so that the interaction mechanisms can be precisely evaluated.

The precipitation state is monitored in situ during constant strain-rate experiments carried out at 160 °C using a dedicated heating and straining stage [29]. We will present the influence of strain rate and initial precipitation state on the precipitate evolution during the experiments.

2. Experimental methods

The specific composition of the received AA7449 plate, provided by Constellium—Centre de Recherches de Voreppe (France), was 8.3% Zn, 2.2% Mg and 1.9% Cu (all in wt.%). Prior to the thermomechanical heat treatments that are the subject of this paper, the material was subjected to the following sequence of heat treatments. It was first solution treated for 6 h at 474 °C, then quenched into cold water and naturally aged for 4 days. The material was then heated at 30 °C h^{-1} to 120 °C, aged 6 h at 120 °C and ramped at 15 °C h^{-1} to 160 °C. This stage corresponds to the first initial stage studied in this work. The second initial state was given an additional 10 h at 160 °C.

SAXS experiments were carried out at the European Synchrotron Research Facility (ESRF) on the BM02/D2AM beamline, at a wavelength of 1.3 Å. The accessible range of scattering vectors was [0.007, 0.4] $Å^{-1}$, and the beam diameter was 200 μm. CCD camera data was corrected for read-out noise, distortion, flat-field, background noise. It was normalized using a reference sample and transmission measurements through calibrated filters. The experiments were carried out with a specially designed tensile heating rig, allowing for in situ SAXS measurements in transmission through the sample during the tensile tests, as has been described in Ref. [29]. The tensile samples had a thickness of 150 ± 2 μm and the gauge length was 4 mm. The two cross-heads were moving symmetrically so as to ensure that the area covered by the X-ray beam (located in the middle of the gauge length) remained the same throughout the tensile test. The deformation was measured by an linear variable differential transducer sensor.

The average precipitate radius was determined using the detailed self-consistent procedure for Guinier radius determination presented in Ref. [30]. Given the presence of a precipitate size distribution and the moderate aspect ratio of the precipitate morphology (see transmission electron microscopy (TEM) images below), the conditions are chosen so that the measured Guinier radius and the average precipitate radius are equal. The precipitate volume fraction was calculated from the integrated intensity of the SAXS signal using the usual extrapolations outside the range of measured scattering vectors, and the same hypothesis on precipitate composition as in Ref. [31].

Specimens for TEM were prepared by mechanical grinding down to 100 μm followed by double-jet electropolishing in a nitric acid/methanol solution working at −20 °C, 15 V. Observations were carried out at 300 kV on a JEOL 3010 microscope.

3. Initial microstructures

We first present the microstructural evolution during strain-free ageing along the two-stage heat treatment at 120 and 160 °C that has been used to generate the initial states for the straining experiments. During this heat treatment, the evolution of the precipitate state was monitored