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Precipitation kinetics in a severely plastically deformed 7075 aluminium alloy

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Abstract

In this paper we report a quantitative study, using small-angle X-ray scattering, of the precipitation kinetics during ramp heating and isothermal ageing in an AA7075 aluminium alloy processed by high-pressure torsion. The precipitation behaviour has been compared with that of the same material processed in a conventional manner and observations are supplemented by transmission electron microscopy for precipitate and grain size characterization using automated crystal orientation mapping. After severe plastic deformation and natural ageing, the material is shown to contain a high density of GP zones. During ageing, the precipitate size distribution becomes bimodal, with small precipitates behaving similarly to those of the conventionally processed material and large ones associated with the crystalline defects and reaching large sizes at considerably lower temperatures and shorter times as compared to the conventionally processed material.

Keywords: Aluminium alloys; Severe plastic deformation; High pressure torsion; Precipitation; Small-angle X-ray scattering

1. Introduction

Severe plastic deformation (SPD) is now a well-established way to make materials with extremely small grain sizes and resulting high strength [1]. Using a great variety of processes (the most studied being equal-channel angular pressing (ECAP) [2] and high-pressure torsion (HPT) [3], alongside many others, such as accumulative roll bonding, cryorolling and multiaxial channel compression), grain size is reduced to a range between 100 and 500 nm, which results in materials with very high strength.

In aluminium alloys, the conventional way of achieving high strength is through fine-scale precipitation. Therefore it is not surprising that studying SPD in precipitation-strengthened aluminium alloys has attracted a large amount of interest in the last 10 years [4–31], with the aim of achieving combined strengthening between small grain sizes and precipitates (a yield strength of 1 GPa has been achieved in an Al–Zn–Mg–Cu alloy processed by HPT [32]) and increased stability in the submicron grain size by precipitate pinning [27].

Studying the combination of precipitation and SPD is a complex topic given the number of parameters that can be changed. Notwithstanding the variety of existing SPD processes, SPD can be carried out on a random solid solution (right after quenching from a solution treatment) or on a microstructure already containing precipitates. It can be carried out at cryogenic temperatures (note that only a limited number of processes allow for this), at room temperature or at temperatures where classical precipitation treatments are carried out (typically 100–200 °C). Note that the low temperature processes can usually only be carried out on solution-treated materials, otherwise specimen fracture during SPD is difficult to avoid. SPD can be followed by a subsequent ageing treatment, with or without

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intermediate solution treatment. And of course the results are alloy-dependent, with studies carried out on five alloy families: 7000 (Al–Zn–Mg–Cu) [4–9,15,24,27,29,31,32], 6000 (Al–Mg–Si) [10,14,17,23,25], Al–Cu-based 2000 [12,18,20,22,26], Al–Cu–Mg-based 2000 [28] and Al–Cu–Li-based 2000 [13,19,21].

Despite this complexity, some general rules can be summarized from the now relatively abundant literature:

- If precipitates are present before SPD, several phenomena can take place [13,14,16,18,20,22]: they can be progressively fragmented and even dissolve during the deformation process, resulting in a state close to a solid solution, or continue to precipitate while being deformed together with the matrix. These processes and the competition between them are highly temperature- and strain-dependent.
- The microstructure resulting from room temperature SPD carried out on solution-treated materials has not yet been fully characterized since the very fine grain structure prevents the observation of extremely small objects with conventional tools such as electron microscopy. Some papers invoke the absence of GP zones following room temperature SPD [6] while others evidence the presence of clusters or GP zones [28,29].
- If precipitates are not present initially and SPD is carried out at elevated temperature, accelerated dynamic precipitation occurs during SPD at a rate one or two orders of magnitude faster than corresponding precipitation in the absence of SPD [8,13,15,23–25,29].
- Precipitation following SPD occurs much faster than precipitation in the coarse-grained (CG) counterparts [7,10,19], and in many cases the intermediate metastable phases are skipped so that the equilibrium phase is formed at much lower temperatures than in conventional ageing treatments [12,18,22,24,26]. Among the reported cases, the extensive formation of θ phase in SPD Al–Cu during a few months at room temperature is particularly remarkable [12]. Many other reports exist on the formation of θ at the grain boundaries instead of the metastable θ phase during medium temperature ageing [18,22,26]. Other systems may, however, behave differently. No extensive η′ phase formation has been reported yet at room temperature following SPD on Al–Zn–Mg–Cu alloys, even though it is observed to form extensively at structural defects during elevated temperature ageing [29,31]. In AlCuLi alloys reports of θ′ and T_1 formation exist, similar to what is found in coarse-grained materials [19,21].
- In parallel to all these phenomena, the mechanical behaviour is usually followed by hardness measurements. After moderately large deformation processes (e.g. cryorolling), materials present a strengthening behaviour similar to that of conventional ageing, but accelerated to shorter times [7]. After SPD, however, the hardness is already very high (~200 HV for most precipitation-strengthened Al alloys) so that no strong further increase can be obtained. Depending on the post-SPD ageing temperature (from room temperature upwards) and the alloy family, in some cases an additional strengthening of ~50 HV is found (Al–Zn–Mg–Cu [4,5] and Al–Cu–Li [19]); in other cases the hardness remains stable and at higher temperatures hardness decreases continuously, because strengthening is reduced by precipitate coarsening, a concurrent reduction in defect density (dislocations, microstrains) and by grain growth [7,9,17,19,26,29,33]. Several authors have proposed strategies to optimize the combination of strength and ductility by acting on the SPD parameters and subsequent ageing treatment [11,21].

Several critical parameters are invoked when discussing the effect of SPD on precipitation, even though there has been a lack of quantitative understanding until now. Diffusion rates many orders of magnitude above the equilibrium ones need to be invoked to account for the rate of formation of the precipitates. This has been attributed to extremely high vacancy concentrations due to the SPD process [34,35], and concentrations of 10^{-5}–10^{-4} have been postulated [34]. However, such numbers are difficult to verify, since the positron annihilation technique, for instance, is sensitive in such materials to all present structural defects and proves to be difficult to interpret based on vacancy solute interactions alone [36]. Additionally, since in many cases precipitation occurs together with some grain growth, it has been postulated that grain boundary motion, sweeping solute from the matrix, may play a strong role in accelerating precipitation [12].

Until now, the study of precipitation phenomena in SPD materials has been almost exclusively carried out using transmission electron microscopy (TEM), with associated indirect techniques such as differential scanning calorimetry (DSC) and X-ray diffraction (DRX), and occasionally atom probe tomography (APT) observations. However, given the very small size of the crystallites present (~100 nm), the presence of high levels of microstrains and a high density of dislocations, characterizing in detail microstructural states including very small precipitates, proves to be extremely difficult [10], and the interpretation of the evolution of such precipitates with time or temperature remains necessarily qualitative, except for later stages of ageing where the objects become sufficiently large to be easily observed.

Small-angle X-ray scattering (SAXS) has been extensively used in precipitation-hardened Al alloys (particularly in 7000 and 2000 series) to obtain a quantitative measurement of precipitation microstructures, in terms of both size and volume fraction, and allowing for in situ measurements along isothermal or non-isothermal heat treatments [37,38]. Since this technique is sensitive to spatial variations of electron density (therefore of chemistry) within the sample, the signal is dominated by the precipitate microstructure, and the contribution of structural defects is usually negligible (except in low-contrast, low-volume fraction