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In situ evaluation of the microstructure evolution during rapid hardening of an Al–2.5Cu–1.5Mg (wt.%) alloy

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Abstract

The kinetics of the microstructural evolution during ageing at 200 °C of an Al–2.5%Cu–1.5%Mg (wt.%) alloy is evaluated using nuclear magnetic resonance and in situ small-angle X-ray scattering. This alloy is known to exhibit "rapid hardening" upon artificial ageing, where 50–70% of the total age hardening increment is reached within minutes at elevated temperatures. It is shown that formation of small solute-rich entities which correspond to Cu–Mg clusters or GPB zones occurs within seconds at 200 °C. These entities have radius ∼0.5 nm, volume fraction ∼2% and Cu concentration ∼30 at.%. Within the stage of constant yield strength (rapid hardening plateau) at this temperature, they are shown to be the dominant constituent of the precipitate microstructure and to be extremely stable in terms of size and volume fraction. The S phase is observed to nucleate within the hardening plateau, but remains a minor constituent until the end of this stage. Based on the quantitative measurement of their size and volume fraction, the obstacle strength of these solute-rich clusters is calculated to be about one-tenth of the Orowan strength. The energy required to shear these clusters (∼0.5 eV) is shown to be compatible with existing atomistic calculations of similar objects.

Keywords: Al–Cu–Mg; Precipitation; Nuclear magnetic resonance; Small-angle X-ray scattering

1. Introduction

Precipitation hardenable aluminium (Al) alloys find widespread use in transport industries because of their desirable combinations of formability, environmental resistance and specific mechanical properties. The 2xxx (Al–Cu and Al–Cu–Mg based) and 7xxx (Al–Zn–Mg based) alloys in particular are used extensively by the aerospace industry. The properties exhibited by these materials are sensitive functions of the precipitation state (type of precipitate, size, shape, volume fraction, nature of interaction with dislocations). The precipitation process itself, as well as the relationship between the precipitate state and the resulting mechanical properties have been topics of continual and intense study almost since the discovery of precipitation hardening [1]. Precipitation occurs by the nucleation, growth and subsequent coarsening of precipitates [2], often through a series of metastable transition phases. Typically, the yield strength progressively increases, reaches a maximum and then decreases with this evolution of the precipitate state. In a general sense, both the precipitation process [2] and the principles of hardening [3–5] have now reached a good level of understanding and maturity, and theoretical models that work relatively well for most purposes are available for both the precipitation process and the resulting yield strength, e.g. [6–15].

However, there are certain Al–Cu–Mg-based alloys whose hardening response to a precipitation heat treatment is sufficiently unusual to continue to attract the interest of researchers. An example of the yield strength evolution of a

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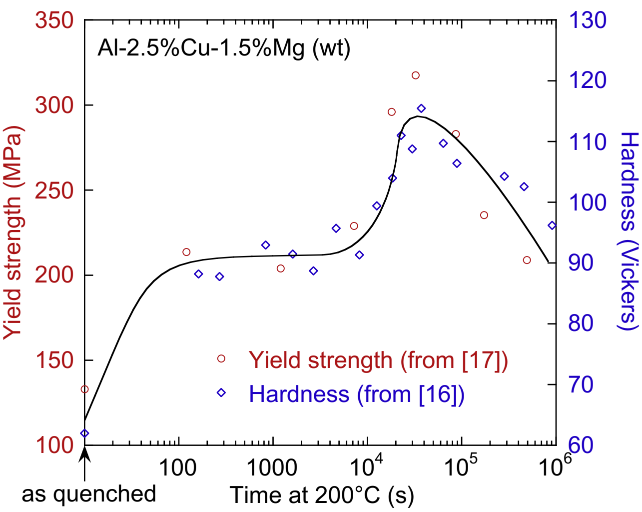

Fig. 1. Evolution of hardness (from Ref. [16]) and yield strength (from Ref. [17]) in an Al–2.5Cu–1.5 Mg (wt.%) alloy as a function of ageing time at 200 °C.

Al–2.5Cu–1.5 Mg (wt.%) model alloy as a function of ageing time at 200 °C after solution treatment and water quenching is shown in Fig. 1 [16,17].

This alloy is a model version of the commercial 2618 alloy (Al–2.3Cu–1.5 Mg–0.2Si–1.1Fe–1.1Ni (wt.%)). There are two distinguishing features of this hardening response. The first is the very rapid increase in strength that occurs within 2 min ageing at elevated temperatures after the solution treatment [18]. As can be seen in Fig. 1, the yield strength after quenching from the solution treatment temperature is 130 MPa, and after 2 min at 200 °C it rises to 220 MPa. This increase is ~50% of the maximum hardening increment achievable at 200 °C and, at lower heat treatment temperatures, this rapid hardening increment can rise to up to 70% of the total hardening increment [16]. Part of the interest in the origin of the rapid hardening increment is that such short ageing times must necessitate limited mass transfer, and the obstacles to dislocation movement responsible for the hardening increment must be very small. The second distinguishing feature of the precipitation hardening response shown in Fig. 1 is the extended yield strength plateau that exists after rapid hardening (from ~2 min) until the second rise to peak hardness (at 200 °C after ~2 h). Similar behaviour is exhibited at lower ageing temperatures (e.g. at 150 °C the plateau extends for 100 h [16]). It would normally be expected that with prolonged ageing the state of precipitation would evolve and, as a result, changes in the resulting yield strength would be observed. This is not the case in this class of alloys.

Motivated by these observations a number of researchers have sought to clarify the sequence of precipitation that accompanies the evolution of yield strength shown in Fig. 1. Bagaryatsky [19] and Silcock [20] were the first to investigate the phase development and, using X-ray diffraction, proposed that, initially, zones consisting of Cu and Mg atoms are formed as rods ~1–2 nm in diameter and 4–8 nm long. A number of different structures for the zones, which are now known as Guinier–Preston–Bagaryatsky (GPB) zones, have been proposed [19,20], but debate remains about their exact structure, and this is an area of current research [21]. Silcock proposed that the second rise to peak hardness (~9 h in Fig. 1) is due to the formation of the equilibrium S (Al_2CuMg) phase [20], and other authors (but not all) concur with this conclusion [22]. With the advent of more and more powerful characterisation tools, the microstructural evolution during and after the rapid hardening has been studied in increasing detail. Using high-resolution transmission electron microscopy (TEM), Ringer and co-workers [23–25] were unable to detect any GPB zones at the short times corresponding to the rapid hardening response in the same alloy composition shown in Fig. 1. Instead, using the atom probe, it was proposed that the initial hardening was due to the formation of Cu- and Mg-containing atomic clusters, and it was suggested that this represented a new form of strengthening (a type of super solid solution strengthening) which was termed “cluster strengthening”. Ringer proposed that GPB zones form from the clusters only at later times in the plateau [23]. The clusters were regarded as being distinct from zones in that “their shape, size, composition, degree of order, orientation and structure, are less defined than a zone” [24]. Such an interpretation of the origin of the rapid hardening response shown in Fig. 1 has not been without controversy [26–29].

It is perhaps not surprising that different interpretations of the microstructural evolution (and as a result the origin of the rapid hardening response) exist. There are a number of different microstructural features that have been identified (Cu–Mg atomic clusters, GPB zones, S phases and its proposed metastable transitional forms) and the relative importance of each is not easily identified. Part of the difficulty is that neither the clusters nor the GPB zones have been precisely defined, the features are extremely small and near the limit of resolution of existing characterisation techniques, the progression of microstructural evolution is inferred from “snapshots” at different stages along the age hardening curve (often in different alloy compositions), and since the nature of the microstructural features appearing at short ageing times is not well known, quantitative testing of whether such features can account for the observed hardening response is not straightforward. One recent exception that does attempt to test the hardening response expected from assumed microstructural features is due to Starink and Wang [30], who use a modulus strengthening model applied to Cu:Mg clusters to rationalise the rapid hardening response observed. The difficulty is that the “effective” modulus of the clusters is not known, and Starink and Wang use this as a fitting parameter to obtain agreement with the experimentally observed strengthening increment.

To clarify the origins of the rapid hardening behaviour in Al–Cu–Mg-based alloys and the extended plateau that is observed in Fig. 1, an in situ characterisation of the