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Improving the ductility of nanostructured Al alloy using strongly textured nano-laminated structure combined with nano-precipitates

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ABSTRACT

A ductile nanostructured Al alloy with desired microstructure is tailored via the precipitation optimization and texture design. The microstructure consists of two-dimensional nano-laminated structure with strong texture and one-dimensional elongated sub-micron structure with intra-precipitations at nanoscale and sub-nanoscale level. The precipitation optimization is realized by the appropriate introduction of its embryo in the NS matrix before aging. The embryo is the solute-rich structures (solute clusters) segregated to dislocations due to strain-induced dissolution of initial precipitations into the matrix. These nano-precipitations not only impede dislocation motion but also accommodate plastic deformation during loading. The textured laminate activates the partial dislocation mechanism in high stacking-fault energy alloys and the extrinsic toughening mechanism during loading. In addition, the laminated structure exhibits significant texture strengthening. Therefore, both nano-precipitations and textured laminated structure are responsible for the simultaneous increase in strength and ductility. Additionally, the initial deformable precipitations play a positive role in microstructural refinement and the refined structure shows significant cluster strengthening, ~20% relative contribution to total strength. This paper provides a new understanding of designing nanostructured materials for achieving high ductility by several toughening strategies, which is expected to be applicable to other age-hardenable alloys and steels as well.

1. Introduction

Nanostructured (NS) materials exhibit extraordinary strength but low ductility, because of their low dislocation accumulation capability. Dislocations emit from one grain-boundary (GB) segment and disappear at another, leaving no dislocations to accumulate inside the grain interior [1,2]. This is confirmed by molecular dynamics (MD) simulations [3–5] and in-situ transmission electron microscopy (TEM) experiments [6,7] in the high stacking-fault energy (SFE) materials, e.g. Al. The introduction of barriers into the NS matrix is one potential approach to simultaneously achieve high strength and ductility [8–16]. However, it is still a challenge to obtain nano-precipitates, which act as the barrier, within NS matrix for the age-hardenable alloys. In the NS Al–Cu alloys produced by severe plastic deformation (SPD), the high-energy GBs absorb vacancies from the grain interior during aging; due to lack of vacancies that assist precipitation nucleation, precipitates tend to nucleate at GBs, which would worsen the properties [17]. If certain structures, i.e. the embryo structures of precipitate, are produced in the NS grain interior before aging, intra-precipitates could develop in NS materials. This has been experimentally proved in NS Mo alloys by introducing a core-shell structure (precipitate embryo) before sintering [14]. Therefore, the way of producing embryo of precipitations is expected to overcome the challenge; however it is still not well understood in NS materials produced by SPD.

It is reported that the embryo should have similar chemical compositions or crystal structure or both with the precipitates. In Al alloy, Guinier-Preston (GP) or Guinier-Preston-Bagaryatsky (GPB) zones or solute clusters satisfy this requirement. It is easy to produce solute clusters or solute-enriched zones during deformation. For instance, carbon-enriched zones which may serve as the embryo of other carbides during annealing are observed in heavily cold-drawn pearlitic steel using atom probe tomography [18,19]. Therefore, the embryo can be realized by the re-

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dissolution of pre-existing second phase into matrix and solute segregation to dislocations during deformation. However, the optimization of embryo is of particular importance role for final precipitations and its mechanical properties, which is remained relatively unexplored. The deformation-induced dissolution of second phases is also founded in Al–Cu alloys [20–22] and Al–Cu–Mg alloy [23]. Such second phases can be classified into two kinds: the deformable particle like θ′ phase in Al–Cu alloys [20–22] and the non-deformable one such as θ phase in Al–Cu alloy [20] and cementite in Fe–C alloys [18,19]. Compared with un-deformable counterpart, the deformable particle dissolves more rapidly and therefore favors the formation of embryo. Interestingly, these un-deformable particles play a positive role in microstructural refinement during deformation [18,19,24]. Similar results are found in un-deformable T-phase [13,25]. On the other hand, whether the deformable particles have similar behavior during deformation? This, if proved true, may further enhance both the strength and ductility due to the existence of precipitate and microstructural refinement.

On the other hand, the transformation of deformation mechanism also plays a critical role in ductility enhancement, in particular, in high SFE metals. For instance, a deformation mechanism, via partial dislocation emission from GBs and leaving SFs/twins within the grain, is found in nano-grained Al [26], whereas {111} < 112 > preferably oriented in coarse-grained (CG) Al [27]. In fact, twins/SFs nucleate when the grain size reduces to a critical value, e.g. 10–15 nm in pure Al [28]. In this case, however, GB-mediated processes can become dominant [29]. Can the partial dislocation mechanism take action to improve ductility in textured NS materials? A laminated structure that severely limits GB processes makes it possible. Recently, the laminated structure in Ni with strong texture shows record hardness of 6.4 GPa [30]. Also, this structure in Ti is reported to exhibit ultrafine-grain strength and CG ductility [31]. Therefore, the textured laminated structure at the nanoscale is expected to achieve an excellent combination of strength and ductility. Simultaneously, the laminated (layered) structure is reported to activate deformation mechanism e.g. non-localized fracture [32] and delamination mechanism [33], which can cause improvement in ductility. Thus, such laminated structure with the preferred orientation is expected to achieve high ductility in NS materials using several toughening mechanisms above.

The main objective of this study is to develop a procedure for achieving high ductility in NS Al2024 alloy by the precipitation optimization and texture design. The precipitation optimization is realized by the appropriate introduction of its embryo in the NS matrix. The textured laminated structure operates several toughening mechanisms and also exhibits texture strengthening. The results provide the new understanding of designing NS materials possessing high ductility, which could have important applications in the age-hardenable alloys and steels.

2. Experimental

The composition of the Al2024 alloy used in this study was Al-4.3Cu-1.5Mg-0.6Mn-0.5Fe-0.5Si-0.3Zn (wt%). Al sheets 1.0 mm thick were subjected to solid solution treatment (SST) at 768 K for 10 h and then immediately quenched into water. Parts of SST samples were subsequently aged at 433 K for three times (10 h, 23 h and 43 h) to obtain precipitates with different features. The rest of SST sample and three kinds of aged samples were cold-rolled (CR) at room temperature (RT) with 80% thickness reduction, thereafter designated as SST-CR, 10-CR, 23-CR and 43-CR, respectively. It is noted that only $<5\%$ reduction was done for each rolling pass to avoid split [34]. Three directions were referred as normal direction (ND), rolling direction (RD) and transverse direction (TD), respectively. Subsequently, re-aging was carried out at 373 K or 433 K for up to 150 h. These samples re-aged at 373 K for 100 h were designated as SST-CR-, 10-CR-, 23-CR- and 43-CR-100, respectively. (The numbers after the "CR" represented re-aging time, similarly hereinafter. The number <10 corresponded to the sample re-aged at 433 K, or it represented the sample re-aged at 373 K.) Vickers microhardness of samples was measured on the RD-TD plane using a FM800 micro-hardness tester to track the evolution of age hardening. A load of 200 gf was applied for a dwell time of 15 s. At least 15 points were taken for each sample. Room temperature tensile tests were performed using an Instron 5565 universal testing machine along RD at a strain rate of $5\times10^{-4}\,s^{-1}$. All tensile specimens were dog-bone shaped, with a gauge length of 15 mm and a width of 4 mm. The length met the minimum size requirement of tensile test; the choice of width provided convenience for TEM and X-ray diffraction (XRD) experiments after fracture. At least two specimens of each condition were tested to verify reproducibility. The XRD patterns were obtained using a Rigaku D/max2500PC diffractometer with Cu Kα radiation at the voltage of 40 kV and current of 200 mA. Two scanning speeds of 6 and 0.6°/min were used for the conventional analysis and careful observation of peak broadening, respectively. The dislocation density of the gauge sections of tensile specimens was measured using the XRD peak broadening before and after tensile tests [12]. After fracture, the surface morphology was observed by a JSM-6010 scanning electron microscopy (SEM). Microstructures were characterized using a JEOL 2100(F) TEM equipped with a high-angle annular dark field (HAADF) detector and a Zeiss Ultra 55 field emission SEM with electron backscatter diffraction (EBSD) detector. After mechanically grinding, samples for TEM and EBSD analyses were prepared using a Gatan Dual Ion Milling System with an Ar+.

3. Results

3.1. Mechanical properties

Fig. 1 displays the Vickers microhardness of rolled Al2024 alloy subjected to different aging treatments. At a re-aging temperature of 433 K, all CR samples show similar trend with aging time (Fig. 1a): hardness decreases after reaching its peak. The SST-CR sample reaches its peak hardness of ~200 after 8 h aging, for instance, and further aging leads to a decrease in hardness. However, the peak shifts to shorter aging times for other aged samples (10-, 23- and 43-CR), leading to a sharp drop in hardness, especially for 43-CR sample. Interestingly, other aged samples show similar peak hardness values (~205), which is higher than that of SST-CR sample. At lowering aging temperature of 373 K, hardness slightly increases initially and then remains stable for SST-, 23- and 43-CR samples, while the 10-CR sample nearly exhibits a linear increase in hardness for up to 150 h (Fig. 1b). Rolling hardens the material; subsequent re-aging leads to a further increase in hardness for CR samples at both the studied temperatures, especially for the three aged samples at lower temperature; 10-CR and its re-aged samples display higher hardness values than other three samples. For example, the hardness of 10-CR sample is ~15% higher than that of SST-CR sample after re-aging at 373 K for 150 h. Therefore, the CG state of 10-CR sample is chosen as the control sample for comparison.

Based on the hardness results, tensile tests are performed on CR samples and their re-aged samples having the peak hardness (Fig. 1) to better understand the mechanical performance. Tensile results are shown in Fig. 2. The control sample shows a tensile strength of ~455 MPa and an elongation to failure of ~11.8%.