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Long-term thermal stability of Equal Channel Angular Pressed 2024 aluminum alloy

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ABSTRACT

The strength of bulk metallic materials can be improved by creating ultra-fine grained structure via severe plastic deformation (SPD). However, the thermal stability of severely deformed materials has been a major issue that restricts their practical use within the industry. Although there are studies on the thermal stability of SPD metals, the long-term annealing response of particularly complex alloys, such as the age hardenable ones, remains undetermined. In the present study, annealing behavior of the single-pass Equal Channel Angular Pressed age hardenable 2024 Al alloy was investigated in the 0.38–0.52 homologous temperature range for up to 1000 h. Microstructures and the corresponding mechanical properties of the samples were determined by transmission electron microscopy, electron back-scatter diffraction analyses, and micro-hardness measurements. After long annealing durations at 80 °C and 120 °C, a secondary hardening was observed whereas a fast softening occurred at 200 °C. At 150 °C, however, a softening followed by a slight secondary hardening was also detected. The increased coarsening rate of S precipitates accompanied with dislocation annihilation was found to be the major cause of the hardness loss. Furthermore, dislocation-rich structure and Mg clusters remaining from the S precipitate dissolution eased the nucleation of Ω precipitates which are responsible for the secondary hardening. It was concluded that below 120 °C the single pass ECAPed Al 2024 components preserve their improved hardness for a prolonged period of time.

1. Introduction

Equal Channel Angular Pressing (ECAP) and high-pressure torsion (HPT) are two promising techniques applied for over 20 years to enhance mechanical properties by attaining ultra-fine grain structure [1,2]. ECAP has been accepted to be more advantageous as compared to HPT when sample size and homogeneity of the structure are considered [3,4]. The application of SPD for age-hardenable alloys requires additional optimization efforts because of the complex dual interactions between plastically deformed structures and the precipitation process. For this reason, in some studies, the age hardening effect was eliminated by furnace cooling or aging prior to ECAP [5,6]. Yet, in other studies the approach was modified to apply solutionizing heat treatment before ECAP with the subsequent aging process [7–9]. In the study by Kim et. al., aging after single pass ECAP accompanied with pre-ECAP solutionizing heat treatment worked effectively to further strengthen 2024 Al alloy [10]. In another study, post-homogenization of the structure due to post-ECAP aging was observed [11].

The high internal energy of severely deformed metals makes the microstructure very prone to alterations which may lead to instability even at low homologous temperatures of 0.3. Such a response was observed in the multi-pass ECAPed pure Cu as a result of discontinuous recrystallization, and lower thermal stability was reported in comparison to the 87% cold-rolled Cu [12]. The thermal stability in severely deformed products was suggested to be enhanced by proper alloying through the drag effect of precipitates, dispersoids or solute segregation of larger atoms to grain boundaries so that the grain coarsening could be suppressed [13]. In a study on alloying effect, 2 h-annealing response of Al-Li alloys after equal-channel angular extrusion or hydrostatic extrusion has been examined, and improvement of the thermal stability with increasing solute content has been observed [14]. 1 h-annealing of the ECAPed 3103 and 1200 Al alloys within the 130–330 °C range showed that the thermal stability could be enhanced by the pinning effect of dispersoids on grain boundaries [15,16]. However, it is very difficult to generalize the effect of precipitates

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on the thermal stability of severely deformed age hardenable Al alloys because of the drastic increase in aging kinetics [3,11,17]. It has been reported that increased kinetics causes a reduction in strength due to early over aging of Al-4%Cu alloy at room temperature after 5 turn HPT [3]. On the contrary, the thermal stability of 8-pass ECAPed Al-0.1%Sc alloy was enhanced after 250 °C/30 h aging due to the Zener pinning of precipitates at grain boundaries [18].

The relationship between the number of ECAP passes and thermal stability is not well established in previous studies. Nevertheless, it has been reported that progressively increasing the number of ECAP passes improves the microstructural stability in terms of grain size and hardness of the ECAPed Al 3103 sheets after annealing at 300–350 °C [19]. On the contrary, in another study, it has been reported that the increased number of ECAP passes reduces the thermal stability of Cu while the addition of a small amount of Zr improved it through precipitate formation [20]. Therefore, there has been an inconclusive debate about whether increasing the number of ECAP passes improves the structural stability or not.

The thermal responses of severely deformed metals have been mostly investigated after short annealing periods. Consequently, they may not represent the actual industrial conditions [3,5,12,14,15,19,21–25]. Significant fluctuations in microhardness values were observed for one turn HPT applied Al and Cu samples after self-annealing at room temperature up to 100 h [23]. Heating of the 6-pass ECAPed 6082 Al alloy samples caused an increase in hardness attributed to the continuous precipitation below 200 °C. Above 200 °C discontinuous recrystallization was reported to initiate in an hour [6]. A sharp hardness drop was observed in Al-Cu-Mg-Si alloy samples processed by post-HPT annealing at 200 °C for a period of 0.5 h [26]. DSC analysis of ECAPed 7075 Al alloy with a rate of 5 °C/min showed that recrystallization of the structure started at 228 °C and ended at 300 °C [18]. In summary, annealing of the severely deformed age-hardenable aluminum alloys below 200 °C may cause early over-aging [3], double-peak age hardening [27], recovery [24] and recrystallization [18,22] as a function of annealing time.

Among one of the few studies on 2024 Al alloys reported that near 200 °C grain growth initiates in one hour, and grain growth rate increases above 200 °C [22]. In the extensive thermal stability study focusing on the grain size stability, it has been concluded that ECAPed 2024 and 7075 Al alloys are thermally stable up to 300 °C [5].

Studies representing the effect of fast aging kinetics on the thermal stability of the solutionized-quenched-ECAPed Al alloys are still lacking. Long-term thermal stability data exists for some commercial Al alloys up to 10,000 h such as 2124 Al alloy in T851 condition that is solutionized, cold worked and artificially aged [28]. A similar approach is essential in ECAPed age-hardenable alloys.

The objective of this study is to investigate the long-term thermal response (up to 1000 h) of ECAPed 2024 Al alloy in the peak aged, and in the bare ECAPed conditions through annealing within the 0.38–0.52 homologous temperature range. It was discovered that there are two different behaviors depending on the aging temperature. A secondary hardening occurred due to omega precipitation at and below 150 °C, and a hardness loss at and above 150 °C due to precipitate coarsening and annealing of the deformed structure. The detailed investigation of the annealing response of the alloy at 150 °C was carried out using TEM and EBSD analysis. The mechanisms of hardness loss and omega precipitation were discussed, and a thermally stable utilization range was proposed.

2. Experimental

Commercial 2024 Al alloy was received in the rod form. Following the solutionizing heat treatment at 495 °C for 1 h all samples were quenched in the ice-salt-water mixture at 0 °C, and then kept at −25 °C before the ECAP process. The samples were ECAPed through a 120° die. The details of this process can be found in the previous study [11]. The sample ECAPed with low back pressure was chosen in order to obtain better thermal stability [29]. A single-pass ECAPed 2024 Al alloy rod was carefully sectioned and two sets were prepared: 1) bare ECAPed, and 2) ECAPed and peak aged at 190 °C for 1 h. The microhardness variations were measured and compared. The lowest annealing temperature was chosen as 80 °C considering the lowest aging temperature found in previous experiments and the upper limit was selected to be 200 °C just above the optimum aging temperature. Two more additional temperatures within this range, specifically 120 °C and 150 °C, were also chosen to monitor the corresponding trend with respect to aging temperature.

The ECAPed sample of 18 mm in diameter and 50 mm in length was cut into slices along the shear angle (~46°) lines as shown in Fig. 1. For each temperature, 200 μm thick slice for TEM analysis and a 50 mm thick slice for micro-hardness measurement were used. The annealing periods were selected as 6, 12, 24, 48 and 168 h in order to observe the precipitation sequence as clearly as possible. The process was then continued with further interruptions until 1000 h. At each interruption, the samples were quenched into water, and then, microhardness measurements were conducted using a scaled map of 15 equal regions as in Fig. 1 and the representative 3 mm diameter specimens were punched for TEM and EBSD investigations.

TEM samples were prepared by conventional dimpling and electro-polishing in an acid solution of 25% nitric acid with 75% methanol at −25 °C. 3 mm diameter samples were ground and electro-polished under the similar conditions for EBSD analysis.

3. Results

3.1. Micro-hardness response

The micro-hardness variations of the ECAPed samples during 1000-h annealing at various temperatures are plotted in Fig. 2(a). At 200 °C, the hardness of the ECAPed sample continually decreases down to 100 HV. When the annealing temperature is 150 °C, peak aging proceeds between 0 and 48 h followed by overaging. The steady decrease in hardness, just as at 200 °C, continues until 450 h of annealing. Interestingly, the hardness drop is interrupted by a pseudo hardening peak at 676 h and the sample maintains its hardness at about 150 HV afterward. Unlike the hardness drop at 150 °C and 200 °C when the annealing temperature is further decreased to 120 °C, a continuous hardening is observed. At 120 °C, the initial hardening was observed after 168 h annealing from 165 to 180 HV followed by a secondary hardening peak between 336 and 1000 h with its maximum at 586 h. The trend for 80 °C annealing is very similar to the one at 120 °C. The hardness increases slightly in the first 336 h from 162 to 170 HV. The secondary hardening event occurred between 336 and 1000 h which raised the hardness up to 195 HV.

The optimized peak aging condition of ECAPed 2024 Al alloy was determined to be 1 h at 190 °C in our previous study [30]. In Fig. 2(b), the micro-hardness variations of ECAPed 2024 Al alloy samples after peak aging are presented. The standard deviations of hardness measurements are slightly less than the ECAPed counterparts shown in Fig. 1(a) due to the homogenization effect of the primary peak aging process. The trend in microhardness change is