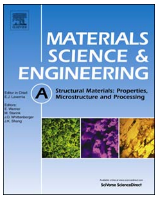
Materials Science & Engineering A 700 (2017) 220–225

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea



Effects of aging on mechanical properties and microstructure of multi-directionally forged 7075 aluminum alloy

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ARTICLE INFO

Keywords:
Aluminum alloy
Aging
Precipitation
Strength
Severe plastic deformation
Multi-directional forging

ABSTRACT

The effects of natural aging for a long period of time (2.4 years) and as well as artificial aging on the mechanical properties and microstructure of multi-directionally forged (MDFed) 7075 aluminum alloys were systematically investigated. MDFing up to a cumulative strain of 0.7 increased yield strength (+96 MPa) and ultimate tensile strength (+94 MPa) mainly due to work hardening. The addition of both natural aging at ambient temperature for 7800 ks and artificial aging at 393 K for 26 ks of the MDFed samples further increased yield strength (+190 MPa) and (+293 MPa), respectively. The microstructure and mechanical properties derived by MDFing were stably maintained for a long period of time during natural aging, even while distinct softening took place soon after the commencement of artificial aging. The number of precipitates in the MDFed samples after natural aging was notably smaller compared with that introduced by artificial aging. The achieved strengths after aging were, therefore, almost comparable.

1. Introduction

7075 aluminum alloy (7075Al) is one of the major types possessing ultimate high strength and is typically employed for structural parts that require a high strength-to-weight ratio performance such as in aircraft and other transportation applications. Improvement in the mechanical properties of this alloy can be achieved by age hardening, which is caused by homogeneously distributed fine precipitates [1]. The most common precipitation process performed during the aging heat treatment in Al-Zn-Mg-Cu alloys is as follows; supersaturated solid-solution state → coherent Guinier-Preston (GP) zones → semi-coherent η' phase → incoherent η phase [2]. Vacancy-rich clusters retained after quick quenching serve as possible nuclei for precipitates [3]. Two-step aging is the most widely employed process for Al-Zn-Mg-Cu alloys with lower temperature aging during the first step of the process enabling homogeneous dispersion of fine GP zones or clusters followed by coarsening or transformation of them in the second step of aging at higher temperatures [4]. Therefore, the control of size and distribution of precipitates are key factors for strengthening in Al-Zn-Mg-Cu alloys.

Another effective method to improve the mechanical properties of Al alloys is grain refinement. In particular, several severe plastic deformation (SPD) methods, which can produce ultrafine grained microstructures [5], have attracted significant attention in recent years. SPD methods were applied to 7075Al and the yield strength was significantly improved to 645 MPa by equal-channel angular pressing (ECAP) [6], 550 MPa by high-pressure sliding (HPS) [7] and ~1 GPa by high pressure torsion (HPT) [8]. In these cases, ultrafine grained (UFGed) structures with average grain sizes from 26 to 600 nm were achieved. To further enhance the strength of UFGed 7075Al, age-hardening should be employed. Aging of SPDed materials at conventional aging temperatures, however, appears less effective because of their thermal instability which causes recovery followed by grain growth [9–11]. Such microstructural changes spoil the mechanical properties of the SPDed materials. The aging process of the SPDed materials, therefore, remains an open research problem [9–11]. Nevertheless, some influential rules are proposed [11]. A lower aging temperature compared with a conventional one would be favorable for age hardening after SPD to prevent recovery and grain growth. However, as far as the authors know there are few researches concerned with the effects of long-term aging on the mechanical properties and microstructure of SPDed aluminum alloys.

Multi-directional forging (MDFing) is one of the SPD processes [12]. MDF is a repeating forging process with changes in the forging axis during MDFing by 90 degrees pass by pass [13]. Among various SPD methods, MDF seems to be most suitable for industrial production since it can be applied to relatively large samples. Multi-axial forging accelerates a dense evolution of microshear bands and their intersections, which leads to the formation of misoriented domains and, therefore, grain fragmentation [14]. It is reported that the achieved grain size by MDFing of 7475 Al alloy at 523 K was about 2 μm [15]. In a similar

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http://dx.doi.org/10.1016/j.msea.2017.06.017
Received 27 December 2016; Received in revised form 2 June 2017; Accepted 4 June 2017

way, UFGed structures in other alloys have been successfully developed by MDFing; average grain size of 17 nm in Cu-Zn alloy by MDFing at 77 K [16]; 230–360 nm and 300 nm in AZ31Mg and AZ80Mg alloys by MDFing under decreasing temperature conditions [17,18]; 60–200 nm average (sub)grain size in a commercial purity Ti by MDFing at ambient temperature [19]; 5–10 nm grain size in SUS316L stainless steel by MDFing at 77 K [20].

As already mentioned above, age-hardening should be one of the essential keys for further strengthening of SPDed alloys. Quite a few studies about the aging behavior of SPDed 7075Al have been reported [21,22]. In the present study, changes in the microstructure and mechanical properties during artificial aging and natural aging of MDFed 7075Al are investigated. In addition, very long-period natural aging is adapted to MDFed 7075Al to evaluate sustainability. Furthermore, the differences in strengthening mechanisms, depending on aging temperature and MDFing cumulative strain, are precisely discussed.

2. Experimental

A hot-rolled plate of 7075Al with a thickness of 15 mm was cut into rectangular shaped samples with dimensions of 15 × 15.8 × 16.6 mm^3 (aspect ratio of 1: 1.05: 1.11) and was solution heat treated (STed) at 763 K for 1 h in a salt bath, followed by water quenching. The average grain size after ST was 50 µm. The sample aspect ratio was theoretically determined for MDFing by employing pass strains of $\Delta\varepsilon = 0.1$. Seven passes of MDFing, i.e., cumulative strain of $\Sigma\Delta\varepsilon = 0.7$ at maximum, was applied to the samples at room temperature (RT) on an Amslar-type mechanical testing machine at an initial strain rate of $3 \times 10^{-3}\,\mathrm{s}^{-1}$. MDFing was conducted right after ST. Samples were isothermally aged at RT (natural aging) and at 393 K (artificial aging).

The change in hardness during aging was measured using a micro-Vickers hardness (HV) tester by applying a load of 300 g for 15 s. Before measurement, sample surfaces were mechanically and chemically polished. Tensile specimens with a gauge length of 6 mm and a cross sectional area of 2.0 × 0.8 mm^2 were discharge-machined. The tensile test was conducted on an Instron-type mechanical testing machine at an initial strain rate of $3 \times 10^{-3}\,\mathrm{s}^{-1}$ at RT. Three samples were tensile tested for each MDFing and aging condition.

Macroscopic structure was observed using optical microscopy after mirror surface finishing by mechanical and electrical polishing, and then etching by the Keller solution. Substructures and precipitates were observed using transmission electron microscopy (TEM; JEOL JEM-2100F) at an accelerating voltage of 200 kV. The TEM foil samples were prepared by twin-jet electropolishing (Struers Tenupol-5) with a solution of 25% nitric acid and 75% methanol at 243 K. Foil thickness was determined from the convergent beam diffraction pattern [23,24]. Number density and particle size of precipitates were evaluated from TEM images using ImageJ software [25].

Phase identification of precipitates and measurement of dislocation density were conducted by means of X-ray diffraction (XRD; Rigaku RINT-2500, Cu-K_α radiation at 60 kV and 300 mA). Dislocation density was estimated by employing the conventional Williamson-Hall relationship [26].

3. Results and discussion

3.1. Changes of mechanical properties by MDF and aging

Fig. 1 shows a series of stress-stain curves obtained during MDFing of 7075Al. It can be seen in Fig. 1 that the flow stress increased rapidly during the pass by pass of MDFing. Yield stress also increased rapidly at the lower cumulative strain region and gradually at the high cumulative strain region with an increasing cumulative strain. No macroscopic structural changes were observed before and after MDFing.

The age-hardening curves of 7075Al MDFed to different cumulative strains of 0, 0.3, 0.6 and 0.7 are presented in Fig. 2. Please note that the time scales of both figures are quite different. The longest natural aging time was 2.4 years. The hardness of the as-STed sample was 125 HV, whereas in the as-MDFed sample it increased up to 150 HV, 170 HV and 175 HV with cumulative strains of 0.3, 0.6 and 0.7, respectively. An increment in hardness during both the artificial and natural aging of the STed sample appeared earlier than in the MDFed samples. Obvious softening took place at the beginning of artificial aging of the MDFed samples. This softening followed by age-hardening resulted in much sharper age-hardening curves compared with those obtained by natural aging. The softening was more significant in the samples MDFed to higher cumulative strains. Therefore, the increment in the hardness of the MDFed samples during artificial aging was retarded due to the accompanied occurrence of softening. In other words, softening induced by recovery exceeded the effect of age-hardening at the beginning of artificial aging. It should be noted that the aging time to attain peak hardness becomes shorter and the reduction in hardness of the MDFed samples took place earlier and more rapidly with increasing cumulative strain. It is interesting to note, however, that the achieved peak hardnesses were almost comparable (i.e. approximately 185 HV) when aged artificially.

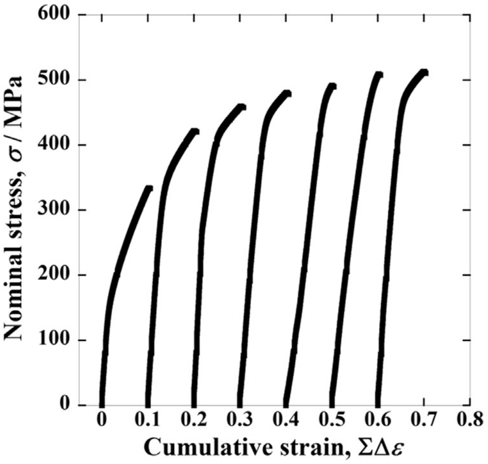

Fig. 1. Typical nominal stress-cumulative strain curves obtained during multi-directional forging of 7075Al alloy at room temperature.

On the other hand, natural age-hardening behavior appears quite different from that during artificial aging. The hardness increment in the MDFed samples by natural aging was rather small and furthermore becomes less significant with increasing cumulative strain. The peak hardnesses of the MDFed samples, however, were almost comparable with those achieved by artificial aging, whereas the amount of age-hardening in the natural-aged STed sample is approximately half of that in the artificially aged one.

Fig. 3 shows the typical stress-strain curves obtained by tensile tests of the samples STed and MDFed to $\Sigma\Delta\varepsilon = 0.7$ all followed by peak-aging. The measured mechanical properties are summarized in Table 1. The yield and ultimate tensile strengths of the STed sample were lowest while total elongation was the largest among the samples tensile tested. Artificial aging at 393 K for 80 ks (see Fig. 2(a), peak-aged) of the STed sample derived a preferable balance of mechanical properties. However, it is well known that a problem of stress corrosion cracking arises in 7075Al prepared by this heat treatment process [4]. The amount of strengthening by natural aging of the STed sample for 7800 ks (peak-aged) was rather small. MDFing of the STed sample increased yield and ultimate tensile strengths almost comparable to those of the natural aged STed sample while total elongation decreased. Aging of the MDFed sample increased yield and ultimate tensile strengths even further. Artificial aging at 393 K for 26 ks (peak-aged) of the MDFed sample recovered ductility to 9% in addition to strengthening the yield and ultimate tensile strengths up to 498 MPa and 540 MPa respectively. In contrast, natural aging of the MDFed sample for 7800 ks (peak-aged)