High-temperature deformation and structural restoration of a nanostructured Al alloy

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We studied the flow stress and microstructural changes of nanostructured Al-6063 alloy produced by mechanical alloying at various temperatures and strain rates. The analysis of flow curves was performed by a constitutive equation, and the stress exponent and activation energy were determined as functions of strain. The deformation mechanisms were elaborated through microstructural observations by electron backscattering diffraction and transmission electron microscopy. Coarsening of the subgrains and grain growth upon deformation was monitored and related to the Zener–Hollomon parameter.

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Interest in the field of nanostructured (NS) materials has grown dramatically in the last decade due to their unique microstructures and mechanical performance [1]. Bulk NS metals are frequently produced by severe plastic deformation (SPD) techniques, such as equal channel angular extrusion [2], high-pressure torsion [3] and mechanical milling followed by hot consolidation methods [4]. NS materials exhibit higher strength and lower ductility and formability compared to their coarse-grained (CG) counterparts [5]. In spite of significant studies on the room-temperature properties of NS materials, their mechanical properties at elevated temperatures, particularly restoration mechanisms involved in microstructural changes occurring during the deformation, have been reported only rarely. Horita et al. [6] and Park et al. [7] showed that the fine grain structure of SPD-processed Al alloys could be stable up to 300 °C (~0.5Tm), whereas consistent grain growth occurred at higher temperatures. Ma et al. [8] observed superplastic behavior in an ultrafine-grained Al alloy through grain boundary sliding. The deformation mechanism of NS aluminum alloys at elevated temperatures may be controlled by dislocation climb as well [9].

In general, NS materials have a large area of grain boundaries and therefore a large stored energy, and are intrinsically unstable with respect to grain growth during the high-temperature deformation operation [10]. Therefore, it is imperative and useful to study the deformation behavior of NS materials and to determine the restoration mechanisms dependent on temperature, strain and strain rate. As a follow-up to our recent work on room-temperature mechanical behavior of NS-Al6063 alloy [11], we herein report the microstructural evolution of the alloy during high deformation at different temperatures and strain rates. The effects of thermomechanical parameters on the flow behavior and dynamic restoration mechanisms are presented and discussed.

Details of the processing method of the NS Al alloy can be found in Ref. [12]. Briefly, gas-atomized Al-6063 powder, with a chemical composition of Al–0.64Mg–0.67Si–0.32Fe–0.2Cu (in wt.%), was utilized. Mechanical alloying was performed in an attrition ball mill (Union Process, OH, USA) for 20 h under an Ar atmosphere. Stearic acid (1.5 wt.% was added as process control agent (PCA). The powder was degassed at 400 °C, compacted in an aluminum container and finally extruded at 450 °C with an extrusion ratio of 14:1. Cylindrical specimens with diameter of 4 mm and an aspect ratio of 1.2 were machined

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from the extruded billet. Uniaxial compression test was performed on a Caster and Thermomechanical Simulator (Fuji Electronic Industrial Co., Ltd, Japan) under a high-purity Ar atmosphere in a temperature of 300–450 °C and a strain rate range of 0.01–1 s⁻¹. Quartz plates were used for reducing the friction between the specimen and anvils. A soaking time of 3 min at the testing temperature was considered for temperature stabilization. To preserve the microstructure after deformation, the specimens were instantly quenched with a cooling rate of ~70 °C s⁻¹.

The microstructural studies before and after deformation were carried out by electron backscattering diffraction (EBSD; Hikari, EDAX, NJ), using a field emission gun scanning electron microscope (Helios NanoLab DualBeam, FEI, Oregon), and transmission electron microscopy (TEM; JEM-2100F STEM, Japan).

Figure 1 shows true stress–true strain curves of NS Al-6063 at various temperatures and strain rates. The true stress was corrected for friction according to the slab analysis of the compression of a cylinder [13]. The flow behavior dependent on the temperature and strain rate is similar to that of CG aluminum alloy, as reported elsewhere [14]. As seen, the flow stress expeditiously increases to a maximum value and then gradually decreases with the true strain. This behavior is due to a dynamic competition between the work hardening caused by interaction, pile-up and tangling of dislocations, and the work softening may be caused by dynamic recovery (DRV), subgrain coarsening, dynamic precipitation, dynamic recrystallization (DRX) and texture softening [15].

The microstructural features of the alloy before and after hot deformation at some selected conditions are shown in Figure 2. In EBSD inverse pole-figure (IPF) maps, low-angle boundaries (LABs) (misorientation between adjacent grains of 2°–15°) and high-angle boundaries (HABs) (misorientation larger than 15°) are shown with white and black lines, respectively. The IPF image of the as-extruded material reveals an elongated grain structure with an aspect ratio of 2.9 (Fig. 2a). As shown in Figure 2d, the microstructure consists of grains with a wide size distribution in the ultrafine size range (>95%) and an average value of 500 nm. While the microstructure consisted of a high fraction of HABs (fHAB ≈ 0.78), some of the larger grains contain subgrains (Fig. 2a and g). Consequently, a high value of the average misorientation angle was measured (θave ≈ 33°). Figure 2j illustrates a representative TEM image of the microstructure. A high density of dislocations is observable within larger grains while nanometric precipitates are distributed in the microstructure. Figure 2b and c shows that a significant change in the grain structure occurred after hot deformation. The elongated grains were mainly disappeared and nanostructured and ultrafine equiaxed grains were formed after deformation at 300 °C and 1 s⁻¹ (Fig. 2b). The size distribution of grains became narrower but the average grain size was slightly increased (Fig. 2e). Moreover, the values of fHAB and θave were declined (Fig. 2f).

In order to determine the restoration mechanisms involving in the deformation behavior, the following hyperbolic sine law between the flow stress (σ), temperature (T) and strain rate (ı) was utilized [16]:

\[
A[\sinh(ı\sigma)]^{n} = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = Z
\]  
(1)

where Q is the deformation activation energy, R is the gas constant, n is the stress exponent, Z is the Zener–Hollomon parameter, and A and z are material constants. The stress multiplier for the highest correlation coefficient for the linear correlation between ln ı and In sinh(ıσ) was determined to be z = 0.06 MPa⁻¹, which is close to the value obtained for the CG Al alloys [16]. The values of n and Q at different strains (ı) were determined from the slope of ln ı vs. ln sinh(ıσ) plot and Rn ln sinh(ıσ) vs. 1/T, respectively. An example of the procedure is shown in Supplementary Figure 3a and b for ı = 0.5. Figure 3 depicts the variation of activation energy and stress exponent with the true strain. Such dependency has also been observed for a number of CG Al alloys, such as AlFe₈.₅V₁.₃Si₁.₇ [17]. Data fitting yields:

\[
n = -16.7ı^{2} - 10^{-8}ı^{4} + 9.2ı^{3} + 3ı^{2} - 4.7ı + 3.8
\]

(2)

\[
Q = 2441.7ı^{5} + 8.3ı^{4} + 1737.1ı^{3} - 232.6ı^{2} - 323.3ı + 298.4
\]

(3)

Although these equations are curve fitting expressions and might not have a physical basis, an average value of 2.9 ± 0.2 and 228 ± 21 kJ mol⁻¹ could be considered for n and Q, respectively, considering that the variation of Q reflects changes in n, as explained else-

Figure 1. True stress–strain curves of NS Al-6063 alloy at various temperatures and strain rates.
where [14]. Meanwhile, values in the range of 155–205 kJ mol$^{-1}$ for the activation energy of CG Al–Mg–Si alloys of various purities at pre-aged or annealed conditions have been reported [16]. The higher activation energy for the nanostructured Al-6063 can be attributed to the microstructural features (nanostructured grains, ultrafine grains and subgrains) that limit the cross slip and climb processes, especially at the initial stages of deformation. The higher activation energy can also be linked to the occurrence of DRX. The progressive accumulation of dislocations in LABs increases their misorientation, leading to the transformation of LABs into HABs. As the straining progresses, the subgrains, grains and precipitates coalesce, easing dislocation motion so that the activation energy falls (Fig. 3).

Figure 4a shows variation in the size of subgrains ($d_S$) and recrystallized grains ($d_R$) as functions of 1n Z. Although lower values for $d_S$ and $d_R$ were obtained compared to conventional Al alloys [18–20], these are comparable with those reported for SPD-processed Al alloys with similar Z parameters [21]. Similar to CG Al alloys [22], linear relationships in semi-logarithmic scale can be established between the grain structure and $d_S$ and $d_R$ with the steady-state flow stress ($\sigma_{ss}$) in logarithmic scale is illustrated in Figure 4b. Similar to conventional alloys [23], the graph shows that the relationship between the size of subgrains and recrystallized grains with $\sigma_{ss}$ during hot working can be expressed as $\sigma_{ss} = Kd^{-m}$. However, analysis of the data yielded m values of 2.56 and 1.04 for subgrains and recrystallized grains, respectively, which are higher than those reported for the hot deformation of micro-crystalline alloys [24,25].
The results indicate that significant changes in the microstructure of the NS alloy occur during the hot deformation, although fine particles are distributed in the matrix (Fig. 2). As reported elsewhere [12], these particles are mainly Al₈Fe₂Si, Al₅FeSi and Al₈Mg₃Fe-Si₂, with a total of 1.8 vol.%. According to the theory of Zener pinning, the volume fraction \( V_f \) of particles with a diameter of \( d_P \) required to stabilize a microstructure with an average grain size of \( D \) should be \( V_f = 4d_P^3 / 3D \) [1]. By considering \( d_P = 62 \text{ nm} \) [12], the required \( V_f \) to stabilize the nanosize grains was estimated to be 16.5%. Therefore, effective grain boundary pinning cannot be afforded by the particulates. It is pertinent to point out that, upon mechanical milling and in the presence of PCA, carbides and oxides can be formed and distributed within the Al matrix, and will contribute to the dispersion strengthening [4,12]. We evaluated this effect by analyzing the threshold stress requiring for dislocation motion at elevated temperatures [26] for NS Al-6063 and obtained an approximate value of 1 MPa. This observation reflects the minor influence of these particles, which have on the hot deformation behavior.

In conclusion, we studied the deformation behavior and microstructural changes of a nanostructured Al-6063 alloy across a wide range of temperatures and strain rates. The stress exponent and activation energy were correlated to strain with a fifth-order polynomial curve. An apparent activation energy of \( 228 \pm 21 \text{ kJ mol}^{-1} \) was obtained. The coarsening of the subgrains, recrystallized grains and precipitates during hot deformation, especially at low \( Z \) values, was shown. Deformation at \( Z > 3.4 \times 10^{17} \text{ s}^{-1} \) resulted in a refinement of grain structure without a significant coarsening of subgrains.

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.scriptamat.2012.02.026.