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Development of Ultra-fine Grained Structure in an Al-5.4%Mg-0.5%Mn Alloy Subjected to Severe Plastic Deformation

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It was shown that extensive grain refinement takes place in an as-cast Al-5.4%Mg-0.5%Mn-0.1%Zr alloy subjected to severe plastic deformation (SPD) under multi-directional forging up to a true strain of ~9 at 250 and 300°C. At a strain of ~3, the microstructural evolution is mainly characterized by the formation of new grains along original boundaries and the development of well-defined subgrains within interiors of initial grains. Upon further straining the misorientation of deformation-induced boundaries increases; new grains appear homogeneously both within grain interiors and along original boundaries. The kinetics of transformation of low-angle grain boundaries (LAGBs) into high-angle grain boundaries (HAGBs) tends to slow down with decreasing temperature from 300 to 250°C at intermediate strains; the formation of a grained structure shifts from $\Sigma\varepsilon$~9 to $\Sigma\varepsilon$~6 with decreasing temperature from 250 to 300°C. The resulted grain size evolved in this alloy was slightly less than that produced in an Al-6%Mg-0.35%Sc by severe plastic deformation at similar conditions. In addition, in the alloy belonging to Al-Mg-Zr system the formation of a fully recrystallized structure was found at lower cumulative strain in comparison with the alloy belonging to Al-Mg-Sc system. This unusual difference associated with the fact that the Al-5.4%Mg-0.5%Mn-0.1%Zr alloy was initially subjected to solution treatment at a relatively low temperature of ~360°C.

Keywords: *aluminum alloy, microstructure, dynamic recrystallization, multi-directional forging, severe plastic deformation*.

1. Introduction

There is currently a significant commercial interest in the development of non-heat treatable aluminum alloys with a submicrocrystalline (SMC) structure for structural applications [1]. It is well-known that extensive grain refinement down to the submicrometer range leads to increased hardness and strength of these aluminum alloys at room temperature; ductility retains at sufficiently high level. At high and intermediate temperatures, the fine grained Al-Mg alloys may exhibit extraordinary high superplastic ductilities at high strain rates, typically in the range of ~10^{-2}-1 s^{-1} [1]. It is now well established that a substantial reduction in the grain size of aluminum alloys can be attained through severe plastic deformation (SPD) [2]. Several SPD techniques, such as equal channel angular pressing (ECAP) [3,4], accumulative roll bonding (ARB) [5,6] and multi-directional forging (MDF) [7], are mostly used for processing of aluminum alloys to produce SMC structures. These methods can impose a large amount of plastic deformation required for the occurrence of grain refinement in aluminum alloys.

The general features of the microstructure evolved during SPD in different aluminum alloys were examined in various works. It was reported [2] that the formation of a SMC structure can occur by the extension and compression of the initial grain boundaries followed by the discontinuous formation of transverse high-angle boundaries HAGBs. Other investigations have emphasized the role of continuous dynamic recrystallization (CDRX) in grain refinement under SPD of aluminum alloys at elevated and high temperatures [3,7,8]. This process associates with the formation of low angle grain boundaries (LAGBs) at low strains and their gradual transformation into high angle ones with further

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straining due to accumulation of lattice dislocations [3,7-10]. New grains result from the gradual increase in misorientation between subgrains during plastic deformation [3,7,8]. It is worth noting that CDRX was found to be the mechanism of extensive grain refinement during ECAP [7,8] and MDF [9] of Al-Mg-Sc containing nanoscale coherent Al_3Sc particles. It is associated with the fact that the coherent dispersoids provide the excellent stability of deformation-induced arrays of LAGBs that is a prerequisite condition for the completion of CDRX process [7-10]. As a result, these alloys can be processed in semi-finished products with a very high strength and sufficient ductility. This unique combination of strength and ductility, which typically have opposing characteristics, is attributed mainly to the formation of the ultra-fine grained structure under SPD. In the same time the typical commercial alloys of 5XXX series belong to Al-Mg-Mn system and contain low amount Cr or Zr, additionally. It is extremely important to develop a commercial viable route of thermomechanical (TMP) processing that can provide the formation of a fully recrystallized structure in semi-finished products from these alloys.

The present study was initiated to demonstrate the potential of MDF in producing a SMC structure in a commercial Russian Al-5.4%Mg-0.5%Mn-0.1%Zr alloy designated as 1561 and denoted here as 1561 Al. Specific attention was paid to the effect of pressing temperature and strain on the microstructural evolution in the 1561 Al subjected to MDF in an attempt to develop a simple processing method for extensive grain refinement in large scale billets of the 1561 Al that is highly important for commercial use in shipbuilding industry.

2. Material and Experimental Procedure

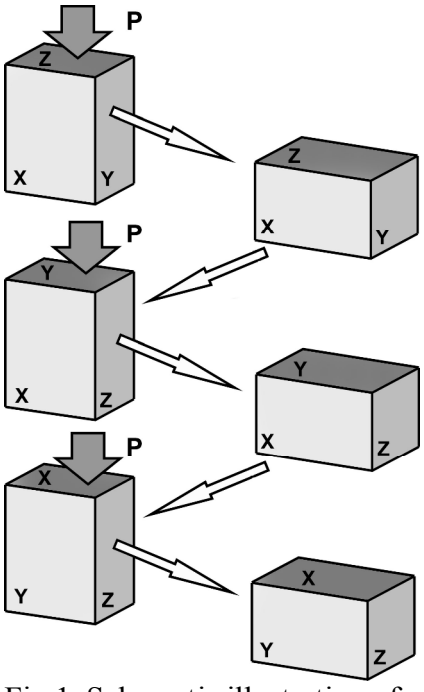

Fig.1. Schematic illustration of a one step of MDF consisted of a three sequent compressions.

The commercial 1561 Al with a chemical composition of Al–5.43%Mg–0.52%Mn–0.1%Zr–0.12%Si–0.014%Fe (in weight %) was manufactured by direct chill casting and, then, aged at 360°C for 6 hours. Next, the 1561 Al was machined to rectangular billets with dimension of $16 \times 14 \times 10$ mm. The multi-directional forging of the 1561 Al was carried out at temperatures of about 250 and 300°C at an initial strain rate of $\sim 10^{-2}$ s^{-1} using an Instron 300LX testing machine. A powder of boron nitride was used as a lubricant. The billets were compressed to a true strain (ε) of ~ 0.5 at each pass of MDF. The loading direction (P) was changed to 90° from pass to pass along three orthogonal directions (Fig.1). Each step of MDF consisted of three sequent changes of compression axis from *x* direction to *y* direction and to *z* direction (Fig.1) with a cumulative strain of ~1.5. New step of the MDF deformation started by compression along *x* axis again. The samples were deformed to several cumulative strains ($\Sigma\varepsilon$) of ~ 3, 6 and 9. Between passes the samples were reheated to the deformation temperature for ~10 min. This short dwelt time was necessary to prevent static recovery. Finally, the samples were water quenched.

Following MDF, the specimens for microstructural observations were cut from the central area of the forged billets parallel to *X*–plate. For the electron-backscattering diffraction (EBSD) analysis these specimens were lightly electropolished to give a strain-free surface. EBSD orientation maps were recorded using a FEI Quanta 600FEG scanning electron microscope equipped with a high-resolution electron backscatter diffraction analyzer. The arbitrary area was automatically scanned with a step size of ~0.1 μm. In the data presented, HAGBs were defined as

θ≥15° in misorientation and LAGBs as 3°<θ<15°. HAGBs and LAGBs are depicted in EBSD maps as black and white lines, respectively.

3. Experimental Results

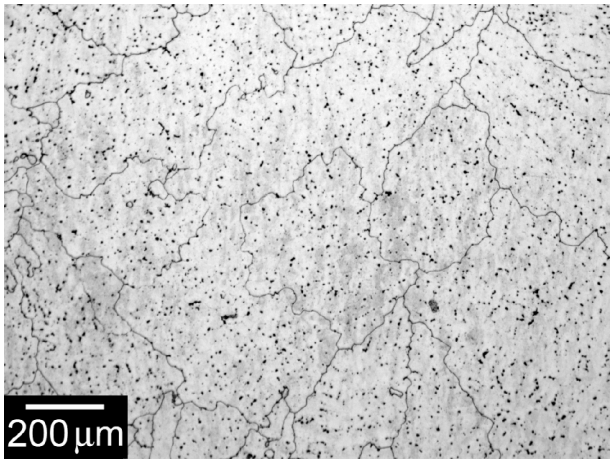

Fig. 2. Microstructure of the 1561 Al after solution heat treatment at 360°C.

A granular structure with an average grain size of ~300 μm was found in the 1561Al after solution heat treatment (Fig. 2); numerous particles of Al_6Mn phase could be observed. Typical EBSD maps of the deformation structures developed in the 1561 Al processed by MDF to a strain of ~ 3 at temperatures of 250 and 300°C are shown in Fig 3a and 3b, respectively. Figure 4 shows strain dependence of the average size of newly developed grains, the average misorientation angle, the portion of HAGBs and the fraction of recrystallized grains at 250 and 300°C. It is clearly seen that the new equiaxed grains outlined by HAGBs alternated with the original grains containing recovered subgrains within their interiors. New grains mainly evolved in vicinity of original boundaries at both temperatures. In spite of the apparent similarity of the developed stricture the strain dependencies of the portion of HAGBs and the fraction of recrystallized grains suggest that the rate of grain refinement is highly accelerated at 300°C (Fig. 4a). The size of new developed grains and subgrains in unrecrystallized areas increases with increasing temperature. At 250°C, the average size of newly developed grains was ~0.58 μm. This size increases to ~0.93 μm with increasing temperature to ~300°C (Fig. 4b).

Further straining to Σε ~6 leads to significant changes in microstructure developed at 250°C (Fig. 3c). The thickness of original grains and subgrains tends to approach the size of new grains (Fig. 3c); extensive elongation of initial grains takes place with strain. On the other hand, at 300°C a uniform formation of new equiaxed grains takes place due to the transformation of LAGBs into HAGBs within the areas of coarse initial grains (Fig. 3d). The new grains are dominant in the microstructure evolved at 300°C. It is interesting to note that some grain growth of the recrystallized grains developed at Σε ~3 takes place with increasing cumulative strain to ~6 both at 250 and 300°C (Fig. 4b). At 250 and 300°C, the portion of HAGBs and the fraction of recrystallized grains increase with increasing strain (Fig. 3b). However, at 250°C, the process of grain refinement occurs with low rate in comparison with 300°C. Despite the appearance of some coarse grains the fraction of new grains of ~0.84 and fractions of HAGBs of ~ 0.73 suggest almost completed recrystallization at a temperature of 300°C.

At 300°C, following plastic straining to a cumulative strain of ~9 leads to no considerable changes in microstructure; there is a slight increase in the fraction of unrecrystallized subgrain areas (Fig. 3f) which may be associated with restoration processes occurring during intermediate annealing between passes of MDF. On the other hand, at 250°C, the plastic deformation to a strain of ~ 9 leads to the formation of almost uniform SMC grained structure (Fig. 3e). Most of the deformation-induced boundaries have high-angle origin (Fig. 3e). As a result, most of crystallites being true grains are entirely delimited by HAGBs. Therefore, at 250°C, a well-defined recrystallized structure is evolved (Fig. 3e); the fraction of recrystallized grains of ~0.93 exceeds the fraction of grains developed at 300°C. At 300°C, the resulted grain size was ~ 60 pct. higher than the grain size developed at 250°C

(~ 0.58 μm). Thus it can be concluded that the MDF of the 1561 Al to a cumulative strain of Σε~9 at 250°C provides the formation of a fully recrystallized structure with a grain size less than 1 μm.

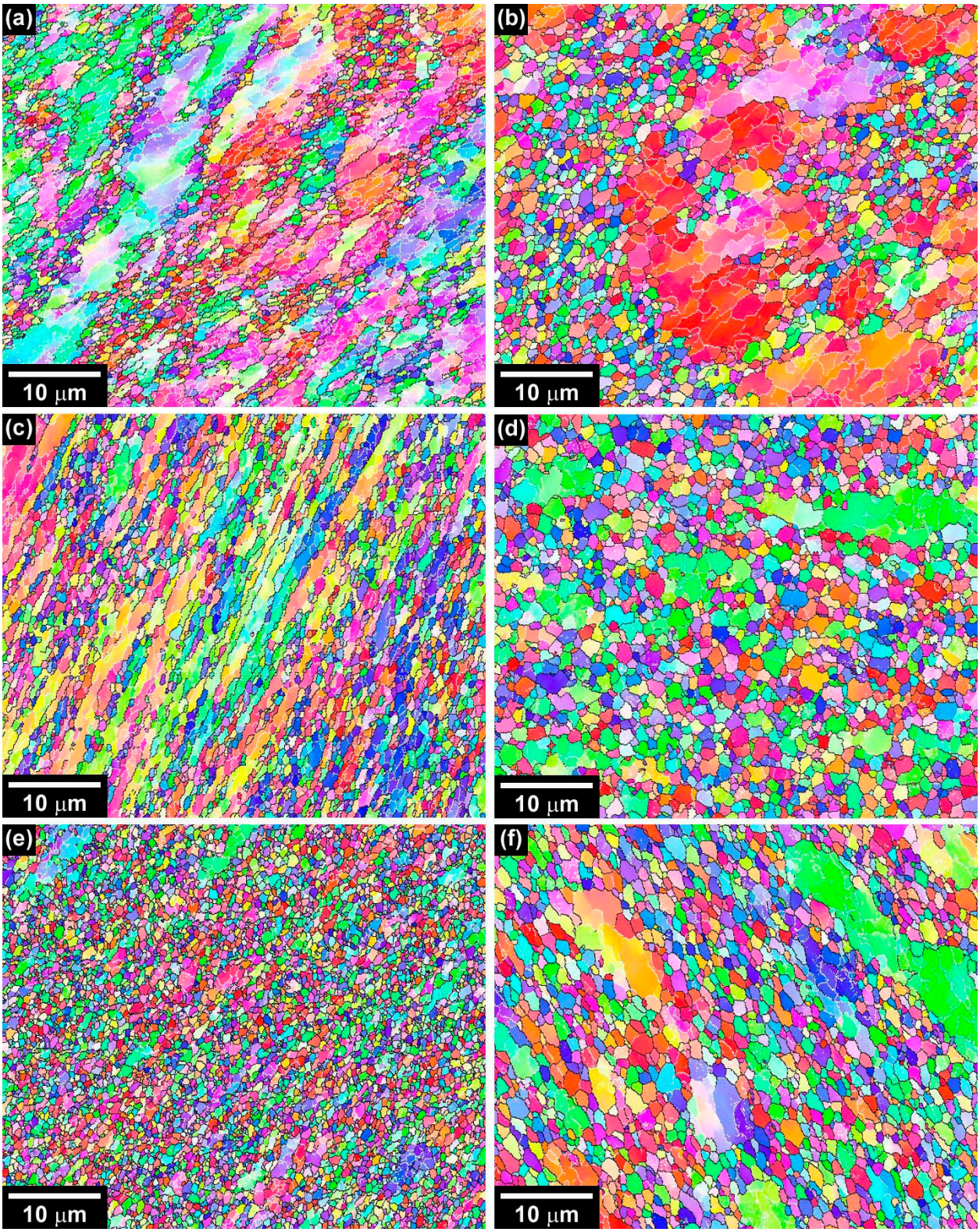

Fig.3. Typical EBSD maps of the 1561 Al processed by MDF to cumulative strain Σε~ 3 (a, b), Σε~ 6 (c, d) and Σε~ 9 (e, f) at temperatures 250°C (a, c, e), 300°C (b, d, f).

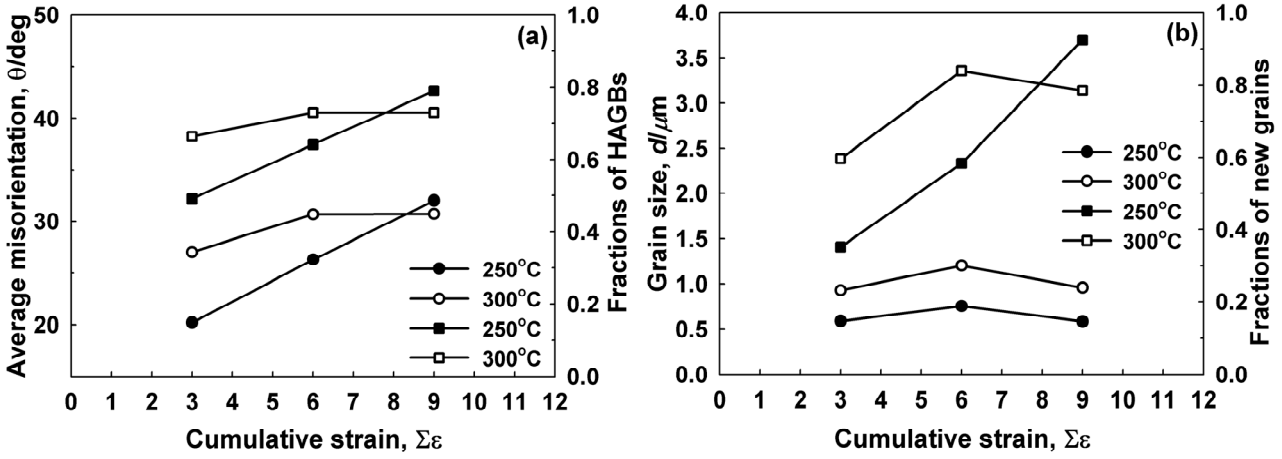

Fig. 4. Effect of cumulative strain on (a) the average misorientation angle (circle symbols) and the fractions of HAGBs (square symbols); (b) the average size of recrystallized grains (circle symbols) and the fractions of new grains (square symbols).

4. Discussion

Inspection of experimental data shows that extensive grain refinement in the 1561 Al occurs through CDRX, which was elsewhere found to be operative in an Al-6%Mg-0.35%Sc alloy designated as 1570Al [7-10]. Regularities of CDRX process in the 1561 Al and 1570Al are almost the same. CDRX consists of two sequential processes: the formation of three-dimensional arrays of LAGBs and the gradual transformation of LAGBs into HAGBs. The first stage of CDRX was not detected in the 1561 Al due to the fact that this stage occurs at low strains which were not examined in the present work. It is obvious that at $\Sigma\varepsilon > 3$ the recrystallized grains persistently replace subgrains through continuous transformation of their boundaries to HAGBs. Upon processing to a cumulative strain of ~3, the average misorientation rapidly increases to 20° and 27° for 250 and 300°C, respectively (Fig. 4a). Increasing temperature promotes the dislocation rearrangement within the interiors of initial grains due to acceleration of diffusion processes. Mobile dislocations move across subgrains and are trapped by deformation-induced LAGBs resulting in an increase in their misorientation. This can result in the formation of HAGBs [11]. At 300°C, the kinetics of transformation of LAGBs into HAGBs increases with strain attaining saturation at $\Sigma\varepsilon$~6. It seems that this fact is associated with significant boundary migration at this temperature. The fraction of HAGBs of ~ 0.84 exceeds a threshold value of ~ 0.6 for conventional grained structures [2]. This suggests that this structure is a granular one, and it is possible to conclude that the almost fully recrystallized structure developed at 300°C under MDF to a moderate cumulative strain of ~6 in the 1561 Al. Decreasing deformation temperature to 250°C reduces the rate of CDRX; the formation of a fully recrystallized structure is observed after a cumulative strain of ~9.

It is interesting to note that the grain size evolved in the 1561 Al is less than that in the 1570 Al. It is in contrast with previous data [4-11]; it was shown that the formation of the ultrafine grained structure in Al-Mg alloys is highly facilitated by the presence of nanoscale particles of $Al_3(Sc,Zr)$. In the 1561 alloy the incoherent nanoscale particle of Al_3Zr precipitated during solution treatment at a relatively low temperature of 360°C. No high temperature solution treatment at a temperature of 500°C was performed. As a result, the Al_3Zr particles retain their nanoscale size. Perhaps, the formation of three-dimensional arrays of LAGBs has no effect on the coagulation of this dispersoids as in the 1570 Al [7]. This is why the resulted grain size in the 1561 Al was less than that in the 1570 Al [7-10].

In addition, in the 1561 Al the formation of a fully recrystallized structure was found at lower cumulative strain in comparison with the 1570 Al. Perhaps, it is caused by the fact that high volume fraction of coherent $Al_3(Sc,Zr)$ hinders the formation of a subgrain structure at lower strains in the 1570Al. It seems that the 1561 Al subjected to solution treatment at intermediate temperatures contains volume fraction of the Al_3Zr dispersoids that is an optimal one for the extensive grain refinement. Easy evolution of ultrafine grained structure under SPD of the 1561Al is attributed to the optimal size and incoherent nature of nanoscale dispersoids. Thus, the combination of solution treatment at intermediate temperature with MDF provides the formation of submicrometer scale grains in the 1561Al which contains no Sc additions.

5. Summary

The present study demonstrates the feasibility of achieving grain refinement in the commercial 1561 aluminum alloy. It was established that MDF is a very effective processing method to introduce SMC grain size (~1 μm) into bulk billets of the 1561 Al in as-cast state. At temperatures of ~ 250 and 300°C, extensive grain refinement takes place in the 1561 Al subjected to MDF. The average crystallites size increases with increasing temperature from ~0.58 μm at 250°C to ~0.96 μm at 300°C. Increasing temperature accelerates the transformation of LAGBs into HAGBs. A fully recrystallized structure was observed at $\Sigma\varepsilon$ ~6 in samples subjected to MDF at 300°C. Decreasing MDF temperature to 250°C shifts the formation of a SMC structure to $\Sigma\varepsilon$ ~9.

Acknowledgements

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