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The strength and thermal stability of Al–5Mg alloys nano-engineered using methods of metal forming

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ABSTRACT

In this work, nano-engineering approach was used to increase the strength of Al-5 Mg alloys (5xxx series). These nano-engineering methods were: (a) plastic consolidation of powders at elevated temperature and (b) hydrostatic extrusion. The microstructural factors influencing their properties were characterised. The results indicate that the alloys can be significantly strengthened by both hydrostatic extrusion and consolidation of powders when compared to cold rolling. Both processing routes results in ultrafine grained structures which together with dislocation and solid solution strengthening bring about high strength (yield strength exceeds 400 MPa). In addition to efficient strengthening, plastic consolidations impart exceptional thermal stability, which can be attributed to the presence of nano-sized oxides and an increased fraction of high angle grain boundaries.

1. Introduction

Commercial aluminium alloys have a wide range of industrial applications because of their good mechanical properties and low density. Nevertheless, there are continuing efforts to further increase their strength-to-density ratio by employing various strengthening mechanisms. The strengthening of the 5XXX series Al–Mg alloys is mainly caused by the formation of a solid solution. As such alloys are not heat-treatable, they exhibit only moderate increase in strength, which is sufficient for such applications as petrol tanks, pressurised cryogenic vessels, marine structures and fittings, automotive trim and architectural components. Traditionally, their mechanical properties have been improved by work hardening, using processes such as cold rolling.

In recent years, new opportunities have arisen in this respect with the development of methods for grain size reduction below 1 μm or even down to 100 nm. Such an approach can be viewed as nano-engineering. In practice, the reduction of grain size to submicron or nanoscale can be achieved by following either (1) a bottom-up approach such as inert gas condensation, consolidation of nanopowders, electrodeposition or crystallisation from the amorphous state or (2) a top-down approach to grain size refinement. The reduction in grain size of a metallic alloy brings about an increase in strength which was predicted by the well-known Hall–Petch relationship and confirmed experimentally [1–6]. Simple estimation shows that technically pure aluminium may exhibit the yield strength of 500 MPa if its grain size is reduced to 25 nm. However it has been demonstrated that Hall–Petch relationship is valid until a certain critical value of grain size below which a decrease in mechanical strength is observed [7].

In the case of Al–Mg alloys, an interesting option is to combine grain boundary strengthening with cold work hardening as precipitation strengthening is not efficient [8]. Such an attempt has been already explored by using the various methods of severe plastic deformation (SPD), such as Equal Channel Angular Pressing [9,10], High Pressure Torsion [11], Cyclic Extrusion Compression [12] and Multi Axial Forging [13].

In the present work, we consider the viability of two methods, namely hydrostatic extrusion (HE) and plastic consolidation (PC) of nanopowders, in terms of strengthening effect of 5XXX series aluminium alloys. HE and PC methods are of significant importance because they were already proved as efficient to produce large quantities of material with ultrafine grained and nanocrystalline structure in the form of rods and wires with a diameter of a few millimetres and a length of several tens of centimetre can be produced [14,15]. In an attempt to explore the outcome of strengthening by these methods, one should also take into account thermal stability of produced alloys. The high density of defects, in this case grain boundaries and dislocations, accumulated in the material to increase the strength, increase the susceptibility to recovery, recrystallisation and grain growth.

2. Experimental

Two types of nano-engineering samples of Al–5Mg alloys were used for the experimental work

(a) 3 mm 5483 rods obtained by HE at ambient temperature with true strain of 3.8;

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Table 1
Chemical compositions of the alloys.

Chemical composition (wt%)	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Others
5182 (CR)	max 0.20	max 0.35	max 0.15	0.2–0.5	4.0–5.0	max 0.10	max 0.25	max 0.10	max 0.05
5083 (PC)	max 0.30	max 0.40	max 0.10	0.4–1.0	4.0–4.9	max 0.25	max 0.25	max 0.10	max 0.05
5483 (HE)	max 0.20	max 0.15	max 0.05	0.55–1.0	4.5–5.0	max 0.20	max 0.20	max 0.05	Zr max 0.20

(b) 5 mm 5083 rods fabricated by PC of rapidly solidified flakes, the cooling rate exceeded 10,000 K/s, followed by hot extrusion at a temperature of 420 °C with an extrusion ratio of 25.

The results obtained were compared to conventionally cold rolled (CR) 5182 alloy (4 mm thick sheets subjected to 50% reduction by CR, corresponding to a true strain of 0.7). The magnesium content was 5 wt% in all the alloys. The chemical composition of the alloys is given in Table 1 which shows that the amounts of other elements showed the typical variation of commercial aluminium alloys.

The mechanical properties of the samples were determined by microhardness measurements under a load of 100 g and by tensile tests carried out at ambient temperature at strain rate of $10^{-4}\,s^{-1}$. In order to evaluate the thermal stability, the as supplied samples were annealed at different temperatures (23 °C, 100 °C, 200 °C, 300 °C, 400 °C) for 1 h.

The microstructures were examined by TEM observations and quantified in terms of the grain size described by the diameter of a circle of the area equal to the surface area of a given grain. The misorientation angles of grain boundaries were determined using the Kikuchi line patterns of adjacent grains. A fully automated EBSD system (provided by HKL Corporation) installed in a HITACHI SU-70 scanning electron microscope was used in the case of CR sample, but for the HE and PC samples, the resolution of this method was too low. In these cases, Kikuchi line patterns were obtained in the transmission mode from convergent beam diffractions. These patterns were subsequently used to calculate the crystallographic orientation of individual grains and the misorientation across grain boundaries was determined by KILIN and DES software developed at AGH University of Technology. To identify the chemical compositions of precipitates and possible oxide segregation at the grain boundaries EELS (Electron Energy Loss Spectrometry) analyses were used, which is particularly suitable for investigating the presence of light elements such as oxygen.

3. Results

The results of microhardness measurements and tensile tests are presented in Table 2. The highest hardness and mechanical strength were possessed by the samples subjected to hydrostatic extrusion. They are 50% greater than that of the commercially supplied cold rolled sheets. A significant improvement in strength has also been obtained for PC samples whose properties are higher by 30% than that for CR specimens. Although the highest mechanical strength was obtained for HE sample, the best plasticity was exhibited by the PC sample. In general, nano-engineered samples exhibited a much better combination of properties (strength and ductility) than the conventional cold-rolled material.

The results of mechanical properties measurements are fully consistent with the microstructures of the samples illustrated in Fig. 1. The HE sample, which possessed the highest microhardness and strength, has a grain size well below 1 μm, with an average size c.a. 200 nm. It should be noted that despite such a small size, the grain interiors contain a relatively high density of dislocations (estimated at $10^{16}\,m^{-2}$), which have been retained during HE because of the very high strain rate applied at the ambient temperature. The microstructural investigations revealed that the PC samples exhibit a submicron grain size, with an average size of c.a. 500 nm. However, in this case, a much lower density of dislocations (about $10^{13}\,m^{-2}$) is observed in the grain interiors. Such a small density of dislocations is consistent with the fact that the consolidation extrusion was carried out at 420 °C. The presence of second phase particles should also be noted for PC samples. Finally, prime microstructural element of the CR samples is dislocations, which form a banded structure with dislocation walls spaced at 550 nm.

Table 2
Mechanical properties of 5XXX aluminium alloys obtained by various fabrication methods.

Sample	Microharness HV0.1	Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation to failure (%)
CR	106	300	380	9.8
HE	140	450	545	13
PC	127	410	465	15.5

The quantitative data on the grain size as well as grain boundary misorientation angles in the CR, HE and PC specimens are summarised in Table 3. For the purposes of the following discussion these angles have been grouped as

(i) low angle, where the misorientation angle is less than 5°;
(ii) medium angle, with a misorientation angle between 5 and 15°;
(iii) high angle, where the misorientation angle is greater than 15°.

The results given in Table 3 show that the distribution of the misorientation angles for the HE sample was relatively wide, with about 40% of boundaries being high angle, 50% medium and 10% low angle grain boundaries. In contrast, the PC sample attained a grain structure in which more than 90% of the grain boundaries were of the high angle type. It should be realised that conventional rolling does not bring about the formation of new grains and, as a result, low angle dislocation boundaries are dominant in the microstructure.

The plot of average hardness and average grain size against the annealing temperature is given in Fig. 2, which shows the remarkable thermal stability of PC samples. The average microhardness remains unchanged up to a temperature of 300 °C and the average grain size increased only slightly after annealing at 400 °C for 1 h. The grain size of the HE sample changed significantly after annealing at 300° and significant grain growth, with the average grain size increased to 9 μm, was caused by annealing at 400 °C. The lowest thermal stability was shown by the CR samples, which become fully recrystallised after annealing for 1 h at 300 °C and after annealing at 400 °C for 1 h the alloy possessed an average grain size of about 20 μm.