Ultra-fine grained Al-Mg alloys with superior strength via physical simulation

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Abstract. The Al 5xxx alloys are widely used in form of sheets in marine, transport, and chemical engineering and, thus, they are often have to undergo hot/cold rolling as the final metal forming operations. Recent investigations have demonstrated that ultra-fine grained (UFG) Al 5xxx alloys have a significant potential for industrial applications due to their improved mechanical properties and enhanced corrosion resistance. However, the development of hot/cold rolling routes for the UFG Al alloys is very time consuming due to numerous experimental trials and very expensive due to higher cost of the UFG processed materials. In this work, physical simulation of cold rolling is applied to the UFG Al 5083 alloy obtained via equal channel angular pressing with parallel channels to analyze the effect of cold rolling on the microstructure and microhardness of the material. The cold rolling parameters are chosen based on the outcomes of physical simulation and the UFG Al 5083 alloy is successfully subjected to cold rolling resulting in superior mechanical strength of the material. It is concluded that physical simulation can significantly increase the efficiency of experimental work on development of thermo-mechanical processing routes.

1. Key requirements

Aluminium alloys are the most used non-ferrous material, so they play a very important role in modern engineering. Al alloys have been widely used in various sectors of engineering due to their good mechanical properties, high corrosion resistance, along with good weldability, machinability, and relatively low cost [1, 2]. Over last two decades, much progress has been made in fabrication of the ultra-fine grained (UFG) Al alloys and study of their mechanical, physical and chemical properties for advanced structural and functional use [3, 4].

In the family of the Al alloys, the non-heat treatable Al alloys of the 5XXX series (Al-Mg-Mn alloys) are one of the most attractive ones for various applications in marine and transport engineering and in the chemical industry [1, 2]. There is a significant body of research on grain refinement in these Al alloys via cryomilling of powders and their further consolidation (i.e. 'bottom-up' approach) [5-10]. Another approach to refine the microstructure in the Al alloys of the 5XXX series is application of severe plastic deformation (SPD) techniques, i.e. the so-called 'top-down' approach [11, 12]. It has been demonstrated that the latter one is the most flexible way for the nanostructural design in the Al alloys for further improvement of their properties [3, 4, 13], but less research on SPD processing of the Al alloys of 5xxx series has been done [10, 11]. The equal channel angular pressing (ECAP) [14] is the most popular SPD technique for grain refinement in the 5XXX Al alloys in order to significantly improve their mechanical strength, but at the expense of their ductility. Usually, billets having a square or cylindrical cross section are subjected to ECAP processing. The 5XXX Al alloys often have to undergo rolling as the final metal forming operation since they are often used in form of sheets. Thus, the hot/cold rolling routes have to be developed for the final metal forming operations of the 5XXX Al alloys. However, these metal forming operations can significantly affect the microstructure of the UFG 5XXX Al alloys and, thus, significantly...
affect their final mechanical and functional properties. It should be also noted that the development of hot/cold rolling routes for the UFG metallic materials are very expensive due to their much higher cost, compared to the coarse-grained counterparts, and time consuming due to the numerous experimental trials required for determination of the optimal processing parameters.

Physical simulation of metallurgical processes is an advanced approach to increase the efficiency of experimental work and to reduce the amount of material required for development of novel metallurgical processing routes. In physical simulation, a small specimen is subjected to the same thermo-mechanical profile that the real material has to undergo during processing at large industrial scale [15, 16]. Rolling of large billets at industrial scale can be reproduced by plane strain compression testing of small specimens in the thermo-mechanical simulator, since the material is deformed under similar stress state. If physical simulation is accurately performed, its outcomes can be readily transferred from lab to the large scale production process.

The main goal of the present work is to apply physical simulation of cold rolling for development of the optimal cold rolling route for fabrication of the UFG Al5083 sheets with superior mechanical strength.

2. Material and processing

An Al-4.4%Mg-0.7Mn-0.15Cr (Al5083) alloy was selected as a material for this study. A hot rolled plate with a thickness of 100 mm was received. The as-received material was subjected to annealing at 350°C for 2 h. Afterwards, the plate was machined into bars with diameter of 20 mm and length of 100 mm. The bars were subjected to equal channel angular pressing with parallel channels (ECAP-PC). The ECAP-PC technique is a modification of a conventional equal channel angular pressing (ECAP). Detailed description of the ECAP-PC technique can be found in the earlier publications [17, 18]. The intersection angle between the parallel channels and the channel connecting them was 110° and the distance between the parallel channels was 20 mm. Each bar was heated up to 100°C in the furnace and soaked for 15 min followed by its lubrication. Afterwards, the bar was subjected to ECAP-PC processing pass in the die heated to 100°C. The punch speed was 6 mm/s. The bars were subjected to 7 ECAP-PC passes. The strain produced in each ECAP-PC pass was ~ 1.6, so the total cumulative strain induced into each sample was ~ 11.2.

3. Experimental procedures

The microstructure of the supplied coarse-grained material was analyzed using optical microscope Olympus GX41. Samples for optical microscopy studies were prepared using standard metallographic technique. They were polished to the mirror-like surface using colloidal silica at the final stage. To reveal the grain structure, the samples were etched using the Keller reagent.

To study microstructure of the ECAP-PC processed material, transmission electron microscopy (TEM) analysis was carried out using a JEOL-2100 microscope equipped with a digital camera OLYMPUS D71. The microscope was operating at 200 kV. Samples for TEM study were cut out from the longitudinal section of the ECAP-PC processed samples and samples after physical simulation, and mechanically ground to thickness of 100 µm. The TEM foils were prepared by twin jet electro-polishing in Tenupol-5 using chemical solution which consisted of 30% of Nitric acid and 70% of methanol. Electro polishing was performed at -25°C at an operating voltage of 12 V. TEM observations were made in bright and dark field imaging modes. Selected area electron diffraction (SAED) patterns were recorded from the areas of interest using an aperture of 1 µm nominal diameter. To determine the average
grain/subgrain size, the linear intercept method was employed and at least 200 grains/subgrains were analyzed. The XRD measurements were performed using Rigaku Ultima IV diffractometer using CuKα radiation (20 mA and 30 kV). Coherent domain size (CDS) and elastic micro-distortion level $\langle \varepsilon^2 \rangle^{1/2}$ were calculated for each materials condition via Rietveld refinement method using the MAUD software [19]. Dislocation density $\rho$ was estimated using Eq. 1 [20],

$$\rho = 2\sqrt{3}\langle \varepsilon^2 \rangle^{1/2} / (D \times b) \tag{1}$$

where $b$ is the Burgers vector ($b = a\sqrt{2}/2$ for fcc metals) and $D$ the size of the coherent scattering domain (CSD).

Figure 1. a) Schematic presentation of plane strain compression test; b) schematic presentation of a sample after plane strain compression test.

A thermo-mechanical simulator GLEE BLEE 3800 was employed to perform physical simulation of cold rolling via plane strain compression testing. Figure 1 illustrates the schematic presentation of plane strain compression testing. It is a laboratory testing method which allows to measure the stress-strain response of metallic materials at given strain rate and temperature with constraint similar to that in industrial plate rolling [21, 22]. Small plates having dimensions of 20x15x5 mm³ were machined from the ECAP-PC processed rods. The 'rolling direction' (RD) in the plates for physical simulation coincided with the pressing direction in the ECAP-PC processed samples (Fig. 1b). The testing was performed at room temperature with strain rate of $10^6$ s⁻¹. The plates were subjected to plane strain compression testing to reduction ratios of 10%, 20%, 30%, 40%, 50%, 60% and 75% with 10% reduction ratio per each step (and 5% reduction ratio at the last step). The break time between plane strain compression steps was ~30 s which is relevant to that between rolling passes in real cold rolling of the material. The true stress $\sigma$ during plane strain compression testing was calculated as [23]

$$\sigma = \frac{F}{wb}, \tag{2}$$
where \( F \) is the load, \( w \) the width of the anvil (Fig. 1a), and \( b \) the specimen width (Fig. 1a). The true compressive strain was estimated as [23]

\[
\varepsilon = \ln\left( \frac{h_0}{h} \right),
\]

where \( h_0 \) is the initial thickness of the plate and \( h \) the thickness of the plate during plane strain compression testing.

The real cold rolling of the UFG Al5083 alloy was performed in a roll mill with a roll diameter of 120 mm at a set rolling speed of 15 rev/min.

![Figure 2. Microstructure of the Al5083 alloy](image)

4. Results and discussions

4.1. Microstructure of the Al5083 before and after thermo-mechanical processing
Microstructure of the as-received hot rolled Al 5083 alloy after annealing is shown in Fig. 2(a,b). It can be described as a homogeneous microstructure consisting of grains elongated in the rolling direction and having a length of 300...400 µm and aspect ratio of 2...3. These coarse grains consist of smaller equiaxed cells with thin dislocation walls as boundaries (marked by black arrows on Fig. 2b). The interior of cells is free of dislocations and their size is in the range of 0.5..1 µm. Such microstructure was developed during hot rolling of the material and its further annealing. Lattice dislocations generated during hot rolling rearranged so as to form such stable configurations because of the enhanced activity of cross-slip and climb during hot rolling, and further annealing led to annihilation of the statistically stored dislocation in the interior of cells via their climb [24]. The rod type AlMnCr dispersoids having a length in the range of 400...600 nm and aspect ratio of ~5 are also observed in the microstructure. These dispersoids are typically present in the Al-Mg-Mn alloys (Fig. 2b) [2].

ECAP-PC processing of the material led to formation of a very homogeneous UFG microstructure (Fig. 2c,d). The ultra-fine grains have mainly an equiaxed shape and their size is in the range of 250...600 nm with the average size of 480 nm. Lattice dislocations are seen in the interior of most of the ultra-fine grains. The X-Ray measurements have clearly demonstrated that dislocation density after ECAP-PC processing has increased by two orders of magnitude (Table 1). The ECAP-PC processing resulted in fragmentation of the rod-shape dispersoids into fine spherical dispersoids having a size of 50...100 nm. Fragmentation of the rod-shape dispersoids during ECAP-PC occurs as a result of shearing processes [25]. It should be also noted that the ECAP-PC processing reduced the CSD size by a factor of 3 (Table 1) due to formation of much finer microstructure as well as due to the significant increase of lattice dislocation density (Table 1).
Evolution of microstructure in the UFG Al5083 alloy during plane strain compression testing at room temperature is illustrated in Fig. 3. Plane strain compression testing with reduction ratio of 10% (compressive strain ~0.11) results in elongation of some ultra-fine grains along the 'rolling direction' (Fig. 3a), though most of them retain their equiaxed shape. Some areas of micro-shear banding (MB) are observed in the microstructure, which are marked by white arrows on Fig. 3b. Higher dislocation density is observed in the interior of ultra-fine grains, it increases by one order of magnitude (Table 1). Further increase of true compressive strain to reduction ratio of 50% (compressive strain ~0.69) results in formation of complex bi-modal microstructure consisting of areas containing significant amount of MBs and areas where elongated ultra-fine grains prevail (Fig. 3c). X-Ray measurements show that dislocation density in the UFG Al5083 alloy tends to increase with increasing compressive strain (Table 1). It should be noted that evolution of the microstructure during plane strain compression testing is significantly different from that observed during cold rolling of the coarse-grained Al-Mg alloys that was reported in earlier publications [26, 27].

| Reduction ratio | CSD [nm] | $\rho$ [m^{-2}] |
|-----------------|---------|-----------------|
| Hot rolled + annealed | 0 | 480 ± 34 | 1.01 x 10^{14} |
| | 10% | 109 ± 12 | 9.85 x 10^{13} |
| | 30% | 100 ± 17 | 1.34 x 10^{14} |
| Ultra-fine grained | 50% | 90 ± 9 | 2.48 x 10^{14} |
microhardness values are in a good accordance with the flow stress values determined from plane strain compression testing. Such a significant increase of mechanical strength can be related mainly to dislocation hardening (due to significant increase of dislocation density in the material) (Table 1).

Based on the outcomes of the microhardness measurements in the samples undergone physical simulation, it can be concluded that further cold rolling of the UFG Al5083 alloy with reduction ratio of 75% should dramatically increase its mechanical strength.

The UFG Al5083 sample was successfully subjected to cold rolling to reduction ratio of 75% (Fig. 5). No cracking was observed in the cold rolled samples. The cold rolled Al5083 alloy showed microhardness 165±6.4 Hv. It should be noted this value is very close to that predicted by physical simulation (163±6.9 Hv). Preliminary tensile tests of the cold rolled sheet showed superior mechanical yield strength of 523 MPa, ultimate tensile strength of 555 MPa and elongation to failure of 5.2%. Current research activities are focused on further investigation of mechanical behavior of the UFG Al5083 sheet.

![Figure 4](image4.png)

**Figure 4.** Evolution of microhardness in the UFG Al5083 alloy during plane strain compression testing.

![Figure 5](image5.png)

**Figure 5.** View of the UFG Al5083 strips after cold rolling with reduction ratio of 75%.

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