Influence of RCS on Al-3Mg and Al-3Mg-0.25Sc alloys

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Abstract. An influence of repetitive corrugation and straightening (RCS) was studied on Al-3Mg and Al-3Mg-0.25Sc alloys up to eight passes. Each pass consist of a corrugation and followed by straightening. This has resulted in introducing large plastic strain in sample, and thus led to formation of sub-micron grain sizes with high angle grain boundaries. These sub grain formation was eventually resulted in improved mechanical properties. The average grain size of Al-3Mg-0.25Sc alloy after 8 passes yielded to ~0.6µm. Microhardness, strength properties were evaluated and it suggests that RCS was responsible for high hardness values as compared to the as cast samples. The microhardness values after RCS were 105 HV and 130 HV for Al-3Mg and Al-3Mg-0.25Sc alloys, respectively. Similarly, ~ 40% improvement in tensile strength from 240 MPa to 370 MPa was observed for Al-3Mg and Al-3Mg-0.25Sc alloys after RCS process. Al-3Mg and Al-3Mg-0.25Sc alloys exhibited maximum strength of 220 MPa and 370 MPa, respectively. It is concluded that RCS process has a strong influence on Al-3Mg and Al-3Mg-0.25Sc alloys for obtaining improved mechanical properties and grain refinement. In addition to RCS process and presence of Al₃Sc precipitates in Al-3Mg-0.25Sc alloy had a significant role in grain refinement and improved mechanical properties as compared to Al-3Mg alloy.

Keywords: Al-Mg-Sc alloys; RCS; Microhardness; Strength; Plastic deformation

1. Introduction

Severe plastic deformation (SPD) procedures are attractive methods for producing ultrafine grained (UFG) metals in bulk form [1]. Aluminium alloys with magnesium as the major alloying element constitute a group of non-heat treatable with medium strength, high ductility, excellent corrosion resistance, and weldability. In Al based alloys, it is generally difficult to reduce the grain size below 10µm through the conventional recrystallization process following thermomechanical treatments [2]. But recently it has been shown that in Aluminum-Magnesium alloys, the Hall-Pitch relationship remains valid at sub-micron grain size [3]. Several super plastic aluminum alloys have been developed but Al-Mg based alloys belong to the group of most widely used for super plastic forming [4]. Wrought Al-Mg alloys are used as structural materials in marine, automotive, and aerospace applications. Scandium (Sc) is considered as one more promising alloying element in aluminium
alloys, and it has been shown that small Sc addition influences the microstructure and can improve the corrosion resistance and mechanical properties [5]. Further development in strength can be achieved through the ultra-fine grain (UFG) refinement by employing relatively less known plastic deformation process, like repetitive corrugation and straightening (RCS) [6], which can be potentially upgraded into a continuous process in comparisons to a well-known equal channel angular processing (ECAP) [7], equal channel angular extrusion (ECAE) [8], high pressure torsion methods (HPT) [9], accumulative roll bonding (ARB) [10], constrained groove pressing (CGP) [11] etc. The repetitive corrugation and straightening (RCS) is an alternative SPD technique that can not only create bulk nanostructured material free of contamination and porosity, but can also be easily adapted to large-scale industrial production. Daryoush Emadi et al. [12] have reported that grain refinement effect of Sc (up to 0.17 wt. %) the limited solubility of Sc in Al-rich matrix forms the Al$_3$Sc a ternary phase particle along with other alloying elements at inter dendritic region which gives the collective effect on the mechanical properties of A319 alloy. Tensile properties (UTS, YS, and % El) of A319 alloy drop by the addition of Sc in the T6 heat treated condition. J. Y. Huang et al. [13] for high purity copper bar (99.9% Cu) reported the RCS first time, they have observed the existence of non-equilibrium grain boundaries and equilibrium grain boundaries, and reduction in grain size from 750 µm to 0.50 µm. In RCS process, a work-piece is repetitively bent and straightened without significantly changing the cross section geometry of the work-piece, during which large plastic strains are imparted into the materials, which leads to the refinement of grain size and homogeneity in the microstructure and also increases hardness and strength of the processed alloy [14]. A basic RCS cycle consists of two steps: corrugation and straightening. The corrugation is carried out in an corrugation die which is the discontinuous version of RCS process, and further to continue with the cycle the straightening is accomplished by pressing the corrugated work-piece between two flat platens. Thus this impedes dynamic recovery at lower deformation and consequently improves the grain refinement in bulk metals.

The present work reveals the effect of RCS on mechanical properties of Al-3Mg and Al-3Mg-0.25Sc material processed by repetitive corrugation and straightening method. Al-3Mg and Al-3Mg-0.25Sc bars of required dimensions were processed for 8 passes using specially designed and developed corrugating with straightening setup.

2. Experimental materials and procedure

Al-3Mg and Al-3Mg-0.25Sc alloys were selected as starting materials and its composition is shown in Table1.

| Alloys          | Composition wt. % |
|-----------------|-------------------|
|                 | Mg    | Si    | Fe    | Cu    | Zn    | Mn    | Ti    | Cr    | Sc    | Al    |
| Al-3Mg          | 3.2   | 0.08  | 0.16  | 0.02  | 0.025 | 0.003 | 0.03  | 0.002 | ---   | Bal.  |
| Al-3Mg-0.25Sc   | 2.96  | 0.09  | 0.15  | 0.02  | 0.002 | 0.003 | 0.02  | 0.001 | 0.25  | Bal.  |

Al-3Mg and Al-3Mg-0.25Sc alloys were homogenised by heating in the furnace at 450°C temperature and then cooled in the furnace about 12 h. In the present study the RCS process is done on Al-3Mg and Al-3Mg-0.25Sc alloys of dimension 85×12×12 mm$^3$ size. Samples were bent in a corrugated die and then straightened in the flatten die. Samples were processed for 1 to 8 passes either rotation between passes or by rotating the samples by 90°, each pass is repetitive bent (corrugated) and straightened. The RCS process imparts high plastic strain in the material when the sample subjected up to eight pass, thereby it leads to improvement in the grain refinement of both the alloys and subsequently increase in the microhardness and strength in the samples, but in case of Al-3Mg-0.25Sc the refinement of grain size, microhardness and strength better because the scandium plays very important role in grain refinement and other properties compared to Al-3Mg alloy. A schematic representation of RCS process is shown in figure 1.
As cast and RCS processed samples of Al-3Mg and Al-3Mg-0.25Sc alloys, Vickers’s microhardness was recorded along longitudinal direction on each sample using a METATEK hardness tester. Measurements were recorded at incremental distance of 10mm using a load of 0.5kgf and a dwell time for each measurement of 10sec for each indentation. The tensile strength test for as cast and processed alloys were carried out using FIE Make Universal Testing Machine, UNITEK 9550.

The as cast microstructure of Al-3Mg and Al-3Mg-0.25Sc alloys were observed through optical microscopy, scanning electron microscopy and the RCS processed samples were cut at the edge region for TEM studies for Al-3Mg-0.25Sc alloy. The TEM of JEOL JEM 2100 instrument operating at 200 kV was used. The disks of 3 mm were punched from the RCS sample and then mechanically ground below 1 µm and electro jet polished, etched using a per chloric acid-ethanol mixture.

3. Results and discussion

3.1 Microstructural studies

The light images of as cast Al-3Mg and Al-3Mg-0.25Sc are shown in figure 2. In both cases, the grain size is equiaxed. However, Fig.3b exhibits fine Al3Sc precipitates. The average grain size of Al-3Mg alloy in as cast condition is ~80 µm and substantially reduced to ~28µm after addition of Sc. This result clearly demonstrates that Sc addition has significant grain refining effect on Al-3Mg alloy. To understand the distribution of precipitates in Al-3Mg-0.25Sc alloy and to compare with Al-3Mg alloy, SEM studies are made and electron images are shown in figure 3 a, b.
Al-Mg materials derive their strength primarily from solid solution strengthening by Mg, which has a substantial solid solubility in aluminum. However, in order to obtain strength levels approaching the regime of the precipitation hardening alloys, high Mg levels are required. Such high levels of Mg pose processing challenges and can increase the susceptibility of the alloys to stress corrosion cracking. The addition of Mg influences the stacking-fault energy and thus the strength, the recovery and the recrystallization characteristics of Al. The main reason for increasing strength is the formation of Al-Mg intermetallic particles which reinforce the alloy. Some of the corrosion studies on Al-Mg alloys suggest that increasing the Mg content will add to strength. However, when Mg% is raised above ~4%, the corrosion resistance of the alloy will gradually decrease. As a result, the present work is limited to 3Mg content in Al-Mg system. The compositional analysis of Al-3Mg alloy has been checked through EDX results (EDX pattern not shown here). The results confirm that Mg content 3.15%. However, the chemical analysis showed 3.1 wt. %. This is due to that the minor alloying elements such as Fe, Si were not considered during EDX results and while totalling to 100%, higher content of Mg (3.15) is obtained.

Aluminum forms a thermodynamically stable Al$_3$Sc phase with scandium additions. Sc has a very low solid solubility in Al, approximately 0.4 wt. % at the eutectic temperature. For this reason, solutionizing near the eutectic temperature followed by quenching to re-precipitate fine, dispersed Al$_3$Sc particles would be largely ineffective. Fine Al$_3$Sc precipitates that are coherent with the matrix are expected to contribute to the alloy strength through dislocation particle interactions, as is the case in nickel-based super alloys. Addition of magnesium provides both solid solution strengthening and increases the lattice parameter of the aluminum matrix, which provides a better match with Al$_3$Sc and further decreases the driving force for coarsening of the Al$_3$Sc particles. An addition of Sc content and its influence on Al alloys is studied elsewhere and clearly showed that 0.25 wt. % is sufficient for obtaining optimum mechanical properties [15]. It is well known that a scandium addition is effective in promoting grain refinement and a resistance to recrystallization due to the formation of nanoscale Al$_3$Sc precipitates that enhance the elevated temperature properties [15]. Furthermore, processing by RCS produces even greater hardness in the Al–Cu–Sc alloy with an average hardness value of ~180 Hv after RCS through 4 passes when a rotation of 90° is included between each pass. Although the Al–Cu–Sc alloy responded well to RCS processing with a considerable strength improvement up to four passes, attempts to continue the processing to higher numbers of passes were unsuccessful because the sample developed cracks so that further processing led to failure.

During RCS, heavy plastic strain induced in each successive pass. To obtain a homogeneous structure with high angle grain boundaries, the number of RCS processes was given up to eight. The grain size is reduced from micron to submicron levels and subsequently responsible for increased hardness and strength values. Figure 4 shows the development of the microstructure in Al-3Mg-0.25Sc alloy after RCS eight pass with rotation of the sample.
Figure 4. TEM images of Al-3Mg-0.25Sc alloy after 8 RCS passes, a) without rotation and b) with rotation.

Figure 4a and 4b shows that the grain sizes are refined to the sub-micrometre level during RCS process up to 8 passes for Al-3Mg-0.25Sc alloy. Figure 4a represents TEM image of Al-3Mg-0.25Sc alloy without any rotation and a few elongated grain can be seen. The average grain size is ~720 nm. In case of same alloy processed with RCS up to 8 passes with rotation is responsible for ultra-fine grain structure with equiaxed grain structure. The average ultrafine grain size was ~600 nm. It is also observed in figure 4b that Al₃Sc precipitates (indicated by arrows) in the range of 90-110 nm are uniformly distributed in the Al matrix.

3.2 Microhardness measurements

The mechanical properties of the two experimental alloy such as Al-3Mg and Al-3Mg-0.25Sc are discussed. The hardness measurement was taken in as cast and RCS processed alloys.

Figure 5. Variation of microhardness in as cast condition and RCS processed Al-3Mg and Al-3Mg-0.25Sc alloys

The effect of a grain size reduction due to RCS and Sc addition to the Al-3Mg alloy is shown in Figure 5. The grain size of the cast Al-3Mg-0.025Sc alloy was refined to 600 nm with the addition of 0.25% Sc which is very significant considering the small amount of alloy addition. It is also interesting to note that the RCS process alone is capable of refining the grain size of the Al-3Mg-0.25Sc alloy from 28 to 0.6 µm and also it improves the strength values. Hardness measurements are considered over length of 90 mm corresponding to the maximum length of the sample. For alloy Al-3Mg as cast condition the hardness was ~ 40 Hv and in the RCS processed up to eight pass with...
rotation exhibits 105 Hv due to strain hardening effect in the alloy as shown in the Fig.5. Similarly, in case of as cast Al-3Mg-0.25Sc alloy exhibited a hardness of ~ 45 Hv and in the RCS processed sample, the hardness values are increased to ~ 130 Hv. These results demonstrate that higher level of hardness in RCS processed alloys is basically due to refinement in grain size and large plastic strain imparted in the samples and presence of Al3Sc precipitates.

3.3 Tensile strength

Table 2 shows the tensile strength values of Al-3Mg and Al-3Mg-0.25Sc alloys before and after RCS process.

| Alloys          | Before RCS (MPa) | After RCS (MPa) |
|-----------------|------------------|-----------------|
|                 | UTS   | YS    | UTS   | YS    |
| Al-3Mg          | 150   | 110   | 220   | 165   |
| Al-3Mg-0.25Sc   | 240   | 175   | 370   | 250   |

It can be seen from this table that UTS value of Al-3Mg alloy is increased from 150 MPa to 220 MPa which corresponds to an increase of ~ 40%. Similarly the YS value is also increased from ~ 110 to ~ 165 MPa. In the case of the Al-3Mg-0.25Sc alloy, the reduction in grain size was responsible for increased UTS value from ~ 240 to ~ 370 MPa which corresponds to an increase of ~ 50%. It is also understood that the ultrafine-grained structure of the Al-3Mg-0.25% Sc alloy is thermally more stable by comparison with the Al-3Mg alloy due to the finer precipitates which are highly stable and introduce Zener pinning of grain boundaries and hence improved mechanical properties.

4. Conclusions

RCS processing through eight passes reduces the grain size from ~8000 to ~800 nm for Al-3Mg alloy. For Al-3Mg-0.25Sc alloy also, the grain size reduced from ~2800 to ~600 nm and increases the yield stress and the ultimate tensile strength by factors of ~2 by comparison with the as-cast Al-3Mg alloy. The reduction in grain size in Al-3Mg is due to solid solution strengthening of Mg in Al-Mg system. Further reduction in grain size in Al-3Mg-0.25Sc alloy is due to the grain refinement effect arising from the presence of fine Al3Sc precipitates. Processing of Al-3Mg-0.25Sc samples by RCS showed further improvement in microstructure and tensile properties as compared to the Al-3Mg alloy after similar RCS process.

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