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Mechanical property enhancement due to plastic deformation prior to peak-age hardening in an Al-Mg-Si aluminium alloy

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Abstract

The effect of plastic deformation prior to peak age-hardening on mechanical properties of an Al-Mg-Si-Cu aluminium alloy was investigated. The alloy was prepared by DC casting and then hot extruded into flat strips of 3 mm in thickness, which were immediately quenched after extrusion to obtain a supersaturated solid solution state. The extruded and quenched flat strips were either left naturally-aging for a controlled short period of time or pre-aged before deformation. Plastic deformation was performed at room temperature by cold rolling or stretching to a strain of 4% or 8%. Hardness measurements and tensile testing were performed to obtain the mechanical properties of the alloy under different processing conditions. Scanning electron microscopy techniques, including electron backscattered diffraction (EBSD), were conducted to characterize microstructural features such as the dislocation density and crystallographic texture. Experimental results showed that the combination of pre-ageing and deformation by stretching resulted in a significant increase in the mechanical properties after the final peak ageing treatment. The interactions between dislocations and pre-ageing induced solute clusters and fine precipitates during deformation are considered to have increased the dislocation density in the material and promoted heterogeneous nucleation during final age hardening treatment.

Keywords

Al-Mg-Si-Cu alloys, Plasticity, Dislocation, Precipitation Hardening, EBSD.

1. Introduction

The effect of work hardening on 6xxx aluminium alloys has been well studied [1]. Its application in the processing of aluminium alloys has become common practice in manipulating properties based on intended purpose [2]. 6xxx aluminium alloys are also age-hardenable and thus a thermomechanical process, which comprises an ageing and a work hardening step or any combination of them, can be used to further enhance the mechanical properties of these alloys, such as strength and ductility [3]. Each step can be altered in order to give advantageous properties and thus understanding the effect of processing conditions and material performance at each step can aid in optimising the process. There have been studies that have looked into the effect of medium and high strain deformation via cold rolling [4] and severe plastic deformation [5], etc. However, these studies are not directly relevant or applicable in a manufacturing scenario. Since in manufacturing processes such as panel forming, the degrees of deformation are relatively low, it is therefore important to carry out observations of low strain deformation and understand how these small strains affect the final mechanical properties of these alloys directly or through thermomechanical processing. Recent work by Ryen et al. [6] and Zhong et al. [7] investigated the effect of pre-aging on the work hardenability of a 6xxx series aluminium alloy. In their work they showed how this pre-ageing step is advantageous through formability tests.

In a manufacturing setting, 6xxx aluminium alloys are commonly used to manufacture automotive parts and components given their lightweighting potential. They are formed to take the shape of the desired parts. Since this process introduces mechanical deformation to the material, if the contribution of these kinds of deformations to the final mechanical properties are well understood, the forming of the vehicle parts can be considered and incorporated into the entire thermomechanical process of production. This study aims to investigate the effect of this thermomechanical treatment on the mechanical properties of 6xxx aluminium alloys and correlate these enhancements to microstructural features and texture changes.

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2. Experimental details

An Al-Mg-Si-Cu aluminium alloy, provided by Constellium as 3 mm thick extrusion flat strips, was used as the starting material in this study. The alloy was prepared by DC casting and homogenization, and then solution heat-treated on the press followed by water quenching. The extruded alloy has a fully recrystallised grain structure and was left at room temperature for 48 hrs before any artificial ageing treatment was applied. In the designed thermomechanical processing schedule, a pre-ageing treatment was carried out at 170°C for 4 h. The pre-aged alloy was then deformed by either cold rolling or stretching to strain levels of 4% and 8%, respectively, before being finally peak aged, which is termed as the full process. The stretching was applied using an Instron 5569 Testing machine and rolling on a ϕ300×150 two-high rolling mill.

For hardness testing, the samples were cold mounted with the normal direction (ND) – extrusion direction (ED) exposed to allow hardness testing across the transverse direction (TD). Five indents were made per sample and the hardness was averaged over the five samples with the standard deviation being calculated from these datapoints. The hardness testing was carried out on a Wilson 432SVD Vickers Hardness Tester. The tensile samples were cut out of the extruded strip along the extrusion direction and were machined to have a gauge length of 50mm (see dimensions in Figure 1). They were tested on an Instron 5569 Tensile Tester. Five (5) samples were tested for each condition to allow an average and standard deviation to be calculated.

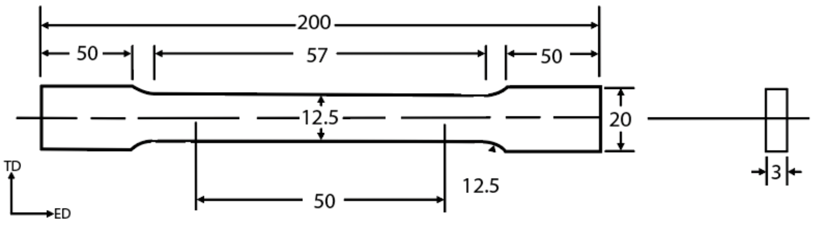

Figure 1. Tensile sample geometry; all numbers are in mm.

For microstructural examination, the samples were prepared following standard metallographic procedures and electropolishing. Electropolishing was conducted at -30°C for 60 s in a solution of 30% nitric acid in methanol. Microstructural examinations were carried out using scanning electron microscopy (SEM), including electron backscattered diffraction (EBSD). The SEM system used was the Zeiss Supra 55-VP FEGSEM. For EBSD measurements, the operating accelerating voltage was 20KV with an aperture of 120um and a working distance of 15mm. The system was set to high current. The step size used for all measurements was 0.13µm. For all micrographs and EBSD maps presented in this paper, the viewing direction is TD with ED (or rolling and stretching direction) vertical and ND horizontal.

3. Results

3.1 Ageing Kinetics

A preliminary ageing treatment was performed at 170°C for various times of up to 72 h to determine the conditions for pre-ageing and peak ageing. The result is shown in Figure 2.

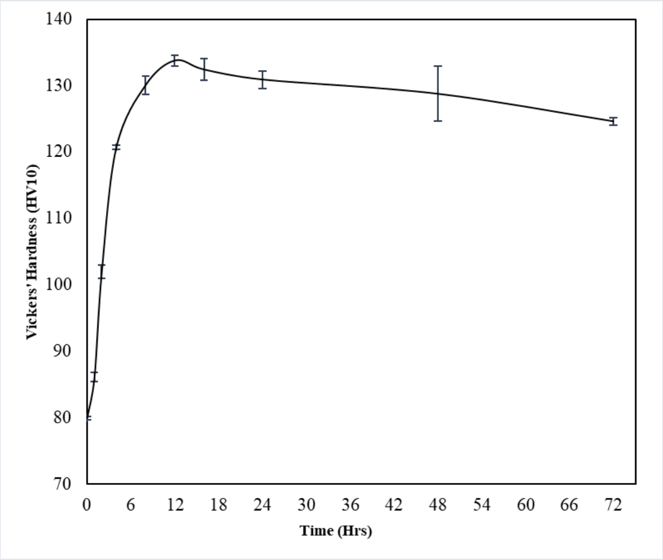

Figure 2 – Hardness response of Al-Mg-Si-Cu alloy at 170°C.

It can be seen that at the peak hardening time, the hardness was measured to be 133.76 HV10. The pre-ageing condition, which is an underaged state, was selected as the point when the hardness was about the half of the peak hardness, with a hardness of 120.72 HV10.

The pre-ageing condition was selected based on the hardness curve at 170ºC close to the mid-point in hardness increase due to precipitation hardening. The medium increase in hardness represent the point where there is sufficient interactions between precipitate/clusters and dislocations but there is solute left in solution to provide further hardening after deformation. At low hardness increments the density of precipitates/clusters are low to provide much interactions even though there is sufficient solute in solution while at higher hardness increments there is enhanced interactions between precipitates/clusters but there is limited solute available for further hardening.

3.2 Mechanical performance

Hardness was measured for the as-extruded material and samples subjected to various thermomechanical processing. This allowed the comparison to be made between mechanical performance under different processing conditions, highlighting the effect of the thermomechanical processing as given in Table 1. It can be seen that the application of just stretch or roll deformation results in a moderate increase in hardness, approximately 5.6% for 4% and 13.8% for 8% for both deformation modes, respectively. For the as-extruded alloy, there was no apparent difference in strengthening effect between stretching and rolling.

Table 1. Vickers Hardness results at various processing states.

Condition	Strain Mode	Strain Level, %	Hardness (HV10)
T4 *	None	0	64
Preaged	None	0	121
Peak Aged	None	0	135
T4 + D	Stretch	4	68
T4 + D	Stretch	8	73
T4 + D	Roll	4	68
T4 + D	Roll	8	73
Full Process	Stretch	4	142
Full Process	Stretch	8	143
Full Process	Roll	4	135
Full Process	Roll	8	137

*T4 state is the condition with limited natural ageing without any thermal or mechanical processing after solution heat treatment and quenching; Pre-aged condition: 170°C; Peak ageing condition; Full Process includes pre-ageing, deformation and the final peak ageing.

Tensile testing was performed to investigate the effect of deformation on the mechanical response of the alloy. Figure 3 shows typical engineering stress-strain curves for the alloy in the as extruded, pre-aged and peak aged states, and after the full process. A summary of the tensile testing results is given in Table 2. The results showed that the application of 8% strain via cold rolling and stretching resulted in an increase in strength, compared to the peak aged condition (4.9% increase for rolling and 15.5% for stretch in yield strength), in most cases with limited decrease in ductility. For the same amount of strain, stretching displayed more significant effect in strengthening than rolling.

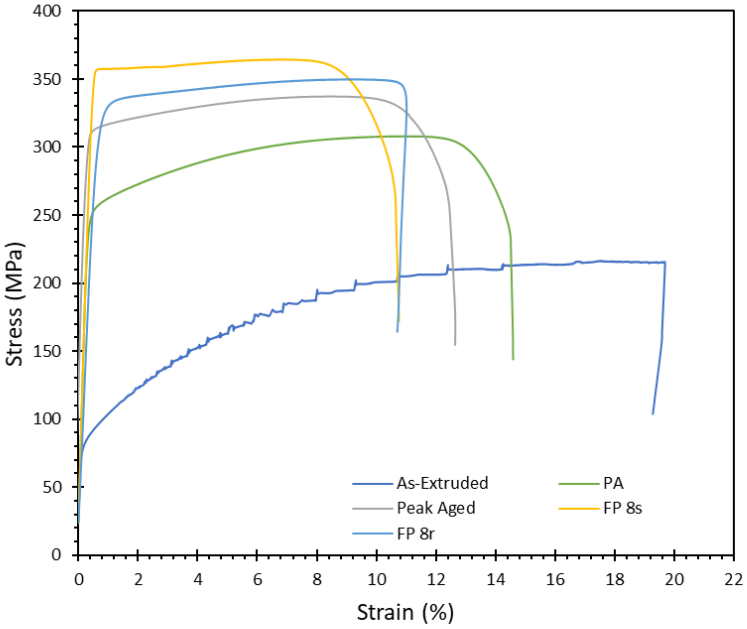

Figure 3. Tensile engineering stress-strain curves for the alloy as-extruded, pre-aged and peak aged and after the full process. PA in the figure being the Preaged condition, FP 8s being the Full Process with 8% Stretch and FP 8r, the Full Process with the 8% Roll.

Table 2. Tensile testing results showing the yield strength (YS), ultimate tensile strength (UTS), uniform elongation and elongation at break for the alloy subjected to different thermomechanical processing.

Temper	**Strain Mode**	**Strain Level**	**YS (MPa)**	**UTS (MPa)**	**Uniform Elongation (%)**	**Elongation at Break (%)**
T4	None	0	88.0	211.2	17	19
Preaged	None	0	249.1	307.3	11	15
Peak Aged	None	0	309.0	334.4	8	13
T4 + D	Stretch	4	173.9	251.7	17	19
T4 + D	Stretch	8	186.2	228.7	11	15
T4 + D	Roll	4	168.8	255.1	15	19
T4 + D	Roll	8	178.0	253.4	13	16
Full Process	Stretch	4	337.9	349.3	6	11
Full Process	Stretch	8	356.9	364.5	5	10
Full Process	Roll	4	303.8	331.5	7	12
Full Process	Roll	8	324.2	348.0	6	11

3.3 Deformation structure

The deformation structure of the alloy after rolling and stretching was examined under SEM using electron back scattered imaging, with the main concern being the homogeneity of the rolled sheets. During stretching, material flow was unconstraint apart from a small part near the end of the sheet for gripping. In rolling there can be a slight strain gradient from sheet surface to the centre due to surface friction [8], but at low strain levels it is not considerably manifested in the sheets. The SEM analysis showed that for the strain levels applied for this work, deformation occurred uniformly across the sample thickness for both stretching and rolling. Fig 4a shows that the image quality (IQ) in each grain is identical as there is no deformation in the material. Grain subdivision occurred in the 8% rolled sample as shown in figure 4b, and the initial grain structure is changed through introduction of strain and therefore some fragmentation within the grains can be seen as a result. However, the variations in contrast are localised and the global deformation structure is uniform as there is no image contrast gradient across the whole structure.

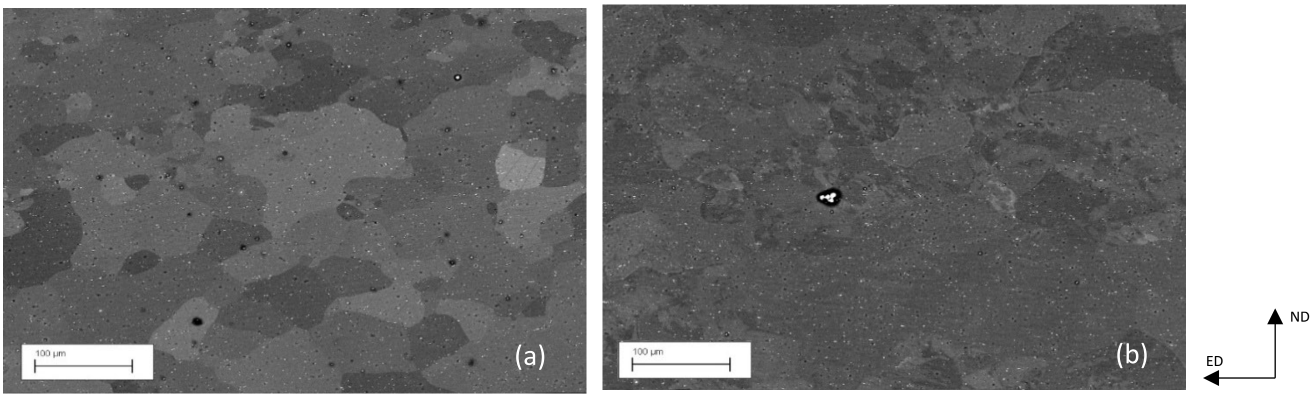

Figure 4: Back Scattered electron micrographs: a) as-extruded and b) 8% cold rolling.

Figure 5 shows the image quality (IQ), inverse pole figure (IPF) and Kernal average misorientation (KAM) EBSD maps of the starting material in the as-extruded condition as the reference for the undeformed state. It can be seen from the EBSD maps that the grain structure is equiaxed and dominated by grains with an orientation close to <001> direction. The misorientation within individual grains is low although there is a variation in contrast in the KAM map due to the application of 0.4% stretching after extrusion for straightening and noises during data acquisition.

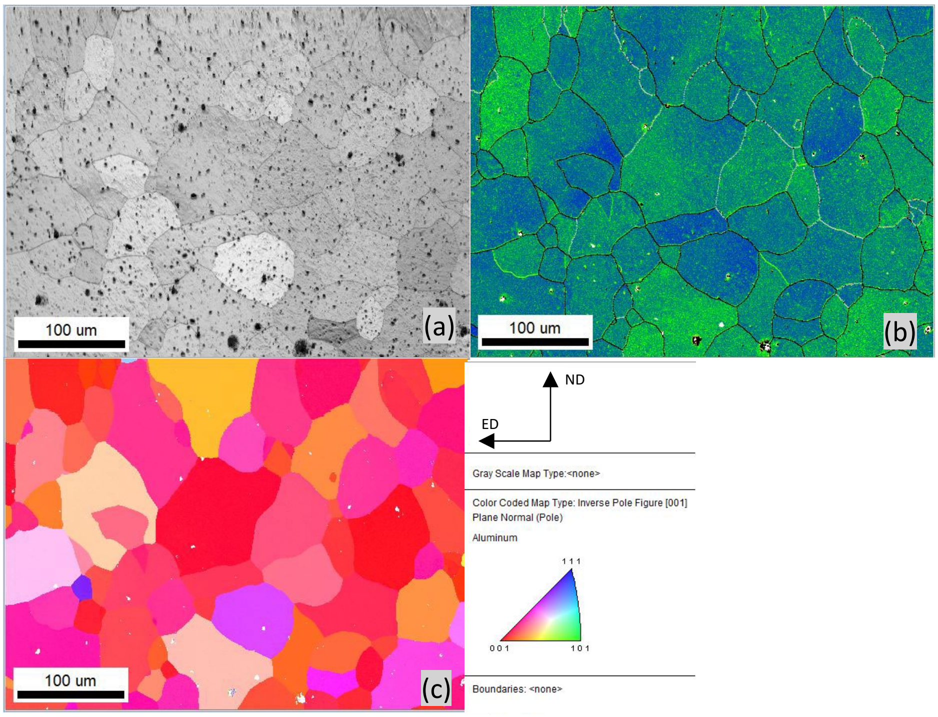

Figure 5: EBSD maps showing the microstructure for the as-extruded alloy: a) IQ, b) KAM and c) IPF.

The KAM maps in Figure 6 show local misorientations for the sample subjected to pre-ageing and 8% stretching (Figure 6b), in comparison to the sample stretched to 8% without pre-ageing (Figure 6a). The only processing difference between the samples is pre-ageing. We observe that pre-ageing has led to an increased dislocation density as these misorientations are substantially caused by the storage of dislocations during deformation [9].

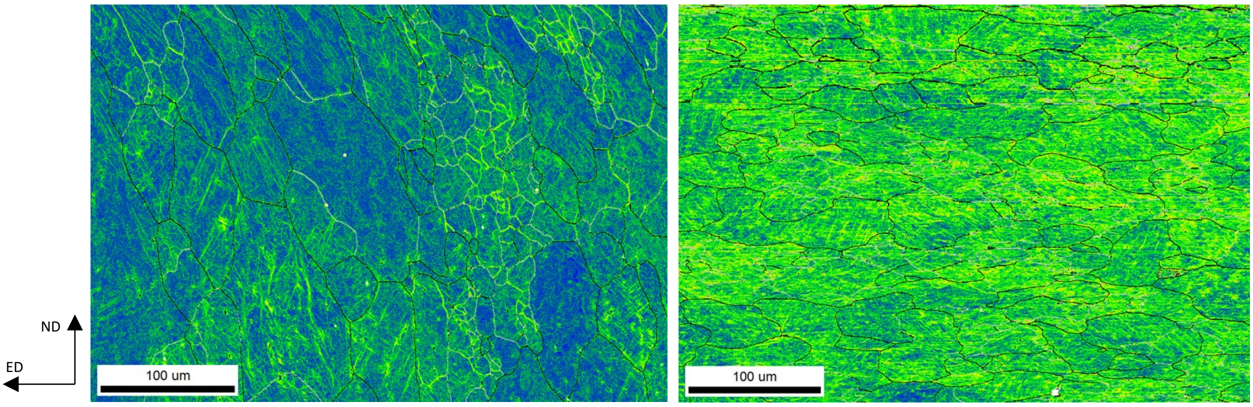

Figure 6: EBSD KAM maps showing the effect of pre-ageing on the development of dislocation structure during deformation: a) as extruded alloy 8% stretching and b) pre-aged 8% stretching.

4. Discussion

For the as-extruded alloy, the effect of the age hardening treatment alone was significantly greater than the effect of straining. This is mainly due to that the strain levels applied were low and that the alloy has a relatively low work hardenability (relevant data not included due to limit in space). Here there was no significant difference between rolling and stretching as deformation mode is concerned. Major differences occurred when the pre-ageing was added to the thermomechanical sequence. The combination of pre-ageing and deformation produced apparent positive impact on the mechanical properties after final ageing, in particular at the strain level of 8% by stretching. With 8% stretching after pre-ageing, an increase in the yield strength from 309 MPa to 357MPa was observed (Table 2), which is significant. This may be explained by the pre-ageing enhanced dislocation density during deformation as shown in Figure 5, which could promote heterogeneous nucleation and possibly increase the number density of precipitates in the final ageing treatment. It is reasonable to assume that the interactions between dislocations and the solute clusters and GP zones formed in the pre-ageing may play a strong role in dislocation multiplication and pinning during deformation. It is interesting to see that stretching showed more outstanding effect than rolling and the reason behind this is not fully clear. For the extruded strip samples selected, both rolling and stretching were essentially performed in plane strain deformation. A major difference is that during rolling material flow occurs only within the volume between the arc length of the rolls that are in contact with the sheet material, whereas in stretching material flow is not limited to any particular region. Another difference is that friction plays a role in the surface in rolling while it does not exist in stretching. These differences can affect the development of the crystallographic texture and the homogeneity of dislocation structures locally and globally. The texture components developed during rolling and stretching to 8% were determined from the EBSD data as shown in Figure 7, in comparison to those for the as-extruded condition. A strong cube texture was detected for the as-extruded condition (Figure 7a), which was slightly elongated along the rolling or stretching direction after deformation. However, no significant differences were found in the type of texture between rolling and stretching. More investigation is required to fully understand the different performances between rolling and stretching during this thermomechanical processing.

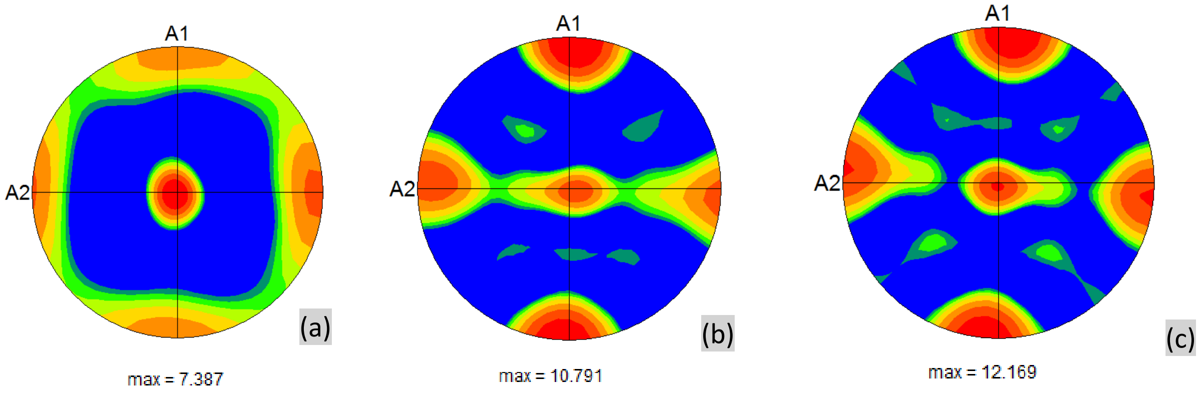

Figure 7. 001 pole figures (calculated from EBSD data) showing strong <001> texture for a) as-extruded, b) 8% stretched and c) 8% rolled (A1 – TD, A2 – ED).

5. Conclusions

In this study, the effect of small plastic deformation in a thermomechanical processing schedule designed for an Al-Mg-Si alloy has been investigated. It was found that:

1) The combination of pre-ageing and plastic deformation has significantly increased the mechanical properties of the Al-Mg-Si alloy. With the application of pre-ageing at 170°C for 4 h and 8% stretching, the yield strength of the material increased from 309 MPa obtained under conventional peak ageing treatment to 357 MPa under the same final ageing condition, with limited compromise in ductility.
2) The stretch deformation mode exhibited an advantageous performance in this pre-ageing followed by a cold deformation step in the studied thermomechanical processing schedule, although the exact reasons are unclear.
3) The enhanced mechanical performance can be attributed to the increased dislocation density due to the interactions between dislocations and pre-ageing induced clusters and fine precipitates during deformation, which is expected to promote dislocation multiplication and pinning while assisting heterogeneous precipitation nucleation in the final ageing.

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References

[1] F.J. Humphreys, P.S. Hurley. Orientation Averaging of Electron Backscattered Diffraction Data. J. Microsc. 201 (2001) 50

[2] J.F. Nye. Some Geometrical Relations in Dislocated Crystals. Acta Metall. Vol. 1 Issue 2 (1953) 153-162

[3] N.A. Fleck, M.F. Ashby. The Role of Geometrically Necessary Dislocations in Giving Material Strengthening. Scripta Materialia 48 (2003) 179-183.

[4] D.P. Field. Analysis of Local Orientation Gradients in Deformed Single Crystals. Ultramicrosc. 103 (2005) 33-39

[5] E.A. Mortsell. The Effect of Elastic Strain and Small Plastic Deformation on Tensile Strength of a Lean Al-Mg-Si Alloy. Metals (2019), 9(12), 1276.

[6] O. Ryen. Precipitation, Strength and Work Hardening of Age Hardened Aluminium Alloys. Mat Sci and Eng 89 (2015) 012013.

[7] H.Zhong. Influence of Pre-Aging on the Stretch Formability of Al-Mg-Si Automotive Sheet Alloys. Mat. Sci. and Eng. A, Vol. 697 (2017) 79-85.

[8] H.Liu. Effects of Different Tempers on Precipitation Hardening of 6000 Series Aluminium Alloys. Trans. Nonfer Met Soc China 17 (2007) 122-127.

[9]. D.P. Field, P.B. Trivedi, S.I. Wright, M. Kumar. Analysis of local orientation gradients in deformed single crystals. Ultramicroscopy 103 (2005) 33–39.