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Microstructural evolution and observed stress response during hot deformation of 5005 and 6022 Al alloys

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Abstract

A proper understanding of the relationships that connect processing conditions, microstructural evolution and mechanical properties is important for any materials scientist to improve productivity and product quality. The objective of this work is to experimentally and statistically analyze the effect of physically measurable features of the starting microstructure on the yield stress of 6022 Al alloy. Quantitative parameters obtained from microstructure characterization and stress analysis were analyzed by a multiple regression analysis technique to determine the relative influence of various microstructural parameters on the observed stress response. The geometrically necessary dislocation (GND) density was determined to be the most important measured parameter affecting the yield stress. Experimental and statistical analysis showed a linear relationship between the yield stress and square root of the GND density.

Keywords: Statistical analysis; Aluminum alloys; Microstructure evolution; Geometrically necessary dislocations

1. Introduction

Modeling the microstructure evolution and the macroscopic stress response during deformation has been a focus of research for many years. There are two challenging tasks that must be overcome to achieve a good degree of success in this field. One is to perform accurate microstructural analysis of the deformed material and to develop quantitative parameters that are representative of the microstructural heterogeneity. The other is to develop a physically based model that incorporates these microstructural parameters.

Many attempts on microstructural modeling in processing of Al alloys use the internal state variable approach, which relates the property of interest with the microstructural variables [1–5]. The generalized constitutive equation for the flow stress response during thermomechanical processing of Al alloys can be written in the form:

$$\sigma = f(\dot{\varepsilon}, T, S_i) \tag{1}$$

where $\dot{\varepsilon}$ is strain rate, T temperature and S_i includes a number of parameters describing chemistry, dislocation structure, particle morphology, etc. Some of these microstructural parameters evolve with strain at a rate which is governed by the characteristics of the starting microstructure and externally imposed deformation conditions. Such evolution equations for precipitates and dislocation structures as a function of processing conditions are included in various yield strength models developed for Al alloys [5–8]. However, the complexity of real microstructure at the subgrain scale and the interactions between microstructural features are often ignored. Physically based constitutive models which incorporate the observed microstructural features should offer the potential for predictive capability rather than interpolation of experimental results, as is common with phemenologically based models. Common measures of microstructures such as the size and shape of grains, phase fractions, and texture have been incorporated in various constitutive models; however the effect of dislocation substructure or lattice curvature has not often been used in the constitutive models except in a phenomenological framework.

The present study is an effort to incorporate experimentally determined density of geometrically necessary dislocations (GNDs) into a statistically formulated model of deformation response. GNDs are defined as dislocations needed to accom-

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modate the difference in crystal lattice rotation produced by different slip system activity from point to point within a given grain [9–12]. Recently GNDs have received lot of attention due to experimental evidence showing dependence of length scale on the material behavior; however no specific attempt has been made to relate the experimentally measured GNDs to the stress response. It is possible to get an estimate of GND density from spatially specific orientation measurements such as those obtained from automated EBSD analysis. Some other microstructural parameters that describe misorientation within a grain are grain orientation spread (GOS) and grain average misorientation (GAM). GOS is a measure giving the algebraic average of the misorientation angle between all points (whether adjacent or otherwise) within a given grain. GAM is a second measure that gives the algebraic average of the misorientation between all points and their nearest neighbor measurement points. Even though all of the above three microstructural parameters (GND density, GOS and GAM) provide a scalar number that describes the misorientation within a grain, computation of GND density provides additional information about the spatial distribution of densities of individual dislocation types that are required to support lattice curvature during deformation. Orientation spread within a grain arises, when stressed, because of differential rotation of crystallite regions (both in terms of angle and direction) within a grain. Development of in-grain orientation spread affects the mean free paths of dislocations and therefore alters the stress required for deformation. Other microstructural features measured were grain size, inter-particle spacing, volume fraction of precipitates, and radius of precipitates.

Following are the major areas of investigation in the current paper:

i. Compare the experimentally observed microstructural evolution during annealing and deformation of 5005 and 6022 Al alloys.
ii. Investigate the effect of processing parameters such as strain, strain rate and temperature on the stress–strain behavior and microstructural evolution.
iii. Investigate the effect of GND and other microstructural parameters on the observed stress response.
iv. Develop a statistical model that predicts the yield stress of 6022 alloy as a function of experimentally measured microstructural features.

2. Experimental procedures

Flat samples with dimensions of 25 mm × 10 mm × 4 mm were cut from hot rolled plates of 5005 and 6022 Al alloys and annealed at 520 °C for 30 min. Three samples (to form each batch for hot deformation) were placed on top of one another and deformed using channel die compression such that the direction of material flow is parallel to the direction of rolling. Various combinations of processing parameters (i.e. temperature of 250, 350 and 450 °C, strain of 10%, 20% and 30%, and strain rate of 0.01, 0.1 and 1 s^{-1}) were applied during hot deformation to generate a variety of microstructures. Microstructural analysis on the hot deformed samples was done along the long transverse cross-section using a scanning electron microscope (SEM) equipped with EBSD. Texture analysis was performed using EBSD scans with a step size of 10 μm and the dislocation structure analysis was done using a step size of 0.2 μm. Backscatter electron imaging, which provides compositional contrast, was used to detect precipitates in AA6022. Image analysis software was used to characterize precipitates in AA6022 in terms of inter-particle spacing, area fraction and radius of precipitates. Three miniature sized dog-bone samples (gage length: 10 mm and thickness: 2 mm) were prepared from each batch of hot deformed samples and room temperature tensile testing was performed to failure with a constant crosshead speed of 0.005 in./s. Statistical regression analysis was performed using MINITAB-14.0 (a commercial statistical and graphical analysis software package from Minitab Inc.) with an input of parameters from microstructural characterization and stress analysis.

3. Results and discussion

The major focus of this paper is to quantitatively understand the influence of experimentally determined GND density on the observed stress response during deformation. We chose two commercially used Al alloys 5005 and 6022 for this study with a measured chemical composition shown in Table 1. It is known that in AA5005, Mg forms a substitutional solid solution with Al, whereas in AA6022 precipitates of Mg_2Si form in an Al matrix. In the following part of the paper, the first three sections are devoted to the comparison of experimentally observed microstructural evolution and stress–strain behavior of 5005 and 6022 alloys and the last two sections are devoted to the development of a yield strength model for 6022 alloy. The GND density is determined to be the major microstructural parameter, sufficient enough to represent all characteristics of dislocation structures, affecting the yield strength.

3.1. Characterization of starting materials

Hot rolled plates of 5005 and 6022 showed elongated grain structures with an average aspect ratio of 0.63, indicating preferential elongation of grains in the direction of rolling, which is expected during deformation. The dense dislocation substructure in both alloys can be evidenced from the high values of grain orientation spread and grain average misorientation. The average grain orientation spreads were 3.97° for 5005 alloy and 2.95° for 6022 alloy and the grain average misorientations were 2.37° for 5005 alloy and 1.97° for 6022 alloy. Both alloys were annealed at 520 °C for 30 min. The purpose of annealing was to recrystallize the microstructure and reduce the total dislocation content. The average grain orientation spread reduced to 0.91

Table 1
Measured chemical compositions (in wt.%) of 5005 and 6022 Al alloys

Alloy	Mg	Cu	Si
5005	0.7–1.1	0.05	0.3
6022	0.55	0.056	1.1

and grain average misorientation reduced to 0.84 after annealing for both alloys. This could be attributed to recrystallization phenomena, where dislocation cells are removed by the formation and growth of new, strain-free grains.

3.2. Effect of processing parameters

Industrial processing of metals and alloys requires application of a wide range of processing parameters such as strain, strain rate and temperature, which influence the microstructure evolution and the kinetics of deformation. Various approaches have been used in the past in the area of microstructural modeling with the length scales ranging from atomistic, to slip system activity, to grain structure and continuum level. It is important to have statistically reliable and experimentally verifiable information about the microstructural evolution and stress response as a function of processing parameters to validate such models. Such understanding is important to define mechanisms driving microstructural evolution during thermo-mechanical processing and can be applied to develop processes that produce materials with a microstructure just good enough for a desired application. This section discusses the effect of processing parameters on the stress response and texture evolution of both alloys.

Fig. 1 contains a plot of the Zener–Holloman parameter (temperature modified strain rate, $Z = \dot{\varepsilon}\exp(U/RT)$) versus flow stress obtained during channel die deformation for both alloys [13]. In the above equation $\dot{\varepsilon}$ is applied strain rate, R the gas constant, T the absolute temperature and the activation energies, U (obtained from commonly selected values for Al alloys) used for the calculation were 156 and 140 kJ/mol for 6022 and 5005 alloys, respectively. In accordance with earlier experimental observations and theoretical understanding, it can be seen from Fig. 1, that higher flow stress is observed for samples deformed at high Z (i.e. low T and high $\dot{\varepsilon}$). Also the plot describes the effect of alloy chemistry on the deformation behavior of the two alloys at high temperatures. Trivedi et al. [14] studied the room temperature small strain (up to 10%) deformation behavior of AA5005 and AA6022 and observed that AA6022

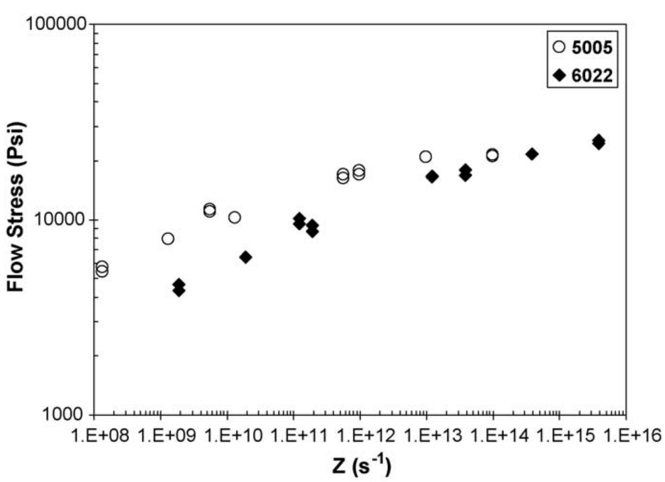

Fig. 1. Plot showing flow stress obtained during channel die compression as a function of Z for both 5005 and 6022 alloys.

generated a higher increase in dislocation structure (and correspondingly flow stress) with strain than AA5005. However a reverse effect is observed from Fig. 1 during hot deformation in the sense that, at low values of Z (i.e. at low $\dot{\varepsilon}$ and/or high T) AA5005 showed higher flow stress than AA6022. This suggests that under the current experimental conditions, solid solution hardening observed in AA5005, is more effective in resisting deformation, especially for samples deformed at low Z values (i.e. at high T and low $\dot{\varepsilon}$), than precipitation hardening in AA6022. The difference in stress–strain behavior during channel die compression between the two alloys can be due to the difference in starting microstructure and/or their alloy content. Since both alloys were fully annealed before hot deformation, AA6022 has a distribution of coarse overaged particles, which are easily bypassed by dislocations and hence do not significantly, contribute to strengthening. Solute atoms in AA5005 are in solid solution even during high temperature deformation and therefore possess higher ability of resisting deformation at higher temperatures.

Fig. 2 contains the (0 0 1) pole figures showing predominantly cube texture of AA6022 after hot deformation at various processing parameters. In contrast AA5005 showed (Fig. 3) an ND rotated cube texture under similar deformation conditions due to the presence of large grains having a rotated cube orientation in all the samples. Since both the alloys were subjected to similar deformation conditions, the difference in texture evolution is due to the difference in alloy content of the two alloys.

3.3. Effect of microstructure

As mentioned earlier mechanical response of a polycrystalline material strongly depends on the details of microstructure. This section is devoted to the influence of starting microstructure (specifically density of GND) on the different stress responses observed during tensile testing. Texture evolution after channel die deformation was similar under all deformation conditions for both alloys – predominantly cube for 6022 alloy and predominantly rotated cube for 5005 alloy. Therefore the effect of texture on stress response during subsequent tensile deformation was neglected.

Fig. 4 shows the variation in the 0.2% yield stress, obtained during room temperature tensile testing, with square root of GND density. Calculation of GND density was done from EBSD scans of step size 0.2 μm using the normal equation lower bound approach proposed by El-Dasher et al. [15]. It can be seen that with increasing GND density the stress required for plastic deformation for both alloys increases. Such a direct relationship between dislocation density and flow stress has been suggested by various modelers [16] with an equation of the type

$$\sigma = \sigma_0 + \alpha GbM\sqrt{\rho_f} \quad (2)$$

where σ is the macroscopic stress response, σ_0 the friction stress, M the Taylor factor, G the shear modulus, α constant and ρ_f is the density of forest dislocations. Forest dislocations in the above equation include both GNDs and statistically stored dislocations (SSDs), formed by statistical mutual trapping of

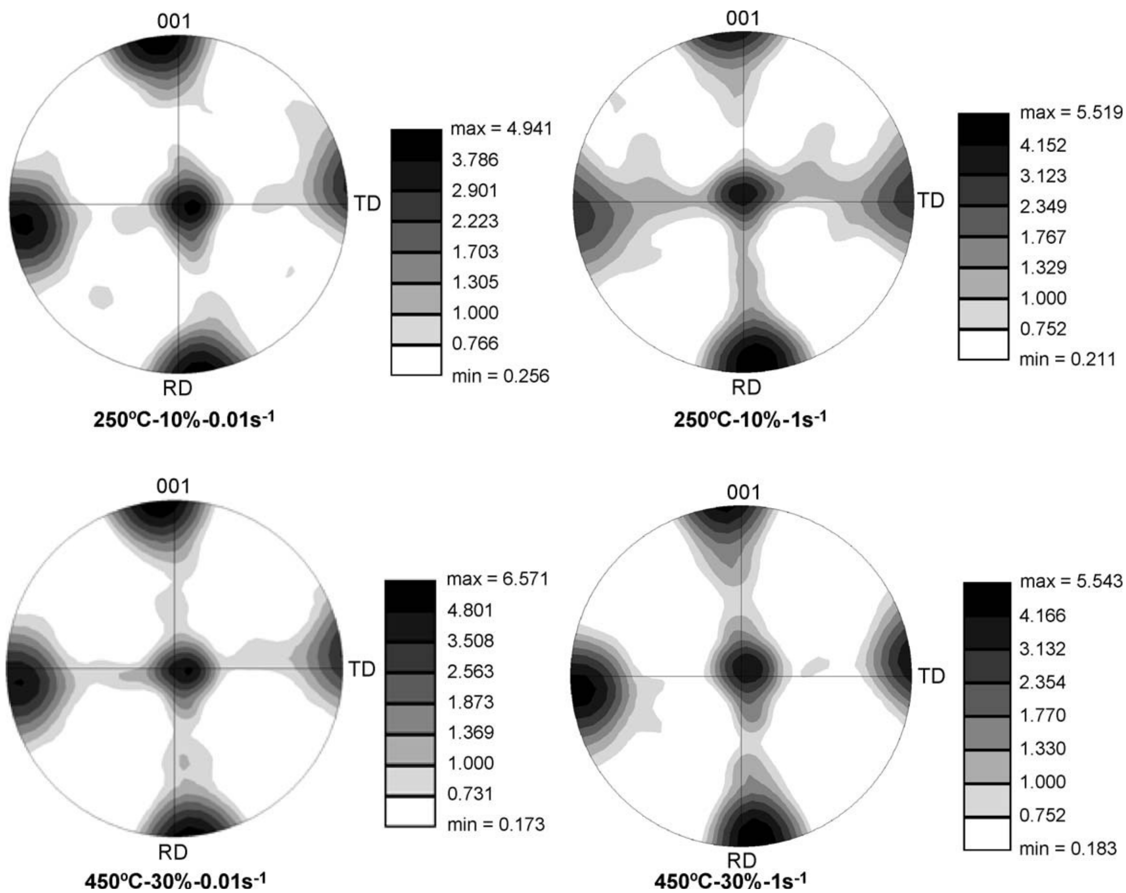

Fig. 2. (0 0 1) Texture pole figures of 6022 alloy showing predominantly cube texture for samples deformed under various processing conditions.

dislocations such as dislocation dipoles. In the current analysis we have ignored the effect of statistically stored dislocations on the stress response because of difficulties associated with determining the content of SSDs. It is known that at high temperatures Al alloys tend to form well-organized cell structures separated by dislocation cell walls creating small misorientations across them. Dislocations along those cell walls develop lattice curvature and contribute to GNDs. In the current experimental conditions most of the microstructure consists of those cell walls (and consequently the microstructure should be dominated by GNDs) and therefore it may be reasonable to ignore the effect of SSDs on the stress response. In the present study it is assumed that SSDs and GNDs scale similarly, so measuring one or the other will yield the desired relationships. The effect of GNDs on the stress response is evident in Fig. 5, which shows the stress–strain curves obtained during RT deformation for both alloys plotted as a function of GND density.

Influence of GNDs on stress response was particularly studied by Hansen and Juul Jensen [17] and they suggested that strengthening due to GND follows a Hall–Petch type equation:

$$\sigma_{\mathrm{GNB}} = K_1\sqrt{Gb/D_{\mathrm{GNB}}} \tag{3}$$

where K_1 is constant with units of square root of stress (Ex. $(\mathrm{MPa})^{1/2}$) and D_{GNB} is the spacing between geometrically necessary boundaries (GNBs). TEM investigation showed that GNBs are produced by GNDs at medium to high strain and that they have preferred crystallographic orientation. With increasing strain, misorientation across GNBs increases and the spacing between them decreases due to accumulation of GNDs. It is also experimentally verified that these boundaries can have large misorientations across them (10–15°) and are capable of restricting glide. Such observations clearly explain the effect of GND density on the stress response observed in the current analysis (Figs. 5 and 6).

3.4. The model

Many experimental studies on metals and alloys suggest that the macroscopic flow strength of the material is given by

$$\sigma = \sigma_0 + \sigma_{\mathrm{d}} \tag{4}$$

where σ_0 is the friction stress and σ_{d} is the strength contribution due to dislocation structure. In the case of precipitation hardening systems (such as in AA6022 in the present analysis) or solid solution hardening systems (such as AA5005), there should be an additional term describing their effect in Eq. (4). A linear addition of strength contribution due to the matrix, precipitates and solid solution has been suggested by various modelers [18–20]. Depending on the type of systems (precipitation hardening or solid solution hardening), the flow stress relationship

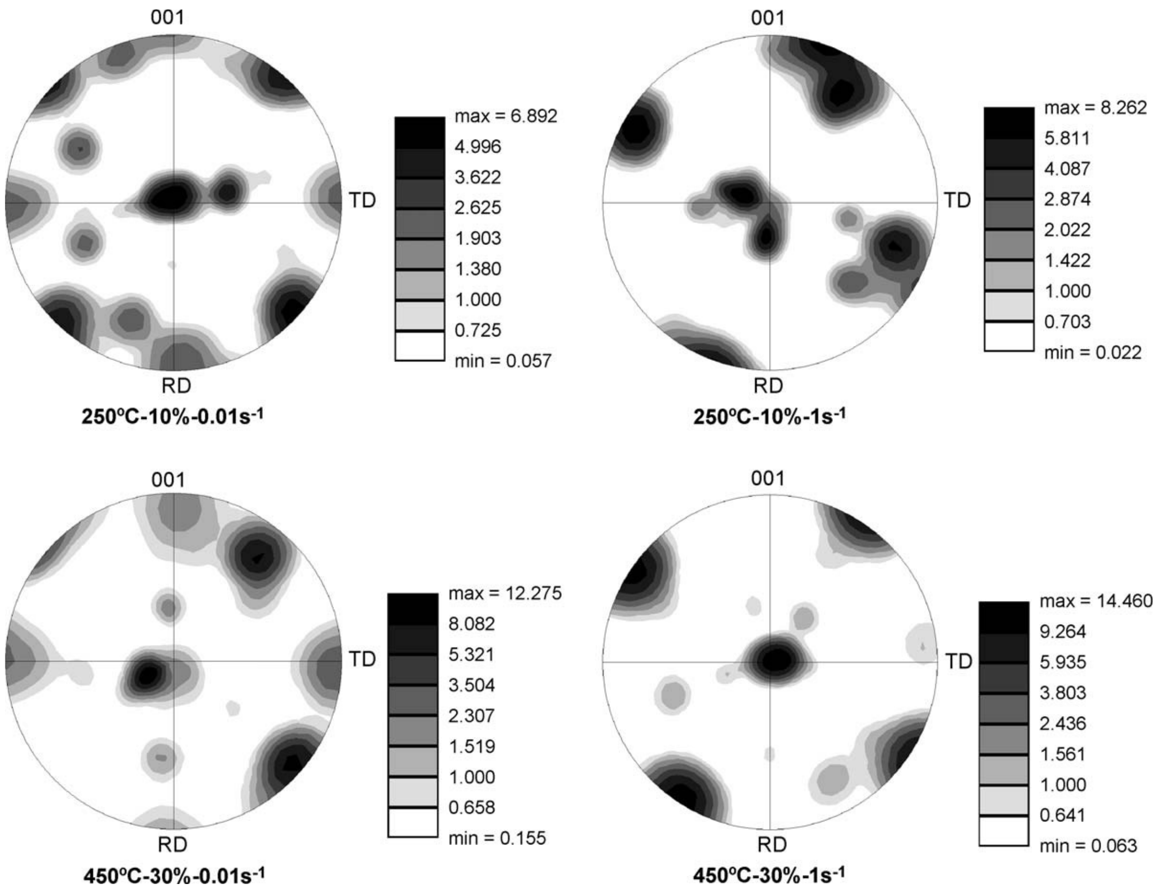

Fig. 3. (0 0 1) Texture pole figures in 5005 alloy showing predominantly rotated cube texture for samples deformed under various processing conditions.

then becomes

$$\sigma = \sigma_0 + \sigma_d + \sigma_p \text{(for precipitation hardened systems)} \tag{5}$$

and

$$\sigma = \sigma_0 + \sigma_d + \sigma_{ss} \text{(for solid solution strengthened systems)} \tag{6}$$

where σ_p and σ_{ss} are the strength contribution due to precipitates and solid solution, respectively. It is known based on a large amount of experimental data of metals and alloys that the strengthening due to dislocation structure is related to the dislocation density by the relation

$$\sigma_d = \alpha(\dot{\varepsilon}, T) MGb\sqrt{\rho_T} \tag{7}$$

where α is a material parameter, G the shear modulus, b the Burger's vector and ρ_T is the total dislocation density [16]. As mentioned earlier the total dislocations can be divided into two different categories: statistically stored dislocations (SSDs) and geometrically necessary dislocations (GNDs) and so Eq. (7) becomes

$$\sigma_d = \alpha(\dot{\varepsilon}, T) MGb\sqrt{\rho_{SSD} + \rho_{GND}} \tag{8}$$

Also, based on current experimental observations, SSDs and GNDs scale similarly and therefore assuming that the effects of SSDs and GNDs are proportional, we get

$$\rho_{SSD} + \rho_{GND} = \rho_{GND}(p+1) \tag{9}$$

where p is a proportionality constant. Including $\sqrt{(p+1)}$ in the material constant α, Eq. (8) becomes

$$\sigma_d = \alpha MGb\sqrt{\rho_{GND}} \tag{10}$$

To describe the effect of precipitates (σ_p) we are considering the model proposed by Deschamps and Brechet [21] such that the strengthening due to particles follows the relationship

$$\sigma_p = \frac{M\bar{F}}{bL} \tag{11}$$

where $\bar{F}$is the mean obstacle strength and L is the average inter-particle spacing. The above equation assumes homogeneous distribution of particles such that dislocations have to pass through all the obstacles to cause deformation. Depending on the initial characteristics of the precipitates, $\bar{F}$ and L will evolve with aging time and processing conditions. For coherent fine particles, the obstacle strength $\bar{F}$is dependent on particle radius and for coarse and overaged particles, obstacle strength $\bar{F}$ is constant. Deschamps and Brechet further developed Eq. (11) for the case of all precipitates being bypassed by the dislocations (which is the case in AA6022 in the current work) such that

$$\sigma_p = \frac{kMGbf_v^{1/2}}{\bar{R}} \tag{12}$$

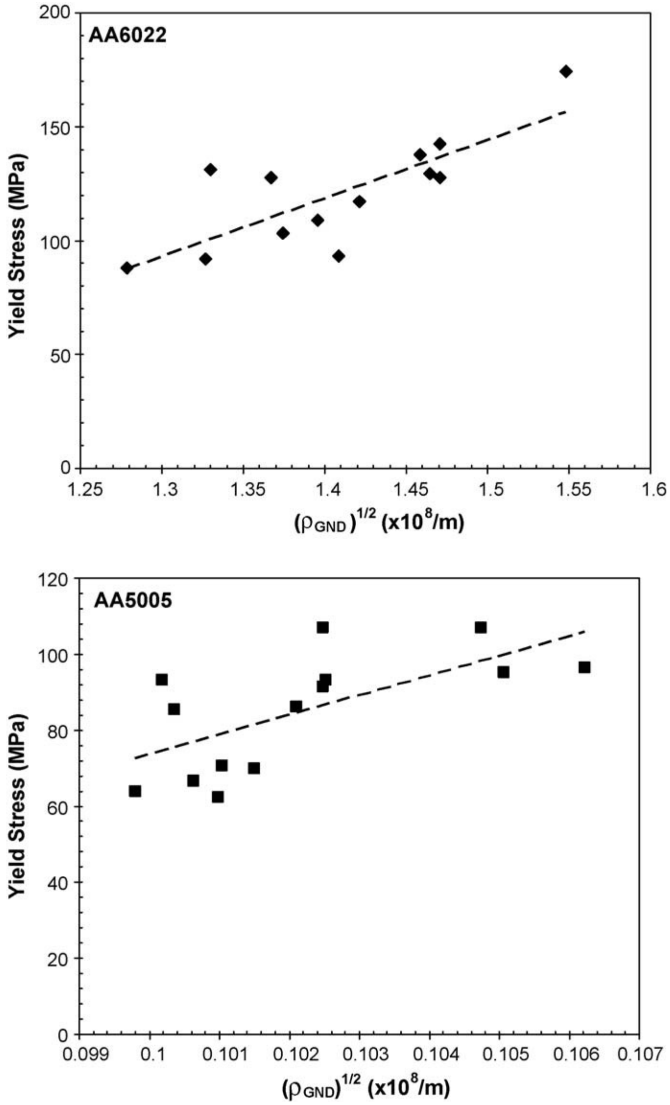

Fig. 4. Plots showing variation in 0.2% yield stress (MPa) obtained during tensile testing with square root of the GND density ($\times 10^8\ \mathrm{m}^{-1}$) for (a) 6022 alloy and (b) 5005 alloy.

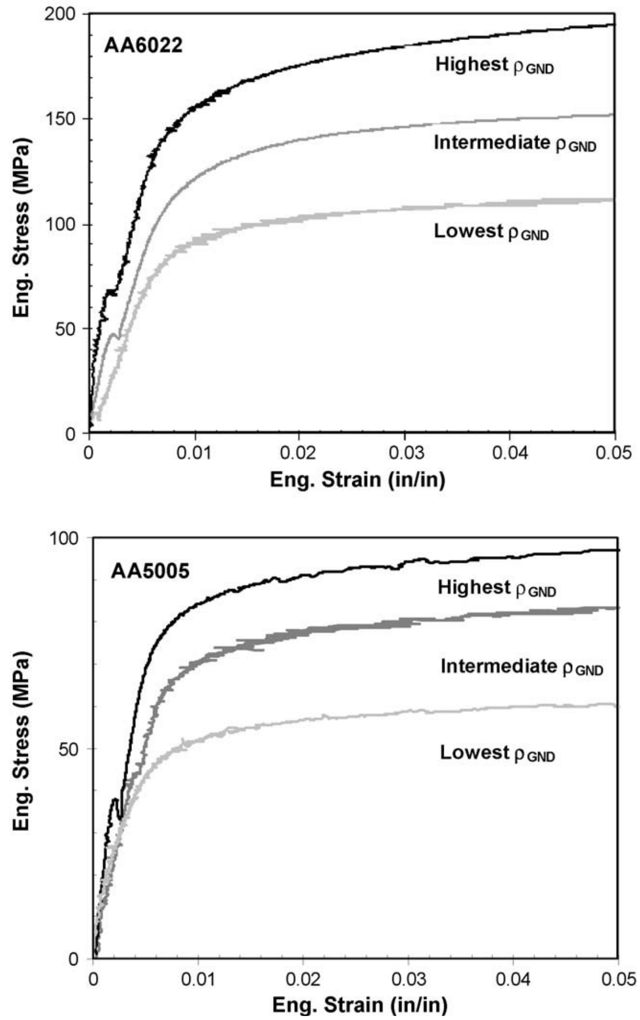

Fig. 5. Stress–strain curves obtained during tensile testing for samples with different GND density.

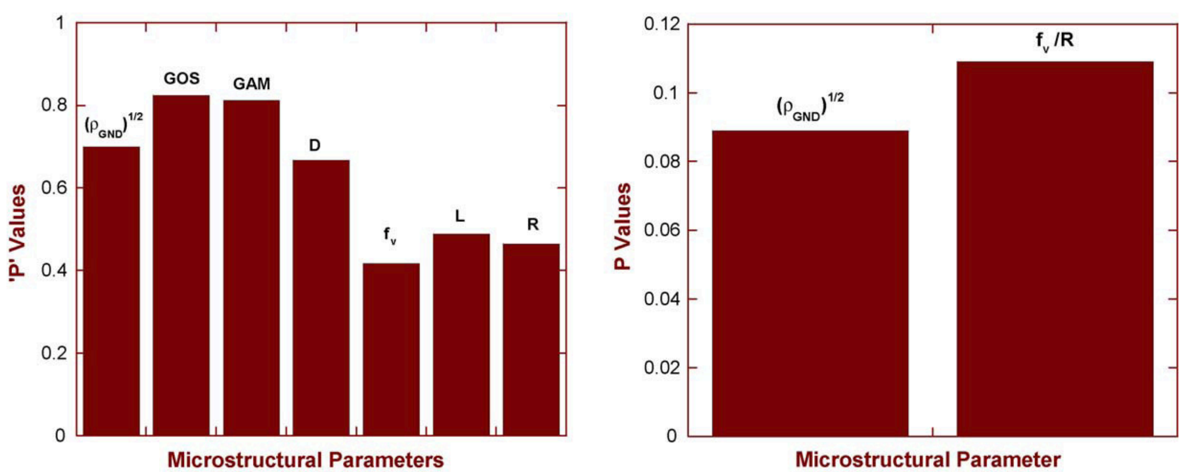

Fig. 6. Summary of regression analysis showing influence of microstructural parameters on the yield stress of 6022 alloy.

where k is constant (usually 0.6–0.7), G the shear modulus, f_v the volume fraction of precipitate particles, and $\bar{R}$ is the mean radius of precipitates. Incorporating the effect of dislocation and precipitate structures into Eq. (5), we get

$$\sigma = \sigma_0 + \alpha MGb\sqrt{\rho_{\mathrm{GND}}} + \frac{kMGbf_v^{1/2}}{\bar{R}} \tag{13}$$

It is known that the concentration of solutes in solid solution will decrease with an increase in volume fraction of precipitates (f_v). The precipitates observed in AA6022 are overaged, and therefore the matrix should contain a minimal amount of solute atoms in solution. Regardless of the solid solution content, these effects are included in the constant α and in f_v in Eq. (13).

3.4.1. Regression analysis

Microstructures of materials can be described by various structural parameters and each variable can potentially have a dominating effect on certain properties exhibited by the material. Therefore the selection of a microstructural variable of importance should depend on the desired property (response variable). One of the ways to extract the information about which microstructural features are dominant is by using statistical analysis. In the current paper we use multiple regression analysis to investigate the effect of various experimentally measured microstructural parameters on the response variable. Regression analysis is a method that can be used to quantify the relationship between two or more predictor variables (X) and a response variable (Y) by fitting a line or plane through all the points such that the fitted line or plane minimizes the sum of the squared deviations of the points from the fitted values [22]. The response variable chosen was 0.2% yield strength determined from room temperature tensile testing.

Various microstructural parameters measured were grain size, grain orientation spread, grain average misorientation, density of geometrically necessary dislocations, and some of the precipitates characteristics such as average inter-particle spacing, area fraction of precipitates, and mean radius of precipitates. A similar parametric study in single crystal Ni was performed by Horstemeyer et al. [23] using the analysis of variance (ANOVA) technique with an input of data from MD simulation. He divided various parameters such as crystal orientation, strain rate, temperature, deformation path, x, y, and z dimensions into different levels in the analysis. In the current paper processing parameters such as strain rate and temperature were not included in the analysis because stress parameters (forming response variable in the regression analysis) were obtained from room temperature and constant strain rate experiments. In the current regression analysis we included the actual values of quantitative parameters that describe different microstructural features rather than dividing them into various levels to allow more degrees of freedom (which is desired to obtain least error mean square).

3.4.2. Application to AA6022

When all the microstructural parameters such as grain size, density of GND, grain orientation spread, grain average misorientation, average inter-particle spacing, average particle radius and area fraction of precipitates were incorporated in the regression analysis, large 'P' values were obtained for all the parameters (Plot 6a). This indicates that such combinations of microstructural parameters fed into the analysis are ineffective in describing the stress response due to existing correlations between them. The matrix of correlation between various microstructural features was determined and it was observed that there exists a relatively strong correlation between density of GND and GOS, GOS and GAM, density of GND and inter-particle spacing. Various combinations of microstructural parameters were fed into the regression analysis to determine major parameters that influence yield strength. The regression equation that best defines the relation and the one that gave minimum 'P' values for corresponding microstructural parameters was

$$\sigma_y(\mathrm{MPa}) = -111 + 0.000001\sqrt{\rho_{\mathrm{GND}}}(\mathrm{m}^{-1}) + 0.000045\frac{\sqrt{f_v}}{\bar{R}}(\mathrm{m}^{-1}) \tag{14}$$

It is interesting to note that the regression equation obtained for the yield stress (Eq. (14)), by choosing the parameters that give minimum 'P' values, is similar to the one proposed by Deschamps et al. in Eq. (13). 'P' values obtained during the regression analysis (Plot 6b) suggest that the GND density explains more variation in the yield strength than the characteristics of particles. This in fact is believable and precipitates do not significantly affect the yield stress because precipitates in AA6022 are overaged and so dislocations can bypass them easily. The strength contribution due to precipitates is directly related with volume fraction of precipitates suggesting that with increase in volume fraction of precipitates in the matrix, the number of obstacles to dislocation motion also increases, which eventually increases the stress required for plastic deformation. In the current analysis the strength contribution due to dislocation structures is considered to be only due to the GND density and we have ignored the effect of other dislocation structures such as cell-size, cell-shape, SSDs, cell-wall misorientation, etc. However it can be seen from the statistical analysis that under the current experimental conditions, GND density alone could satisfactorily represent the overall strength contribution due to dislocation structures. We attempted to use dislocation cell-size, to represent D_{GNB} (in Eq. (3)) in the regression analysis. Cell-size was measured using EBSD analysis by changing the grain definition to 0.5° misorientation and substituted for GND density in the regression analysis. A large 'P' value was obtained for cell-size in the regression analysis indicating that the cell-size was not a major parameter influencing the yield stress of the alloy. This could be because of fairly constant cell sizes, in the range of 2.0–3.0 μm, obtained in all hot deformed samples. It is possible to obtain such an equilibrium cell-size during hot deformation and so the effect of applied stress during deformation will increase misorientation across cell-walls due to accumulation of dislocations, keeping cell-size relatively constant.

3.4.3. Determination of coefficients

If we consider the average Taylor factor as 2.73 (calculated for grains with cube orientation since the texture of 6022 alloy is predominantly cube), shear modulus (G) of 6022 alloy as 26 GPa and magnitude of Burger's vector as 2.86×10^{-10} m, the material constant α is determined to be 0.05. The material constant α is a function of strain rate and temperature and when strengthening of materials is due to pure dislocation–dislocation interaction (without considering the effect of precipitates), the value of α is proposed to be 0.3 [6,16]. As expected, we obtained a lower value of α because of additional strength contribution due to dislocation–precipitate interactions included in the current analysis. Various yield strength models (regions of small strain) developed for Al alloys used M value as 2, and they suggested that since the material is in early stages of plastic deformation, grains are not fully constrained and so the homogeneous stress hypothesis can be applied [21,24]. Thus, reducing the value of M to 2.0 will further increase the value of the material constant α to $\approx$0.1.

4. Conclusions

In general, the yield strength model developed for 6022 alloy is similar to the model proposed by Deschamps and Brechet [21]. Statistical analysis and experimental observations showed a linear relationship between yield stress and square root of GND density. It was observed that GND density alone can sufficiently represent the strength contribution due to dislocation structures and samples with higher GND density observed higher flow stress. The current work supports the trend of increasing interest in the scientific community towards quantifying the GND density in deformed samples and incorporating it into a physically based model. However it is known that microstructure evolves during the process of plastic deformation and an additional equation(s) describing the evolution of GND density should be determined. Even though no attempt has been made in determining the evolution of GND density, a general form for the evolution of GND can be written of the form

$$\frac{\partial \rho_{\mathrm{GND}}}{\partial \gamma} = f(\sigma, \dot{\varepsilon}, T, S_i) \qquad (15)$$

Also the strength contribution due to dislocation structures depends to some extent on the interaction between dislocation and precipitates, which in turn is dependent on the characteristics of precipitates. In the current analysis we have distribution of coarse overaged precipitates and in order to capture the dislocation–precipitate interaction effectively it is important to have different precipitate morphologies in the matrix. Future work in this effort should also include determination of the strength contribution due to precipitates responsible for peak of hardness.

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