

Modeling and experimental study of static recovery and mechanical behavior of AA5052 alloy during cold-working and subsequent annealing

M. Seyed Salehi¹ and N. Anjabin^{2*}

¹Department of materials science and engineering, K. N. Toosi University of Technology, Tehran, Iran
²Department of materials science and engineering, School of engineering, Shiraz University, Shiraz, Iran

Abstract: In the present study, the mechanical behavior of AA5052 aluminum alloy during cold-working and subsequent isothermal annealing in a temperature range of 225-300 °C was investigated, using the uniaxial tensile test data. It is found that by increasing the annealing time and temperature, the material yield strength is decreased. The microstructural investigations of the annealed samples show that the grains are elongated and there is no evidence of recrystallization. Hence, recovery is the main restoration phenomenon during the annealing treatment. The work hardening behavior of the alloy during cold working is modeled using a dislocation density based modeling approach and the softening behavior of the deformed samples during subsequent annealing is modeled by applying a kinetics equation relating the yield strength to the annealing parameters. The kinetics equation is a nonlinear differential equation and it is solved numerically by employing Runge-Kutta-Fehlberg (RKF) integration scheme which is coupled with Gauss-Newton nonlinear optimization technique to obtain the material constants of the model. The numerical results are validated using the experimental flow data.

Keywords: Kinetics of static recovery, AA5052 aluminum alloy, Cold working, Isothermal annealing, Nonlinear regression

1. Introduction

During the plastic deformation of aluminum alloys, two competing phenomena, i.e. work hardening and work softening occur concurrently. These phenomena are affected by the initial structure of the deforming material and deformation conditions such as deformation temperature, strain rate and amount of plastic strain [1, 2]. The presence of second phase particles, grain boundaries, solute atoms and dislocation forest act as barriers to the movement of mobile dislocations, which results in dislocation accumulation. The storage of dislocation is the main reason of work hardening, during cold working. Moreover dislocation accumulation increases the internal stored energy of the material and provides the driving force for the dynamic restoration process during deformation (i.e. dynamic recovery) which results in work softening. Kocks and Mecking [3] and then Estrin and Mecking [4], have proposed a model based on dislocation evolution to study these phenomena by relating the variation of average dislocation density to the imposed plastic strain.

The strain energy stored during cold working may result in the occurrence of static restoration processes such as, static recovery and/or static recrystallization, during subsequent annealing treatment [1]. In the alloys with high stacking fault energy, such as aluminum and its alloys, recovery predominates instead of recrystallization during the annealing treatments and so, investigation of the static recovery phenomenon in these materials is important. During the static recovery, the internal stored energy and the material flow stress are decreased gradually by decreasing the density of point defects, and annihilation and rearrangement of dislocations [2]. In order to experimentally quantify the kinetics of static recovery, the changing of some

physical or mechanical properties of the material such as electrical resistivity, hardness or yield strength are considered [2, 5-9]. Here, the variation of yield strength during annealing treatment is used to estimate the amount of recovery progress, $R(t)$ [2]:

$$R(t) = \frac{\sigma_d - \sigma(t)}{\sigma_d - \sigma_a} \quad (1)$$

where, σ_d , σ_a and $\sigma(t)$ are yield stress of as-deformed material, fully annealed material and material after annealing duration t . Different semi-empirical and phenomenological models were proposed for prediction of recovery kinetics in the metals and alloys. Kuhlmann *et al.* [7], presented a logarithmic relationship between annealing time and recovery progress parameter. In this model, it is assumed that the stored energy is increased during deformation due to dislocation storage and then degraded during static recovery by thermally activated mechanisms. Friedel and Smoluchowski [6], supposed that flow stress is equal to the internal stress of the dislocation structure in the crystal lattice and a similar logarithmic model proposed for recovery kinetics. Nes [8], revised the recovery models by taking into account the effects of dislocation density, internal stress between dislocations, annealing temperature and material properties. Verdier *et al.* [9], proposed a model for prediction of yield strength during static recovery of aluminum alloys on the basis of a relaxation of the internal stresses by thermally activated dislocation motion.

In the present study, the kinetics of recovery after cold rolling is investigated for AA5052 aluminum alloy. For this purpose, as-deformed samples are isothermally annealed at different temperatures and durations and then the yield strength of the annealed samples is measured. The flow behavior of material during cold-working are obtained by employing the Kocks-Mecking (K-M) [3, 4] constitutive equation, and the recovery progress during annealing are predicted by a mathematical kinetics model based on Verdier [9] recovery model.

2. Experimental Procedure

The chemical composition of AA5052 Al-Mg alloy used in this work is presented in Table 1. The as-received material was supplied in the form of rolled-sheet having thickness of 4 mm, and annealed at 550 °C for one hour to remove the metallurgical history and make a homogeneous microstructure. In order to study the work hardening behavior of the alloy, uniaxial subsize tensile specimens (gage length 25 mm, gage width 6 mm, and thickness 2 mm) were prepared from the annealed material according to ASTM E8M standard [10] and the tensile test is carried out at room temperature. To investigate the static recovery kinetics, the annealed materials were cold rolled to 30% reduction, and then tensile specimens were prepared in the rolling direction. The specimens were isothermally annealed at temperatures of 225, 250, 275 and 300 °C for different durations from 3 minutes to 3 hours in molten salt bath. The specimens were then quenched in cold water, and the uniaxial tensile test at room temperature, with a strain rate of $6.4 \times 10^{-3} \text{ s}^{-1}$ was done to measure the offset yield strength (0.2% proof strength). For microstructural investigations, the samples were characterized by optical microscopy after surface preparation and electroetching using the Barker's reagent (5ml HBF₄ and 200ml H₂O) [11].

3. Modeling Approaches

a) Cold work modeling

During the plastic deformation of the aluminum alloys, dislocation density is changed as results of competing between the rate of dislocation storage and dislocation annihilation due to dynamic recovery [1, 2, 12]. The effects of these phenomena on the dislocation density evolution can be described by the following equation proposed by K-M model [3, 4],

$$\frac{d\rho}{d\varepsilon_p} = k_1\rho^{\frac{1}{2}} - k_2\rho \quad (2)$$

where ρ is the average total dislocation density, ε_p is plastic strain, k_1 and k_2 are material constants. The first term in Eq. (2) implies the dislocation accumulation and the second term accounts for dynamic recovery. By integrating Eq. (2), the average total dislocation density can be obtained as a function of plastic strain.

$$\rho = \left(\frac{k_1}{k_2} - \left(\frac{k_1}{k_2} - \rho_0^{\frac{1}{2}} \right) \exp\left(\frac{-k_2 \varepsilon_p}{2}\right) \right)^2 \quad (3)$$

where, ρ_0 is the dislocation density of the fully annealed material. The dislocation contribution to the flow stress of the deforming material can be related to the dislocation density using the following equation:

$$\sigma = \alpha M G b \rho^{1/2} \quad (4)$$

where α is a positive constant, M is the average Taylor factor, G is the shear elastic modulus, b is the length of Burgers vector. The contribution of other strengthening mechanisms such as lattice resistance, solid solution hardening, second phase hardening and grain size effects on the flow stress are taken into account by adding σ_0 to the Eq. (4). By substituting the dislocation density from Eq. (3), the flow stress is obtained as:

$$\sigma = \sigma_0 + \alpha M G b \left(\frac{k_1}{k_2} - \left(\frac{k_1}{k_2} - \rho_0^{\frac{1}{2}} \right) \exp\left(\frac{-k_2 \varepsilon_p}{2}\right) \right) \quad (5)$$

Using Eq. (3) and (5), the average total dislocation density and the yield strength of the material after cold-working may be obtained.

b) Modeling of static recovery

Recovery is a thermally controlled process, where the dislocation motion and annihilation are mainly controlled by recovery temperature. The energy barrier for dislocation motion in crystal lattices depends on the dislocation resistance against slip. Hence, the energy barrier can be related to the yield stress during the annealing process. According to the model proposed by Verdier [9], the rate of yield stress variation ($\frac{d\sigma}{dt}$) during recovery may be obtained by the following equation.

$$\frac{d\sigma}{dt} = \frac{-2(1+\nu)\sigma^2 \nu_D}{G\alpha^2 M^3} \exp\left(\frac{-Q_r}{k_B T}\right) \sinh\left(\frac{\Omega_r \sigma}{k_B T}\right) \quad (6)$$

where k_B , ν , ν_D , Q_r and Ω_r are Boltzmann constant, Poisson's ratio, Debye frequency, activation energy and activation volume of recovery, respectively. According to this equation, rate of reduction of yield stress depends on the annealing temperature and the material yield strength. By increasing the annealing temperature and yield strength of as-deformed material, the rate of sub-structural changes during annealing treatment is increased and cause more decrease in yield strength. Using Eq. (1), the recovery progress is related to the rate of yield stress variation by;

$$\frac{dR}{dt} = \frac{-1}{\sigma_d - \sigma_0} \frac{d\sigma}{dt} \quad (7)$$

So,

$$\frac{dR}{dt} = \frac{2(1+\nu)\sigma^2 \nu_D}{G\alpha^2 M^3 (\sigma_d - \sigma_0)} \exp\left(\frac{-Q_r}{k_B T}\right) \sinh\left(\frac{\Omega_r \sigma}{k_B T}\right) \quad (8)$$

To calculate the rate of recovery progress by Eq. (8), the material constants Q_r and Ω_r should be determined. This differential equation is nonlinear and the material constants should be obtained using a numerical method. Here, the RKF method [13] is used to numerically integrate Eq. (8). Also, the Gauss-Newton method is applied to fit the model to the experimental data. Based on Eq. (8), the recovery progress is a function of Q_r and Ω_r parameters as well as annealing temperature and time.

$$R = R(T, t, Q_r, \Omega_r) = R(T, t, \boldsymbol{\beta}), \quad \boldsymbol{\beta} = [Q_r, \Omega_r]^T \quad (9)$$

To find the desired values of $\boldsymbol{\beta}$ in different annealing conditions, the sum squared error (SSE) which is the discrepancy between the predicted and measured values of recovery progress parameter, should be minimized. The SSE for different annealing conditions may be defined as follows:

$$SSE = \mathbf{r}^T \mathbf{r} \quad (10)$$

$$\mathbf{r}(\boldsymbol{\beta}) = \mathbf{R}(\boldsymbol{\beta}) - \mathfrak{R}$$

where \mathfrak{R} and \mathbf{R} are the measured and predicted recovery progress values for the material annealed at different conditions, respectively. The optimized values of Q_r and Ω_r may be obtained by minimizing the SSE relative to $\boldsymbol{\beta}$. To solve this nonlinear optimization problem, the Gauss-Newton method [13] is used. In this algorithm, first an initial guess was made for $\boldsymbol{\beta}$, and then the following iterative procedure is used to update $\boldsymbol{\beta}$.

$$\begin{aligned} \boldsymbol{\beta}^{n+1} &= \boldsymbol{\beta}^n + \lambda \Delta \\ \Delta &= -(\mathbf{J}^T \mathbf{J})^{-1} \mathbf{J}^T \mathbf{r} \end{aligned} \quad (11)$$

where λ is a positive number, and \mathbf{J} is the Jacobean matrix.

$$J_{ij}(\boldsymbol{\beta}^n) = \frac{\partial r_i(\boldsymbol{\beta}^n)}{\partial \beta_j} \quad (12)$$

Equation (11) is iteratively solved until the convergence criteria are satisfied. In order to use this optimization technique, the values of predicted recovery progress parameter, $\mathbf{R}(\boldsymbol{\beta})$, and Jacobean matrix at each iteration for different conditions of annealing temperature and duration are needed. The recovery progress parameter is obtained by using the RKF method with the initial condition of $R(T_i, t = 0, \boldsymbol{\beta}) = 0$. The Jacobean can be obtained numerically by employing the central difference scheme as follows:

$$\begin{aligned} J_{i1}(\boldsymbol{\beta}) &= \frac{\partial R(T_i, t_i, Q_r, \Omega_r)}{\partial Q_r} = \frac{R(T_i, t_i, Q_r + \delta Q_r, \Omega_r) - R(T_i, t_i, Q_r - \delta Q_r, \Omega_r)}{2\delta Q_r} + O(\delta Q_r^2) \\ J_{i2}(\boldsymbol{\beta}) &= \frac{\partial R(T_i, t_i, Q_r, \Omega_r)}{\partial \Omega_r} = \frac{R(T_i, t_i, Q_r, \Omega_r + \delta \Omega_r) - R(T_i, t_i, Q_r, \Omega_r - \delta \Omega_r)}{2\delta \Omega_r} + O(\delta \Omega_r^2) \end{aligned} \quad (13)$$

where δQ_r and $\delta \Omega_r$ are small positive values.

4. Results and Discussion

Figure 1 shows the stress-strain curve of fully annealed sample obtained from uniaxial tensile test at room temperature. Also, the data predicted by the K-M model are shown in this figure. The constants of work hardening (k_1) and work softening (k_2) terms in the K-M model are obtained by fitting on the experimental flow data of AA5052 alloy as 1.9×10^8 and 8.52, respectively. As could be seen from Fig. 1, a good agreement is found between the modeling results and the values obtained by the experimental test. So, the K-M model can be successfully applied to obtain the yield strength of as-deformed material, which is used to precisely estimate the initial condition of the cold-worked material before annealing. The experimental stress-strain curves of AA5052 alloy after 30% reduction by cold rolling, and isothermally annealed at different temperatures of 225, 250 and 275 °C for different annealing durations are shown in Fig. 2. According to these figures, a restoration phenomenon occurs during annealing and causes decreasing in flow stress and increasing in fracture strain of the samples. The microstructure of as-deformed and cold worked and annealed sample for one hour at 300 are depicted in Fig. 3. According to Fig. 3a, during cold working the grains are elongated in the rolling direction (RD). After annealing at 300 °C for an hour, the shape of the grains is still elongated as depicted in Fig. 3b. The non-recrystallized grain structure and changing of the material properties during annealing at the temperature below 300 °C indicate that the

recovery is the main restoration phenomenon. Hence, the recovery models can be used to predict the yield strength and recovery progress during static annealing treatment. Fig. 4 shows the variation of material yield strength obtained from uniaxial tensile test as a function of annealing temperatures and durations. From this figure, at a given annealing temperature yield strength is reduced by increasing the annealing duration. The kinetics of recovery accelerates by increasing the annealing temperature and it causes more decrease in yield strength. The materials constants were used in the kinetics models of Eq. (6) and Eq. (8) for prediction of flow stress and recovery progress are presented in Table 2. The material constants Q_r and Ω_r are obtained using the regression of yield strength predicted by Eq. (6) on the experimental data with the combination of Gauss-Newton optimization technique and RKF numerical integration method. In Fig. 5 variations of the root mean squared error and the activation energy and activation volume are shown during the model regression. The initial values used for the activation energy and activation volume are 160 kJ/mol and 15 b^3 , respectively. From Fig. 5, the converged data are obtained after about 14 iterations, which show the rapid convergence of the used nonlinear regression approach. The converged values for activation energy and activation volume for static recovery of AA5052 alloy are 194 kJ/mol and 18.98 b^3 , respectively. The determined activation energy in this study is close to the values reported by Verdier *et. al* [9] ($174\text{-}203 \text{ kJ/mol}$) and Poole *et. al* [14] (214 kJ/mol) for Al-Mg alloys. However, these values are rather higher than the activation energy of self-diffusion of aluminum [15] (122 kJ/mol), and the activation energy for the diffusion of Mg in Al [16]. The values of the activation energy for Mg drag in dislocation climb or glide mechanisms are in the range of 115 to 136 kJ/mol [16, 17]. In fact, Mg can decrease the stacking fault energy of aluminum and raise the apparent activation energy which has been discussed by Verdier *et al* [9]. Moreover, the activation energy of the dislocation mobility in Al-Mg alloys depends on the concentration of the drag element [18] and the plastic strain prior to the annealing [9].

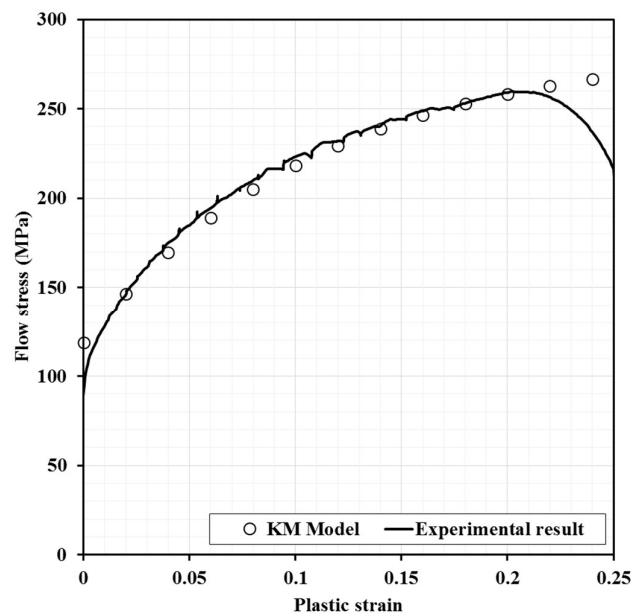


Fig. 1. Comparison between the stress-strain curve predicted by the K-M model and experimental result at room temperature.

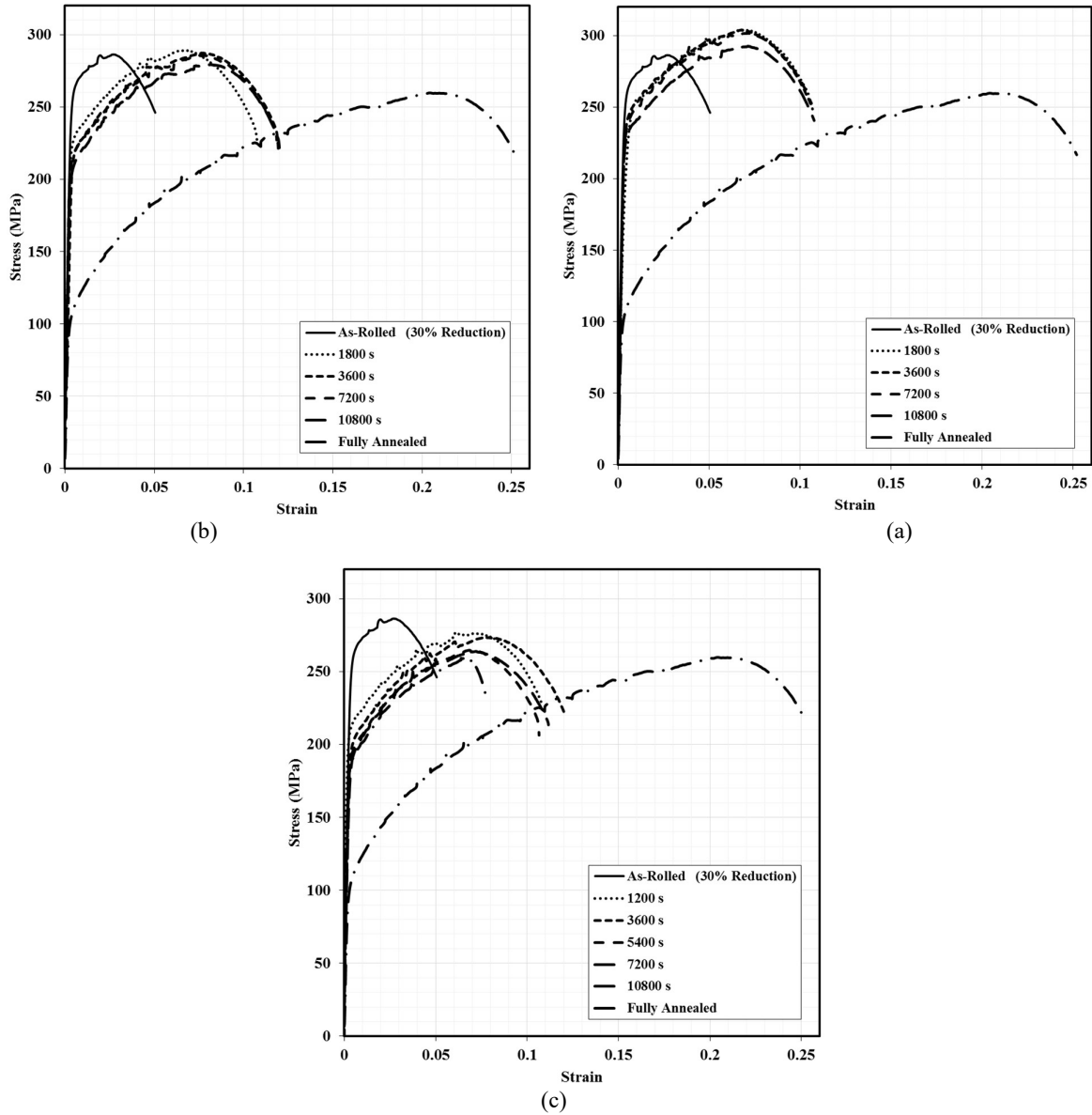


Fig. 2. Experimental true stress-strain curves of AA5052 alloy after 30% cold rolling, and then annealing for different durations at various temperatures: a) 225 °C, b) 250 °C, and c) 275 °C.

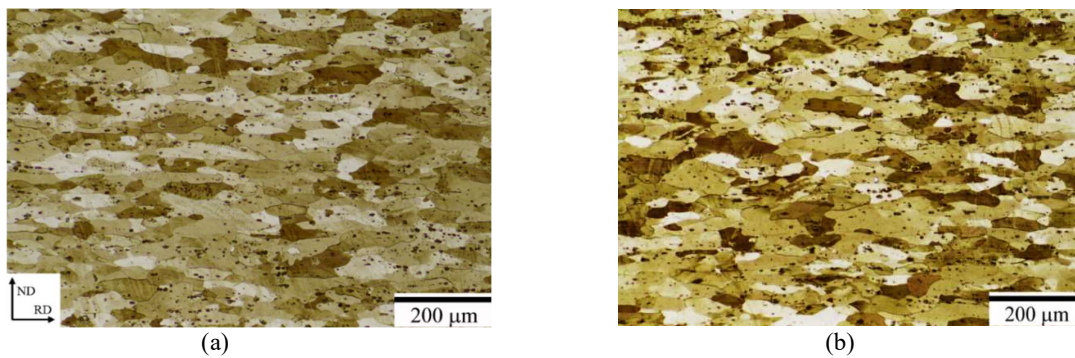


Fig. 3. Microstructure of AA5052 alloy (a) after cold working and (b) after cold working and annealing at 300°C for one hour.

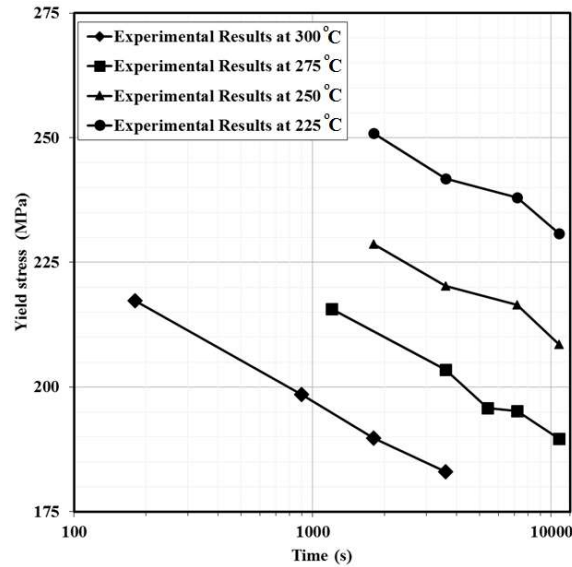


Fig. 4. Yield strength (0.2% proof strength) of cold rolled material during subsequent annealing at different durations and temperatures.

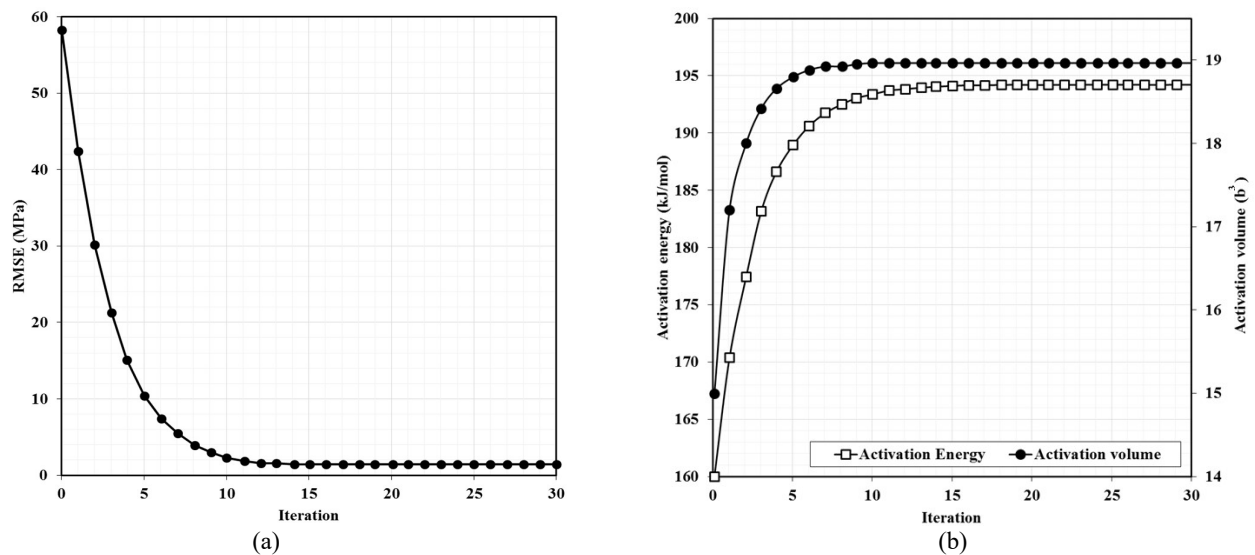


Fig. 5. Variation of a) root means squared error (RMSE) and b) the values of activation energy and activation volume during nonlinear regression analysis.

Figure 6 shows the model prediction for recovery kinetics at different annealing durations and temperatures in comparison with the experimental results. A good agreement between experimental results and model predictions is seen, giving confidence in the recovery model. As can be found in this figure, the recovery kinetics intensifies with raising the annealing temperature. The smooth increase of the recovery progress as a function of annealing time appears in this figure, implies that the dislocation density is gradually reduced by time during annealing.

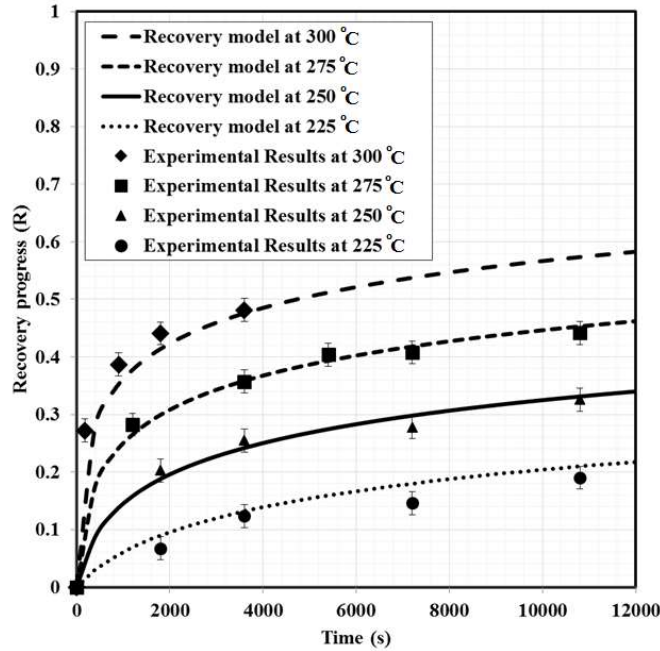


Fig. 6. Comparison between the evolution of recovery progress parameter predicted by the theoretical model and experimental results.

The effect of prior cold work on the recovery progress (R) after annealing for one hour at different temperatures is evaluated by the kinetics model and depicted in the Fig. 7. As shown in this figure, the recovery progress is increased by raising the annealing temperature, and the amount of prior cold plastic deformation. Truly, the recovery is a thermally activated process and the micro-mechanisms of this phenomenon is dominantly controlled by temperature. Increasing the annealing temperature raises the rate of recovery and so result in more material softening.

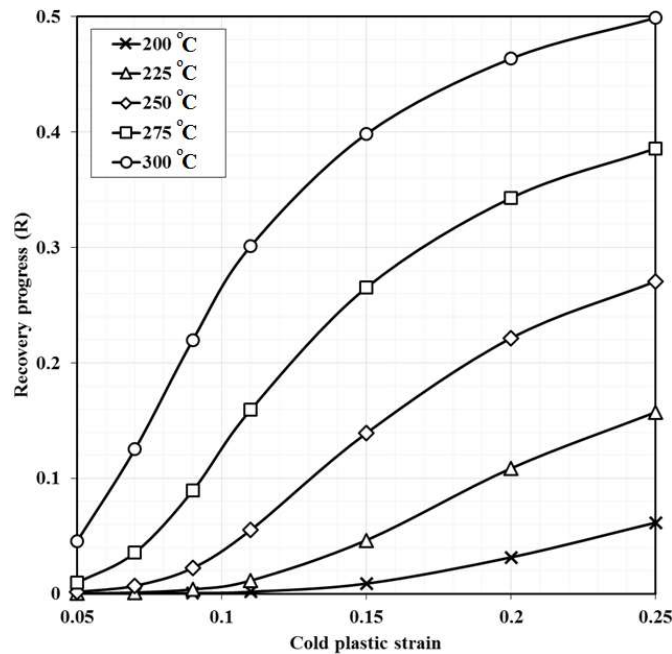


Fig. 7. Evolution of recovery progress parameter during annealing at different temperatures for one hour versus amount of prior plastic strain.

Also, by increasing the plastic strain, dislocation density and consequently the internal stored energy is increased, which it obviously increases the driving force of recovery. The effect of annealing times on the rate of recovery (dR/dt) during annealing at 250 °C for different prior plastic deformation is shown in the Fig. 8. As could be seen, recovery occurred very rapidly at the initial stage of the annealing treatment, and then the rate of static recovery is considerably decreased as annealing treatment proceeds. Also by increasing the amount of prior plastic deformation, the rate of recovery is increased noticeably, which is due to the higher values of the internal stored energy.

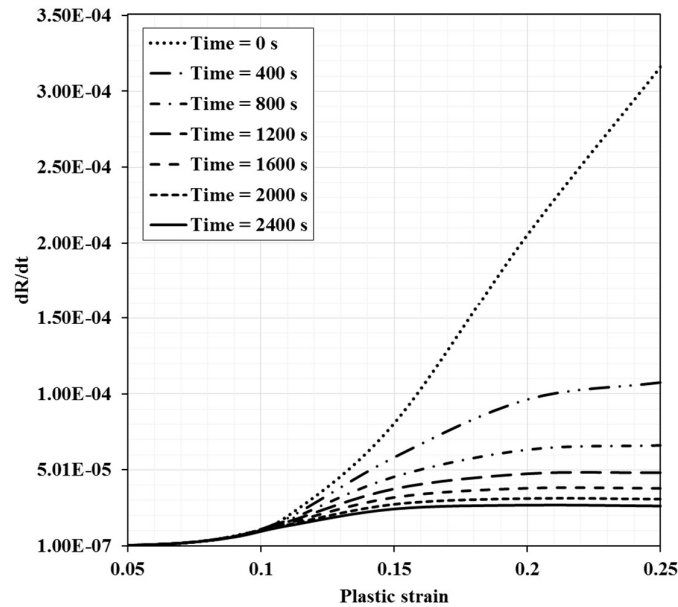


Fig. 8. Changing of the recovery rate versus prior plastic strain for different annealing times at 250 °C.

Table 1. Chemical composition of AA 5052 alloy (wt. %)

Al	Mg	Si	Fe	Mn	Cu	Cr	Zn
Base	2.38	0.248	0.345	0.121	0.037	0.168	0.037

Table 2. The material constants used in the model

Parameter	b (nm)	M	G (GPa)	ν	ν_D
Value	0.286	2.8	21	0.33	1.53×10^{13}

5. Conclusion

Work hardening and softening behavior of AA5052 alloy during cold work and subsequent annealing treatment were studied via uniaxial tensile test data and numerical modeling. The obtained main results are as follows:

- (1) The K-M dislocation density based model is used to predict the flow stress of the fully annealed material under the cold working condition. The predicted work hardening and softening coefficients of the K-M model for AA5052 alloy are 1.9×10^8 and 8.52, respectively.
- (2) The yield strength of the cold worked samples is decreased during the subsequent annealing treatment. Microstructural investigation shows that there is no evidence of recrystallization, and recovery is the main restoration mechanism up to annealing temperature of 300°C.

(3) Verdier's recovery kinetics model is successfully applied for modeling the static recovery of the alloy. The values of activation energy and activation volume obtained by a coupled nonlinear optimization technique and nonlinear differential equation solver are $18.98 b^3$ and 194 kJ/mol, respectively.

(4) From the modeling results, by increasing the amount of plastic deformation prior to annealing and annealing temperatures, the recovery progress parameter and also the rate of static recovery increased.

5. References

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مدلسازی و بررسی آزمایشگاهی بازیابی استاتیک و رفتار مکانیکی آلیاژ AA5052 حین کار سرد و عملیات آنیل بعدی

مجید سید صالحی^۱ و نوذر انجبین^{۲*}

^۱تهران، دانشگاه صنعتی خواجه نصیرالدین طوسی، دانشکده مهندسی و علم مواد.

^۲شیراز، دانشگاه شیراز، دانشکده مهندسی، بخش مهندسی مواد.

چکیده: در تحقیق حاضر به بررسی رفتار مکانیکی آلیاژ آلومینیم AA5052 با استفاده از نتایج آزمون کشش یک محوری حین کار سرد و عملیات آنیل همدمای بعدی در محدوده دمایی $225-300\text{ }^{\circ}\text{C}$ پرداخته شده است. نتایج نشان می‌دهد که با افزایش دما و زمان آنیل استحکام تسلیم ماده کاهش می‌یابد. بررسی‌های ریزساختاری نشان می‌دهد که پس از آنیل، نمونه‌ها همچنان دارای دانه‌های با مورفولوژی کشیده شده‌اند و نشانه‌ای از وقوع پدیده تبلور مجدد مشاهده نمی‌شود. بنابراین در شرایط فوق بازیابی مکانیزم غالب بازگشت حین عملیات آنیل می‌باشد. رفتار کارسختی آلیاژ حین کار سرد با استفاده از یک مدل مبتنی بر دانسیته نابجایی پیش بینی شد. همچنین نحوه نرم شدن آلیاژ تغییر شکل یافته، حین عملیات آنیل بعدی با استفاده از یک رابطه سینتیکی که تنش تسلیم را به پارامترهای آنیل مرتبط می‌سازد، مدلسازی شد. برای حل معادله سینتیکی که یک معادله دیفرانسیل غیرخطی است از روش انتگرال‌گیری عددی رانگ-کوتا-فلبرگ به همراه روش بهینه یابی غیرخطی گوس-نیوتن برای یافتن ثوابت ماده در مدل استفاده شد. نتایج عددی بدست آمده با استفاده از نتایج آزمایشگاهی مربوط به رفتار سیلان ماده صحت‌گذاری گردید.

واژه های کلیدی: سینتیک بازیابی استاتیک، آلیاژ آلومینیم AA5052، کار سرد، آنیل همدمای، برازش غیرخطی.