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APPLICATION OF RECOVERY MODEL TO THE ANNEALING BEHAVIOUR OF COLD ROLLED ALUMINIUM.

J.A.Sæter[1] and E.Nes[2]

1. SINTEF Materials Technology, N-7034 Trondheim, Norway.
2. The Norwegian Institute of Technology, Department of Metallurgy, N-7034 Trondheim, Norway.

Abstract

Recovery of mechanical properties during annealing of cold rolled aluminium has been modelled based on a microstructural representation comprising two elements, (i) the cell/subgrain structure (size δ) and (ii) the dislocation density (ρ) within the subgrains . These two microstructural elements are treated as independent internal state variables and the recovery of flow stress obtained by adding the time dependent contributions due to subgrain growth ($\sigma \propto 1/\delta(t)$) and dislocation network growth ($\sigma \propto \sqrt{\rho(t)}$). The model has successfully been applied in the interpretaions of recovery observations in commercial purity aluminium cold-rolled to different strains prior to anneling.

Introduction

A model for the recovery of mechanical properties in deformed metals has recently be presented by Nes[1] and Furu et al [2]. The background theory for this model rests on the assumption that during annealing of a pure metal, or stable solid solution, the substructure can be adequately described by a few time dependent microstructural elements, the two most important ones being the cell/subgrain size $\delta(t)$ and the dislocation density in the cell interior, $\rho(t)$. based on this microstructural description several possible approaches can be taken in order to calculate the flow stress, as discussed in detail in Ref.1. Two interesting treatments being the composite theory due to Mugrabi [3,4] and Pedersen et al.[5] and a modification [1] of the original Kuhlmann-Wilsdorf [6,7] link length model. However, as shown in Ref.1 both approaches predicts a relationship of the form

$$\tau(t) = \tau_i + \alpha_1 Gb\sqrt{\rho(t)} + \alpha_2 Gb\frac{1}{\delta(t)} \qquad (1)$$

where τ_i is a friction stress, G is the shear modulus, b is the Burgers vector and α_1, α_2 are constants.

It is convinient to express this relationship in terms of a normalized parameter, namely the fraction residual strain hardening, R(t), which is defined as follows:

$$R(t) = \frac{\sigma(t) - \sigma_i}{\sigma_0 - \sigma_i} \qquad (2)$$

where $\sigma(t)$ is the instantaneous flow stress and σ_0 and σ_i are the as deformed and friction stresses respectively. Combination of Eqs. 1 and 2 gives:

$$R(t) = f_1\sqrt{\frac{\rho(t)}{\rho_0}} + f_2\frac{\delta}{\delta(t)} \qquad (3)$$

where

$$f_1 = \frac{\alpha_1 MGb\sqrt{\rho_0}}{\sigma_0 - \sigma_i}, \quad f_2 = \frac{\alpha_2 MGb}{\delta_0(\sigma_0 - \sigma_i)}, \quad f_1 + f_2 = 1$$

ρ_0 and δ_0 are the average dislocation density and average cell size at time t = 0 respectively. It follows that f_1 and f_2 are the fractional contributions to the recoverable flow stress prior to annealing due to the distributed dislocations and subgrain structure, respectively.

The annealing out of the inter-cell dislocations was treated by Nes[1] as a network growth problem with thermaly activated glide of jogged screw dislocations being the rate controlling reaction. In this case the dislocations anneal out according to a reaction law of the form

$$\left(\frac{\rho}{\rho_0}\right)^{1/2} = 1 - \frac{kT}{A_1}\ln\left(1 + \frac{t}{\tau_1}\right) \qquad (4)$$

where $A_1 = \alpha_4 Gb^3$ (α_4 is a constant of order unity) and τ_1 is a relaxation time parameter:

$$\tau_1 = \frac{\sqrt{\rho_0}\, A_1 \nu_D C_1}{kT}\exp\left(-\frac{U_i - A_1}{kT}\right)$$

where υ_D is the Debye frequency and U_i is the activation energy. The climb of the jogs are in commercial purity aluminium expected to be controlled by solute drag (notably iron), i.e. U_i is the interaction energy between the solute and the jog. C_1 is a constant which needs to be determined experimentally. For more details, see Ref.1.

It follows from [2] that the kinetics of subgrain growth is obtained by solving the following equation:

$$\frac{d}{dt}\left(\frac{\delta}{\delta_0}\right) = \frac{2b\nu_D C_2}{\delta_0}\left(\exp - \frac{U_i}{kT}\right)\sinh\frac{l_2 A_2}{\delta_0 kT}\left(\frac{\delta_0}{\delta}\right) \qquad (5)$$

where $A_2 = \alpha_5 Gb^3$, l_2 is the separation of solute atoms along the boundary dislocations and α_5 is a constant of order 0.3. For more details on subgrain growth, see Ref.2.

By combining Eqs. 3-5 the effect of recovery on mechanical properties is obtained. In the following the predictions of this model will be tested experimentally by studying the static softening in cold rolled commercial purity aluminium.

Experimental

The material investigated is commercial pure aluminium with the composition: 0.17wt% Fe, 0.05wt% Si. The material was DC-cast and homogenized at Hydro Aluminium Research Center and processed at Pechiney CRV. The processing was done to obtain a low content of iron in solid solution. The grain size prior to cold rolling was 30 μm. The cold rolling was carried out on a laboratory rolling mill to true strains of ε=0.5 and 3.0. The annealing experiments were carried out in salt baths with subsequent water quenching. The specimens were annealed in the temperature range from 160°C to 320°C. At the highest temperatures the time to recrystallization is very short and consequently, the heat-up time in a salt bath becomes relevant in the time measurements. Furu et al. has shown [2] that in case of short annealing times samples of minimum thickness should be used in combination with stirring. In the present study samples of 1 mm. thickness were used, which should give heat-up times of about 1 sec. The softening reaction was followed by hardness measurements. A Vickers hardness instrument with a 1 kg load was used, each hardness number represents the average of at least 6 measurements. In the present investigation a Fisher Sigmascope has been used in order to follow variations in conductivity. It is then possible to detect precipitation reactions which may occour during annealing. A Phillips EM 400 Transmission Electron Microscope was used to measure the subgrain-size in the as deformed material. Foils were taken from the long transverse section, defined by the rolling direction and the sheet plane normal.

Experimental Results

Isothermal annealing at a range of temperatures from 160°C to 320°C of specimens cold rolled to ε=0.5 and ε=3, resulted in the softening behaviour illustrated in Figs. 1 and 2. With increasing annealing temperature, the positions of similar strength are displaced to shorter times. It is interesting to see that the low-strained material has a different annealing response compared to the high-strained material. For the low-strained material the softening have more character of a two-step process, where the speed of softening is rather slow at the beginning of the recoverey period, and the speed increases quickly after the transition point. Microstructural investigations shows that the material strained to ε=0.5 is about 10% recrystallized at a Vickers hardness of 29 and at a hardness of 33 for the material strained to ε=3. The arrows on the figures indicates when the material is about 10% recrystallized.

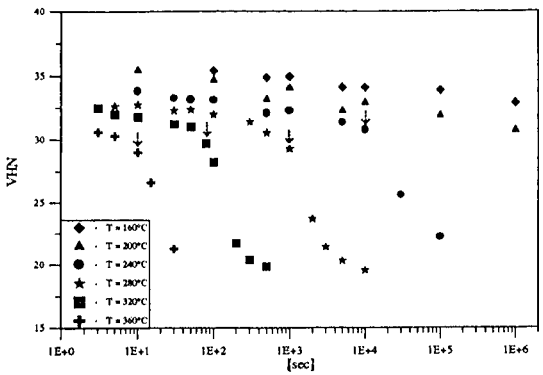


Figure 1. Hardness measurements as a function of annealing time for material strained to ε=0.5.

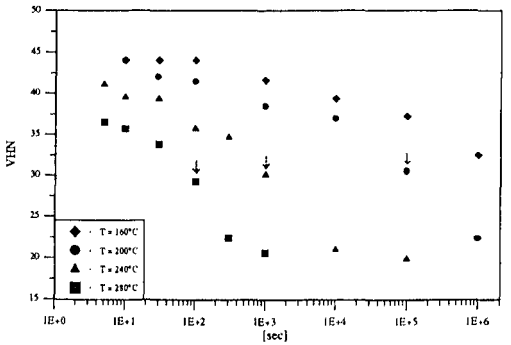
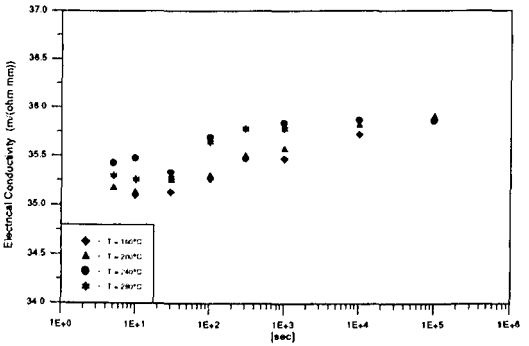
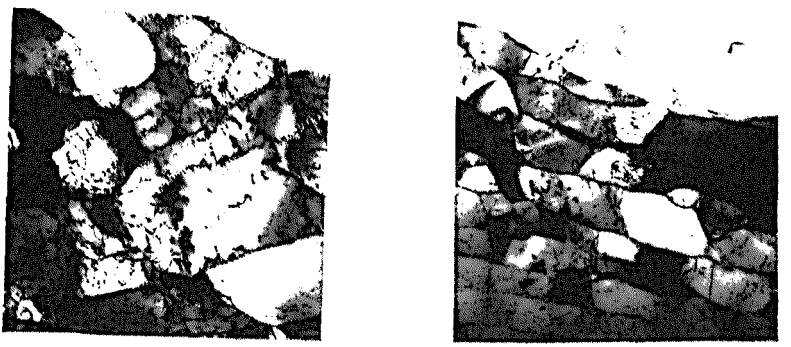


Figure 2. Hardness measurements as a function of annealing time for material strained to ε=3.



Figur 3. Electrical conductiviy as a function of annealing time for material strained to ε=3.



a) **R.D.** **b)**

Figure 4. TEM micrographs of the material in as cold rolled condition, a) ε=0.5 b) ε=3.

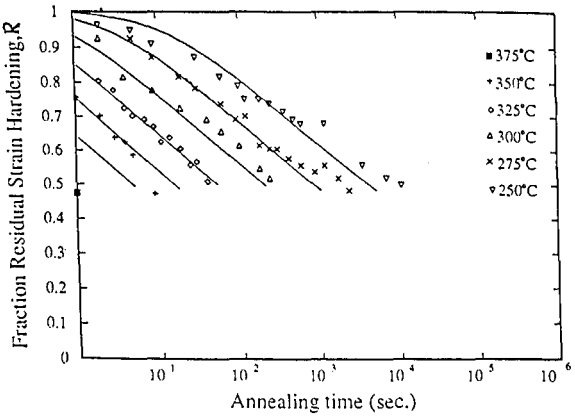


Figure 6. Fractional residual strain hardening, R, versus annealing time for a selection of temperatures at ε=3. From Furu et. al. [2].

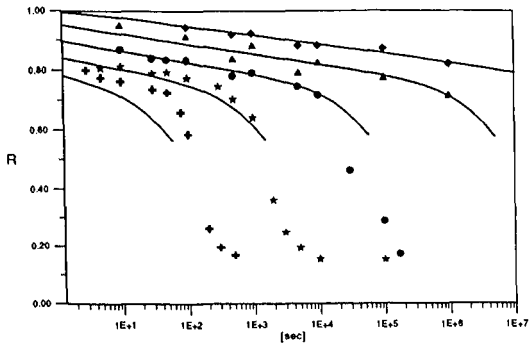


Figure 7. Fraction residual strain hardening, R, versus annealing time for a selection of temperatures at ε=0.5.

compared to the present results: 35.0 mΩ/mm² or an estimated solid solution content of 0.03 wt%. This difference has a clear effect on the kinetics (relaxation time) and the slope of the curves. By comparing the results of Fig. 5 and Fig. 6, it can be seen that at T=275°C and R=0.7, the curves from the work by Furu are displaced about an order of magnitude towards longer annealing times. The change in solid solution content changes also the slope of the curves, which reflects different sizes of the activation volumes involved. As $V_a \equiv l_1 bh = l_1 b^2/\theta$, the slope change requires a change in l_1 from $2.7 \cdot 10^{-7}$ m to $1.3 \cdot 10^{-7}$ m. l_1 is here the separation of solute atoms along the migrating ledge dislocation. This is a difference by a factor 2. If this difference is due to iron in solid solution, this implies a difference of about ΔFe_{ss}=0.025 wt% which corresponds to the estimated difference in solid solution content between the two materials.

When the strain is as low as 0.5, the model also has to account for the dislocations inside the cell structure, as shown in figure 7. By combining the equations for the annealing out of

The variation in conductivity has been followed with the results given in Fig.3. High purity aluminium has an electrical conductivity, κ, at room temperature of approximately 36.7 m/(Ω mm^2). According to Altenpohl [8], the connection between electrical conductivity and the solid solution content of alloying elements (in wt%) of interest is:

$$1/\kappa = 0.0267 + 0.032Fe + 0.0068Si \qquad (6)$$

In this study, the conductivity increases from 35 m/(Ω mm^2) in deformed condition to about 36.0 m/(Ω mm^2) in fully recrystallized state, i.e. a decrease in the iron content in solid solution of approximately 0.025 wt%. The solute content in the as deformed condition is estimated to about 0.03 wt%.

The micrographs on Fig. 4 illustrate the substructure in the as deformed condition for the two strains. It can be seen that the substructure in the ε=0.5 case can be characterized as a vaguely defined equiaxed cell structure, while at a strain of ε=3 the substructure is better described as sharp "pancake"-shaped subgrains. A characteristic difference between these two substructures is that in the low-strained material the cell structure also contains a relatively high dislocation density within the cells. At higher strains, the subgrains appear virtually dislocation-free. The subgrain size was measured to be 1.5 μm at a strain of 0.5 and 0.6 μm at a strain of 3.

Application of Recovery Model

In Fig. 5 the results given in Fig. 2 are replotted in terms of the fraction residual strain hardening, R vs log t. Here it is assumed that the subgrains are dislocation-free, and the decrease in hardness is due only to growth of the subgrains which implies that f_i therefore becomes zero in Eq.3. The fitting of the model to the experimental results are obtained by selecting l_i = $2.7 \cdot 10^{-7}$ m and using an activation energy, U=200 kJ/mole.

It is interesting to compare these results with those obtained on a similar alloy by Furu et. al. [2] shown in Fig.6. The main difference between the two allyos is the solid solution content (mainly due to iron), the conductivity from Furu et. al. is 34.1 mΩ/mm^2 (estimated solid content

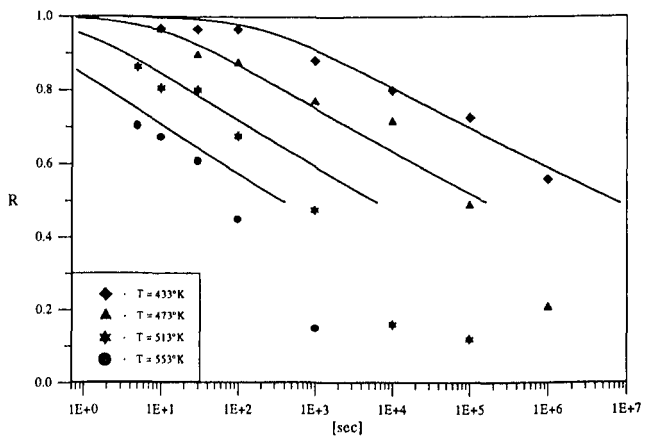


Figure 5. Fraction residual strain hardening, R, versus annealing time for a selection of temperatures at ε=3.

dislocations (eq.4) and subgrain-growth (eq.5), the predicted softening becomes as illustrated in Fig. 7, which also contains the experimental results. The correlation is reasonably good. The fractional contribution to the flow stress due to the dislocations inside the cells has been selected to be f_i=0.2. The calculated activation-energy for the movement of the dislocations is U=160 kJ/mole. The model predicts that the annealing out of dislocation is a dominating process in the first part of the recovery-process, while subgrain growth becomes important in the later stages.

Conclusion

A recovery-model which is based on the growth of the subgrains and the annealing out of dislocations inside the cells has succefully been applied to static recovery in a commercial purity aluminium alloy deformed to different strains. The model demonstrates that in highly deformed material, subgrain growth is the dominating process, while in material deformed to small strains one also have to consider the annealing of dislocations inside the cells.

Acknowledgments

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