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EFFECT OF PRE-ANNEALING AND PRE-COLD WORKING ON ISOTHERMAL ANNEALING OF
(Al + 0.25 wt pct Fe)

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Abstract: Pre-annealing and pre-coldworking were found to have considerable effects on the isothermal characteristics of the deformed (Al + 0.25 Fe). The activation energy of relaxation ranges between 0.38 and 0.72 eV., depending on the thermal and mechanical history of the alloy. The activation energy per unit recovery $\partial Q/\partial R$ was found to decrease linearly with shear strain $\bar{\gamma}$ according to the relation

$$\frac{\partial Q}{\partial R} = -\alpha \bar{\gamma} + \beta .$$

This might be due to excess stored energy given by pre-coldworking. The activation energy per unit recovery per unit shear $\partial^2 Q/\partial R\, \partial \bar{\gamma}$ was found to increase with pre-annealing temperature with a relatively slower rate beyond 350 °C. This might be attributed probably to the Al_3Fe and Al_6Fe phases that form at temperatures beyond 350 °C.

1. Introduction

Aluminium-iron alloy did not receive enough attention about its annealing and cold working mechanisms, although it is widely used in industry due to its high strength properties over pure aluminium. Attia *et al*[1]) studying the isochronal annealing of deformed (Al + 0.25 wt pct Fe) reported that its annealing spectra was composed of three annealing stages. The first stage occured in the temperature range from 25 °C to 100 °C, denoting the formation of complex vacancy-iron aggregates. The second stage was a multimechanism stage that occured in the temperature range from 100 °C to 350 °C. The third stage took place above 350 °C where the excess dissolving iron precipitated in the form of Al_3Fe.

The present work aims to investigate the effect of pre-annealing and pre-cold working on the isothermal annealing of (Al + 0.25 Fe) in the temperature range 200° — 350 °C at intervals of 50 °C. Samples were pre-annealed at temperatures 250°, 350° and 550 °C. The choice of these pre-annealing temperatures was based on the variation of solubility of iron in Al. Samples were cold worked by means of the torsional deformation method in which Kovacs and Nagy²⁾ characterized the deformation by the total equivalent shear strain ($\bar{\gamma}$) according to the equation

$$\bar{\gamma} = \frac{2\pi}{3}\frac{ND}{L} + \frac{3\,\Delta L}{L_0},$$

where N is the number of turns twist, L and L_0 are the initial and the instantaneous lengths of the wire of diameter D, while $\Delta L/L_0$ is the tensile strain that accompanies the torsion strain.

2. *Experimental*

The material used in this investigation was a commercial pure aluminium, of chemical composition 0.25 Fe, 0.05 Si, 0.02 Mg and 0.005 Cu wt pct and small traces of Mn and Ca. The test samples were in the form of wires of 0.5 mm diameter and 25 cm long. These wires were pre-annealed, in a silica tube evacuated to about 10^{-5} mm Hg, for 5 hours at 250°, 350° and 550 °C. Three degrees of plastic torsional deformation of average shear strain ($\bar{\gamma}$) of 0.24, 0.68 and 1.13 were given to the pre-annealed samples. After deforming in the twisting machine the wire was immediately clamped in the electrical circuit where its resistance at room temperature ($R_{r,t}$) was measured. An a.c potentiometric circuit of sensitivity $\pm 1 \cdot 10^{-5}\ \Omega$, similar in principle to that of Chiotti³⁾ and others⁴⁾ was used.

Since the present work requires the measurements of electrical resistivity after different heat treatments, a tubular furnace of diameter 7 cm and length 35 cm, was constructed to suit this purpose. The furnace surrounds a pyrex glass tube (Fig. 1.) containing the test sample, clamped in U shape,⁵⁾ the potential and current leads, and a calibrated thermocouple. The furnace was provided with a thermoregulator such that the temperature inside the tube could be maintained constant for several hours to within ± 2 °C. Temperature uniformity throughout the part occupied by the sample was better than ± 1 °C.

To ensure a closer control on the annealing temperature, the glass tube containing the sample was first introduced into an auxiliary cylindrical fur-

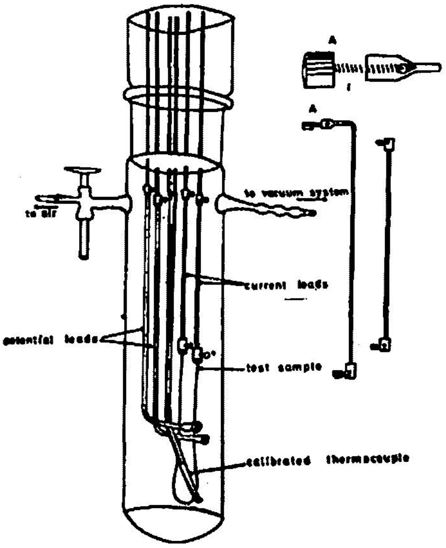

Fig. 1. Pyrex glass tube with sample holder, calibrated thermocouple and potential & current leads.

nace maintained at temperature higher than the desired annealing temperature. In less than one minute the specimen reaches the required annealing temperature. The glass tube was then immediately transferred to the annealing furnace where the heat treatment was continued. In this way the approach to the annealing temperature was much enhanced.

The temperature at which the measurements were made was not of prime importance,[4] but it was necessary that the temperatures of the specimen wires be measured to high precision (better than 0.1 °C). To achieve this in the water bath at room temperature, efficient stirring was necessary. A cylindrical Dewer flask which served as a constant temperature bath was used for the measurements at room temperature. In order to avoid temperature fluctuations in the room temperature bath, the specimen assembly was brought nearly to the correct temperature in an intermediate water-bath before placing it in the room temperature bath for measurements.

3. Results

A typical set of isothermal annealing curves are shown in Figs. 2, 3, 4, 5 and 6 representing the percent change in resistivity $\Delta\rho/\rho_o$ % ($\rho_o - \rho_t/\rho_o$ %) with annealing time (t) for samples pre-annealed at 250°, 350° and 550 °C and then cold worked by average shear strain ($\bar{\gamma}$) of 0.24, 0.68 and 1.13. Isothermal

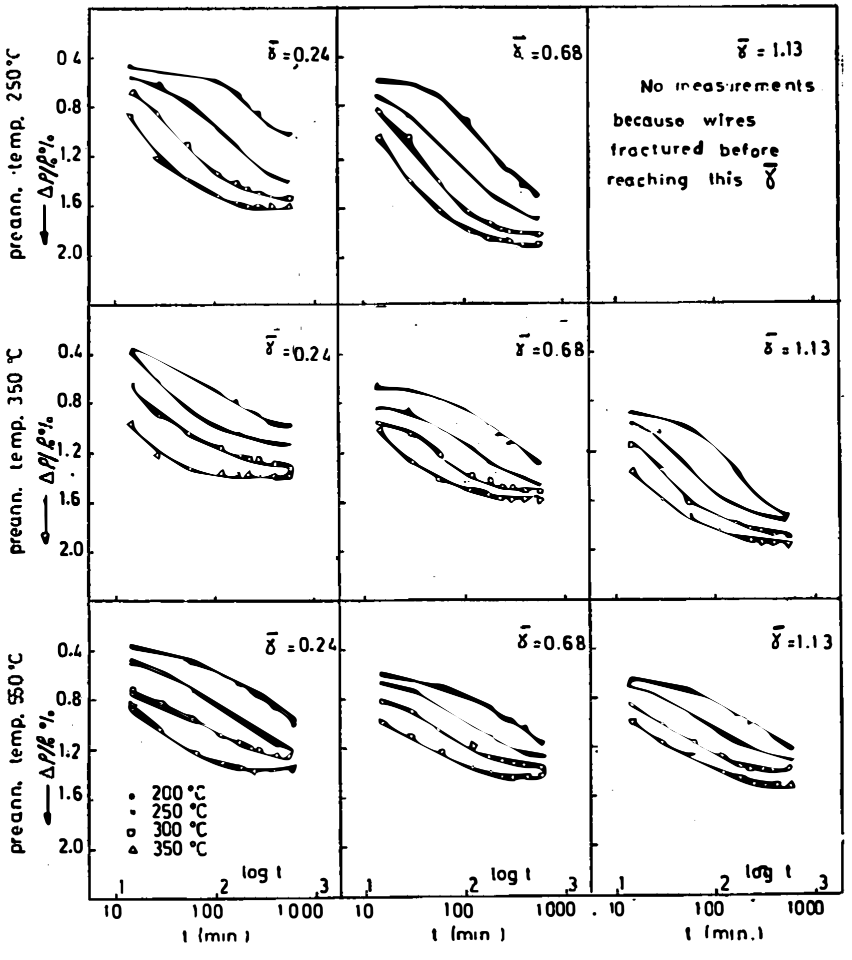

Fig. 2. The effect of annealing temperature on the change of $\Delta\rho/\rho_0$ % with time. Pre-annealing temperature and degree of cold work $\bar{\gamma}$ are kept constant.

annealing temperatures were 200°, 250°, 300° and 350 °C. Here ρ_0 is the resistivity after deformation and $\rho_0 - \rho_t$ denoted as $\Delta\rho$ is the change in resistivity due to thermal treatments. From these figures it was possible to deduce that:

- the percentage change in resistivity $\Delta\rho/\rho_0$ % which is taken here as a measure for the recovery, increases with annealing time till it reaches an asymptotic value after relatively long time. Notice that $\Delta\rho/\rho_0$ % increases downwards on the y axis in all curves;
- reaching the asymptotic value for each annealing temperature was enhanced by raising the annealing temperature;

— the amount of recovery $\Delta\rho/\rho_0$ % attained was always increasing with the degree of pre-cold working as shown in Fig. 3; and

— for the same $\bar{\gamma}$ and at the same annealing temperature, the amount of recovery increases the lower the pre-annealing temperature, as shown in Figs. 4, 5 and 6.

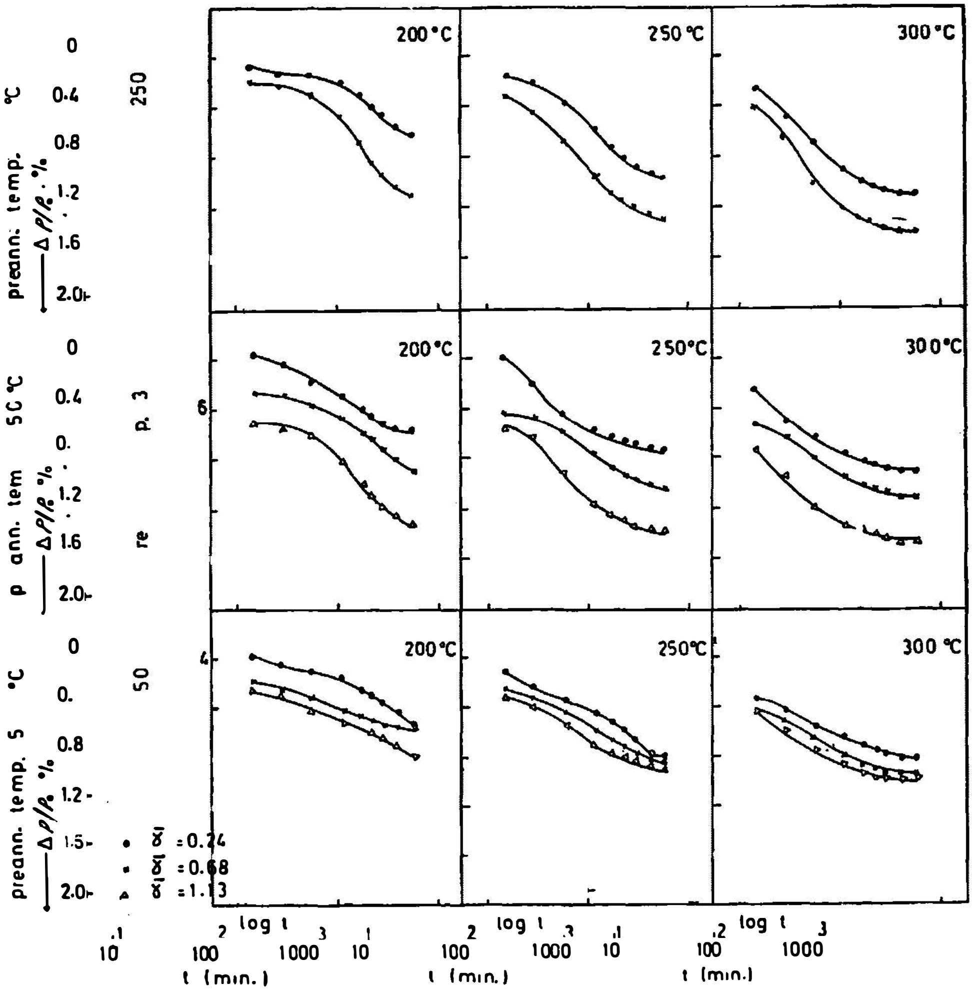

Fig. 3. The effect of pre-cold work $\bar{\gamma}$ on the change of $\Delta\rho/\rho_0$ % with time. Pre-annealing temperature and isothermal annealing temperatures are kept constant.

Activation Energy. The activation energy Q of the relaxation process was determined on the basis of the assumption that recovery was controlled by an Arhenius type equation

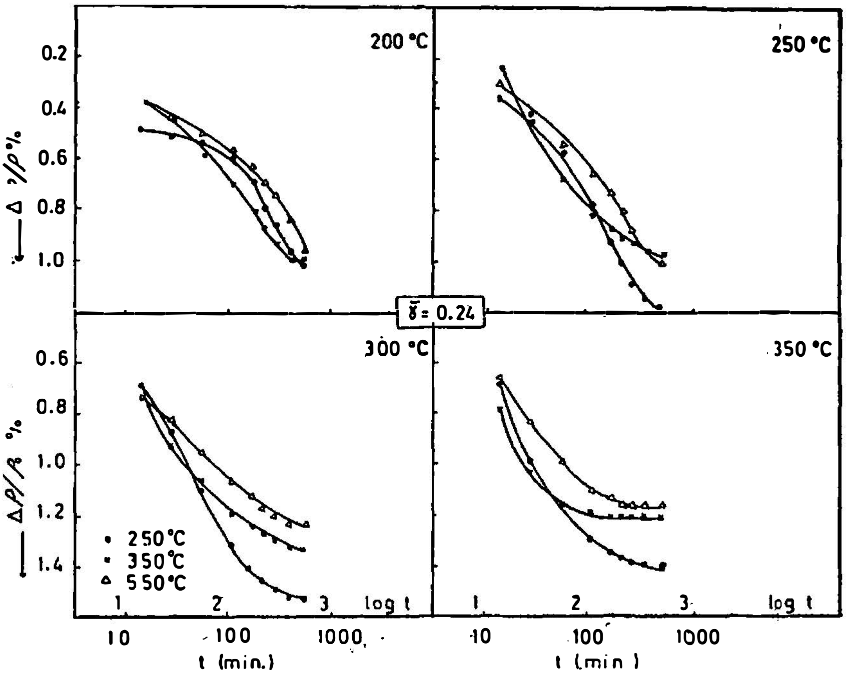

Fig. 4. The effect of pre-annealing temperature on the change of $\Delta\rho/\rho_0$ % with time. Degree of cold work = 0.24 and isothermal annealing temperature is kept constant.

$$t \exp. (-Q/kT) = \text{const.},$$

where t and T represent the corresponding times and temperatures required to reach a certain degree of recovery $\Delta\rho/\rho_0$ %. Fig. 7. shows the linear change of log t versus $1000/T$ for different values of $\Delta\rho/\rho_0$ %. This is repeated for the three values of the pre-cold working $\bar{\gamma}$ and the three pre-annealing temperatures. The values of the calculated activation energies are shown on each line.

Plotting the activation energy Q versus $\Delta\rho/\rho_0$ % (taken as recovery R) for every $\bar{\gamma}$ and every pre-annealing temperature, as shown in Fig. 8, it was possible to deduce that:

- the more the recovery attained the more the activation energy required;
- the rate of change of activation energy with recovery percentage $\partial Q/\partial R$ decreases with increasing $\bar{\gamma}$ as shown in Fig. 9.;
- the rate of change of activation energy with recovery $\partial Q/\partial R$ increases with pre-annealing temperature as shown in Fig. 10. Fig 9. also shows

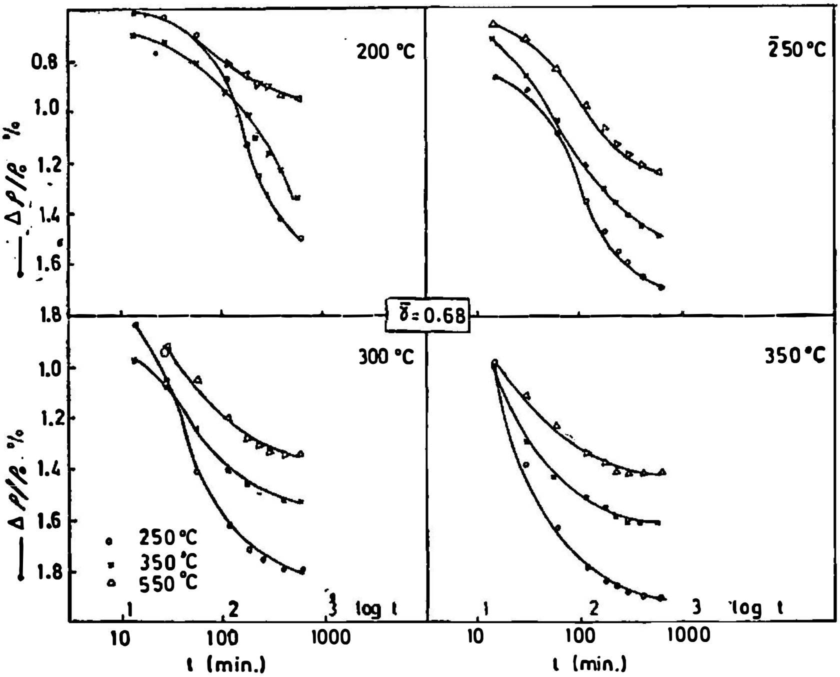

Fig. 5. The effect of pre-annealing temperature on the change of $\Delta\rho/\rho_0$ % with time. Degree of cold work = 0.68, isothermal annealing temperature is kept constant.

the variation of $\partial Q/\partial R$ with γ for every pre-annealing temperature. A linear function of the type

$$\frac{\partial Q}{\partial R} = -\alpha\bar{\gamma} + \beta$$

could be obtained to correlate the activation energy per unit recovery with the pre-cold working at a constant pre-annealing temperature. The Table lists the values of the constants α and β for every pre-annealing temperature.

Table

Pre-annealing temperature °C	α	β
250	0.083	0.120
350	0.375	0.515
550	0.400	0.580

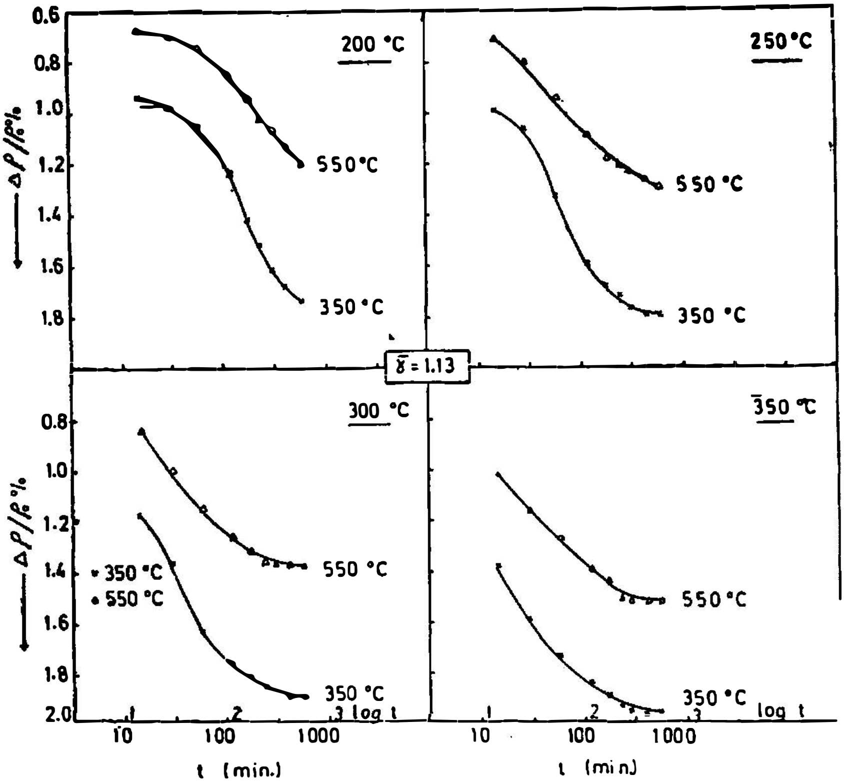

Fig. 6. The effect of pre-annealing temperature on the change of $\Delta\rho/\rho_o$ % with time. Degree of cold work = 1.13, isothermal annealing temperature is kept constant.

Fig. 10 shows the variation of α ($\alpha = \partial^2 Q/\partial R\,\partial\bar{\gamma}$) with the pre-annealing temperature. From this figure it could be observed that $\partial^2 Q/\partial R\,\partial\gamma$ increases with the pre-annealing temperature. The rate of increase is faster before 350° and slower after 350° up to 550 °C.

4. *Discussion*

Dependence of the amount of recovery on the annealing temperature. Keeping the average shear strain and the pre-annealing temperature constant for every set of samples annealed at different annealing temperatures, the increase of the annealing temperature gives more thermal energy which in turn increases the amount of vacancies annealed out, and also the amount of clustering. The scattering of conduction electrons decreases spontaneously

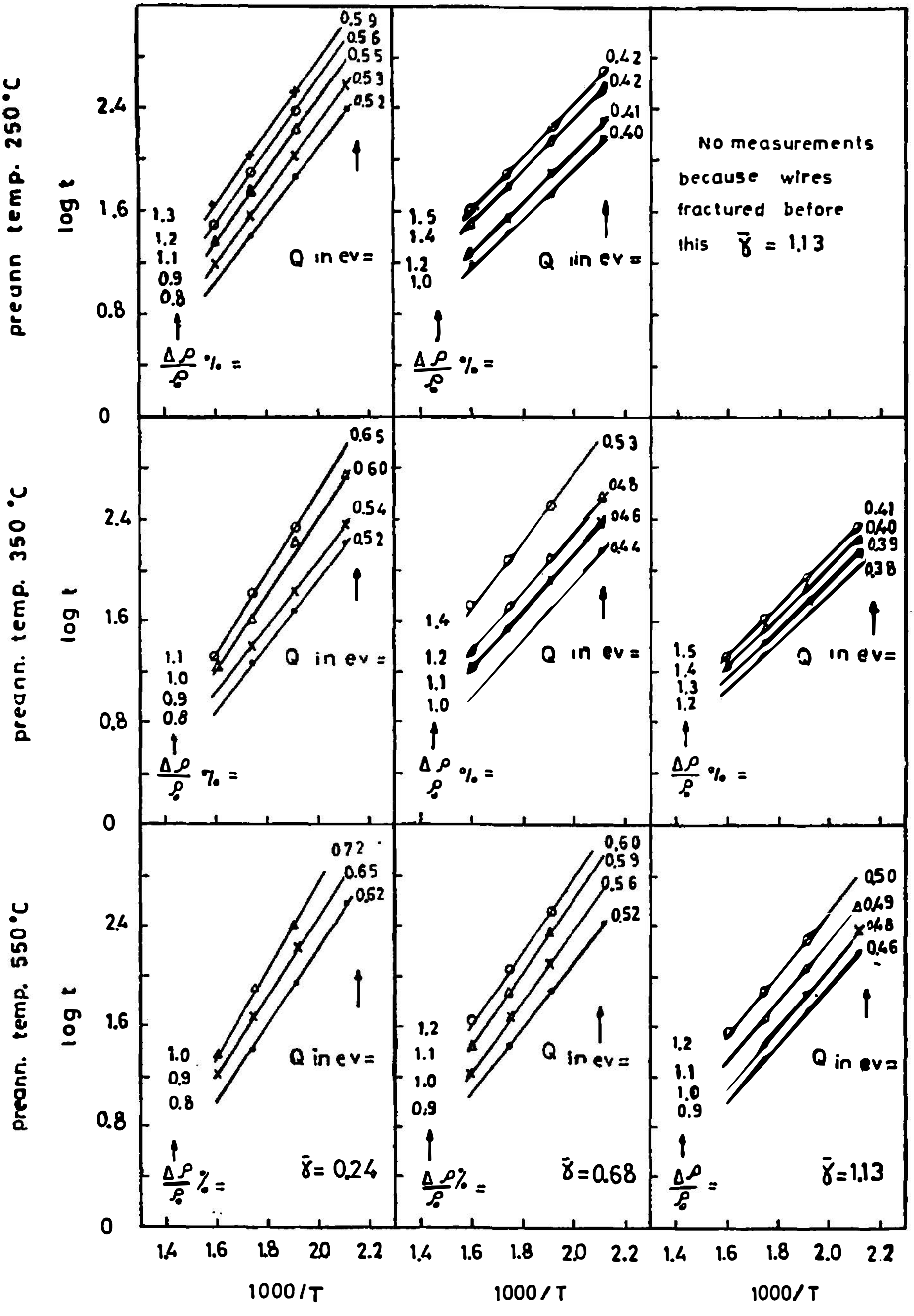

Fig. 7. Determination of activation energy for various $\Delta\rho/\rho_o$ % for each pro-anne-aling temperature and cold work.

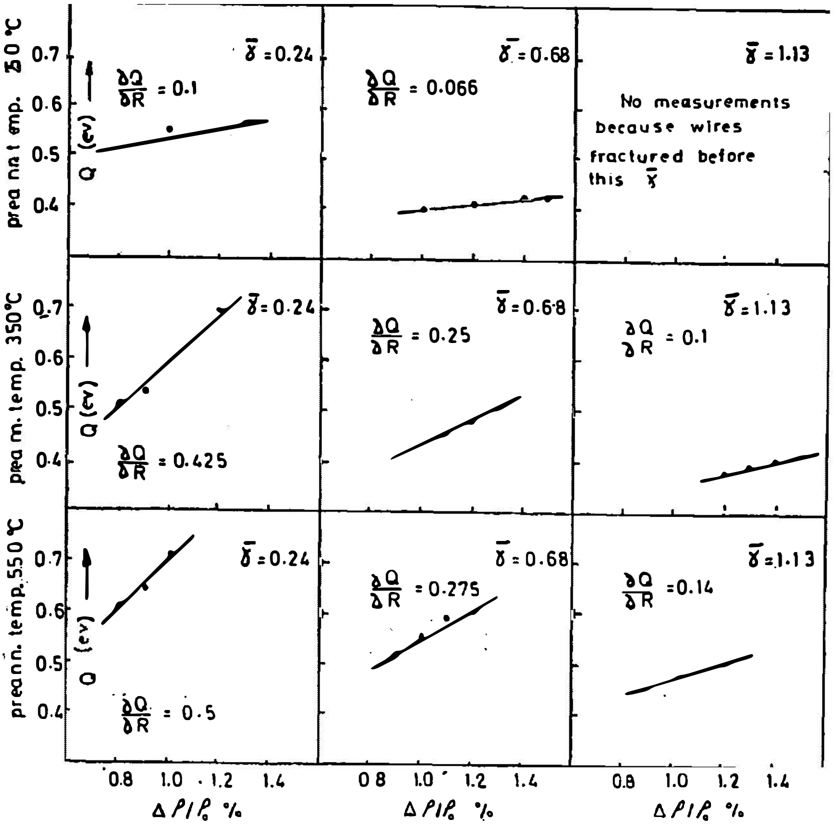

Fig. 8. The effect of $\Delta\rho/\rho_0$ % on activation energy Q, keeping pre-annealing temperature and degree of cold work constant.

with decrease of vacancy concentration[6] and accordingly resistivity decreases and thus the amount of recovery increases.

The increase of activation energy with the amount of recovery. When metals are plastically deformed point defects, point defect aggregates and dislocations are produced. Due to the presence of impurities in commercial Al, a significant degree of interaction and binding is expected between solute Fe and Si and the point defects present in the Al matrix. There is much evidence available that impurity atoms do play an important role in the annealing mechanism, if they are present in sufficiently high concentrations. Damask and Dienes[7] illustrated how monovacancy-impurity binding may influence the annealing of excess monovacancies to fixed sinks in the presence of impurity solute atoms. The activation energy required for this mechanism could vary between monovacancy migration energy and the sum

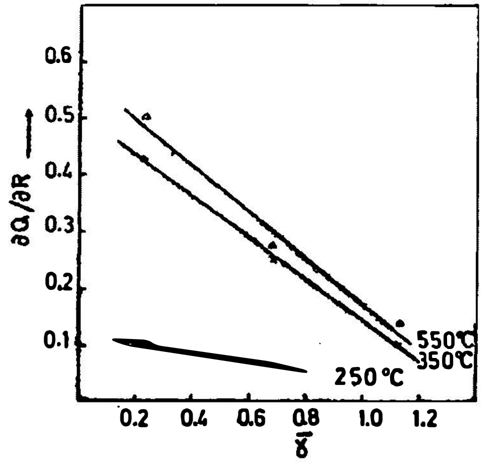

Fig. 9. The effect of $\bar{\gamma}$ on $\partial Q/\partial R$.

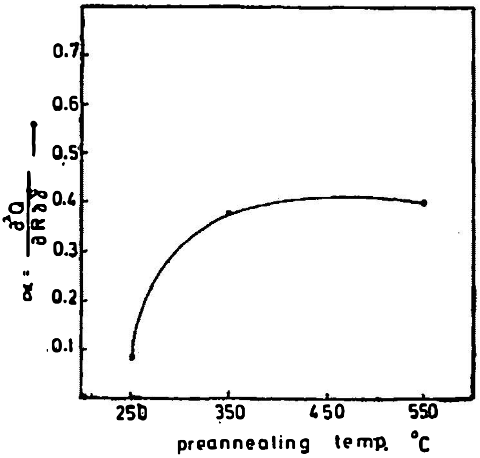

Fig. 10. The effect of pre-annealing temperature on the rate of change of $\partial Q/\partial R$ with $\bar{\gamma}$.

of that energy with the energy of the monovacancy-solute binding. The actual value was dependent in each case upon the solute concentration and the magnitude of the monovacancy-solute binding energy. When recovery occurs these different defects, that were already present before annealing were annealed out in successive steps according to their ease of action. Increasing number of imperfections annealed out implies more energy of activation, Since during recovery different mechanisms occur, beginning with annealing of interstitials, divacancies, vacancies, dissociation of clusters and annihilation of dislocations. These different mechanisms were found to have increasing amount of energy. This may explain the increase of activation energy with percentage recovery.

The dependence of recovery and activation energy on the pre-cold working. The observed increase in recovery as $\bar{\gamma}$ increases as the same pre-annealing temperature as shown in Fig. 3. may be discussed as follows: as the amount of pre-cold work increases, the number of dislocations of opposite signs will also increase, and when the dislocations are close together, the interaction energy is greater. This means that the heavier the cold work applied, the geater is the rate of recovery.

Concerning the activation energy, it is clear that increasing the amount of cold work gives rise to a more substantial strain energy stored in the lattice so it needs less amount of external energy. This may explain the decrease of activation energy per unit recovery with the increase of pre-cold work $\bar{\gamma}$ as shown in Fig. 9.

The dependence of $\partial^2 Q/\partial R\,\partial\bar{\gamma}$ on the pre-annealing temperature. The process of annealing helps the metal to get rid of the internal strains and imperfections already, existing before annealing. As the pre-annealing temperature is raised, the internal stored energy is reduced and more imperfections are annealed out. Therefore it could be deduced that the lower the pre-annealing temperature the higher the residual energy stored in the sample. Accordingly the samples that are deformed by the same amount of cold work $\bar{\gamma}$ are different in the residual stored energy. Thus the lower the pre-annealing temperature the lower the activation energy required per unit recovery and unit cold work. This explains the increase of $\partial^2 Q/\partial R\,\partial\bar{\gamma}$ with pre-annealing temperature. This increase is faster below 350 °C and slower between 350° and 550 °C. This decrease in the rate of change of $\partial^2 Q/\partial R\,\partial\bar{\gamma}$ with pre-annealing temperature above 350 °C could be explained on the basis of the relatively higher solubility of Fe in Al at temperatures higher than 350 °C. This high solubility of Fe in Al is responsible for the precipitation hardening due to the precipitation of the new phases Al_3Fe[8, 9, 10, 11] and Al_6Fe.[12] This precipitation hardening might be responsible for the relative decrease in the rate of change of $\partial^2 Q/\partial R\,\partial\bar{\gamma}$ with pre-annealing temperature above 350 °C compared to the same rate below the same temperature.

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UTJECAJ PRETHODNOG OTPUŠTANJA I PRETHODNOG HLADNOG RADA NA IZOTERMNO OTPUŠTANJE

(Al + 25% t. d. Fe)

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Sadržaj

Ispitao se je utjecaj prethodnog otpuštanja i prethodnog hladnog rada na izotermno otpuštanje (Al + 25% t.d. Fe). Uzorci u obliku žice prethodno su otpuštani na temperaturama od 250°, 350° i 550 °C a zatim su podvrgnuti uvijanju uz prosječne napone smicanja 0.24, 0.68 i 1.13.

Utvrđeno je da aktivaciona energija relaksacije, vrijednost koje se kretala od 0.38 do 0.72 eV, zavisi od termičke i mehaničke historije materijala.

Aktivaciona energija po jediničnom oporavljanju, $\partial Q/\partial R$, linearno je opadala sa hladnim radom, dok je za brzinu njenog opadanja sa hladnim radom, $\partial^2 Q/\partial R\,\partial\bar{\gamma}$, utvrđeno, da zavisi od temperature prethodnog otpuštanja prije deformacije.

Na temperaturama višim od 350 °C precipitacija Al_3Fe odgovorna je za otvrđivanje,, koje smanjuje brzinu promjene $\partial^2 Q/\partial R\,\partial\bar{\gamma}$ sa temperaturom prethodnog otpuštanja.