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Quenched-in Lattice Defects in Pure Aluminium

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Abstract: Prepared specimens of pure aluminium (99.999%) in the form of thin sheets of 100μm thickness were used for electrical resistivity measurements. Samples were quenched at different temperatures ranging from 373 K to 723 K for 30 minutes. Samples were also annealed for a constant time of 30 minutes at different temperatures (373 K to 673 K). It was observed that resistivity of pure aluminium increases with increase in temperature. The effect of annealing and quenching on electrical resistivity had also been observed. It was found that the room temperature resistivity increases with increase in quenching temperature but decreases after subsequent annealing at various temperatures. Increase in resistivity after quenching was found to be due to creation of defects and imperfections such as vacancies and dislocations etc. Decrease in resistivity after annealing can be attributed to recovery and recrystallization processes.

Key words: Quenched-in, Annealing, Defects, Resistivity, Deformation

Introduction

The mechanical properties such as flow stress, hardness and ductility of metals and alloys recover monotonically towards the values characteristic of the fully annealed stage during the process. Obviously the cells, developed during deformation prior to the annealing process, grow in size during the recovery stage and presumably set the stage for eventual recrystallization events in metals or alloys (Tseng and Varma, 1992: Bay *et.al,* 1992).

Electrical resistivity (a specific characteristic of metals/alloys) data can be useful to understand various phenomena in metals and alloys. For example the resistivity measurements provide an easy and an inexpensive method for study of phase transitions with change of temperature in crystalline and amorphous metals/alloys (Rana and Ansari, 1995: Ansari *et.al,* 1994: Rana *et.al,* 1999). Similarly residual resistivity measurement is one of the most simple and sensitive method for studying impurities, defects and other structural changes (Rosenberg, 1963).

When the specimen is heated up to certain suitable temperature, atoms in the specimen starts moving within certain limits about their mean position. If the specimen is quenched, the atoms freeze at their present position therefore defects and vacancies are produced in the specimen and if temperature is low enough, the equilibrium concentration of vacancies will be frozen in the sample (Thompson, 1969). The rapid quenching of a close-packed metal from a high temperature should "freeze in" large numbers of lattice vacancies (single and possibly pairs) which are in thermal equilibrium at high temperature. In close packed metals vacancies are favored as the defects rather than interstitials atoms, because they required as much energy as interstitials (Huntington, 1953: Huntington and Seitz, 1942: Brooks, 1955). The relative concentration of vacancies present after quenching may be obtained by measuring the increase in electrical resistivity, which the vacancies produce. The changes in the quenched material are so fast that it is in a state of strain that may cause surface or internal cracks. The strains set up in a quenching process are relieved completely during annealing (White, 1988).

Annealing of a specimen may affect either density of current carriers or their mobility. In metals and alloys the carrier density (free electron) remains unaltered by annealing. However, annealing increases mobility by relieving internal stresses and by decreasing disorder in the lattice (Raghavan, 1992: Cahn and Haasan, 1983). Therefore, the effect of annealing on metals and alloys is to decrease their resistivity. If the lattice were perfect, the electron waves would be transmitted through the lattice without scattering or with no resistance.

In the present investigations, pure aluminium (99.999%) was chosen to study the variation of electrical resistivity with temperature. Specimen of pure aluminium were heated to a high temperature and then quenched in water. Water was chosen for the quenching medium because it gave a rapid, reproducible quench and because it produced very little deformation. Furthermore, effect of annealing on electrical resistivity was also studied.

Materials and Methods

Samples of pure aluminium (99.999%) in the form of thin sheets of thickness 100μm were obtained from KFA Juelich, Germany. Specimens having different dimensions (length, width and thickness) were used to measure electrical resistivity by well-known four-probe method (Ansari *et.al,* 1994: Ansari, 1990: Rana *et.al,* 1990). Specimens were quenched at different temperatures ranging from 373 K to 723 K for 30 minutes. The annealing was performed at different temperatures (373 K, 423 K, 473 K, 523 K, 573 K and 623 K) for 30 minutes. Electrical resistivity was then measured after each quench and anneal. This research work was conducted at Department of Materials Science, Bahauddin Zakariya University, Multan, Pakistan during the year 1999-2000.

Results and Discussion

In general, it is observed that electrical resistivity of pure aluminium increases linearly with increase in temperature in the observed range. This result is in good agreement with the theoretical observations that pure metals at normal temperature show a linear relationship with the increase of temperature corresponding to the relation (Victor, 1976: Van Block, 1980: Theraja, 1981):

$$\rho(T) = \rho_1[1 + \alpha(T - T_1)]$$

Where ρ_1 and $\rho(T)$ are resistivities at temperatures T_1(K) and T(K) respectively and α is the temperature coefficient of the resistance. This increase in resistivity of metals is due to more thermal agitation of atoms and structural disorder to diffract and scatter the electrons at high temperatures (Williams *et.al*, 1976).

The plot of resistivity Vs temperature (Fig. 1) shows a linear increase in resistivity with increasing temperature. In case of aluminium ρ_1 = 2.5 μΩ-cm at T_1 = 273 K and temperature coefficient of resistance for the temperature range 273 K to 673 K was found to be 4.75 x 10^{-9} / K increase of temperature. Therefore, the behavior of electrical resistivity of aluminium as a function of temperature in the linear range can be expressed as:

$$\rho_{AL} = 2.5 + 0.011875\ (T - 273)$$

Electrical resistivity measured before and after quenching was compared to different quenching temperatures. It was found that after quenching the resistivity increased almost linearly with the increase of quenching temperature as clear from plot of ρ Vs T_Q (Fig. 2). The observed resistivity increased after quenching was found to follow the theoretical results (Huntington, 1953). For the range of quenching temperatures studies, it was found that resistivity increase on quenching could be described by the relation:

$$\Delta\rho = A \exp\ (-E_F / k\, T_Q)$$

Where Δρ is the increase in resistivity, A is constant, E_F is the energy required to form the defect, k is the Boltzmann constant and T_Q is quenching temperature. The justification for calling E_F, the energy of formation of defect, comes from statistical mechanics. It gives an expression similar to above equation for the equilibrium concentration of defects having formation energy E_F, in a solid at temperature T_Q. If it is assumed that defects are retained during the quench, above equation follows immediately.

To check the goodness of fit of data to above equation, the data was plotted as lnΔρ Vs $1/T_Q$. The points were almost found to fall on a straight line as clear from the plot of lnΔρ Vs $1/T_Q$ (Fig. 3). The values of E_F and A were obtained from the slope and intercept of the line respectively (Bauerle and Koehler, 1957: Koehler *et.al*, 1957).

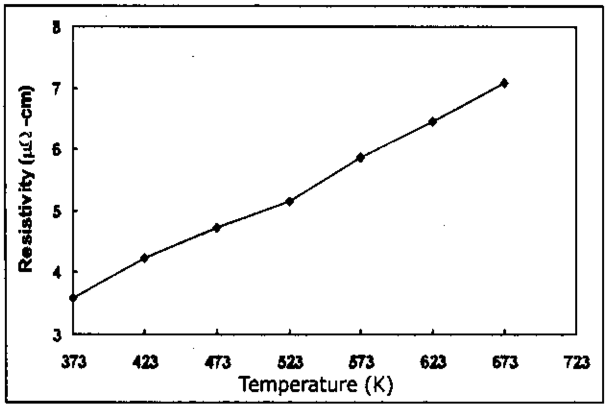

Fig. 1: Plot of Electrical Resistivity as a function of temperature

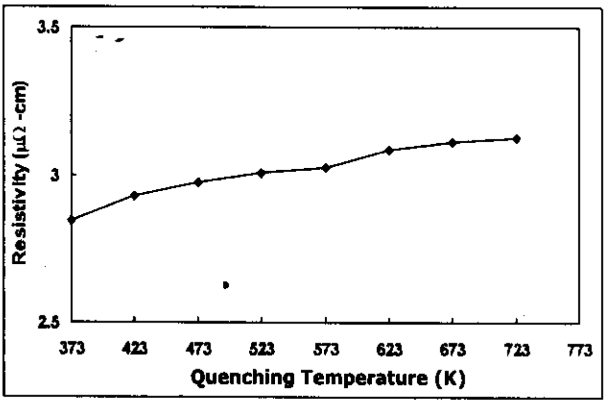

Fig. 2: Plot of Electrical Resistivity after quenching as a function of quenching temperature

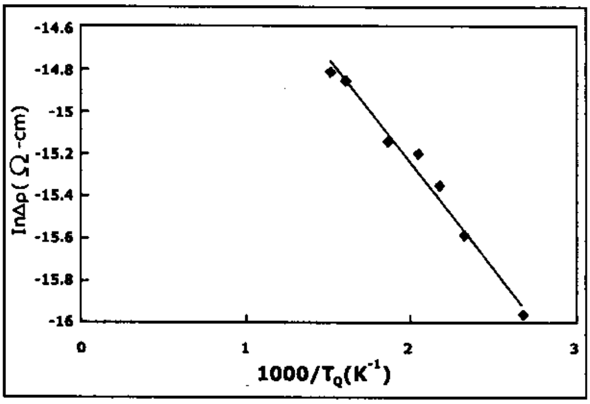

Fig. 3: Plot of lnΔρ as a function of reciprocal quenching temperature

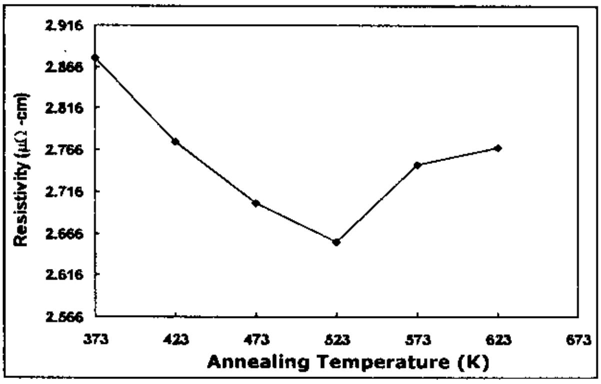

Fig. 4: Plot of Electrical Resistivity as a function of temperature after annealing

The values of E_F and A calculated from the experimental data are as under:

$$E_F = 0.7845 \text{ eV} \qquad A = 2.96 \times 10^{-4} \ \mu\Omega\text{-cm}$$

The plot of In Δ Vs $1/T_Q$ (Fig. 3) also shows that resistivity increases linearly with quenching temperature. The experimental results were found in good agreement with the observed data by TEM or resistivity measurements (Smallman, 1976; Federighi, 1965).

Some discrepancies could be due to the fact that the quenching process was not so fast and all heat treatments were made in the presence of air. Another possible source of more resistance increase on quenching could be the impurities initially present in the specimen. It is conceivable that the resistance increase found on quenching might be due to impurities and defects produced during quenching such as vacancies.

The room temperature resistivity measured after annealing decreases slowly with the increase of annealing temperature as shown in plot of resistivity Vs annealing temperature (Fig. 4). It is clear that the room temperature resistivity against different annealing temperatures decreases exponentially.

The decrease in resistivity of pure aluminium after annealing at certain elevated temperatures is due to recovery and recrystallization processes. During the recovery stage, the decrease in stored energy and electrical resistivity is accompanied by only a slight lowering of hardness and the greatest simultaneous change in properties occurs during the primary recrystallization. The recovery process describes the changes in the distribution and density of defects with associated changes in physical and mechanical properties, which take place in worked crystals before alteration of orientation occurs (Samuels, 1988).

In recrystallization stage, the deformed or quenched lattice is completely replaced by a new unstrained one by means of a nucleation and growth process. Thus most of the defects produced during some processes or previously present in the sample are removed due to these recovery and recrystallization processes. When the sample is annealed, most of the defects such as vacancies and dislocation are partially removed and the sample becomes more periodic, crystalline and ordered. Moreover, stresses and some other defects, which could be produced while sample cutting and preparations, were also removed during annealing process. Therefore, resistivity decreases after annealing because of more orderness and structural periodicity of the material. These results are, however, in good agreement with the theoretical data (Warren and David, 1959) showing a small discrepancy, which may be due to the certain experimental error.

Conclusion

- Electrical resistivity increases linearly with the increase in temperature.
- Electrical resistivity measured at room temperature increases by nearly 0.068% after quenching with the increase of quenching temperature, which is almost a very small increase.
- Electrical resistivity measured at room temperature decreases after annealing with the increase in annealing temperature, which shows almost an exponential decrease.
- Electrical resistivity also depends upon the nature of the material.

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