Evaluation of Lattice Defect Density in Deformed Ti by Precise Measurement of Electrical Resistivity

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Abstract

Several metallic materials have been developed for many purposes by alloying and controlling microstructure. From the viewpoint of materials recycling, several properties should be controlled by the latter in simple alloys. Then, observation and evaluation of lattice defects such as vacancy, dislocation and grain boundary are very important for understanding microstructure development during thermo-mechanical treatments. The purpose of this study was to establish a method for estimating density of lattice defects in cold rolled and annealed Ti by a precise measurement of electrical resistivity. Pure Ti plates were cold rolled at room temperature. Bar shaped specimens were cut from the plates. Electrical resistivities at 77 K (liquid nitrogen) and 300 K were measured by a direct current four-point method with a constant current of 100 mA. The accuracy of temperature control at 300 K was 0.1 K in silicone oil. Basically the electrical resistivities gradually increased with increasing a reduction of thickness. The density of dislocation was determined to be $2\times10^{14}$ m$^{-2}$ in the 15–80% CR specimens

Key words: Electrical resistivity, DC four-point method, Lattice defect, Titanium, The Matthiessen’s empirical relationship

1. Introduction

Several metallic materials have been developed for many purposes by alloying and controlling microstructure. From the viewpoint of materials recycling, several properties should be controlled by the latter in simple alloys. Furthermore, mechanical property in metallic materials is sensitive to microstructure and involved lattice defects. To understand the density and the behavior of lattice defects such as vacancy, dislocation and grain boundary is very important for materials design. Recently, new observation techniques have been developed in several fields e.g. 3D tomography in transmission electron microscopy (TEM). These analyses provide useful detailed information.

Basically, electrical resistivity is sensitive to phase transitions and change in densities of lattice defects. Up to now, electrical resistivity measurement has been carried out for various metallic materials to investigate transformation behavior, and/or to evaluate densities of several lattice defects and so on. Therefore precise measurement of electrical resistivity must also provide valuable information on lattice defects as well as the new developed techniques.

The purpose of this study was to detect lattice defects in pure Ti after conventional cold rolling (CR) and annealing by precise measurement of electrical resistivity. In addition, Matthiessen’s plot was created. It gives electrical resistivity from electrical resistance without measuring dimension of specimen.

2. Experimental procedures

2.1 Cold rolling and annealing

A commercial purity titanium (ASTM grade 2) sheets, 2 mm in thickness, 60 mm in width and 100 mm in length were used in this study. The chemical composition of the material is shown in
Table 1 Chemical composition of the commercial purity titanium studied (mass%)  

| C   | H   | O   | N   | Fe  | Ti  |
|-----|-----|-----|-----|-----|-----|
| 0.08| 0.013| 0.20| 0.03| 0.25| Bal.|

Table 1. The sheets were deformed at room temperature to aimed rolling-reduction by 0.02–0.06%. This process showed relative-small rise in temperature. The reduction in thickness was selected to be 15, 30, 50, 70 and 80% in this study. Bar shaped specimens (1.5 × 1.5 × 40–60 mm) along the rolling direction (RD) were cut from the plates as shown in Fig. 1. Then small Ti plates were welded as terminals for measuring electro-motive force in electrical resistivity measurement. In the measurement, lead wires for the potential contact were spot-welded to the terminals. Annealing was carried out for such specimens by direct dipping in nitrate bath held at 573–773 K. After annealing, the specimens were quenched immediately into ice-water. This method makes it possible to detect small changes in electrical resistivity, because the distance between two potential contacts (L) of the specimen does not change in the whole process from cold rolling to annealing.

2.2 Electrical resistivity measurement

Electrical resistivity measurement was carried out in order to observe plastic deformation and recrystallization behaviors. Electrical resistance at temperature T (Ω\text{T}) was measured by a direct current (DC) four-point method with a constant current of 100 mA. Cross sectional area (S) and distance between two potential contacts (L) of the bar shaped specimen were measured by micrometer and measuring microscope, respectively. Then electrical resistivity (ρ\text{T}) was obtained as follows:

\[ \rho_T = (S/L)\Omega_T \]  

(1)

Electrical resistivity is sensitive to temperature. Particularly in titanium and its alloys, the temperature dependence of electrical resistivity is very strong\(^3\). Therefore, attention needs to be paid to temperature control. In this study, measurement temperatures were selected to be 77 K (in liquid N\text{2}, ρ\text{77}) and 300 K (ρ\text{300}). The measurement at 300 K was carried out in dimethylpolysiloxane which is one of silicone oils. The accuracy of temperature control at 300 K was 0.1 K during the measurement.

3. Matthiessen’s empirical relationship

Electrical resistivity of multi-component dilute solid solutions at temperature T (ρ\text{T}) is represented by following equation:\(^4\):

\[ \rho_T = \rho_P + \Sigma \Delta \rho_i \cdot C_i, \]  

(2)

where ρ\text{P} is the resistivity of ideally pure solvent, \(\Delta \rho_i\) is the contribution to resistivity per unit concentration of “i” th solute or lattice defect at temperature T and \(C_i\) is its concentration.

In general, dimension of specimen must be measured in order to obtain electrical resistivity from electrical resistance. Fortunately, there is a procedure to evaluate the resistivity of specimens without measuring the dimension. The procedure is called the Matthiessen’s empirical relationship:\(^2\). This relation can be also employed for evaluating the errors in average dimensions due to bending and surface roughness of specimens. In this study, electrical resistivities at 300 K and 77 K were measured. Since the temperature dependence of the contribution to resistivity per unit concentration is ignorable for the lattice defects, the following relation can be obtained from Eq. 2:

\[ \rho_{300} - \rho_{77} = \rho_P - \rho_{77}, \]  

(3)

where \(\rho_{300}\) and \(\rho_{77}\) are the resistivity of specimens in various states such as cold rolled, recrystallized. From Eq. 3, the relation between the resistivity at 77 K, \(\rho_{77}\), and the resistivity ratio \(R = \rho_{300}/\rho_{77}\) can be described as follows:

\[ \rho_{77} = (\rho_P - \rho_{77})/(R-1). \]  

(4)

In this equation, the term of \(\rho_P - \rho_{77}\) is a constant and R is the ratio of resistance, \(R = \Omega_{300}/\Omega_{77}\) \((\equiv R_{300}/R_{77})\). Thus, the resistivity at 77 K of specimen can be evaluated by measuring the resistance at 300 K and 77 K without measuring the dimension of specimen.
4. Results and Discussion

4.1 Change in electrical resistivity of pure Ti by cold rolling

Figure 2 shows optical microstructures of the as received and the 80% CR specimens. The starting materials had equiaxed grain with average grain size of 11 μm (Fig. 2(a)). Although the microstructures for 15–70% reduction in thickness are not shown here, elongated initial grains involving deformation bands were observed. In the 80% CR specimen, the initial grain boundaries were completely disappeared (Fig. 2(b)). Many lines attributed the deformation structure can be confirmed parallel to the rolling direction (RD).

Basically electrical resistivity of metallic materials increases by introducing impurities and alloying elements or lattice defects such as vacancy, dislocation, grain boundary and so on. A large amount of lattice defects were introduced into the specimens during the CR process. Figure 3 shows changes in resistivities as a function of true strain. The resistivity at 77 K, \( \rho_{77} \), increased monotonically with increasing true strain. On the other hand, the resistivity at 300 K, \( \rho_{300} \), increased with true strain up to \( \varepsilon = 0.42 \) and it decreased once. Then it monotonically increased again. The mechanism of this abnormal phenomenon is not yet clear. However it might be related to heterogeneous deformation originating in hexagonal structure of Ti.

The density of dislocation was evaluated by Eq. 2 and the contribution to resistivity per unit concentration of dislocation in pure Ti under the assumption that the increment of resistivity was due solely to the dislocation density. This assumption seems to be reasonable because almost vacancies must be vanished just after finishing the CR process at RT. The calculated dislocation density is shown in Table 2. The density of dislocation was gradually increased with reduction of thickness in the order of \( 10^{14} \text{ m}^{-2} \).

In general, tensile strength is known to increase with the square root of dislocation density. Similarly, electrical resistivity is proportional to dislocation density as shown in Eq. 2. Therefore, the increment of Vickers hardness must be proportional to the square root of the increment of electrical resistivity because Vickers hardness is proportional to tensile strength. The increments of hardness were plotted versus the square root of the increment of \( \rho_{77} \) (Fig. 4). However the \( \Delta HV \) showed parabolic increase against the \( \Delta \rho_{77}^{1/2} \). The \( \Delta HV \) vs. \( \Delta \rho_{77}^{1/2} \) might show linear relation in highly strained region. At least, hardness or tensile

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**Table 2** Dislocation density calculated from increment of electrical resistivity

| Reduction (%) | Dislocation density/10^{14} \text{ m}^{-2} |
|--------------|---------------------------------|
| 15           | 2.9                             |
| 30           | 3.8                             |
| 50           | 4.6                             |
| 70           | 6.0                             |
| 80           | 7.4                             |

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**Fig. 2** Optical micrographs of the as received (a) and the 80% cold rolled (b) specimens.

**Fig. 3** Changes in resistivity at 77 K (\( \rho_{77} \)) and 300 K (\( \rho_{300} \)) by cold rolling as a function of true strain.

**Fig. 4** Hardening in the cold rolled Ti versus the increment of electrical resistivity.
4.2 Electrical resistivity change in cold rolled Ti by annealing

In order to investigate recovery and recrystallization process, heat treatments were continuously-carried out at 573 K, 673 K and 773 K for the 80% deformed specimens. Figure 5 shows changes in resistivities of the 80% CR specimens during heat treatments. The resistivity gradually decreased and saturated in the heat treatments at 573 K and 673 K. In the treatment at 773 K, on the other hand, the resistivity had been slightly decreasing. The final level of resistivity is almost the same as the original.

4.3 Matthiessen’s empirical relationship

The resistivities at 77 K were plotted versus $1/(R-1)$ as shown in Fig. 6. Open squares and half-filled squares are for the CR and the annealed specimen, respectively. Both plots show linear relationships. The relations showed different gradients but it is very small. Although attention may need to be paid to the difference in gradient, the resistivity can be evaluated in the present pure Ti from the resistance of $\Omega_{77}$ and $\Omega_{300}$ by using the gradient ($\alpha$) and intercept ($\beta$) terms of these relationships.

5. Summary

The precise measurements of electrical resistivity were carried out for the cold rolled and annealed pure Ti. The following conclusions were reached.

1. The resistivity increased with increasing the reduction in thickness. However, it is not yet saturated by the conventional cold rolling up to about 80%.

2. The density of dislocation was calculated to be $10^{-14}$ order from the resistivity; it was gradually increased with increasing reduction of thickness.

3. The Matthiessen’s plot was created for pure Ti by using the cold rolled and annealed specimens.

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