Measurement of Electrical Resistivity during Tensile Deformation of Pure Ti

Kousuke FUJITA, Masato UEDA* and Masahiko IKEDA

Kansai University, Faculty of Chemistry, Materials and Bioengineering, Osaka 564-8680, Japan

Abstract

The observation and evaluation of lattice defects such as vacancies, dislocations, and grain boundaries are very important in materials design. Electrical resistivity measurement is superior to electron microscopy for obtaining average microstructural information, including density and type of lattice defects. The purpose of this study was to estimate changes in electrical resistivity during the tensile deformation of commercial-purity (CP) Ti. The electrical resistivity of a cold-rolled Ti sheet was measured at 77 K ($\rho_{77}$) and 300 K ($\rho_{300}$) along the rolling direction (RD) using a direct current (DC) four-point method to determine Matthiessen’s empirical relationship, $\rho_{77} = \alpha/(R - 1) + \beta$, $R = \rho_{300}/\rho_{77}$. Plots of $\rho_{77}$ versus $1/(R - 1)$ showed a linear relationship, and the values of $\alpha$ and $\beta$ were determined to be 0.5266 and −0.0024, respectively. Changes in $\rho_{77}$ during tensile deformation were estimated by substituting the resistance ratio $R$ into Matthiessen’s empirical relationship. In the elastic deformation region, no remarkable change in the resistivity was observed. Therefore, the dislocation density did not change significantly. However, the resistivity did increase drastically near the yield point.

Key words: Electrical resistivity, Tensile deformation, Titanium

1. Introduction

The mechanical properties of materials are generally determined by their microstructure, which includes lattice defects in addition to composition. Thus, understanding the density, character, and behavior of lattice defects such as vacancies, dislocations, and grain boundaries is very important in materials design. Recently, new observation techniques have been developed in several fields, such as 3D tomography in transmission electron microscopy (TEM), orientation imaging microscopy, and positron annihilation. These methods provide useful and detailed information regarding microstructure and lattice defects. The electrical resistivity is sensitive to phase transitions and changes in the density of lattice defects. Electrical resistivity measurements have been employed with various metallic materials to investigate their transformation behaviors and to evaluate the densities of several types of lattice defects. As the specimen volume for the analysis is $10^8$ times that for TEM, average information can be obtained from this larger volume. In addition, in-situ measurements can easily be carried out, such as during deformation or heat treatment. Therefore, the precise measurement of electrical resistivity can also provide valuable information on lattice defects as well as the new developed techniques.

Electrical resistivity can generally be measured using a well-shaped cylindrical or tabular specimen. The dimensions of the specimen must be accurately measured in order to obtain the electrical resistivity from four-point DC electrical resistance measurements. This means that the resistivity cannot be measured in irregularly shaped specimens. However, resistivity can be estimated using the empirical relationship.

The purpose of this work was to determine Matthiessen’s empirical relationship by cold rolling, and to estimate changes in electrical resistivity during tensile deformation in commercial-purity titanium.
2. Experimental procedure

2.1 Cold rolling

Commercial-purity titanium (ASTM grade 2) sheets, 2 mm thick, 60 mm wide, and 100 mm long, were used in this study. The chemical composition of the material is shown in Table 1. The sheet was cold-rolled at room temperature (RT) to aimed rolling-reduction by a reduction of 0.02–0.06% per pass. This process resulted in a relatively small increase in temperature. Samples were reduced in thickness by 15, 30, 50, 70, and 80%. Flat bar-shaped specimens (1.5 × 1.5 × 40–60 mm) along the rolling direction (RD) were cut from the sheets. The dimensional error in the cross section of 1.5 × 1.5 mm was less than ±2.5 μm. Small Ti plates (1 × 1 × 5 mm) were spot-welded as terminals for measuring the electromotive force using a direct current (DC) four-point method. In this measurement, Mo lead wires for the potential contact were spot-welded to the terminals.

2.2 Tensile deformation

Tensile specimens (gauge: W2 × L50 × T1 mm) were cut from CP Ti sheets, as shown in Fig. 1. The specimens were deformed with a crosshead speed of 0.025 mm/min at room temperature. Then, tabular specimens were prepared from the gauge and shaped by mechanical polishing. The dimensional error in the cross-sections was less than ±2.5 μm.

2.3 Electrical resistivity measurements

Electrical resistivity measurements were carried out in order to observe the plastic deformation behavior. The electrical resistance at temperature \( T \) (\( \Omega_T \)) was measured by a DC four-point method using a nanovoltmeter (Keithley 2182A) with a direct current/alternating current (DC/AC) current source (Keithley 6221) with a DC constant current of 100 mA. The thermoelectromotive forces at all contacts were canceled out by changing the current polarity of the measurements.

The cross-sectional area \( S \) and the distance between the two potential contacts \( L \) of the bar-shaped specimen were measured by a micrometer and a measuring microscope, respectively. We refer to the ratio of \( S \) to \( L \) as the size factor, \( S/L \). The electrical resistivity at temperature \( T \) (\( \rho_T \)) was obtained as follows:

\[
\rho_T = (S/L)\Omega_T. \tag{1}
\]

Electrical resistivity is sensitive to temperature, and the temperature dependence of electrical resistivity in pure titanium is particularly strong\(^9\). Therefore, care must be taken to control the temperature. In this study, the measurement temperatures selected were 77 K (in liquid \( N_2 \)) and 300 K. Measurements at 300 K were carried out in dimethylpolysiloxane, a silicone oil. The temperature of the oil was controlled using a refrigerated/heated circulator and monitored with highly accurate probes. The accuracy of the temperature control at 300 K was ±0.1 K during the measurement.

2.4 Matthiessen’s empirical relationship

The electrical resistivity of multi-component dilute solid solutions at temperature \( T \) (\( \rho^S_T \)) can be represented by the following equation\(^10\):

\[
\rho^S_T = \rho^P_T + \sum \Delta \rho^i_T \cdot C_i, \tag{2}
\]

where \( \rho^P_T \) is the resistivity of the ideally pure solvent, \( \Delta \rho^i_T \) is the contribution to resistivity per unit concentration of the \( i \)-th solute or lattice defect at temperature \( T \), and \( C_i \) is its concentration or density.

In general, the dimensions of the specimen must be measured in order to obtain the electrical resistivity from electrical resistance. Fortunately, Matthiessen’s empirical relationship can be used to estimate the resistivity of a specimen without measuring its dimensions\(^11\). This relationship can also be employed to evaluate the errors in average dimension due to bending and surface roughness of the specimens. This study measured electrical resistivities at 300 K and 77 K. Assuming that the temperature dependence of resistivity per unit concentration is negligible for lattice defects, the following relationship can be obtained from Eq. (2):

\[
\rho_{300} - \rho_{77} = \rho^P_{300} - \rho^P_{77}, \tag{3}
\]

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

$$\rho_{77} = \frac{(\rho_{p300} - \rho_{p77})}{(R - 1)}. \quad (4)$$

The $(\rho_{p300} - \rho_{p77})$ term is constant, and $R$ is the ratio of the resistances, $R = \Omega_{300}/\Omega_{77} (=\rho_{300}/\rho_{77})$. Although Eq. (4) indicates a straight line through the origin, the measured relationship has an intercept on the $\rho_{77}$ axis in plots of $\rho_{77}$ versus $1/(R - 1)$. Therefore, Matthiessen’s empirical relationship can be described as follows:

$$\rho_{77} = \alpha/(R - 1) + \beta. \quad (5)$$

The values of $\alpha$ and $\beta$ can be determined from resistivity measurements with well-shaped specimens.

3. Results and Discussion

3.1 Determination of Matthiessen’s empirical relationship

Figure 2 shows a grain boundary map and a (0001) pole figure obtained by electron backscatter diffraction (EBSD) for an as-received CP Ti sheet. The starting materials had equiaxial grains with an average grain size of 11 μm.

The electrical resistivity of metallic materials increases with the introduction of impurities, alloying elements, or lattice defects such as vacancies, dislocations, or grain boundaries, as shown in Eq. (2). Figure 3 shows changes in resistivity as a function of the true strain $\varepsilon_t$. 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 $\varepsilon_t = 0.4$, and then decreased before increasing again. This characteristic change in $\rho_{300}$ is most likely due to the developed texture. These increases can be attributed to lattice defects introduced by the cold rolling. A large number of dislocations were introduced into the specimens during the CR process.

The resistivity at 77 K, $\rho_{77}$, is plotted versus $1/(R - 1)$ for the CR specimens in Fig. 4. All plots show a linear relationship, with correlation coefficients close to 1. The values of $\alpha$ and $\beta$ were determined to be 0.5266 and −0.0024, respectively. Using this relationship, the electrical resistivity at 77 K, $\rho_{77}$, can be estimated from the resistances at 300 K and 77 K, as shown in Eq. (4).

3.2 Estimation of electrical resistivity during tensile deformation

Figure 5 shows a scheme for the evaluation of resistivity at 77 K, $\rho_{77}$, after tensile deformation. All of these measurements were performed using the same tensile specimen. First, a tensile deformation was applied to a determined strain at room temperature. After unloading, the specimen was kept at room temperature for 30 min to remove any vacancies introduced by the deformation.
Then, electrical resistance measurements were carried out at 77 K and 300 K. Substituting the resistance ratio $R$ into the Matthiessen’s empirical relationship, the electrical resistivity at 77 K was obtained. By repeating this deformation and measurement process, the pseudo-change in electrical resistivity caused by tensile deformation was obtained.

Figure 6 shows stress-strain curves and corresponding electrical resistivities during tensile deformation. In this figure, strain represents the total applied strain, including the elastic strain. In the elastic deformation region, no remarkable change in electrical resistivity was observed, indicating that the dislocation density remained relatively unchanged. However, the electrical resistivity increased drastically around the yield point, indicating that many dislocations were introduced. The electrical resistivity was almost saturated at a strain of 1.0, even when additional deformation was introduced. Reaction of dislocations and/or crystal rotation might occur at this stage. The dislocation density at saturation was calculated to be $9 \times 10^{14}$ m$^{-2}$, based on the electrical resistivity. Although further study is needed, electrical resistivity was successfully estimated in specimens with ever-changing shape using Matthiessen’s empirical relationship.

4. Conclusions

Precise measurements of electrical resistivity were carried out for cold-rolled and tensile-deformed commercial-purity Ti. The following conclusion can be drawn.

(1) The resistivity increased as the thickness was reduced. This increase can be attributed to dislocations introduced by plastic deformation.

(2) Matthiessen’s plot was created for CP Ti in the cold-rolled state. All plots showed a linear relationship, with correlation coefficients close to 1. The values of $\alpha$ and $\beta$ were determined to be 0.5266 and −0.0024, respectively.

(3) Changes in electrical resistivity during tensile deformation were evaluated using the Matthiessen’s empirical relationship. Many dislocations were introduced near the yield point.

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