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Determination of dislocation densities using transmission electronic microscopy and positron annihilation spectroscopy

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1. Introduction

A dislocation is a lattice line defect that defines the boundary between slipped and unslipped portions of the crystal. In crystalline materials, the plastic behavior is related directly to the presence of dislocations. Also, the amount of dislocations within materials is an important factor to determine the strength of materials. Much effort has been devoted to the direct investigation of dislocations. The density of dislocation is defined as the number of dislocation lines that intersect a unit area in the crystal. Usually, the density ranges from 10^{11} to 10^{12} dislocations/cm^2 in deformed metal crystals. Among the methods available for estimating the dislocation density, transmission electron microscopy (TEM) is a power tool to investigate the dislocation structures in deformed metals and to derive parameters to describe them.

Positron annihilation lifetime spectroscopy (PALS) is a sensitive method for investigating open-type defects which include vacancies, vacancy agglomerates, and dislocations. The measured positron annihilation lifetimes of a sample are linked to the size of the defects and the relative intensities of each defect are related to the defect concentrations. The present paper is intended to quantify the dislocation densities for cold-worked stainless steel samples by using two different methods, TEM observation and PALS analysis.

2. Experimental

The material used for this study is commercial-grade stainless steel (SS) 316. The chemical compositions of SS316 are listed in Table 1. The samples were prepared by a conventional cold-working at room temperature with different percentages of a deformation. They were cold worked by 20%, 40%, 60% and 80%. In this section each technique used to quantify the dislocation density is described.

Table 1. Chemical compositions of SS316

Cr	Ni	C	Mn	Si	P	S	Mo	Fe
16	12	0.08	2	1	0.045	0.03	2.5	Bal

2.1 TEM Analysis

The dislocation density was determined by using a 200kV JEM-2000FX TEM. First, the specimens were mechanically polished down to a thickness of 100μm and were prepared for thin discs with a 3-mm diameter. Then, these discs were thinned by an electrochemical method such as double jet technique (Struers Tenupol 5). The electrolyte is a mixture of 70% methanol, 20% butylcellosolve, and 10% perchloric acid. From the TEM images, the dislocation density was calculated by the line intersection method which is based on the superimposition of a grid consisting of horizontal and vertical lines on the TEM image. The dislocation density, ρ is given by;

$$\rho = \frac{1}{t}\left(\frac{\sum n_v}{\sum L_v} + \frac{\sum n_h}{\sum L_h}\right) \qquad (1)$$

where Σn_v and Σn_h are the summation of the number of intersections of vertical and horizontal grid lines with dislocations, and ΣL_h and ΣL_v the total length of horizontal and vertical grid lines, respectively. The magnitudes of the total length of the horizontal (ΣL_h) and vertical (ΣL_v) lines are determined from the grid, while the thickness, t of the observed area for the TEM specimen is determined from the convergent beam electron diffraction technique [1].

2.2 PALS Measurement

The PALS measurements were performed at room temperature by means of a fast-fast coincidence timing spectrometer. We employed a ^{22}Na β^+-source of about 1 MBq and collected more than one million counts for each test. The positron lifetime can be measured by detecting the time difference between the birth γ-radiation of the ^{22}Na β^+-source and one of the annihilation γ-quanta with energy of 511 keV. The scheme of the positron lifetime measurement is shown in Fig. 1. The time resolution of the system is 260 ps in full width at half maximum. The positron lifetime data was analyzed by subtracting the source components and background. All the spectra were decomposed into two lifetime components by using the PASFIT program.

When there are two types of defects in the lattice (vacancies and dislocations), we have to apply a three-component fit to PAL spectra. In this case, it is probable to obtain poor statistics due to the uncertainty about the analyzed lifetimes. Accordingly, we decompose the spectra using only two lifetimes with fitting parameters τ_1(short), τ_2 (long), and I_2. In interpreting the lifetime data, the τ_1 component was assumed to be weighted average of annihilation in the dominant bulk as well as a shorter lifetime trap (ex. dislocations), whereas the τ_2 component is related to the relatively big-sized traps such as vacancy clusters. From the analysis of the standard trapping model, we can derive the dislocation density with proper information on the positron trapping coefficients.

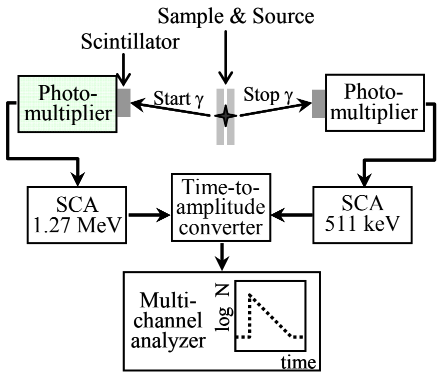

Fig. 1. Scheme of the positron annihilation lifetime spectroscopy (SCA: single-channel analyzer).

3. Results

The TEM images of SS316 samples with different levels (0, 20, 40, and 60%) of cold rolling are shown in Fig. 2. Unlike others, there are few dislocations seen in Fig. 2(a). After sufficient deformation, small subgrain structure is created and dislocation clusters are distributed all over the regions. The results of calculated dislocation densities are listed in Table 2. Comparing the density between 0% and 20% cold-worked sample, the density increases by five orders of magnitude due to the reduction of thickness. After 20% cold-working, the density does not increase significantly but goes to saturation, up to 7.5 x 10^{15} m^{-2}.

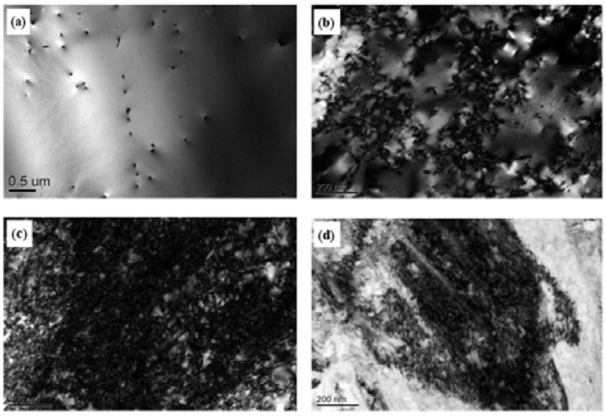

Fig. 2. TEM micrograph of SS316 (a) 0%, (b) 20%, (c) 40%, (d) 60% cold-working.

The PALS lifetime data are shown in Fig. 3. Short (τ_1) and long (τ_2) annihilation lifetimes are derived from all samples using the standard trapping model (STM). The measured lifetime, τ_1 ranges from 145 to 160 ps, which are characteristic of combinations of defect-free Fe (~110 ps), dislocation (~140 to 170 ps) or mono-vacancy (~180 ps) sites. The long lifetime, τ_2 increases with the amount of cold-working, while its intensity becomes small. It is believed that the big-sized vacancy clusters are created due to the cold-working. The STM can be applied to estimate the dislocation densities from the measured τ_1, τ_2 and I_2 based on the known bulk lifetime (108 ps) and an assumed lifetime in a trap. Here, we assume that τ_1 is attributed to a mixture of annihilation in the bulk matrix and dislocations (~160 ps). The dislocation densities based on this assumption are listed in Table 2. In this calculation, the specific trapping rate for α-Fe of 0.36 x 10^{-4} m^2/s was used [2].

Table 2. Dislocation density of cold-worked SS316 measured from TEM and PALS analysis

Cold-working (%)	Dislocation density (m^{-2})	
	TEM	PALS
20	3.10 x 10^{15}	8.83 x 10^{14}
40	7.80 x 10^{15}	1.99 x 10^{15}
60	6.90 x 10^{15}	4.31 x 10^{15}
80	8.00 x 10^{15}	6.68 x 10^{15}

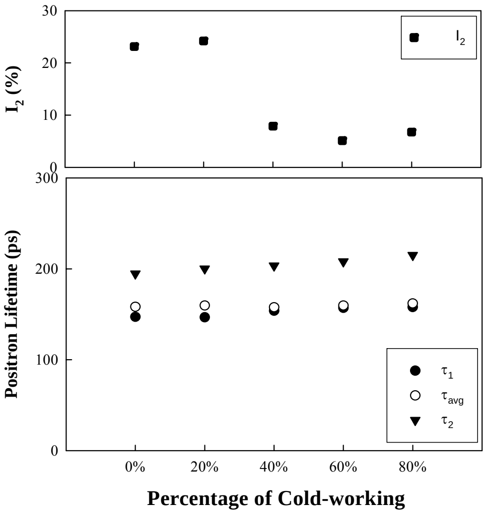

Fig. 2. PALS measured lifetimes and relative intensities for cold-worked SS316.

4. Conclusions

Two different methods, TEM investigation and PALS analysis, were applied to estimate the dislocation density for cold-worked SS316. It is found that two methods are useful in determining dislocation densities and are complementary each other.

REFERENCES

[1] D. B. Williams, C. B. Carter, Transmission Electron Microscopy: a textbook for materials science, Plenum Press, 1996.

[2] J. Cizek, I. Prochazka, J. Kocik, E. Keilova, Phys. Stat. Sol. (a) **178** (2000) 651.