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Positron Annihilation Lifetime Spectroscopy of Vacancy-Type Defects in Cold-Worked Stainless Steel 316

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1. Introduction

Austenitic stainless steels have high ductility, low yield stress and relatively high ultimate tensile strength, when compare to a typical carbon steel. Austenitic steels have a FCC atomic structure which provides more planes for the flow of dislocations, combined with the low level of interstitial elements (elements that lock the dislocate-eeeion chain), gives this material its good ductility.

It is well known that plastic deformation generally creates a large number of dislocations in metals. In addition, open-volume defects such as vacancy clusters are produced simultaneously. The treatment was carried out on type 316 stainless steel as follows; cold working (20, 40, 60, 80 %).

In this study, we investigate the features of open-volume type defects as a function of the degree of deformation for pure iron using PALS. The parameters of interest include the size of vacancy clusters and their number density.

2. Experimental

2.1 Experimental and Sample Examined

The Positron Annihilation Lifetime (PAL) measure-ements were made using a conventional fast-fast coincidence system. The positron lifetime can be measured by detecting the time difference between the birth-γ (1.27 MeV) of the β^+-decay in the source and the annihilation-γ (0.511 MeV) emitted from the sample. The positron sources used for measurement consist of ^{22}Na sandwiched between two Al foils. The positron measurements were made using two-detector fast-fast coincidence PALS. A simplified diagram for the two-detector system is given in Fig. 1. BaF_2 scintillators and photomultipliers with a short rise-time, consisting of both detector modules, are employed to obtain a high time resolution. The voltage pulses from the module are delivered to a CFDD, which determines the time of arrival of the pulse. If the height of the pulse lies within the pre-selected energy window, a standard pulse is sent to fast coincidence. Hence the time between two standard pulses from the two modules is the positron lifetime. Then the time spectrum is stored in a Digital Oscilloscope. The instrumental time resolution of the system is 290 ps of the full width at half maximum (FWHM) The source correction was made using well-annealed iron sample, which has a known positron lifetime of 106 ps. We applied the PALSfit program from the RISØ laboratory to analyze the measured spectra [1]. In performing PALS analysis with the PALSFit package, we used two Gaussian resolution functions to extract positron lifetime information from the lifetime distributions.

We have examined 5-pair stainless steels(SS) 316 sample supplied by Hankook Vacuum Metallurgy Co., Korea. 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.

2.2 Positron Trapping Model of a Single Type of Defect

The positron lifetime is defined as the time difference between the birth of a positron and the annihilation of positron. A thermalized positron in a perfect metal is annihilated with a constant annihilation rate $\lambda_b(=1/\tau_b$, τ_b=bulk lifetime). The positron lifetime of bulk Fe and Cr are known to 106 ps and 118 ps, respectively. In the presence of vacancy-type defect, positrons tend to trap at their sites with a trapping rate κ_d and annihilate with a annihilation rate λ_d (=$1/\tau_d$, τ_d=defect lifetime), which is schematically shown in Fig. 1. The positron decay function can be described as

$$f(t) = I_1 \exp(-\frac{t}{\tau_1}) + I_2 \exp(-\frac{t}{\tau_2}) \qquad (1)$$

where each lifetime and intensities are defined as

$$\tau_1 = \frac{\tau_b}{1+\kappa_d \tau_b}, \tau_2 = \tau_d, \quad . \qquad (2)$$
$$I_1 = 1 - I_2, I_2 = \frac{\kappa_d}{\lambda_f - \lambda_d + \kappa_d}$$

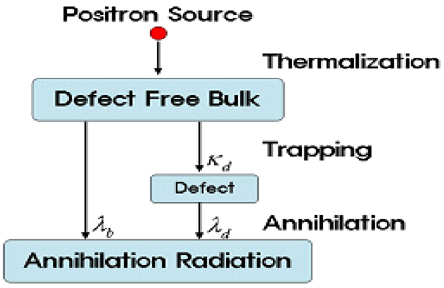

Fig. 1. A schematic of the trapping model includes only one defect type.

From Eq. (2), the trapping rate κ_d can be calculated using experimentally measured PA data.

$$\kappa_d = (I_2 / I_1) \cdot (\lambda_b - \lambda_d) \qquad (3)$$

According to trapping model, the positron trapping probability is directly proportional to the defect concentration. Denoting a defect concentration C_d, the trapping rate κ_d is given as

$$\kappa_d = \mu_d \cdot C_d \qquad (4)$$

where the proportionality constant μ_d is the trapping coefficient.

3. Results and Discussion

The positron lifetime spectra for cold-worked SS316 were decomposed into two components, τ_1 (shorter) and τ_2 (longer). Fig. 2 shows the dependence of positron lifetime on the percentage of cold work. The observed τ_1 for the rolled Fe is longer than that for the bulk Fe (106 ps), but shorter than that for the dislocation defects (165 ps). This result indicates that positrons are primarily trapped at the bulk and dislocations. It can be found that as a result of cold-working in Fe, large size of vacancy clusters is produced. The longer lifetimes τ_2, shown in Fig. 2. In addition, the intensity of the longer lifetime component, I_2 is significant after cold-working, which represent the high number density of vacancy clusters. It is noteworthy that longer lifetime τ_2 decreases with increasing the degree of cold-working. It is probable that the size of vacancy clusters shrinks in cold-working, while the dislocation density becomes higher

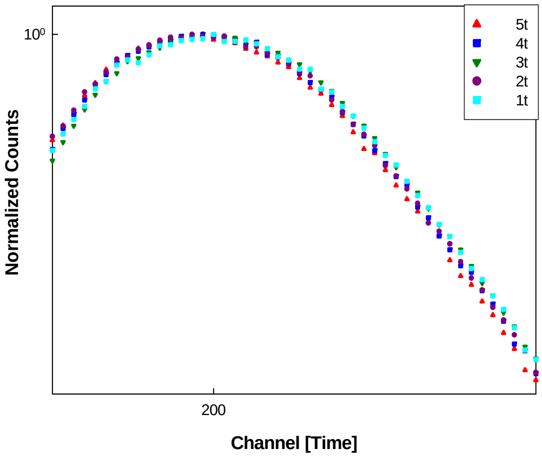

Fig. 2. The measured positron lifetime spectra of Stainless steel 316 alloys.

Table I: Measured lifetime and intensity

Cold-working rate	Bulk (ps)	τ_1 (ps)	I_1 (%)	τ_2 (ps)	I_2 (%)
0 %	108	147	76.85	194.6	23.15
20 %	108	146.7	75.8	200.2	24.2
40 %	108	153.8	92.1	203.4	7.9
60 %	108	157	94.87	208	5.13
80 %	108	158	93.24	215	6.76

Then, we estimated of the single vacancy concentration through Eq. (3) & (4). We used the bulk positron lifetime (τ_b) of the SS 316 alloys from the measured positron lifetime of well annealed SS 316 alloys. Then, the annihilation rate of bulk ($\lambda_b=1/\tau_b$) and monovacancy ($\lambda_d=1/\tau_d$) are calculated. From these data, the positron trapping coefficients for vacancy are determined in SS316 alloys. The trapping coefficient for vacancies in pure Fe was known to be (1.1 ± 0.2) x 10^{15}/s from electron irradiation experiment. Through the combination of the trapping rate and the trapping coefficient, the concentrations of monovacancy for SS316 alloys are estimated. The results are listed in Table II.

Table II: Problem Description

Alloy	trapping rate (κ_d,10^{12}/s)	Concentration (C_d, ppm)
0%	12.412	1.128
20%	13.614	1.237
40%	3.725	0.339
60%	2.407	0.218
80%r	3.341	0.304

4. Conclusions

The positron annihilation measurements were carried out to investigate the vacancy-type defects in SS316 created by cold-working. It is found that a large size of vacancy clusters, as well as dislocations, is produced as a result of cold-working. We also observe that the vacancy clusters do not grow in size in proportion to the amount of cold-working. In contrast, the largest of vacancy clusters becomes smaller and the number density of clusters increases with cold-working. The present positron annihilation study therefore suggests a high sensitivity method for the detection and measurement of open-volume defects present in materials.

REFERENCES

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