Study of microstructural defects in stainless steels with positron annihilation technique and hardness test

Jung-ki Shin *, Yongbok Lee, Junhyun Kwon
Korea Atomic Energy Research Institute, 1045 Daedeok-daero, Yuseong, Daejeon 305-353, Korea
E-mail: jkshin@kaeri.re.kr *

1. Introduction

Investigation of the damaged state of a material is very important for industrial applications. Most mechanical damages start with a change in the microstructure of the material [1].

The positron annihilation lifetime (PAL) techniques have been widely used for studying defects of materials during the last two decades. The PAL can be used as a powerful probe for the measurement of the existence free–volume in materials. 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 [2]. The aim of this study was to investigate the influence of cold-working on the mechanical defects using PAL, and then an attempt was done to establish a correlation between the hardness and the positron annihilation parameters.

2. Experimental and Methods

2.1 Samples Examined

We examined stainless steels 316 samples which were deformed by a cold-rolling. Deformation of the samples was conducted at the room temperature by the conventional rolling. By cold rolling of pure stainless steels 316, five samples with different percentage of deformation were prepared; 0, 20, 40, 60, and 80 %.

The chemical compositions of Stainless steels 316 are given in Table 1.

Table 1. Chemical compositions of SS316 [wt%]

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

2.2 Experimental

A conventional fast-fast coincidence system has been used to measure the lifetime spectra, incorporating two BaF_2 scintillation detectors, two constant fraction differential discriminators, a time to amplitude converter and multi-channel analyzer. The instrumental time resolution of the system is 250 ps of the full width at half maximum (FWHM). The positron sources used for the measurement consist of ^{22}Na sandwiched between two Ni foils. The equipment is kept in a room at a constant room temperature to reduce the electronic drift. Through this PAL system, we can measure the time difference between the appearance of two γ-quanta (start and stop gamma) as like Fig. 1.

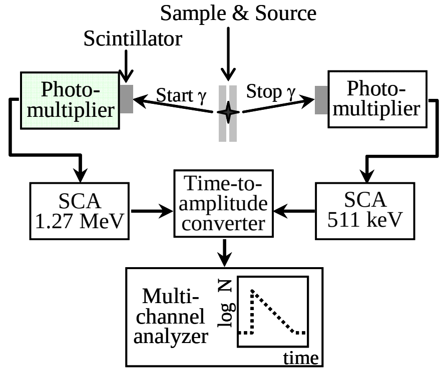

Fig. 1. Scheme of the positron annihilation lifetime spectroscopy (SCA: single-channel analyzer).

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 PALSfit program [3]. Also, we carried out hardness test by Hardness test machine, HM-122, made by Akashi co. 0.5 kg, 15 sec. And then, We compare with micro-vickers hardness numbers and PAL parameters(κ_d) .

2.3 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 λ_b(=1/τ_b, τ_b=bulk lifetime). The positron lifetime of bulk Fe is known to 106 ps, respectively [4]. 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/τ_d, τ_d=defect lifetime), which is schematically shown in Fig. 2. The positron decay function can be described as

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

where each lifetime and intensities are defined as

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

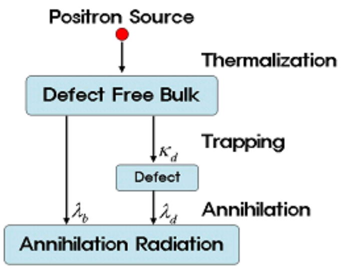

Fig. 2. A schematic of the trapping model includes only one defect type.

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

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

3. Results

The positron lifetime spectra for cold-worked stainless steels 316 were decomposed into two components, τ_1 (shorter) and τ_2 (longer). Fig. 3 shows the dependence of positron lifetime on the percentage of cold work.

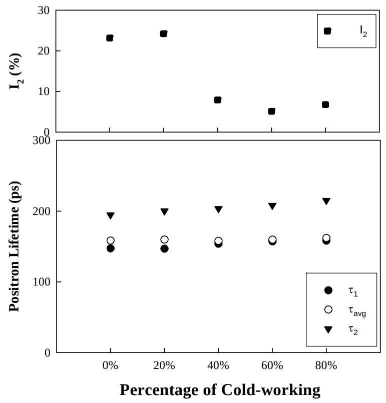

Fig. 3. PALS measured lifetimes and relative intensities for cold-worked SS316.

The observed τ_1 for the rolled Stainless steel 316 is longer than that for the bulk Fe (~110 ps). 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. Also, we calculated trapping rate(κ_d) by equation(3), carried out hardness test using same samples. The results of calculated trapping rates(κ_d) are listed in table 2. Comparing the values between hardness numbers and $1/\kappa_d$, the values increase until 40% cold-worked sample. After 40% cold-working, the values of hardness number and κ_d do not increase significantly but go to saturation, the graphs shown similar shape.

$$\text{Hardness} = a / \kappa_d + b \qquad (4)$$

Where, a and b are constants each 11.24159 and 165.12456.

We find linear relation like (4). The relations of hardness numbers and $1/\kappa_d$ are shown in Fig 4.

Table 2. Trapping rates(κ_d) measured PALS analysis and Hardness test results cold-worked SS316

Cold Working (%)	PALS	Hardness
	κ_d	$HV_{0.5}$
0	0.4608	158.85
20	0.1461	284.70
40	0.0553	339.93
60	0.0513	373.29
80	0.0546	398.16

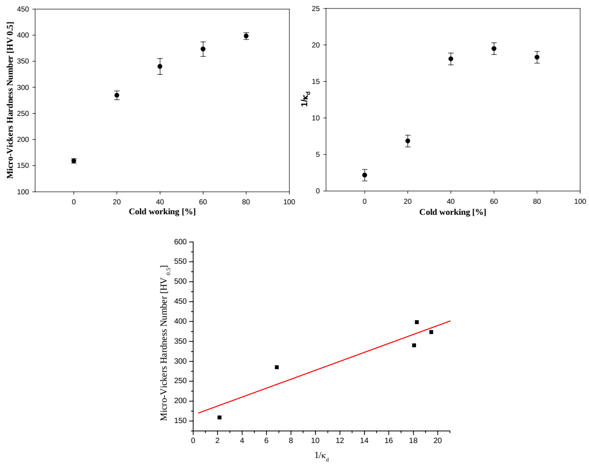

Fig. 4. Relations of calculated $1/\kappa_d$ using PALS parameters and measured hardness numbers for cold-worked SS316.

4. Conclusions

Through the application of the positron annihilation measurement techniques, we investigate the Cold-working induced defect clusters in stainless steels 316. From the PALS, formation of vacancy clusters in the cold-worked materials was confirmed. Then we employed the positron trapping model of a single type of defect. The application PALS provided information on cold-work induced defect production

By combining the trapping rates and hardness numbers, the concentrations of induced defects by cold-working are estimated. From these results, we confirmed that κ_d can be applied to estimate hardness number.

REFERENCES

[1] E.E.Abdel-Hady, Application of the positron Lifetime Spectroscopy to the Study of Microstructural Defects in Steel. (2003).
[2] Tao SJ, J. Chem Phys, 1972, 56, 5499-510.
[3] J.V. Olsen, P. Kirkegaard, N.J. Pedersen, and M. Eldrup, "PALSfit:a computer program for analysing positron lifetime spectra", Risø National Laboratory, Roskilde, Denmark, September (2006)
[4] R.N. West, in: P. Hautojarvi (Ed.), Positron in Solids, Topics in Current Physics, 12, Springer, Berlin, 1979.