Transactions of the Korean Nuclear Society Spring Meeting
Chuncheon, Korea, May 25-26 2006

Positron Annihilation Lifetime Spectroscopy of Vacancy-Type Defects in Cold-Worked Iron

Junhyun Kwon*, Hamdy F. M. Mohamed[+], Yong-Min Kim, Whungwhoe Kim
Korea Atomic Energy Research Institute, 150 Deokjin, Yuseong, Daejeon 305-353
*E-mail: jhkwon@kaeri.re.kr**

1. Introduction

Positron annihilation lifetime spectroscopy (PALS) uses positrons to probe a materials's microstructure. In PALS the lifetime of a positron in a material is determined and the related parameters are measured that reflect the local density of electrons with which the positrons annihilate. PALS can provide the information on the types of microstructural features present in a material, their concentrations, and chemistry. In particular, PALS is the only technique that can sensitively detect vacancy-type defects in a material. Positrons in metals tend to stay longer in an open space where the electron density is relatively low, and to annihilate with the surrounding electrons. By measuring the annihilation gamma rays, we can obtain the information on the annihilation sites.

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. 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 Measurement Technique

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 TAC. Hence the time between two standard pulses from the two modules is the positron lifetime. Then the time spectrum is stored in a MCA. The positron system has a time resolution of 290 ps. The equipment was kept in a room where the temperature was controlled to electronic drift.

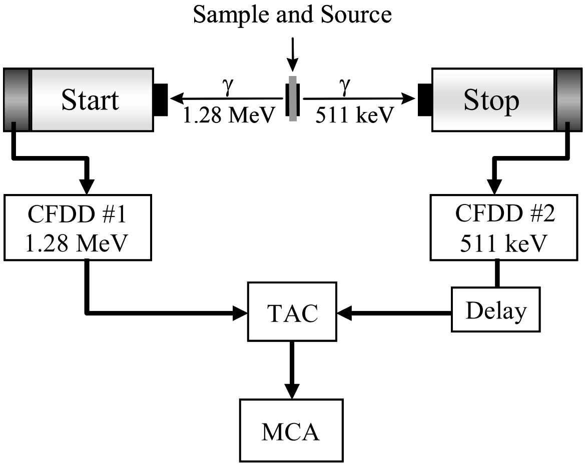

Fig. 1. Scheme of positron lifetime experiment in fast-fast coincidence (CFDD: Constant Fraction Differential Discriminator, TAC - Time to Amplitude Converter, MCA-Multi Channel Analyzer)

We used the PATFIT package to perform the deconvolution of the positron lifetime spectra [1]. The program RESOLUTION was applied to analyze the lifetime distributions corresponding to Fe foil in determining the resolution functions of the system and to determine the fraction of positron annihilations in the source. With the resolution functions, we then used the program POSITRONFIT to conduct the deconvolution of the positron lifetime spectra measured on the test samples.

2.2 Samples Examined

We examined pure Fe 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 Fe, five samples with different percentage of deformation were prepared; 2, 5, 10, 20, and 40 %. For a source correction, well-annealed pure Fe was also tested.

[+] On leave from El-Minia University, El-Minia, Egypt.

3. Results

3.1 Source Corrections

A certain fraction of positrons annihilate in the source itself because the positron source ^{22}Na is covered by thin Al foils. It is, therefore, required to subtract this fraction from the measured raw spectrum. Using the well-annealed Fe sample, we performed a source correction on the assumption that three different components are involved in the lifetime spectra, which include the Al foil itself, salt/surface and positronium [2]. We analyzed a spectrum with 1.6×10^7 total counts and obtained three lifetimes, τ and their intensities, I such as τ / I (Al foils) = 214 ps / 8.77 %, τ / I (salt & surface) = 367 ps / 6.51 %, and τ / I (positronium) = 4461 ps / 0.09 %.

3.2 Effect of Cold-Working on PALS

The positron lifetime spectra for cold-worked Fe 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 (> 5 vacancies) is produced. The longer lifetimes τ_2, shown in Fig. 2, range from 260 to 310 ps. 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.

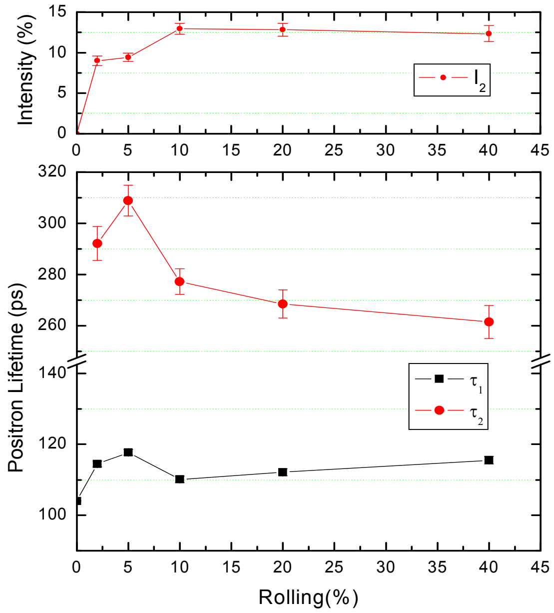

Fig. 2. Dependence of positron lifetimes and intensities on the percentage of cold work in iron.

4. Conclusion

The positron annihilation measurements were carried out to investigate the vacancy-type defects in Fe 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 [3]. 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

[1] P. Kirkegaard, N.J. Pedersen, M. Eldrup, PATFIT-88: A date processing system for positron annihilation spectra on mainframe and personal computers, Riso Nat. Lab., Denmark, 1989.

[2] T.E.M. Staab, B. Somieski, R. Krause-Rehberg, The data treatment influence on the spectra decomposition in positron lifetime spectroscopy Part 2: The effect of source corrections, Nuclear Instruments and Methods in Physics Research A381, p.141, 1996.

[3] H. Ohkubo, Z. Tang, Y. Nagai, M. Hasegawa, T. Tawara, M. Kiritani, Positron annihilation study of vacancy-type defects in high speed deformed Ni, Cu and Fe, Materials Science and Engineering A350, p.95, 2003.