Rolling Effects on Advanced Reduced-Activation Alloy Studied by Positron Annihilation Lifetime Spectroscopy

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1. Introduction

One of the main candidates for reactor structural materials is ferritic/martensitic steel. In the European fusion development agreement, the reduced activation ferritic/martensitic (RAFM) called Eurofer'97 was released, which consisting of Fe-9Cr-1.2W to reduce neutron activation from fusion reactions. Also, Japan developed F82H by slightly changing its chemical composition [1]. KAERI developed a structural material with zirconium added to RAFM steel, called advanced reduced-activation alloy (ARAA). Several types of ARAA steel have been introduced since 2001 as a structural material of the nuclear fusion reactors [2]. The ARAA has been mainly applicable to the test blanket module (TBM) and divertor in the international thermonuclear experimental reactor (ITER). The TBM and divertor can be damaged by fast neutrons because those are exposed to high temperatures, fast neutrons, photons, electrons, and other ions emitted by fusion reactions (Fig. 1).

In the case of nuclear structural materials, the point defect and defect cluster could be generated, and as a result, the condition of the structure material may be damaged and the safety of the material may be impaired. Therefore, in the case of the first wall, it is important to consider the safety of the first wall at the region, where high-temperature plasma is generated, and there should not be many activations due to the neutrons generated by nuclear fusion, and it should withstand high temperatures well. It will be very important to identify the characteristics of the most stable state of the material as the changes in the process.

Positron annihilation lifetime spectroscopy (PALS) has been used for detecting nanoscale defects. Its non-destructiveness and high sensitivity of defects can be applicable to metal alloys, polymers, and semiconductors. By thermomechanical process of metal alloys such as rolling in certain temperature, the microstructure of the alloy samples could be changed, which could be detectable by PALS. In this study, we investigated the rolling effects on ARAA alloy using PALS.

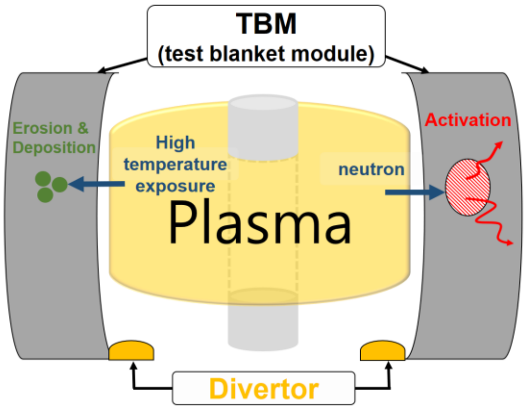

Fig. 1. The structure of the test blanket module (TBM) and divertor on the first wall of the international thermonuclear experimental reactor (ITER) and the effects of nuclear reactions

2. Materials and Methods

2.1 Positron Annihilation Lifetime Spectroscopy

Positron annihilation lifetime spectroscopy (PALS) was used to analyze the defect of ARAA steel in this study. The PALS system consists of high voltage power supply (HVPS), fast plastic scintillators, photomultiplier tubes (PMTs), constant fraction differential discriminators (CFDDs), nanosecond delay, multi-channel analyzer (MCA), and time to amplitude converter (TAC). Figure 2 shows the structure of the installed devices. Two HVPS were connected to the PMT base and a high voltage of -1.9 kV was applied for the gamma energy of the start signal (1.27-MeV) and -2.5 kV for the gamma energy of the stop signal (0.511-MeV). The output signal of the PMT base has been received to two CFDDs. The generated stop signal was shifted in the nanosecond delay to optimize the error and stored as MCA through time amplification in the TAC. The MCA integrated with the analogue to digital converter (ADC) and was sent into 4,096 channels.

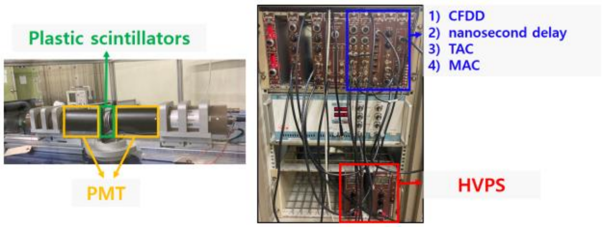

Fig. 2. Positron annihilation lifetime spectroscopy (PALS) system. PMT: photomultiplier tube HVPS: high voltage power supply, CFDD: constant fraction differential discriminator, TAC: time-to-amplitude converter, MCA: multi-channel analyzer.

The positron source used for PALS was the Na-22 radioactive isotope of 30 μCi as a 2.5-μm nickel foil, overlapping like a sandwich on both sides. The 8×8 mm² positron source was positioned between the samples. In order to minimize the air layer between the sample and the source, it was sealed and used. Figure 3 illustrates the sample setting process. For TMP 13C, TMT 32, TMP 19, TMT 20 positron lifetime spectra were obtained more than 2×10^6 counts during 7,200 seconds.

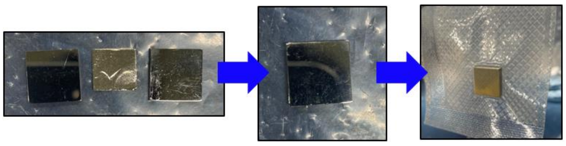

Fig. 3. Sample preparing process for positron annihilation lifetime spectroscopy (PALS). Glared blocks were advanced reduced-activation alloy (ARAA) samples, and light color is the positron source with a nickel foil

The time resolution was about 286 ps in full width at a half maximum (FWHM). The positron lifetime spectra were analyzed by two unfolding modules, *Resolutionfit* and *Positronfit*, in *PALSfit3* [3]. In the sample of metals, the positron lifetime appears in two parameters. The first is a value that takes into account the probability that the penetrates the sample will annihilate in the bulk state and the probability that it will annihilate after being trapped in the free state, and is called the short lifetime (τ_1). The intensity of the short lifetime is indicated by I_1. Second, it is the value in which the positron penetrated into the sample increases in the lifetime of the positron within the defect and annihilates relatively slowly compared to the bulk state, this is called the long lifetime (τ_2). The intensity of the long lifetime is indicated by I_2. The mean lifetime can be expressed through the following equation:

$$\tau_m = \tau_1 I_1 + \tau_2 I_2$$

2.2 Samples

ARAA steel was created by adding 0.01wt% zirconium to Fe-9Cr-1.2W based ferritic/martensitic steels. The basic composition of ARAA steel is described in Table 1. All samples were double normalized for 40minutes at 1000℃ before rolled, and each sample was rolled in a different method. Each sample size was 10×10×1 mm³. Specific sampling methods for ARAA steel are described in Table 2.

Table I. Chemical composition of the advanced reduced-activation alloy (ARAA) steel (wt.%)

Element	Composition (wt%)	Element	Composition (wt%)
C	0.1	V	0.2
Si	0.1	Ta	0.07
Mn	0.45	N	0.01
Cr	9	Ti	0.01
W	1.2	Zr	0.01

Table II. Thermomechanical process of advanced reduced-activation alloy (ARAA) steel

Sample	Annealing and rolling method
TMP 13C	[1]N+N
TMT 32	N+N+15%[2]HR@700℃
TMP 19(34)	N+N+25%HR@700℃
TMT 20	N+N+35%HR@700℃

[1]N: normalizing at 1000℃/40 minute/air-cooling
[2]HR: hot-rolling

3. Results

For pure iron measured in PALS, the short lifetime is known to observe around 110 ps. The short lifetime of the four samples analyzed was observed to be similar to that of pure iron. In the case of EUROFER'97 steel similar to ARAA steel, the short lifetime was analyzed to be less than 100 ps [4]. Figure 4 illustrates the measured short lifetime, and there was no significant difference in the value of the short lifetime between unrolled and rolled. The long lifetime was observed between 250 ps and 310 ps. Similar to what was mentioned earlier, the value of the long lifetime did not differ between rolled and unrolled. Details for each sample were described in Table 3.

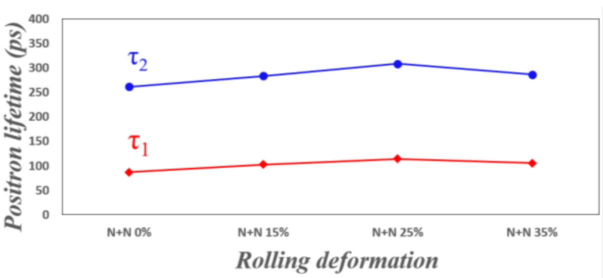

Fig. 4. Short and long lifetime of the samples in different rolling deformation.

Table III. Short lifetime (τ_1), long lifetime (τ_2), the intensity of short lifetime (I_1), the intensity of long lifetime (I_2), and mean lifetime (MLT).

Sample	Lifetime				
	τ_1 (ps)	I_1 (%)	τ_2 (ps)	I_2 (%)	MLT (ps)
TMP 13C	87.0	56.4	261.3	43.6	163.1
TMT 32	102.7	60.1	283.4	39.9	174.9
TMP 19(34)	114.1	66.9	308.3	33.1	178.3
TMT 20	105.7	59.9	286.6	40.1	178.2

Fig. 5 and 6 show the intensity and the mean lifetime of the positron, respectively. In figure 5, it is observed that the positron intensity (I_i) changes when rolling at 25%, and in figure 6, it is observed that the mean lifetime increases with increasing rolling strain and then saturated. In the case of positron intensity, it appears to have changed when rolling 25%, but it is within the error range. As with rolled at 700 ℃ and cold rolled, the mean lifetime shows a similar trend. It is reported that the mean lifetime changes before 9% rolling, but the mean lifetime is saturated after 9% rolling [5]. When rolling up to 9%, dislocations and vacancies are growing, and after 9%, growth is expected to stop and become saturated.

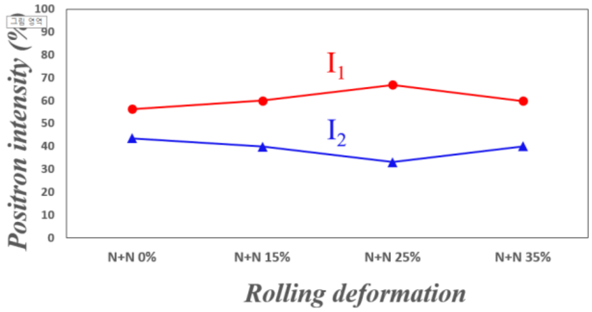

Fig. 5. Lifetime intensity of the samples in different rolling deformation

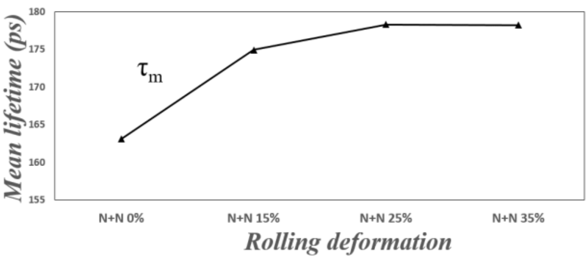

Fig. 6. Mean lifetime (τ_m) of the samples in different rolling deformation

3. Conclusions

In this study, unrolled and rolled ARAA steel was analyzed using PALS. It was observed that when rolling to 9%, the mean lifetime gradually increased, and after 9% rolling, the mean lifetime was saturated. The increase in the mean lifetime before 9% rolling is expected to increase the mean lifetime due to the slow growth of dislocations and vacancy during the rolling process. After 9% rolling, the growth is expected to slow and saturate. As a result of this study, when rolling the ARAA steel, it is expected it is safe to process the rolling so that the percentage of deformation due to rolling is more than 9%, and use ARAA steel rolled more than 9% as a structural material. Furthermore, through this result, it is planned to analyze whether there is a change according to the temperature applied by hot-rolling.

Acknowledgment

This work was supported by Radiation Technology R&D program through the National Research Foundation of Korea funded by the Ministry of Science and ICT (NRF-2017M2A2A6A05018529).

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