Practical Application of PALS in Japan (Sub-Nanometer Material Evaluation)

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1. Introduction

Positron annihilation lifetime spectroscopy (PALS) is a technique that can evaluate atomic-level defects and intermolecular space and pore structures with nondestructive and high sensitivity. PALS is used to evaluate the free volume void size of polymer materials and the degree of fatigue accumulation in the early stages of metal fatigue, and in recent years, material evaluation using PALS has been actively conducted. Since 2010, Toyo Seiko has been working with the National Institute of Advanced Industrial Science and Technology (AIST) to develop a positron lifetime measurement device (PSA) for industrial use. Today, this device has been improved to the point where positron lifetime values can be obtained without requiring specialized knowledge or skills, and it has begun to be used in general industry as well. This article introduces PSA developed by Toyo Seiko and AIST.

2. Issues and solutions in device development

Before Toyo Seiko and AIST developed PSA, positron lifetime measurement method was a technique that was basically only used by a limited number of researchers in legally designated radiation-controlled areas in Japan.

The most significant development was the improvement of ^{22}Na sealed radiation source for positron lifetime spectroscopy by Japan Radioisotope Association (JRIA). Figure 1 shows the current sealed ^{22}Na source. The thickness of the Kapton film was thinned down to 5.0 μm, and the annihilation within the film was significantly reduced. Since radioactivity is less than 1.0 MBq, no controlled area or personnel qualifications are required for handling in Japan. Other points that were important for the commercialization of PSA are shown in Table 1.

3. PSA device

There are three types of PSA, and the standard model is shown in Figure 2. The configuration of the PALS system is shown in Figure 3, and the sample setup (actually just placing the sample on sealed radioisotope) is shown in Figure 4.

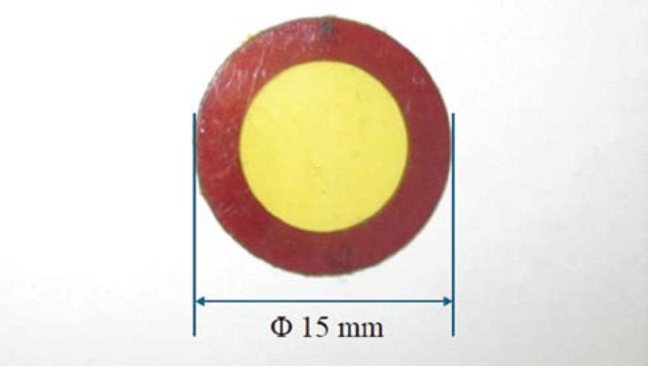

Fig. 1 Sealed ^{22}Na source supplied by JRIA

Table 1 Issues and Solutions

Thema	Issues	Solutions
Preparation of measurement sample	The most common setup for the measurement sample is to sandwich a sealed source film between two measurement samples. So researchers had to deal with sealed sources.	The anti-coincidence method (patented Technology[1]) has been put into practical use, making it possible to perform measurements using a single sample without handling sealed source.
Measurement and analysis program	The equipment is designed for professionals, so it is difficult to use for people who are not familiar with PALS.	We have developed dedicated software with a simple interface that allows measurements and analysis to be completed with just a few clicks.
Positron lifetime standard material	They are available only for polymer and quartz, and are not supplied for semiconductors or metals.	Four types of reference materials are supplied by AIST to suit any user's needs.

Fig. 2 Standard model : PSA Type-LII

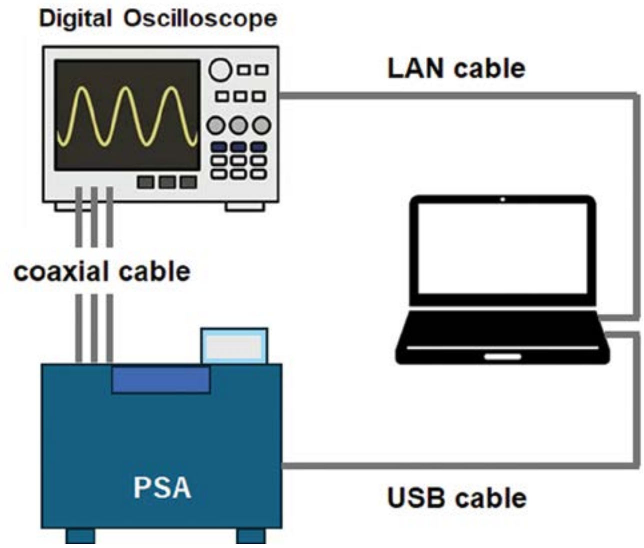

Fig. 3 Configuration of PSA

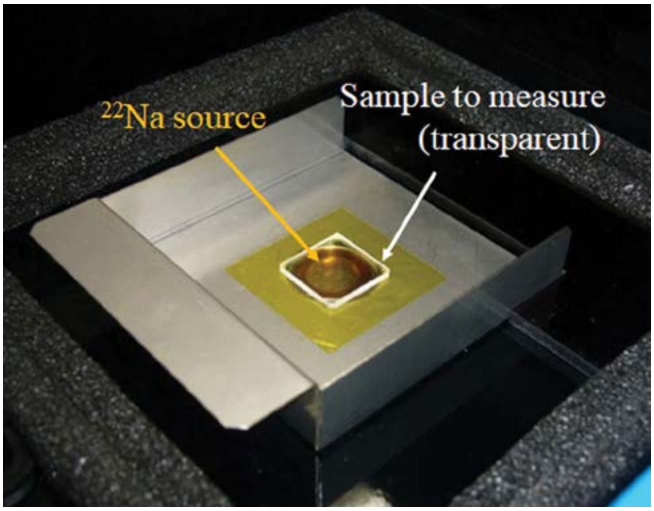

Fig. 4 Sample set-up in PSA

4. Case studies of PSA measurement

① Shot peening on stainless steel [2]

Bright annealed Type 304 stainless steel was shot peened with several levels of coverage and was measured by PSA. The shot peening conditions are shown in Table 2, and the results of the positron lifetime measurements and the FWHM (Full Width at Half Maximum) of X-ray diffraction are shown in Figure 5. As shown, the positron lifetime increases and saturates faster than FWHM. This phenomenon can be explained: PSA evaluates the increase of dislocations, whereas FWHM captures the variation in lattice spacing. In this way, PSA makes it possible to evaluate trends even in cases where X-ray diffraction does not clearly capture the trend of change.

In addition, PSA has the following advantages over X-ray diffraction and other analytical instruments:

- Detects vacancy-type lattice defects (sub-nano size) with high sensitivity. Physical properties can be evaluated using a different approach from residual stress evaluation.
- Evaluates coarse crystals and single-crystal material. Also, liquids can be evaluated.
- Evaluates ten times deeper than X-ray diffraction. Therefore, the quality of shot peening can be evaluated non-destructively.

Table 2 Shot peening conditions

Sample	Type 304 stainless steel (15 x 15 x 3 mm)
Shot media	CCW12 (Φ0.3 mm, 500 HV)
Shot peening machine	Direct air pressure type
Nozzle	Φ10 mm
Pressure	0.20 MPa
Peening time	0, 12, 36, 72, 120 sec

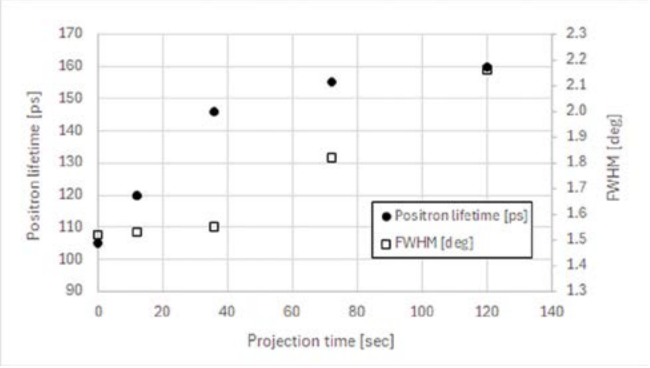

Fig. 5 Changes in positron lifetime and FWHM of shot-peened Type 304

② Monitoring fatigue process [3]
The fatigue process of the rotating bending fatigue specimens Type 316 was monitored by PSA. The shape of the specimen is shown in Figure 6. A single notch was introduced on the surface of the specimen to make it easier to observe the fatigue crack growth from the notch. The positron lifetime was measured at the notch and at the 180° opposite side.

The fatigue damage was non-destructive and sequentially evaluated by PSA. The results are shown in Figure 7. The positron annihilation lifetime increased with increasing fatigue loading cycles. Lifetime at the notch root became longer than that at the smooth section after crack initiation. The precision of lifetime analysis was high enough to detect small plastic zones with high dislocation density at the fatigue crack tips.

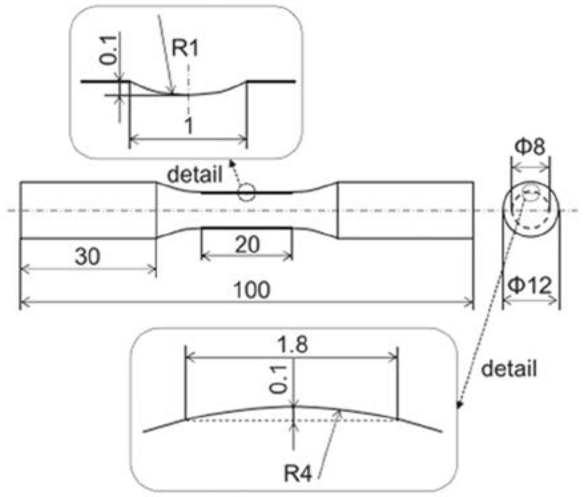

Fig. 6 Fatigue specimen configuration and the detail of notch

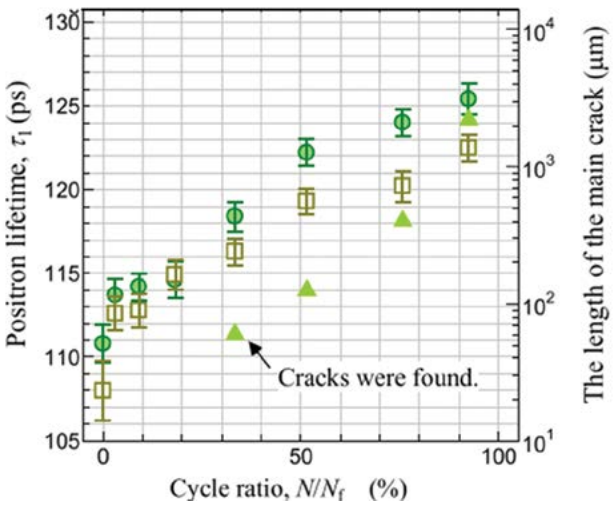

Fig. 7 Positron annihilation lifetime analytical results and crack length

5. Conclusion

In this article, we have described the challenges and solutions for practical application of a positron lifetime measurement device, PSA, which is a measurement method that can evaluate atomic-level defects and intermolecular gaps and pore structures non-destructively and with high sensitivity. In addition, as case studies of measurement of metallic materials, the positron lifetime during shot peened Type 304 and the fatigue process of Type 316 were discussed.

We believe that this device has achieved its goal of practical application in terms of developing the basic components. However, positron lifetime measurement technology is still in the development stage for industrial use, and improvements are being made toward further practical use.

6. Acknowledgements

We would like to thank for the generous cooperation in promoting the industrial use of PSA to JRIA.

Reference

[1] US 8,785,875 B2
[2]N. Uesugi, M. Yamawaki, K. Hattori, Y. Watanabe, "Application of on-site positron annihilation lifetime spectroscopy system as non-destructively shot peening evaluation technique", proceedings of 14th international conference of shot peening (ICSP-14), 2022058.
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