

I-1. PROJECT RESEARCHES

Project 6

Advanced Applications of Materials Irradiation and Characterization Techniques Using High Energy Particles

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OBJECTIVE:

We have developed and maintained various irradiation facilities (KUR SSS for neutrons and KURNS-LINAC targets for electrons) and characterization facilities (positron annihilation spectroscopy and thermal desorption spectroscopy systems) in Institute for Integrated Radiation and Nuclear Science. For stimulating joint-use research projects in this area, this project aims to further advance the development and the improvements of facilities and to study novel irradiation effects.

RESEARCH SUBJECTS:

R7P6-1 Study to improve transport and measurement performance of slow positron beamline

The current performance of the positron beam was examined to confirm the availability of typical analytical techniques after a problem with the positron source occurred.

R7P6-2 Micro-vickers hardness in neutron-irradiated Fe-Cr alloys: Part 2

The progress of phase separation in Fe-Cr binary alloys was evaluated by positron annihilation spectroscopy and correlated with hardness for samples neutron-irradiated at 473K and 573 K.

R7P6-3 Positron and Gamma ray excited light-emissions from ZnO

The radiation-induced luminescence spectra of a ZnO crystal substrate were investigated, specifically comparing emission characteristics when excited by positrons and gamma rays.

R7P6-4 Hydrogen trapping behavior in vacancy type defects in B2-type Fe-Al alloys

Positron annihilation spectroscopy was used with elastic recoil detection analysis to investigate hydrogen-vacancy interactions in the Fe-Al alloys irradiated at KURNS-LINAC.

R7P6-5 Structural changes in Mn-DLC films due to Mn elution

Changes in vacancy-type defects within the DLC film accompanying Mn dissolution were investigated using positron annihilation spectroscopy (PAS) with a slow positron beam.

R7P6-6 Evaluation of density in diamond-like carbon coatings using positron annihilation spectroscopy

DLC coatings were systematically characterized using positron annihilation spectroscopy, with a focus on the relationships among deposition conditions, defect structure, and film density.

R7P6-7 Evaluation of irradiation damage and the effects of heat treatment on Au-Si ion-irradiated SiO₂/Si substrates using positron annihilation spectroscopy

To evaluate the Au-Si ion irradiated damage of SiO₂/Si surfaces before and after annealing, positron annihilation spectroscopy was performed.

R7P6-8 Effects of pulse irradiation by charged particles on damage structures in metals

The effects of pulse irradiation on the defect evolution in metals were evaluated for pulse-ion-irradiated samples and numerical simulations were performed for comparison.

R7P6-9 Development of slow positron beamline using electron linear accelerator

A slow positron beam generated by a 30 MeV electron beam was introduced to a sample chamber; the annihilation gamma-rays were successfully detected by a scintillation and Ge detectors.

Study to improve transport and measurement performance of a slow positron beamline

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INTRODUCTION: Positron annihilation spectroscopy is an important analytical method to detect vacancy-type defects and vacant spaces of materials. Energy-variable mono-energetic positron beams (slow positron beams) are essential to perform depth-dependent positron annihilation spectroscopy of surface layers such as ion-implanted layers or thin films. As moderation efficiencies to obtain slow positron beams are typically low ($<10^{-4}$), intense positron sources are required for practical use. A positron source using pair-creation by gamma-rays from a nuclear reactor have been developed by using Kyoto University research Reactor (KUR) to obtain a slow positron beam for materials analysis. In October 2025, a problem with the positron source occurred and the positron intensity decreased to $>50\%$ of those before the problem. Owing to high radiation levels of the activated source, it cannot be repaired during the yearly operation of KUR. Thus, we investigated the state of the positron beam and examined the availability of analytical techniques without repairing the source.

EXPERIMENTS: Energy distributions of the positron beam from the source were evaluated by changing the chopper bias voltage of a positron pulsing system[1]. Count rates of a Ge detector were used as a measure of the positron intensity. Doppler broadening annihilation radiation (DBAR) and positron annihilation lifetime spectroscopy (PALS) were examined for the positron beam without any extraction voltages. For scintillation detectors used in PALS measurements, three types of scintillation BaF₂ crystals (small $\phi 20 \times 20$, Medium: $\phi 25 \times 25$, Large: $\phi 50 \times 40$ mm) were compared[2]. A Kapton sample was measured at an acceleration energy of 2 keV as a reference material.

RESULTS: We found that electrical connection for the source electrodes (converter, moderator and lens) was inactive. Fig. 1 shows the chopper voltage dependences of positron count rates. Without extraction voltages, positron energies around 10 eV were observed. It can be ascribed to charging-up effects of the electrodes. Fig. 2 compares the count rates for three-types of BaF₂ crystals. Previously used small BaF₂ gave ~ 14 cps but the introduction of the medium BaF₂ increased to ~ 23 cps without serious degradation of the spectra. Appropriate lifetimes cannot be obtained with the large BaF₂ as shown in the previous study[2]. Thus, the medium size BaF₂ was chosen for PALS measurements.

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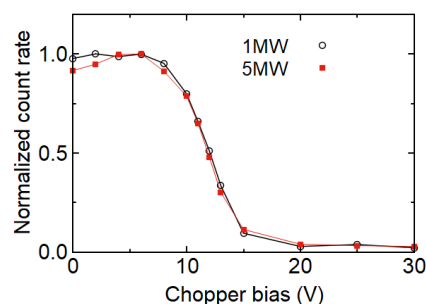


Fig. 1. Normalized count rates as a function of chopper bias voltage.

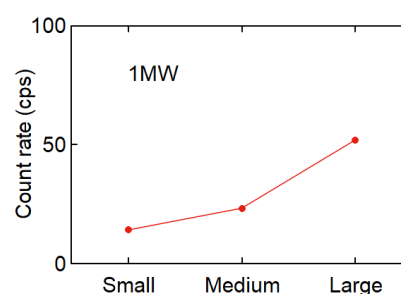


Fig. 2 Count rates of lifetime spectroscopy for different BaF₂ sizes.

Micro-Vickers Hardness in Neutron-irradiated Fe-Cr Alloys: Part 2

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INTRODUCTION: Ferritic stainless and heat resistant steels used as nuclear reactor peripheral materials have high Cr content [1]. In these materials, ductility and toughness remarkably decrease, and hardness and tensile strength increase by aging from 593 to 813 K. This phenomenon is caused by the formation of Fe-rich and Cr-rich phase, and is called 475°C embrittlement [2]. These changes in mechanical properties are an important issue in terms of evaluation of aged deterioration when it was used as a reactor structural material. Positron annihilation spectroscopy (PAS) is very powerful tool to obtain the information of vacancy-type defects (even single vacancies) and precipitates. In Fe-Cr alloys used in this study, positron affinity of Fe is lower than that of Cr [3]. Therefore, we can detect the formation of Fe-rich phase in phase separation of Fe-Cr alloys using PAS. The purpose of this study is to detect the progress of phase separation using PAS, and to obtain the correlation between the hardness and phase separation in Fe-Cr binary alloys irradiated with neutrons at 473K and 573 K.

EXPERIMENTS: Fe-x wt.%Cr (x = 0, 9, 15, 30, 45, 50, 70, 85, 91, and 100) binary alloys were used in this study. The weight of high purity Fe (99.99%) and Cr (99.99%) were measured, and samples were melted by the arc melting. For neutron irradiation, samples with diameters of 3 mm and thickness of 0.25 mm were cut using the wire electric discharge machine. Fe-xCr (x = 30, 45, 50, 70, 85, 91, and 100) were annealed at 1273K for 1h, and Fe-xCr (x = 0, 9, 15) were annealed at 1073K for 1h in a vacuum ($< 4 \times 10^{-4}$ Pa), and then water-quenching was performed for the suppression of phase separation. The neutron irradiation was carried out at the Material Controlled Irradiation Facility (SSS) of Kyoto University Reactor (KUR) [4]. The doses were 0.44×10^{-3} , 0.5×10^{-3} , and 2.1×10^{-3} dpa, at 473 K (0.5×10^{-3} dpa) and 573 K (0.44×10^{-3} and 2.1×10^{-3} dpa). Vickers hardness was measured at room temperature using an HMV-T2 (Shimadzu) with a load of 0.9807 mN (Hv0.1) and a dwell time of 15 s.

RESULTS: The hardness increased after neutron irradiation in all samples. In Fe-xCr (x = 0, 9, and 15), the hardness is highest after irradiation at 473 K. In Fe-xCr (x = 30, 45, 50, 85, and 91), the hardness of samples irradiated at 473 K for 47 h is almost the same as that of samples irradiated at 573 K for 42 h. In Fe-70Cr irradiated at 473 K, it is expected that irradiation-induced defects, especially those present in the Cr-rich phase, have a significant influence on the hardness. In Fe-xCr (x = 15, 30, 45, 50, and 70) irradiated at 573 K for 200 h, the hardness is lower than that after irradiation at 573 K for 42 h. It is expected that long irradiation times lead to significant progress of phase separation, and that the wavelength of Fe/Cr compositional fluctuations may increase. Consequently, dislocations may migrate more easily through the two phases. This possibility should be investigated using thermally aged Fe-Cr alloys.

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- [2] R.O. Williams, Trans. AIME 212 (1958) 497.
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Positron and Gamma ray excited light-emissions from ZnO

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INTRODUCTION: Zinc oxide (ZnO) is a widely studied wide-bandgap semiconductor because of its excellent optical properties and diverse applications in optoelectronics, gas sensors, and scintillator technologies [1]. ZnO is known to exhibit visible defect-related luminescence, such as a broad green band emission. The specific characteristics of this visible emission are highly dependent on the type, concentration, and distribution of native point defects—such as oxygen vacancies or zinc interstitials—present within the crystal lattice. Photoluminescence (PL) spectroscopy, using laser or UV light as an excitation source, has been the standard technique for characterizing these defects. While PL is generally surface-sensitive due to the limited absorption depth of light, different types of radiation such as positron and gamma ray can probe various depths and volumes within the crystal. This brief report investigates the radiation-induced luminescence spectra of a ZnO crystal substrate, specifically comparing emission characteristics when excited by positrons and gamma rays.

EXPERIMENTS: Sample used for the present study is ZnO bulk single crystal substrate. The ZnO substrate was irradiated at room temperature with gamma-rays of 1.17 and 1.33 MeV from a cobalt-60 source and positron source of Institute for Integrated Radiation and Nuclear Science, Kyoto University. The photo-emission measurements were performed by using a charge coupled device (CCD) equipped spectrometers.

RESULTS: The provided spectral data illustrates the luminescence intensity of a ZnO single crystal as a function of wavelength under two distinct excitation sources. The bottom panel displays the luminescence spectrum induced by gamma-ray excitation [2]. This spectrum exhibits a characteristic, broad emission band extending from approximately 400 nm to over 800 nm, with a pronounced peak maximum centered around 550 nm. This broad green-yellow emission is typical for bulk ZnO crystals and is generally attributed to deep-level intrinsic defects dispersed throughout the crystal lattice. In contrast, the top panel shows the luminescence spectrum obtained under positron excitation. While a broad emission band in the green region (500–600 nm) is still evident, the spectral shape and quality differ significantly from the gamma-ray excited spectrum. One possible reason for these observed spectral differences is considered to be the difference in the penetration depths of the respective radiation types. Gamma rays are highly penetrating electromagnetic radiation. They deposit their energy deep within and throughout the bulk of the ZnO crystal. Consequently, the gamma-ray excited spectrum represents the averaged defect luminescent properties of the entire bulk volume. In contrast, positrons interact strongly with matter and possess a significantly shallower penetration depth. When a positron interacts with the sample, it implants and thermalizes primarily within the near-surface region of the crystal. Therefore, the positron-excited spectrum acts as a highly localized probe, reflecting the specific defect structure near the surface.

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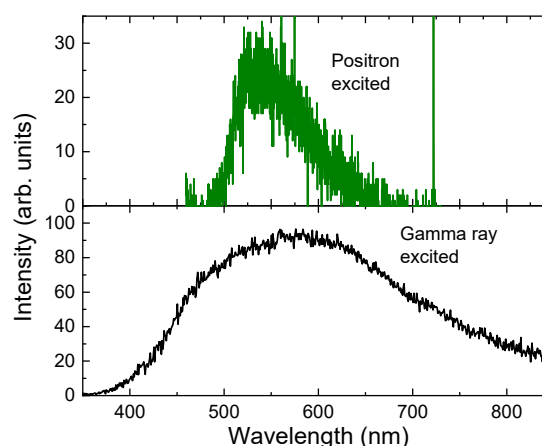


Fig. 1. Radiation-induced luminescence spectra of a ZnO single crystal substrate. The top and bottom panels display the emission spectra under positron and gamma-ray excitation, respectively.

Hydrogen trapping behavior in vacancy type defects in B2-type Fe-Al alloys

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INTRODUCTION: Fe-Al alloy is used as high strength at high temperature material because of its good properties such as specific strength to weight ratio, oxidation resistance. First-principles calculation indicates that not only one hydrogen atom but multiple hydrogen atoms can be trapped in a single vacancy in B2 ordered Fe-Al alloys. However, the interaction between vacancy and hydrogen atoms in this Fe-Al alloy is not cleared yet. We have been investigated this interaction in Fe-Al intermetallic alloy. Previous experiments have reported the changes in the positron annihilation S-parameter distribution and a decrease in positron lifetime, suggesting the possibility of hydrogen trapping at vacancies near the surface of this alloys [1]. To verify whether the changes observed by positron annihilation measurements were due to the trapping of hydrogen into vacancies, elastic recoil detection analysis (ERDA) was performed on the same sample.

EXPERIMENTS: Fe50at.%Al alloy sample was prepared by arc melting method. Sliced samples with the thickness of 0.5 mm were annealed at 1273 K for 20 h and cool down to 973 K slowly and then quenched. These specimens were irradiated with 8 MeV electron up to the fluence of 4×10^{18} /cm² by LINAC at KURRI. After irradiation, hydrogen was injected for the sample at 0.1 mA/cm² in a NH₄SCN solution bath added 0.001 mol/L H₂SO₄ by electro chemical method for 8, 16 and 80 hours. They were examined by using slow positron beam at KUR and ERDA measurement.

RESULTS: Figure 1 shows the hydrogen distribution obtained by ERDA measurement in a sample injected with hydrogen after electron beam irradiation. For comparison, the figure also shows the distribution of S-parameters, which represent the depth distribution related to vacancies measured with slow positrons, corrected for positive and negative inversion, as a solid line of the same color. Since the depth resolution of ERDA is at least 10 nm and the surface position is determined using another sample, the surface here does not necessarily precisely coincide with that for positron analysis. As the hydrogen injection time increases, the peak of ERDA gradually shifts to deeper level, and overall intensity increases. This indicates that hydrogen is penetrating and diffusing in the depth direction. Also, the trend of the hydrogen distribution of S-parameter and ERDA for each charging time are similar. That is, as the charging time increases, the peaks shift to deeper regions and are located at almost the same depth. This suggests that the S-parameter distribution reflects the distribution of hydrogen, and consequently indicates the presence of hydrogen within the vacancies.

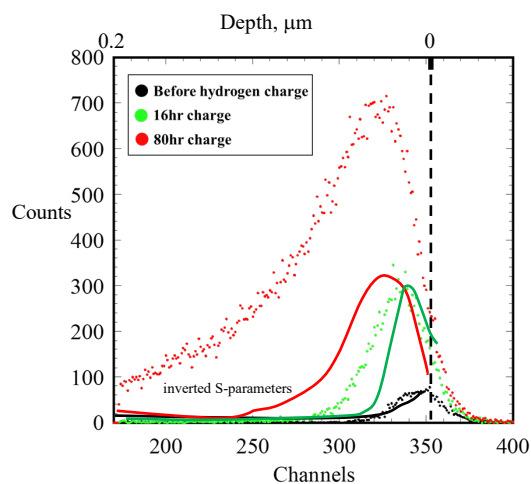


Fig. 1. The depth profile of hydrogen in Fe-Al alloy injected with H after electron irradiation by ERDA. For comparison, the solid line represents the positive/negative inverted S-parameter distribution.

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Structural Changes in Mn-DLC Films due to Mn Elution

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INTRODUCTION: Research on metal-storage DLC films capable of storing bioactive metal elements within metallic implants and supplying them in trace amounts to the body is gaining attention. Understanding the metal release process requires knowledge of the structure of the deposited metal-containing DLC films and how this structure changes due to metal release. Particularly, vacancy-type defects within DLC films containing metals are thought to influence both the amount of metallic elements the DLC film can contain and the amount of metallic elements that are slowly released. In this study, focusing on DLC films containing Mn, which has bone-promoting effects and antioxidant properties, changes in vacancy-type defects within the DLC film accompanying Mn dissolution were observed using positron annihilation spectroscopy (PAS) with a slow positron beam.

EXPERIMENTS: The Mn-DLC film samples were deposited on Si wafers using Ar sputtering with a C/Mn mixed target. The film thickness was 400-500 nm. The Mn-DLC films were immersed in distilled water, removed after 12 h, 24 h, and 48 h, dried, and stored. PAS measurements were performed using a slow positron beam system installed at the B-1 hole of KUR. Positrons generated in the reactor were thermalized, and accelerated up to 30 keV just before the irradiation chamber, and irradiated onto the sample. The γ rays generated by annihilation were observed using a Ge detector.

RESULTS: X-ray photoelectron spectroscopy (XPS) measurements showed the surface Mn composition decreased from 6.6% before immersion to 2.1% after 48 h. Pulsed 2 keV positrons were injected into the Mn-DLC film, and the positron annihilation lifetime was determined by observing the decay of the gamma ray intensity. The positron annihilation lifetime (PALS) increased with immersion time, indicating that the hole-type defects in the Mn-DLC film grew larger. On the other hand, the S-parameters decreased upon immersion. This is considered to be due to changes in the chemical environment around vacancy-type defects.

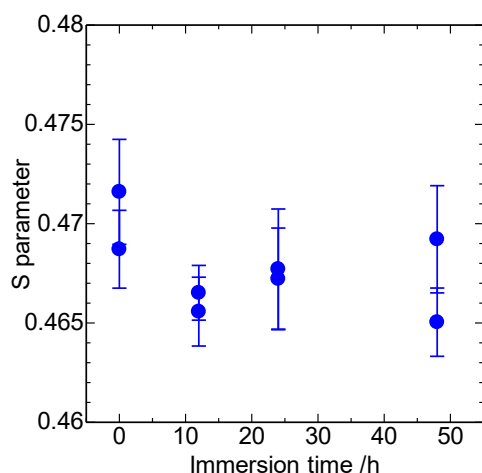


Fig. 1. Dependence of S-parameter of Mn-DLC film on immersion time.

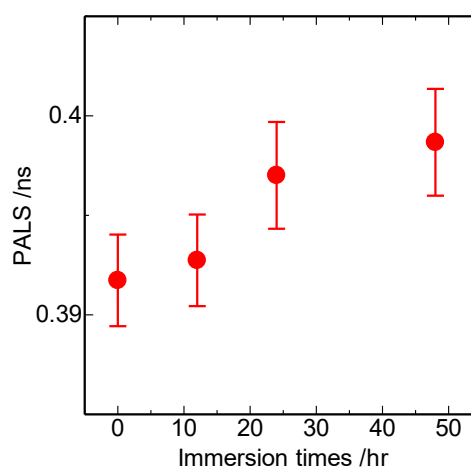


Fig. 2. Dependence of PALS of Mn-DLC film on immersion time.

Evaluation of Density in diamond-like carbon coatings using positron annihilation spectroscopy

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INTRODUCTION: Enhancing the efficiency and durability of mechanical systems has driven increasing interest in carbon-based materials with reduced friction and wear. Among these, diamond-like carbon (DLC) coatings have attracted significant attention due to their favorable tribological behavior combined with high hardness and chemical stability [1]. The properties of DLC are strongly dependent on their atomic structure, which can vary from graphite-like carbon (GLC) to polymer-like carbon (PLC) depending on deposition conditions and fabrication techniques [2]. Such structural variations are governed by factors including hydrogen content and the sp^2/sp^3 ratio, which directly influence the packing density of the carbon network. In addition to the bonding structure, the presence of nanoscale defects and free volume plays a critical role in determining the overall density and associated mechanical properties of DLC coatings [3]. These defects, often associated with open-volume regions, can alter the local atomic arrangement and consequently affect coating performance. Therefore, a precise evaluation of density in relation to defect structure is essential for understanding and optimizing DLC materials. Positron annihilation spectroscopy (PAS) is a highly sensitive technique for probing such open-volume defects and free volume at the atomic scale. In this study, the density of DLC coatings deposited at varying bias voltages is systematically characterized using PAS, with a focus on elucidating the relationships among deposition conditions, defect structure, and film density.

EXPERIMENTS: The DLC coatings were synthesized using a plasma-based ion implantation and deposition (PBII&D) on Si (100) substrates. To control the microstructural characteristics and resulting coating density, negative bias voltages ranging from -1 to -10 kV were adjusted during deposition. The duration time was set to achieve a coating thickness of 1 μm .

RESULTS: Figure 1 presents the variation in coating density and positron lifetime as a function of applied bias voltage. As the bias voltage increased, the density of the DLC coatings decreased, indicating a reduction in atomic packing. Correspondingly, the positron lifetime decreased with increasing density, suggesting a decrease in open-volume defects within the carbon network. This inverse relationship confirms that positron lifetime is sensitive to density-related defect structures in the coatings. The results highlight the strong correlation between deposition conditions, density, and defect characteristics.

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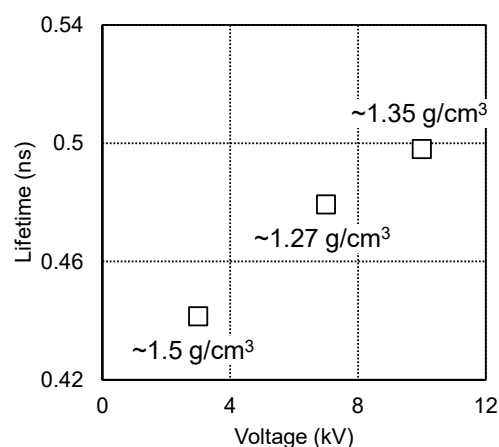


Figure 1. Coating density and positron lifetime as a function of bias voltage.

Evaluation of Irradiation Damage and the Effects of Heat Treatment on Au-Si Ion-Irradiated SiO₂/Si Substrates using Positron Annihilation Spectroscopy

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INTRODUCTION: To evaluate the Au-Si ion irradiated damage of SiO₂/Si surfaces before and after annealing, which might be important to control the formation of Au nanoparticles inside the SiO₂ layer by Au-Si ion irradiation and successive annealing, we have performed the positron annihilation spectroscopy in the present study.

EXPERIMENTS: The ions emitted from the AuSi alloyed metal ion source were irradiated on the substrates using the acceleration voltage of 7 kV at an fluence of $6 \times 10^{15} \text{ cm}^{-2}$. Then the S-parameters obtained from the Doppler broadening for the energy of the γ -ray by the positron-electron annihilation were measured before and after annealing at 1000°C for 45 or 150 min. under atmosphere.

RESULTS: Figure 1 shows the S-parameters obtained for the ion irradiated SiO₂/Si surfaces. For the initial SiO₂/Si surfaces, the S-parameters shows the small peak between the positron energy of 0-4 keV with the peak position around 2 keV, corresponding to the existence of SiO₂ layer on the Si substrate. After the ion irradiation, the S-parameters were significantly decreased in the SiO₂ layer. The reason of the decrease is not known at present, however, almost the same phenomena was reported for the B-ion implanted SiO₂ layer [1]. When the sample was annealed for 45 min., the decrease of the S-parameters was slightly recovered. After the annealing of 150 min., the S-parameters for the SiO₂ layer were almost the same values with the un-irradiated SiO₂ layer, showing the full recover of the irradiated damage in the SiO₂ layer by the annealing. However, the S-parameters for the positron energy below the 4 keV, which correspond to those for Si substrate, were lower than those of the un-irradiated and 45 min.-annealed SiO₂ layers. The results might be indicating the diffusion of the ion-induced defects into the Si substrate from the SiO₂ layer through the SiO₂/Si interface. The relation between the formation of Au nanoparticles inside the SiO₂ layer and such defects is a task for the future.

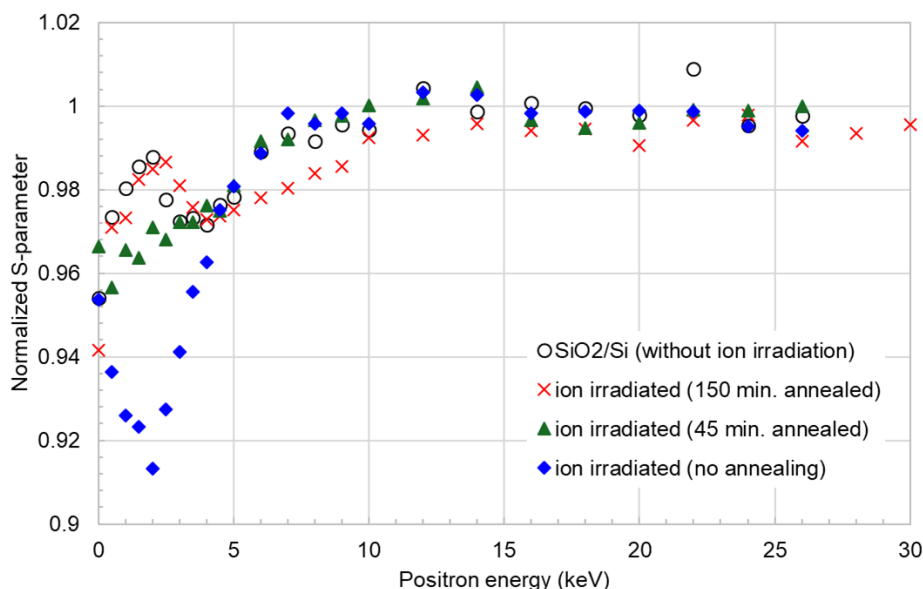


Fig. 1. S-parameters of SiO₂/Si surfaces with and without Au-Si ion irradiation before and after annealing.

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Effects of Pulse Irradiation by Charged Particles on Damage Structures in metals

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INTRODUCTION: Irradiation experiments are crucial for the development of nuclear materials. High-energy charged particles, such as ions and electrons produced in accelerators, are commonly employed in these experiments. The time structures of ion and electron beams from accelerators that use radio frequency or beam scanners are characterized by periodic/discontinuous intensities. The purpose of this study is to clarify the effects of pulse irradiation on the damage defect evolution in metals, by using ion irradiation and simulation.

EXPERIMENTS: Self-ion irradiation of Ni with the accelerating energy of 2 MeV was performed by a tandem accelerator in the Research Center of Ion Beam Technology, Hosei University. The irradiation temperature, total damage, and duty ratio are 573 K, 0.1 dpa and 0.1, respectively. The damage rate of 1 kHz pulse beam, 1 Hz pulse beam and continuous beam are 5.6×10^{-6} , 5.7×10^{-6} and 7.1×10^{-6} dpa/s, respectively. The accumulation of defects after ion irradiation was investigated by the S parameter measurements with positron annihilation spectroscopy in the slow positron beam-line at the Kyoto University Research Reactor at room temperature.

RESULTS: Reaction kinetics were employed to obtain the reaction rates among point defects and their defect clusters. Fig. 1 shows the accumulation of vacancies after 0.1 dpa self-ion irradiated Ni at 573 K with three irradiation conditions: 1 kHz (pulse duration/period = 10^{-4} s/ 10^{-3} s), 1 Hz (0.1s/1s), and continuous beams. Some differences in vacancy accumulation are evident. But the difference of 1 kHz and 1 Hz are 1 % and 5 % of the continuous beam, respectively. The penetration length of the positron varied by changing the accelerating voltage to obtain information at the irradiated area. The S parameter depends on the total number of vacant sites in Ni, including vacancies and irradiation-induced clusters. To avoid the effect of the surface, we chose the area from 300 to 800 nm for the S parameter. As shown in Fig. 2, there exist differences between unirradiated and irradiated specimens. For both simulation and experiment, the effect of 1 Hz irradiation is the highest. But the difference between 1 kHz and 1 Hz irradiation is small in S-parameters compared to the simulation. They are within 0.6 % of the continuous beam. One of the reasons for the difference is that only the residual vacancy concentration obtained by the simulation is used for the comparison. The effects of interstitial type dislocation loops are not introduced. Loops are edge dislocations with a Burgers vector of $1/3[111]$. There exists a dilatational field on one side of the dislocation lines. The detailed comparison with the experiment will require accounting for the effects of loops.

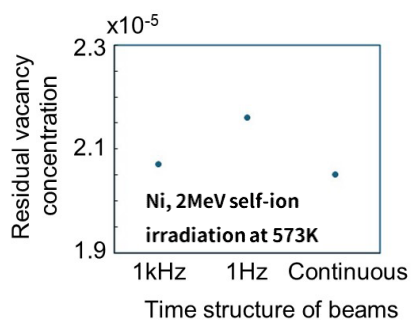


Fig. 1. Simulation of the accumulation of residual vacancies.

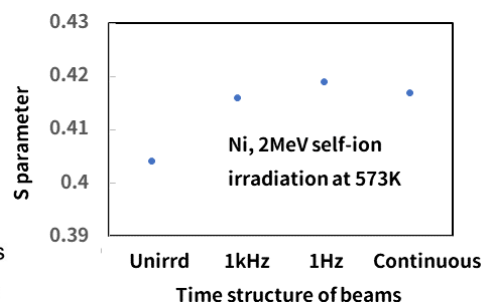


Fig. 2. S-parameter of positron annihilation spectroscopy.

Development of slow positron beamline using electron linear accelerator

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INTRODUCTION: Slow positron beams (energy-variable monoenergetic positron beams) have been widely used to investigate vacancy-type defects in thin films or near-surface layers in ion irradiated materials. Intense slow positron beams can be generated through pair-creation reactions by large-scale experimental facilities such as nuclear reactors and accelerators. In terms of accelerator-based positron sources, so far, radiofrequency-driven electron linear accelerators (LINAC) have been mainly used. In principle, LINAC-generated positron beams are not continuous (i.e., pulse beams), while reactor-based positron beams are continuous. The pulse beams can be potentially applied for unique applications such as pump-probe type measurements [1]. This study aims at such unique applications using KURNS-LINAC.

EXPERIMENTS: A 30 MeV electron beam was used to generate slow positrons by the KURNS-LINAC. A linear storage section was installed to control time structures of the slow positron beam. An original pulse width of 5 μ s can be stretched or delayed by controlling the waveforms applied the entrance and exit electrodes in the linear storage section. The beamline was extended to a sample chamber at a measurement room next to an accelerator target room. The annihilation gamma-rays at a sample holder were detected by a scintillation detector for positron lifetime spectroscopy and a Ge detector for Doppler broadening measurements, respectively.[2]

RESULTS: The narrow pulse width of original positrons cannot be used for the Ge detector, as it saturates the detector operation. Thus, the linear storage section is required for the Doppler broadening measurements. Fig. 1(a) shows the gamma-ray spectrum obtained from a reference sample (Kapton). A 511 keV peak originating from positron annihilation were observed. In addition, three intense peaks can be observed in the range of 1000 – 1500 keV. These peaks are attributed the background gamma-rays from Co-60 and K-40. Such signals increase unwanted background levels around the 511 keV peak. Thus, a gate signal synchronizing with positron release from the linear storage section was applied to a multichannel analyzer used to measure gamma-ray spectra. Fig. 1(b) shows the spectrum with the gate signal. The background signals were successfully reduced. Our experimental results showed that positron pulse widths can be effectively controlled by operating the linear storage section for accelerator-driven original positron pulses.

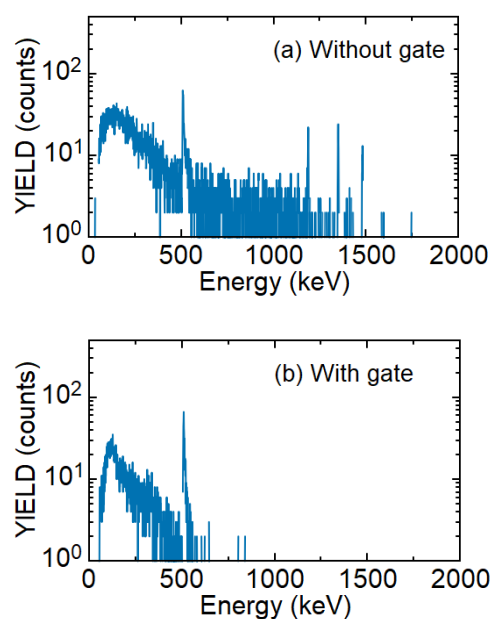


Fig. 1 Gamma-ray spectra obtained with and without gate signals.

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