Nuclear Instruments and Methods in Physics Research B 285 (2012) 18–23

Contents lists available at SciVerse ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

In situ positron beam Doppler broadening measurement of ion-irradiated metals – Current status and potential

T. Iwai [a,*], H. Tsuchida [b]

[a] Nuclear Professional School, School of Engineering, The University of Tokyo, 2-22 Shirakata-Shirane, Tokai-mura, Ibaraki 319-1188, Japan
[b] Quantum Science and Engineering Center, Kyoto University, Uji, Kyoto 611-0011, Japan

ARTICLE INFO

Article history:
Received 29 February 2012
Available online 12 May 2012

Keywords:
Positron beam
Ion irradiation
Doppler broadening
Defects in metals
Cascade damage

ABSTRACT

The positron beam technique has been widely used as a detection tool for vacancy-type defects in solids, especially defects induced by ion irradiation/implantation that are localized near the surface. To extend this technique to other applications, we constructed unique in situ positron beam measurement equipment combined with an ion beam irradiation chamber. This equipment enables us to measure positron beam Doppler broadening during or immediately after irradiation without any sample handling. With this equipment, defect accumulation and evolution in iron and nickel were investigated at room temperature. In addition, the defect accumulation and annealing process was investigated at temperature below stage III to reveal vacancy configurations after cascade, without any thermal migration after the cascade cooling. In this paper, we summarize these results and discuss ideas for future study.

1. Introduction

Lattice defect production by energetic particles such as ions and neutrons in solids is fundamental in many engineering processes, such as material degradation in nuclear power plants (NPPs) and ion implantation in semiconductors. Although the defect production/accumulation processes have received wide interest from the relevant communities, they are not yet fully understood. One reason for this is that, owing to the sub-nm dimensions of the lattice defects, existing direct detection methods for these defects are very limited. Positron annihilation spectroscopy (PAS), one of these direct detection methods, has been widely used during the last few decades [1]. Since positrons are easily attracted and trapped by vacancy-type defects in metals, even monovacancies can be detected if they are of sufficient concentration (~1 ppm in metals). Positrons used as a probe are often aligned to a monoenergetic beam in order to obtain a defined small penetration depth for research on surfaces or defects under surfaces [2]. This method is often called variable energy positron annihilation spectroscopy (VEPAS). Through this method, evaluation of defects as a function of depth (defect depth profiling) can be achieved by varying the positron energy in the range of a few eV to tens of keV. VEPAS is very effective for the detection of ion-irradiated defects localized under surfaces.

In order to more intensely apply VEPAS to the study of ion irradiation/implantation, we constructed a VEPAS system and connected it directly to an ion beam line from the 3.75 MV Van de Graaff accelerator at the High Fluence Irradiation Facility, The University of Tokyo, around the year 2000 [3]. There are several advantages to this approach: (1) this is the only equipment in the world that can conduct PAS during ion irradiation; (2) irradiation and measurement cycles can be easily achieved, without any specimen handling, to obtain detailed dose dependence; and (3) this method is appropriate for low temperature experiments because specimen transfer after the irradiation is not required. A major limitation is the system's incompatibility with lifetime measurements – Only Doppler broadening can be measured. The specifications of the equipment are summarized in Table 1. Using this unique equipment, we performed several experiments regarding defect production/accumulation caused by ion irradiation in metals, mainly iron and nickel. In this paper, we summarize our results, discuss the potential of the equipment, and present ideas for future work.

2. VEPAS system connected to an ion beam line

2.1. Positron beam

The variable energy positron beam equipment is composed of a source chamber that contains a radioisotope positron source and tungsten foil moderator, a magnetic beam transport, and an acceleration tube. In order to keep the specimen at ground potential, the source chamber, the beam transport, and the connected electrical devices (DC current power supply, etc.) are electrically isolated from earth ground; this allows for the application of high voltage

* Corresponding author. Tel.: +81 29 287 8477; fax: +81 29 287 8490.
E-mail addresses: iwai@nuclear.jp (T. Iwai), tsuchida@nucleng.kyoto-u.ac.jp (H. Tsuchida).

0168-583X/$ - see front matter © 2012 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.nimb.2012.05.005

Table 1.
Performance of VEPAS in conjunction with the ion beam

Ion	H^+, D^+, He^+, C^+, O^+, N^+
Ion energy	0.3–3 MeV
Angle between ion beam and positron beam	30°
Positron energy	0.05–30 keV
Positron beam intensity	~10^4 e^+/s
Measurement	Doppler broadening via HPGe detector (ORTEC GEM 20180-P)
Typical count rate	100 c/s at 511 keV photo peak
Sample temperature	12–773 K

up to 30 kV for acceleration of the positrons. A schematic diagram is shown in Fig. 1 [4].

A sealed ^{22}Na positron source of 740 MBq is located in the source chamber with a linear motion feedthrough. Positrons emitted from the source are injected into the adjacent moderator of annealed tungsten foil, and then a small fraction of them are thermalized in the foil. These thermalized positrons are emitted from the surface with energy of several eV according to their negative work function. These slow positrons are extracted by an electric field between the moderator with a bias voltage of +50 V and an extraction grid mesh at earth ground. A magnetic field is applied to the entire source chamber by Helmholtz coils to carry the positrons into the center of the beam transport. The source chamber and the surrounding Helmholtz coils are shielded by lead blocks to suppress the background radiation level around the irradiation and measurement chamber.

The magnetic beam transport consists of two straight aluminum ducts and two 90° elbow aluminum ducts. Conducting wire is wound around the entire transport so that it functions as a solenoid. The applied magnetic field is around 0.01 T throughout the beam transport. This bend structure is effective as a filter that keeps faster positrons away from the measurement chamber. Vertical and horizontal magnetic steerers are installed at the end of the beam transport to adjust the beam position. The magnetic field is applied by three Helmholtz coils from the acceleration tube and the sample chamber. The spatial distribution of the positron beam can be visually observed using a microchannel plate assembly (MCP) with a fluorescent screen located at the sample position. The beam diameter is estimated to be approximately 6 mm, and the beam intensity is estimated to be approximately 10^4 e^+/s. The count rate at the 511 keV photo peak is approximately 100–200 cps as measured by a high purity germanium detector (ORTEC GEM 20180-P) adjacent to the sample position. This detector is

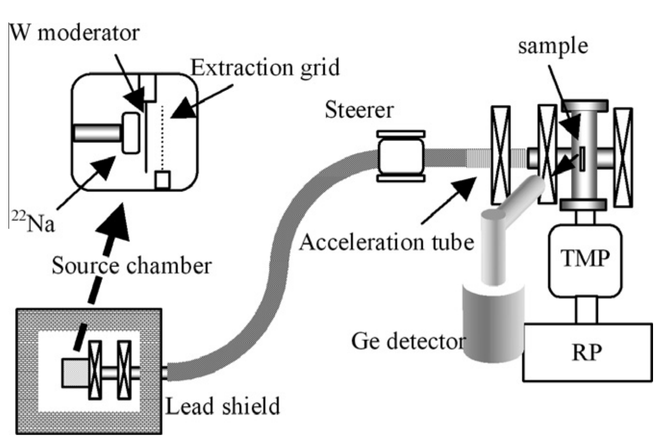

Fig. 1. A schematic of variable energy positron beam connected to ion irradiation chamber at HIT facility [4].

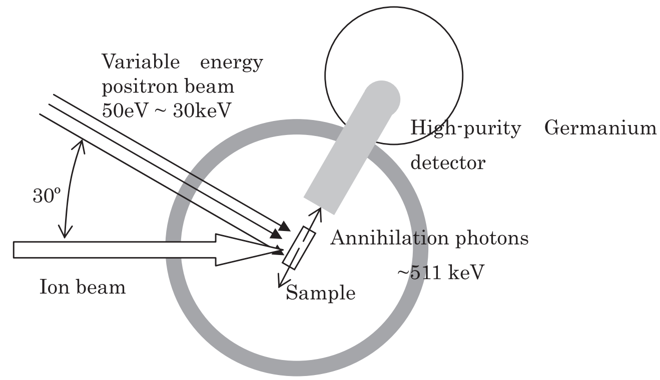

Fig. 2. Experimental configuration of in situ positron beam Doppler measurement [3].

used to measure gamma-ray energy spectra, including Doppler broadening of two photon annihilation. A control program for the data acquisition and beam control runs on a PC near the irradiation and measurement station. The PC is remotely controlled during the irradiation experiments via a remote access program because the irradiation room is locked out when the ion beam is introduced into the room. The system is not currently capable of measuring lifetime or angular correlation.

2.2. Irradiation and measurement station

The positron beam line and the ion beam line are connected to the sample chamber so that both beams merge at the irradiation and measurement station at an angle of 30°. The experimental configuration is shown in Fig. 2 [3]. In most cases, a specimen is set so that the positron beam is injected perpendicular to the specimen surface; the ion beam is tilted at an angle of 30° from perpendicular to the specimen surface. The ion beam is generated by a 3.75 MV Van de Graaff accelerator (Nissin High Voltage KN-3750). The ion energy is 0.3–3.0 MeV, and H^+, D^+, He^+, C^+, O^+, and N^+ can be selected by switching the gas line to the appropriate radiofrequency ion source bottle. The irradiation and measurement station had a heating capability of only 773 K until a new irradiation station incorporating a cryostat (Iwatani HE05 He gas refrigerator-type cryostat) was installed in 2008. This new irradiation station has two irradiation stages, one for low temperature experiments above 12 K and another for high temperature experiments up to 773 K. In addition, the new station is capable of 4-wire resistivity measurements for performing additional experiments on defects.

3. Results

3.1. Defect production in iron

Nuclear engineers are particularly interested in defect production and accumulation in α-iron because the main components of NPPs exposed to substantial neutron irradiation are steels. We performed several experiments on defect production and accumulation in iron at room temperature and at low temperature.

3.1.1. Room temperature

At room temperature, vacancy accumulation in iron caused by irradiation with various ion species was investigated through the monoenergetic positron beam Doppler broadening technique. It is well known that vacancy-type defects in iron can be clearly detected on account of the significant sharpening of the Doppler broadening spectrum, which is parameterized as the S parameter. Various ion