

K. Mori, H. Yoshino, R. Okumura, S. Sato<sup>1</sup>, H. Hiraka<sup>2</sup>,  
K. Iwase<sup>3</sup>, T. Shiono<sup>4</sup> and K. Matsuda<sup>4</sup>

Research Reactor Institute, Kyoto University (KURRI)

<sup>1</sup>High Energy Accelerator Research Organization (KEK)

<sup>2</sup>Neutron Science Center, Korea Atomic Energy Research Institute

<sup>3</sup>Department of Materials and Engineering, Ibaraki University

<sup>4</sup>Graduate School of Engineering, Kyoto University

**INTRODUCTION:** Neutron diffraction is a powerful tool to determine precisely the positions of light elements (e.g., hydrogen and lithium) in solids. This is the main reason why neutron powder diffractometers are critical for structural investigations of energy storage materials, for example, rechargeable lithium-ion batteries and hydrogen absorbing alloys. The B-3 beam port of Kyoto University Research Reactor (KUR) had long been used as a four-circle single-crystal neutron diffractometer (4CND). For the last decade, however, the 4CND was so old that its research activity on neutron science was quite low. Recently, the compact multipurpose neutron diffractometer (CMND) has been installed instead of the 4CND. Here, we report the current status of the B-3 beam port of KUR.

**SPECIFICATIONS:** The CMND has a wide space around the sample, therefore we can easily install any other system. The neutron wavelength,  $\lambda$ , which is monochromatized by the (220) plane of a Cu single crystal (i.e., Cu monochromator), is 1 Å. The old Cu monochromator stage of the 4CND was removed, and then the new one with two goniometers (RA07A-W and SA05B-RM, Kohzu Precision Co., Ltd.) was placed on the B-3 beam port (see, Fig. 1). In addition, the CMND is equipped with a new beam shutter manufactured by the KURRI factory. To cover the detector area of  $6^\circ \leq 2\theta \leq 150^\circ$ , twenty-five <sup>3</sup>He tube detectors (1/2 inch in diameter) are used, where  $2\theta$  is the scattering angle. The distance from the Cu monochromator to the sample is approximately 2 m, and the distance from the sample to the detector is 1.2 m. A detector bank including the twenty-five <sup>3</sup>He tube detectors is placed on an arm of the HUBER-440 goniometer.

**CURRENT STATUS OF B-3:** Paraffin blocks had frequently been used as radiation shielding properties on the 4CND, however the main concern in working was flammability. Accordingly, all paraffin blocks were replaced by nonflammable radiation shielding properties: B<sub>4</sub>C grit mixed with resin and heavy concrete blocks (see, Fig. 2). The CMND is shown in Fig. 3 (under construction). The goniometers (HUBER-430 and -440) and the beam shutter have already been installed. The detector bank will be installed soon.

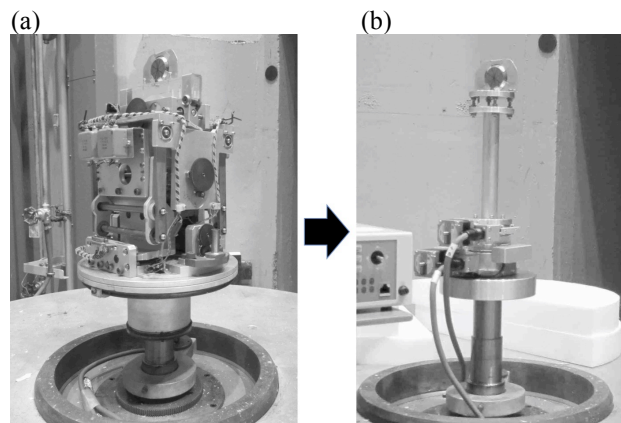


Fig. 1. The Cu monochromator stage: (a) old stage on the 4CND, and (b) new one on the compact multipurpose neutron diffractometer (CMND).

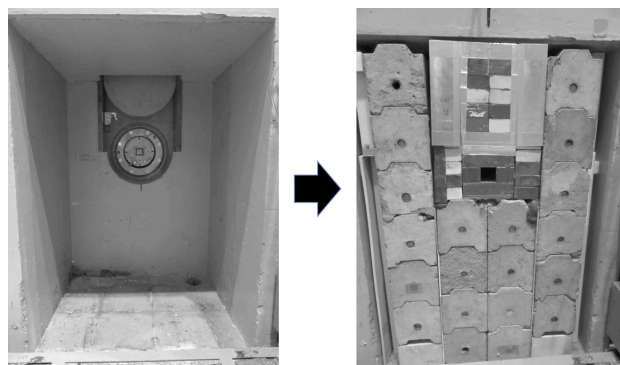


Fig. 2. Radiation shielding properties: B<sub>4</sub>C grit mixed with resin and heavy concrete blocks.

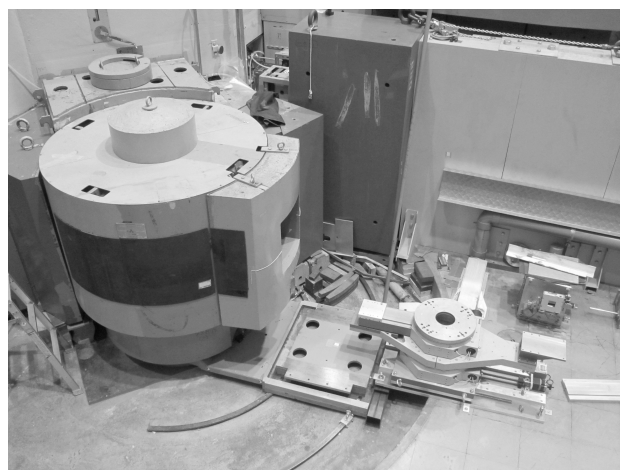


Fig. 3. The compact multipurpose neutron diffractometer, installed at the B-3 beam port (under construction).

M. Hino, S. Takeda<sup>1</sup>, T. Hosobata<sup>1</sup>, Y. Yamagata<sup>1</sup>, T. Oda, H. Endo<sup>2</sup>, N.L. Yamada<sup>2</sup>, J. Guo<sup>1</sup>, S. Morita<sup>3</sup>, M. Furusaka<sup>4</sup> and Y. Kawabata

Research Reactor Institute, Kyoto Univ., Japan

<sup>1</sup> RAP, RIKEN, Japan

<sup>2</sup> IMSS, KEK, Japan

<sup>3</sup> Tokyo Denki Univ. Japan

<sup>4</sup> Grad. Sch. of Eng., Hokkaido Univ., Japan

**INTRODUCTION:** Progress of neutron optical devices is very significant, however, it is still very difficult for neutron aspherical focusing mirror, especially, two dimensional (2D) aspherical focusing supermirror. We proposed new fabrication method for aspherical focusing supermirror with metal substrate [1, 2]. The metallic substrate is robust and ductile, to which able to fabricate steeply curved surface with high form accuracy. It is also applicable to use under high radiation irradiation and high-temperature field, even at a place close to the neutron target and moderator. Furthermore, it is possible to fabricate a large focusing mirror by combining multiple segmented mirrors with mechanical fastening entailing the usage of screw holes and fixture tabs. The big problem was required surface roughness for neutron mirror. The roughness should be smaller than 0.5 nm even for  $m=3$  supermirror coating. Here  $m$  is the maximum critical angle of the mirror in units of critical angle of natural nickel. By using electroless nickel-phosphorus (Ni-P) plating, we almost overcame the problem in case of one-dimensional aspherical focusing supermirror [3]. In this study, we show a performance test of ellipsoidal supermirror with metallic substrates.

**EXPERIMENTS:** We fabricated an ellipsoidal metallic substrates with the Ni-P plating, based on the technology using ultrahigh precision cutting with correction processing, followed by mechanical precision polishing. The manufacturing, polishing and cleaning of the metallic substrate were conducted at RIKEN. The supermirror coating was conducted with KUR-IBS [4]. The neutron experiments were conducted at the BL06 (VIN ROSE) beam port at J-PARC/MLF [5,6].

**RESULTS:** There is two neutron guide tubes at the BL06 and a cadmium pinhole in which aperture size of 1 mm was located at exit of one of the guide tubes. A U-shape slit and an ellipsoidal supermirror of 300 mm length shown in photograph of Fig.1 were placed between the pinhole slit and a 2D neutron detector. The distance between the pinhole slit and the detector is 2.5 m since the semi-major axis of the mirror is 1.25 m. Without the mirror, as shown in Fig 1(a), the intensity image was U-shape and the peak intensity was about 500 counts. With the mirror, as shown in Fig 1(b), the intensity image was circular spot and the spot size was almost equivalent to that of the pinhole. The peak intensity was about 7000 counts and the intensity gain was about 14 in this setup.

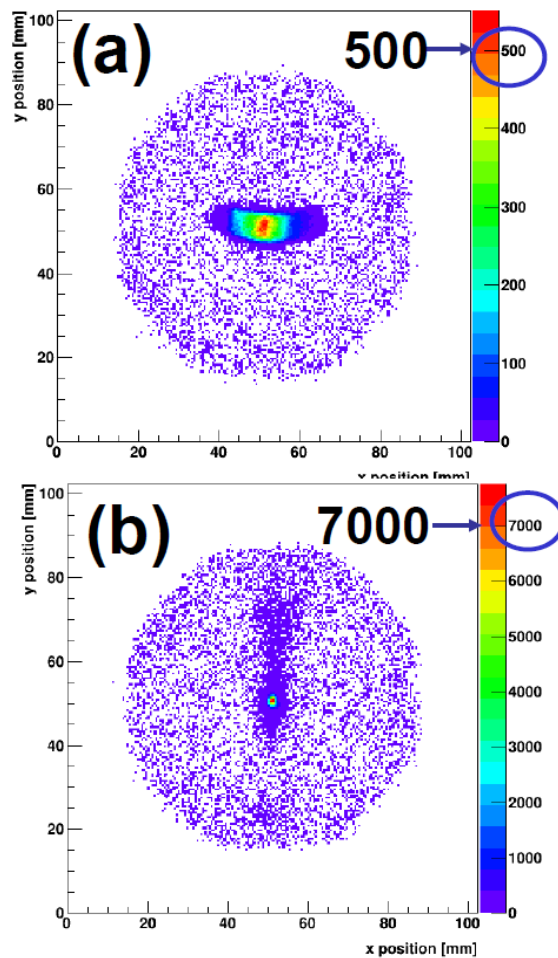
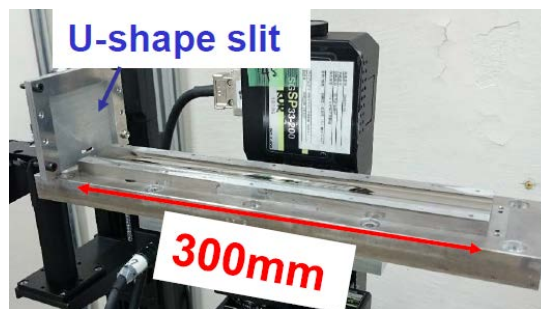


Fig. 1. Photograph of the ellipsoid neutron supermirror at the BL06 at the J-PARC/MLF. Neutron intensity image at detector position (a) without and (b) with the ellipsoid neutron supermirror.

#### REFERENCES:

- [1] J. Guo, *et al.*, Optics Express **22**(2014) 063108.
- [2] J. Guo, *et al.*, Review of Scientific Instruments **86**(2015) 063108.
- [3] S. Takeda, *et al.*, Optics Express **24**(2016) 12478.
- [4] M. Hino, *et al.*, Nucl. Instr. and Meth., **797**(2015) 265.
- [5] T. Oda, Dr thesis, Kyoto University, 2015.
- [6] M. Hino, *et al.*, to be published in JNST

# CO1-3 The Correlation between Microstructural Evolution and Mechanical Property Changes in Neutron-irradiated Vanadium Alloys

K. Fukumoto, T. Onitsuka, K. Tone<sup>1</sup> and Q. Xu<sup>2</sup>

RINE, Univ. of Fukui

<sup>1</sup> Graduate school of Eng., Univ. of Fukui

<sup>2</sup> Research Reactor Institute, Kyoto University

**INTRODUCTION:** Vanadium alloys are candidate materials for fusion reactor blanket structural materials, but the knowledge about the mechanical properties at high temperatures during neutron irradiation is limited and there are uncertainties that may have influenced the results such as the interstitial impurity content of specimens. Recently, material irradiation technology in a liquid metal environment was developed and irradiation experiments in various liquid metal environments can be performed for vanadium alloys. Environmental effects and irradiation effects for mechanical properties should be distinguished independently in order to understand the essential behavior of vanadium alloys during irradiation for fusion reactor application. The objective of this study is to investigate the mechanical properties and microstructural changes of the vanadium alloys, V-4Cr-4Ti alloys during neutron irradiation. In this study, tensile test and microstructural observation were carried out for V-4Cr-4Ti alloys.

**EXPERIMENTS:** The majority of test specimens for this study were prepared from V-4Cr-4Ti alloys. The tensile specimens had nominal gauge dimensions of 0.25mm(t) x 1.2mm(w) x 5mm(l). Before irradiation, all specimens were annealed in vacuum at 1000°C for 2hrs. The specimens were irradiated in Joyo in the temperature range from 450°C to 650 °C with total neutron dose from 0.47 to 2.1 x 10<sup>26</sup> n/m<sup>2</sup>. In the previous study, the ratio of damage level, displacement per atom (dpa) to neutron dose in pure vanadium in Joyo MK-II was 2.5 x 10<sup>-26</sup> dpa/Φ<sub>tot</sub>. The amounts of estimated damage level ranged from 1.2 to 5.3 dpa. Tensile tests were conducted at room temperature and 400°C with various strain rate ranged from 6.7x10<sup>-4</sup> to 10<sup>-2</sup> to obtain the information of strain rate sensitivity at high temperature. SEM observation for fractography after tensile test was carried out in KUR, Kyoto University.

**RESULTS:** Tensile tests for V-4Cr-4Ti alloys were carried out at RT and 400 °C at strain rates between 6.7x10<sup>-4</sup> and 6.7x10<sup>-1</sup>/s. The strain-rate dependence was determined for the lower yield stress σ<sub>LYS</sub>. From the gen-

eral relationship between flow stress and strain rate, the strain rate sensitivity (SRS) parameter is defined as follows;

$$m = \frac{1}{\sigma} \frac{d\sigma}{d \ln \dot{\epsilon}} \quad (1)$$

When the flow stress  $\sigma$  rises with the increase of strain rate  $\dot{\epsilon}$ , i.e.,  $m > 0$ , slip deformation is a thermally activated process. When  $m < 0$ , a barrier strength of obstacle against dislocation slip motion is weakened and leads to reduction of the flow stress, because increasing the strain rate decreases time available for solute diffusion to dislocations. Values of the SRS for the lower yield stress were determined for each irradiation condition and test temperature from a logarithmic fit to the lower yield strength data. Figure 1 shows the SRS of lower yield stress dependence on the test temperature for the irradiated V-4Cr-4Ti in this study and the previous data for V-4Cr-4Ti (US-832665) irradiated in HFBR [1]. The data of SRS,  $m$  in this study shows significantly positive value and larger than the  $m$  values of previous data for US V-4Cr-4Ti alloy irradiated in HFBR. The interaction between interstitial impurities and defect clusters/dislocation is affected by the irradiation damage process after neutron irradiation at 400 °C. Therefore, the dynamic strain aging should be only thermal activation process between impurity and mobile dislocation, and independent of irradiation damage process.

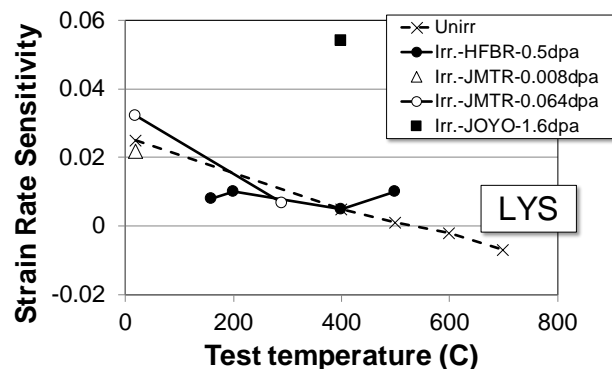


Figure 1 : The strain rate sensitivity (SRS) of lower yield stress dependence on the test temperature for the irradiated V-4Cr-4Ti alloy in this study and the previous data for V-4Cr-4Ti irradiated in HFBR [1].

Reference

[1] A. F. Rowcliffe, S. J. Zinkle and D. T. Hoelzer. J. Nucl. Mater., 283-287 (2000) 508-512

## CO1-4 Preliminary Study of Radiation Resistivity of *Bacillus subtilis natto*

Y. Yanagisawa, T. Chatake<sup>1</sup>, T. Saito<sup>1</sup>, T. Ohsugi<sup>2</sup>,  
H. Sumi<sup>2</sup>

Faculty of Pharmaceutical Sciences, Chiba Institute of  
Science

<sup>1</sup>Research Reactor Institute, Kyoto University

<sup>2</sup>Department of Life Science, Kurashiki University of Sci-  
ence and the Arts

**INTRODUCTION:** *Bacillus subtilis natto* (BSN) is one of famous bacteria in Japan, because Japanese traditional food 'natto' is made of soybeans by fermentation using BSN. Since BSN produces biologically active substances: nattokinase [1], water-soluble vitamin K [2] etc, the detailed mechanism of BSN growth would provide useful information for improvement of quality and quantity of the production of this nutritional food.

The growth of BSN is entirely different from *Escherichia coli* (*E. coli*), which is the most popular bacterium in the field of bacteriology. *E. coli* increase by only simple cell division, and when their population reach the limit, cell division is suppressed. On the other hand, BSN transform themselves to spores like seeds of plants, under their excess pupation. The spores have high resistance to various severe conditions such as starvation, heat, cold, dry, chemical reagents (fungicides). Of course, they have high radiation resistance [3].

The aim of this project is to investigate radiation resistivity of BSN not in its spore state but in its growth state. Generally, the radioresistance of *Bacillus subtilis* is believed to be derived from the formation of spores (sporulation). It would be true, but vegetative cells of BSN might have different property of radioresistance from other bacteria, too.

In our previous study, radioresistance of vegetative cells of *E. coli* was investigated in detail. We tried to establish experimental procedure of the assessment of the radioresistance of vegetative cells of BSN, in the same manner as that of *E. coli*. Here is the first preliminary report of this experiment.

**EXPERIMENTS:** First, cell cloning was carried out using *Bacillus subtilis natto* Miyagino. The obtained clone was confirmed by the fibrin plate method that assesses the fibrinolysis activity of nattokinase, which is abundantly produced in BSN. Prior to irradiation experiments, the growth curve of BSN in LB medium was determined. Glycerol stock (40  $\mu$ L) of the BSN clone was inoculated in 4 mL LB liquid medium in a test tube, and then pre-incubated at 42 °C with the shaking at 1,200 rpm for 16–20 hours. Pre-cultured medium (40  $\mu$ L) was inoculated in 4 mL LB liquid medium, and incubated with the same condition as the pre-incubation. Optical density at 600 nm ( $OD_{600}$ ) was monitored for 8 hours during the incubation.

Irradiation experiment was carried out at the Co-60 Gamma-ray Irradiation Facility of Kyoto University Research Reactor Institute. Incubated medium (500  $\mu$ L) with

$OD_{600}$  of 0.536 was sealed in a 1.5 mL plastic tube. Five samples were prepared, and irradiated with gamma rays at a dose of 0, 50, 100, 200, and 400 Gy at a dose rate of 24 Gy/min. Temperature of the experimental room was 15 °C. After the irradiation, each liquid medium was diluted by LB medium, and plated on standard method agar "Nissui". Colony formation units were counted after 8 hour incubation at 42 °C.

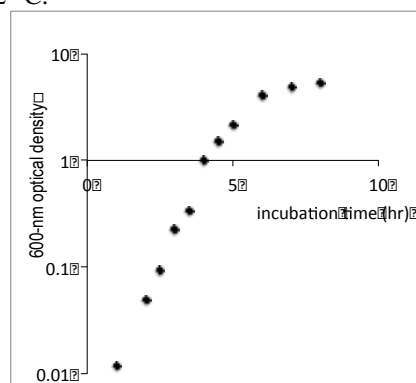


Fig. 1. Growth curve of BSN in LB medium.

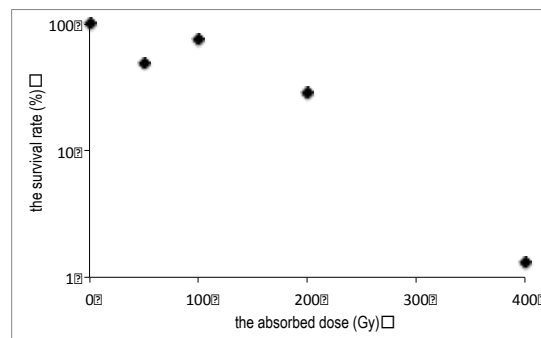


Fig. 2. Cell survival curve of BSN.

**RESULTS:** As shown in Fig. 1, the growth of BSN is in an exponential phase for 1–4 hours from the inoculation. The corresponding  $OD_{600}$  are 0.01–0.98. In this phase, sporulation is thought to rarely occur. The optimum condition for irradiation experiment is estimate to be  $OD_{600}$  = 0.2–0.8 (3–3.5 hours incubation).

Fig. 2 shows our preliminary result of the radioresistance of vegetative cells of BSN in this project.  $D_{10}$  value (10% survival dose) was about 250 Gy. The present result suggests that vegetative cells of BSN do not have so high radioresistance as spore of BSN. On the other hand, we found a few different features in the cell survival curve from that of *E. coli*.

In order to discuss the radioresistance of BSN in detail, improvement of our experimental procedure and repeated irradiation experiments are in progress.

### REFERENCES:

- [1] H. Sumi *et al.*, *Experientia*. **42** (1987) 1110–1111.
- [2] H. Sumi, Y. Yanagisawa, N. Kishimoto, *Nippon Noeikagaku Kaishi* **73** (1999) 599–604.
- [3] P. Setlow, *J. Appl. Microbiol.* **101** (2006) 514–525.