CO1-1

# Trial of mass production of *m=6* neutron focusing supermirrors

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**INTRODUCTION:** Recently new construction project for research reactor is progressing at ”Monju" site at Tsuruga city in Fukui prefecture. Major utilization of the research reactor is for thermal and cold neutron beam utilization for wider utilization [1]. Actual use of the focusing mirror is one of very important to be on par with top level institute. We have established fabrication method for aspherical focusing supermirror with metal substrate [2-3]. 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 filed, even at a place close to the neutron target and moderator. We have realized smooth surface for high-*m* supermirror coating. Here *m* is the maximum critical angle of the mirror in units of critical angle of natural nickel. In this study, we report a status of mass production for high-m neutron focusing supermirrors.

**EXPERIMENTS:** We fabricated ellipsoidal metallic substrates with electroless nickel-phosphorus plating, based on the technology using ultrahigh precision cutting with correction processing, followed by mechanical precision polishing. The first precise manufacturing was conducted at a CNC machine for development of neutron optical devices at workshop of the KURNS. The ultra-precise manufacturing, polishing and cleaning of the metallic substrate were conducted at RIKEN. The supermirror coating was con-ducted with ion beam sputtering machine at the KURNS (KUR-IBS) [3]. Figure 1(a) shows typical photograph of ellipsoidal supermirror deposited on one LOT and silicon substrates. The semi-major and semi-minor axes of the ellipsoidal supermirror were 1250 mm and 65.4 mm, respectively. Eventually, we have fabricated m=6 NiC/Ti(C) supermirrors in which effective number of layers was 9750, where the half of the layers were very thin carbon interlayers. The neutron experiments were conducted at C3-1-2(MINE) port of JRR-3.

**RESULTS:** By adding very thin carbon interlayer between NiC and Ti layers, we have realized m=6 supermirror with high reflectivity. There was a bit difference each LOT because some condition was difference as following: Because cleaning condition was a bit difference between LOT21 and LOT22, and the deposition rate of Ti target of LOT18 was smaller than those of LOT21 and LOT22.

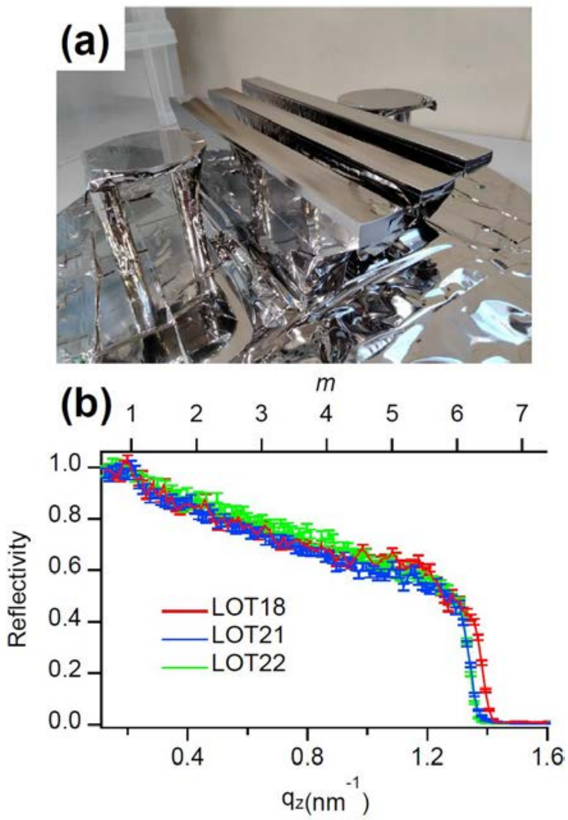


Fig. 1. (a) The photograph of m=6 supermirror deposited on one LOT (three ellipsoidal metal substrates) and two Si wafers (b) Neutron reflectivities of the supermirrors on Si wafers of which LOT number are 18, 21 and 22.

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R5063

CO1-2

# Structural Analysis of Viscosity Index Improver Molecules Using Small-Angle X-ray Scattering

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**INTRODUCTION:** Lubricating oils are necessary for friction reduction and high wear durability of sliding surfaces in machine components, and the development of the best oils is highly demanded by the industry. Viscosity index improver (VII) is a type of additive used to mitigate the decrease in viscosity of lubricating oil due to temperature rise. Classical textbooks say that VII molecules work by changing their equivalent radius in the base oil according to the oil temperature. However, there are few papers investigating the equivalent radius of VII molecules by small-angle X-ray scattering (SAXS) and/or small-angle neutron scattering (SANS)[1], and there is still room for discussion about the behaviour and working mechanism of VII molecules in oil. This study attempted to investigate the radius of gyration of several types of VII polymers in base oil at different temperatures by SAXS, and the behaviour of the VII polymers was investigated and discussed.

**EXPERIMENTS:** To investigate the structure of VII polymer in lubricant, we used a SAXS instrument (NANOPIX, Rigaku) with a Cu-Kα X-ray source (MicroMAX-007, Rugaku) and a semiconductor 2D detector (HyPix-6000, Rugaku). The 1.2 mm-thick aluminium cell with optical windows made of 20 μm heat resistant engineering plastic film (Superio-UT, Mitsubishi Chemical) was used for the measurement. The cell temperature was successively increased to 25, 40, 60, 80 and 100ºC, and the final measurement was carried out again at 25ºC after cooling to check the degeneration of the VII molecule. Comb-shaped poly(methyl acrylate) (PMA) type VII was prepared as a typical one used in engine oil in the study. Squalane was used as a model base oil and the concentrations of PMA in squalane were 0.5 and 2.0 mass%.

**RESULTS:** The SAXS profiles from squalane with 0.5 and 2.0 mass% Comb-PMA type VII at each temperature were shown in Fig. 1. The profiles were obtained by subtracting the SAXS profiles from pure squalane at each temperature. The figure shows that the profiles obtained at low temperature appear to be formed by the addition of two profiles derived from the different structural forms of Comb-PMA on the low-*q* and high-*q* ranges, but as the temperature increases, the profiles on the low-*q* side become relatively smaller. This indicates that comb-PMA type VII undergoes a significant structural change in response to temperature rise, which is linked to its high thickening effect as VII. In order to quantitatively analyze what this morphology looks like, fitting is currently being carried out on the macromolecular model proposed by Pedersen[2].

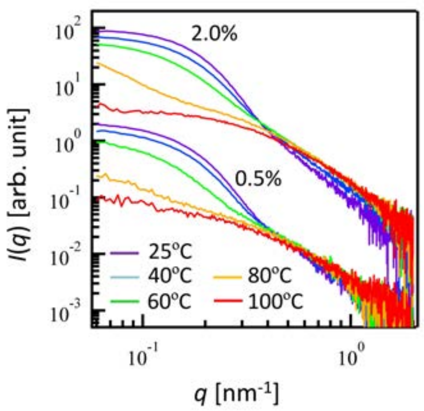


Fig. 1. SAXS profiles from comb-PMA molecules in squalane.

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CO1-3

# Improvement of multilayer mirrors for neutron interferometer

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**INTRODUCTION:** Neutron interferometry is a powerful technique for studying fundamental physics. Numerous interesting experiments [1] have been performed since the first successful test of a single-crystal neutron interferometer [2]. However, the single-crystal interferometer is inherently not able to deal with a neutron that has a wavelength longer than twice its lattice constant. In order to investigate problems of fundamental physics, including tests of quantum measurement theories and searches for non-Newtonian effects of gravitation, the interferometry of cold neutrons is extremely important, since the sensitivity of interferometer for small interaction increases with the neutron wavelength. A large scale of interferometer also has the advantage to increase the sensitivity to small interactions. One of the solutions is an interferometer using neutron multilayer mirrors [3,4]. We can easily control parameters such as Bragg angle, reflectivity, and Bragg peak width by selecting the deposited material and tuning the bilayer thickness and the number of layers.

**EXPERIMENTS AND RESULTS:** We demonstrated a multilayer interferometer for pulsed cold neutrons at the beamline 05 NOP in J-PARC MLF [5]. Figure 1 shows the interference fringes with etalons according to time-of-flight. The phase of interferogram depends on the wavelength of neutrons. We also demonstrated the measurements of neutron coherence scattering length of some nuclei. The better stability of the measurements was demonstrated.

In order to enlarge the number of neutrons, neutron supermirrors which can reflect wide bandwidth of wavelength were developed for interferometer by using Ion Beam Sputtering facility in KURNS. Figure 2 shows the reflectivity of the half mirrors with wide bandwidth. We tried to utilize the mirrors into the interferometer. The geometrical arrangement of the interferometer was confirmed, however, the interferogram was not observed. It is considered that this was due to deformation of the mirror substrates and non-uniformity of the mirror thickness. The results suggested that uniformity of better than a few percent is required. We are continuing to research and develop the neutron mirrors for the interferometer.

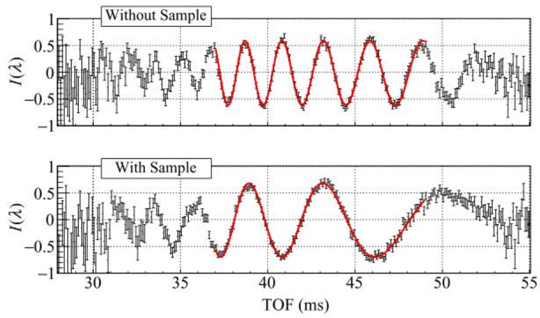


Fig. 1. Interference fringes without and with sample. The phase was changed due to the refraction index of the material.

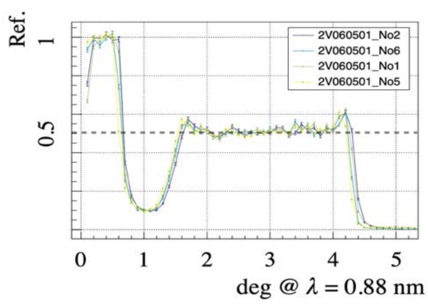


Fig. 2. Reflectivity of the half mirror with wide band of neutron wavelength measured at MINE2 in JRR3.

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R5129

CO1-4

# Development of efficient spin filters for ultracold neutrons

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**INTRODUCTION:** A finite electric dipole moment (EDM) of the neutron implies violation of time-reversal symmetry, and has been searched since 1950s. Searches for a non-zero neutron EDM in high sensitivity constitute stringent tests of theories beyond the Standard Model of particle physics and shed light in the mystery of baryon asymmetry of the universe. In the state-of-the-art neutron EDM experiment, spin-precession frequencies of ultracold neutrons (UCNs), neutrons with kinetic energies on the order of 300 neV or less, are compared under different electromagnetic configurations. One of the key components required for neutron EDM experiments is spin analyzer of UCNs, whose efficiency directly impacts the precision of the spin-precession frequency measurement, thus the sensitivity of the neutron EDM search. Because of extremely low energies of UCNs, magnetized Fe thin films with small coercivities can be used as effective UCN spin analyzers, functioning as filters that selectively transmit the high-field seeking state of UCNs. In this project at the Institute for Integrated Radiation and Nuclear Science, Kyoto University, we have developed sputtered thin Fe films produced with KUR-IBS [1], whose performance have been tested with a cold neutron beam at JRR-3/MINE2, and with pulsed UCNs at J-PARC/MLF.
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**EXPERIMENTS:** During the FY2023, we have applied a new method of Fe sputtering and succeeded in producing Fe film that has significantly small magnetic coercivities than what has been previously developed by ourselves. The coercivity $H_c$ as measured by vibrating sample magnetometry has been reduced from about 20 Oe to 9 Oe, this enables us to design a UCN spin analyzer with small stray magnetic fields, which has numbers of practical merits for building the system including magnetically sensitive components. The new films have been tested with cold and ultracold neutrons during the FY2023. For the test at MINE2, we newly constructed a polarized neutron reflectometry setup with a dedicated θ-2θ stage.
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**RESULTS:** The results of polarized neutron reflectometry measurements of the new Fe film as compared to the previously developed film is shown in the figure. The new film is demonstrated to function as a neutron spin filter at a smaller magnetic field than the previously developed one.
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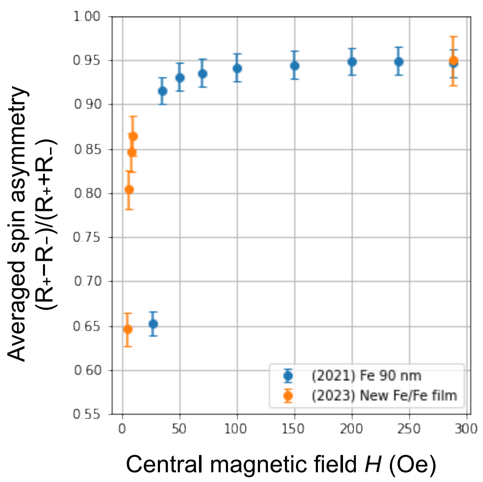


**Fig. 1.** Results of polarized cold neutron reflectometry at MINE2. Averaged spin asymmetry derived from neutron reflectivity of different neutron spins. The new film developed in 2023 functions as a high performance spin analyzer at a smaller applied magnetic field than the previously developed Fe film. The absolute spin asymmetry is limited by the beam polarization.

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R5137

CO1-5

# Development of Cold/ultracold Neutron Detector Using Nuclear Emulsion

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**INTRODUCTION:** We have been developing a cold/ultracold neutron detector using nuclear emulsion for measuring spatial distributions of ultracold neutrons in the Earth's gravitational field [1, 2]. It was fabricated by sputtering a thin layer of $^{10}B_4C$-NiC-C on a silicon substrate and coating it with a fine-grained nuclear emulsion [3]. It is also possible to be used for neutron imaging [4, 5]. For high-spatial-resolution imaging, capability of accumulating tracks with high density is im-portant. In the study in ref. [5], saturation of degree of blackening was observed when track density was more than $3\times10^4$ tracks/(100 μm)$^2$. In this study, we attempted to avoid the saturation by re-ducing the thickness of the emulsion layer of the detector and shortening tracks.

**EXPERIMENTS:** Detectors were fabricated by sputtering thin layers of $^{10}B_4C$(1 μm)-NiC-C on 0.4 mm-thick silicon plates and coating the substrate with the emulsion. The sputtering was done at KURRI by an ion beam sputterer (KUR-IBS) [6]. The coating was done at Nagoya University with two different thickness of usual 10 μm and very small 0.3 μm. Four samples for each were produced. They were exposed to neutrons with wavelength of 0.2 nm at 20 cm downstream of the neutron guide tube in CN-3 beam line. Detection efficiency of the detectors were 1% from the thickness of $^{10}B_4C$ layers and the wavelength. Exposures were conducted with four different times and track density as shown in Table 1. Neutron flux of the beam was $7.6\times10^5$ n/cm$^2$/s when thermal power of the reactor was 1 MW.

Table 1. Sample IDs, thickness of emulsion layer, exposure time and accumulated density of tracks.

| sample IDs and emulsion thickness | | exposure time and thermal power | track density [/(100 μm)$^2$ ] |
|---|---|---|---|
| 10 μm | 0.3 μm | | |
| A-1 | B-1 | 0.37 h (1 MW) | $1\times10^3$ |
| A-2 | B-2 | 3.7 h (1 MW) | $1\times10^4$ |
| A-3 | B-3 | 7.0 h (1 MW) + 6.0 h (5 MW) | $1\times10^5$ |
| A-4 | B-4 | 42 h (1 MW) + 6.0 h (5 MW) | $2\times10^5$ |

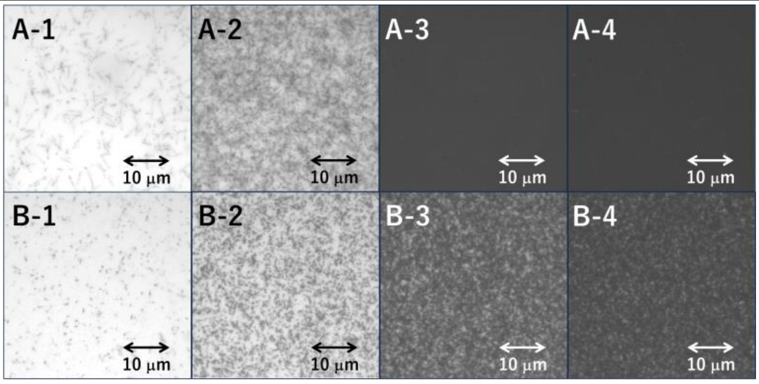


Fig. 1. Micrographs of emulsion samples. Views of A-3 and A-4 were filled with tracks, and degree of blackening saturated, which was not the case in B-3 and B-4.

**RESULTS:** After the exposure, samples were developed at Nagoya University. Their micrographs (Fig. 1) were taken under a manual optical microscope with an epi-illumination system. All of them were taken with the same illumination condition. From comparison between A-1 and B-1, it was seen that tracks were successfully shortened by thinning the emulsion layer. Furthermore, the saturation was not seen in B-3 with exposure density higher than $3\times10^4$ tracks/(100 μm)$^2$.

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CO1-6

# High-temperature test for BGaN semiconductor neutron detectors

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**INTRODUCTION:** The BGaN semiconductor neutron detectors are currently under development at Shizuoka University as promising compact and high-temperature resistant neutron detectors [1]. In this study, we conduct a preliminary experiment to confirm the operability of the BGaN semiconductor neutron detectors in high-temperature environments. Furthermore, through this experiment, technical issues are identified for future development.

**EXPERIMENTS:** In this experiment, we observe the pulsed detection signals for thermal neutron beams irradiated to the BGaN semiconductor neutron detectors installed on a high-temperature hot plate. A picture of the experimental set-up is shown in Fig. 1. The BGaN semiconductor detectors are connected to preamplifier devices (CSP02, ANSeeN Inc.) through high-temperature resistant MI cables, and then, the pulsed signals processed in the preamplifier devices are discriminated at each wave height and rise time in a multi-channel analyzer (ZMCAN-CH04, ANSeeN Inc.). Voltage is applied to the detectors with DC stabilized power supply devices (PA250-0.25A, KENWOOD Co., and PA600-0.1B, TEXIO Technology Co.) In parallel, the waveform of the electrical signals is monitored with an oscilloscope (MSO5074, RIGOL Technologies Inc.). This experiment is performed in the cold neutron beam line (CN-3) [2] in KUR, which has relatively low background noise and can irradiate low-energy neutrons for sensitive detection. Two BGaN semiconductor neutron detectors are irradiated at the same time, and hot plate temperature is set to 300°C. For convenience, they are referred to as Detector 1 and Detector 2 in order from the upstream of the beamline, respectively. Measurement condition is summarized in Table 1.

Fig. 1 A picture of the experimental set-up.

Table 1 Measurement condition

| Items | Detector 1 | Detector 2 |
|---|---|---|
| Type | p-up | p-up |
| BGaN layer thickness | 5 µm | 3.5 µm |
| Diameter of electrodes | 500 µmφ | 500 µmφ |
| Voltage | 75 V | 20 V |
| Reactor power | 5 MW | |

**RESULTS:** As shown in Fig. 2, clear pulsed detection signals were successfully found several times per hour for both detectors. The rise time of the pulse is set to the origin (0µs). This pulsed signal is presumably attributed to neutron-induced reaction in the BGaN layer because the signal is sufficiently large compared to the surrounding electrical noise, and the rising time of the pulse is sharp. These results suggest that the BGaN semiconductor neutron detectors demonstrate the operability at around 300°C at least.

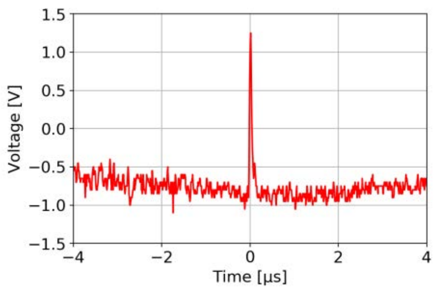


Fig. 2 Voltage time variation around a pulsed detection signal of the BGaN semiconductor neutron detectors.

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