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Position-Encoded Automatic Cell Elevator for BL02, J-PARC MLF

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A sample changer of vertical movement was developed for our top loading cryostat on our neutron spectrometer BL02 at J-PARC MLF. The sample changer, termed “PEACE,” can control reproducibility of the irradiated position using guides made of polyether ether ketone. The variation between the background scattering profiles of three sample positions was found to be less than ±1.6%. This result is reasonable, considering the deviation of sample position of less than ±0.3 mm from the vertical axis.

KEYWORDS: Back scattering spectrometer, QENS, sample changer

1. Introduction

The state-of-the-art high incident-neutron flux and extremely high signal-to-noise ratio of 10^5 of the back-scattering spectrometer BL02 (DNA) of MLF, J-PARC [1] allow us to measure quasi-elastic neutron scattering (QENS) within a relatively short exposure time of 1 h for strong neutron scatterers. A further beampower increase of MLF from the current 500 kW to the designed power of 1 MW could open a new phase of significantly high rate QENS measurements with much shorter exposure time.

In our current set-up, when we change samples, we have to wait a sufficient time for the irradiated sample to radiologically cool. Our standard Al cell becomes a β emitter after neutron exposure and its cooling time can easily be half an hour. If the QENS measurement time becomes shorter, then the rate determining step may be the sample change time. Thus, this calls for the development of a sample changer suitable for BL02 not only to reduce the time or save labor for manual sample loading but also to minimize the waiting time for radiologically cooling an irradiated sample.

A sample changer is more common in the case of small angle scattering (SAS) and powder diffraction, compared with QENS or inelastic neutron scattering (INS). For SANS, there are sample changers commercially available [2]. A monster sample changer installed in the powder diffractometer iMATERIA (BL20 in MLF) can treat several hundreds of samples [3]. Since the measurement time for QENS or INS experiments is generally longer than that of elastic scattering experiments, sample changers available for inelastic scattering instruments are still rare. Among them, IRIS at ISIS, UK [4], developed a sample changer of the vertical elevation type [5]. Cold neutron chopper spectrometer (CSNS) at Separation Neutron Source (SNS), USA, developed a sample changer of the horizontal rotation type (similar to a revolver) [6]. These designs are effective for top-loading cryostats.

An important factor to be considered on sample exchange is the reproducibility of the sample position. Conventional QENS analysis involves often subtraction or convolution between two profiles. However, deviation of the sample position from the scattering center easily affects QENS profile, especially the intensity. In fact, a horizontal shift by 1 mm at the sample position causes a flux deviation of ±7.5% for BL02. Thus, an accurate positioning of the samples in the illuminated area of the beam is an essential requirement for a sample changer design for BL02.

Table I. Specification of PEACE

Sample number	3
Lift speed	3 cm/min
Motor	AC motor
Maximum stroke	30 cm
Position resolution	2 μm/pulse
Expected sample size	ϕ14 mm × 40 mm

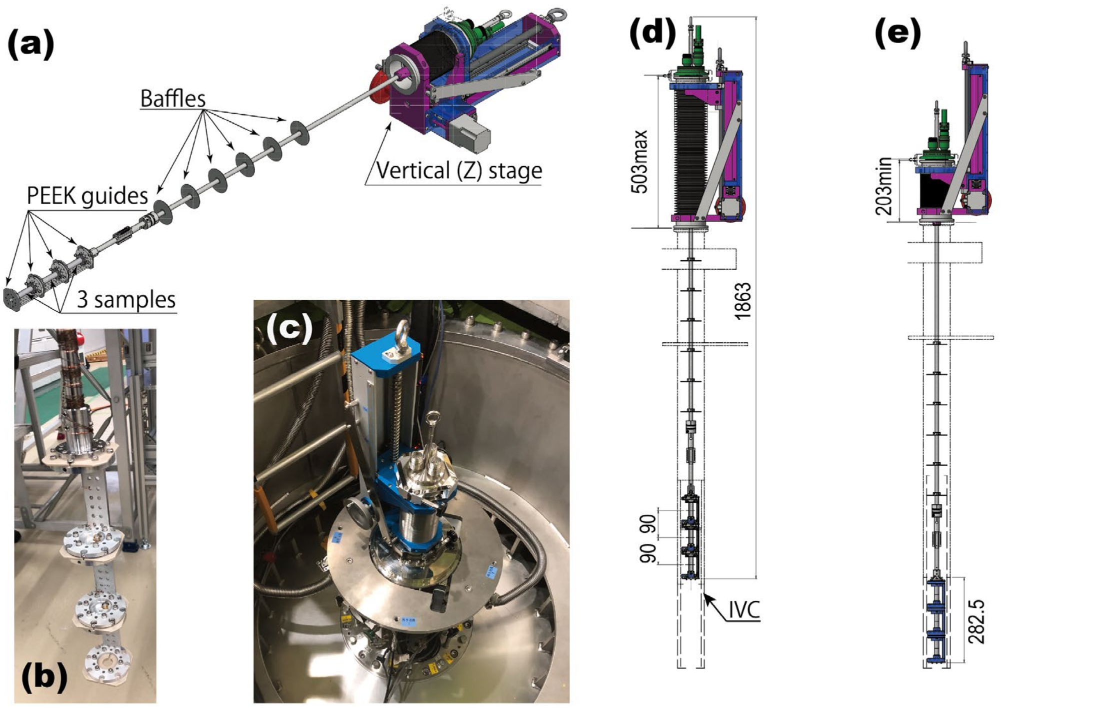

Fig. 1. (a) Schematic of Position Encoded Automatic Cell Elevator (PEACE), (b) image showing sample position, (c) top-view image of Z-stage, (d) image showing the highest position, and (e) image showing the lowest position. All dimensions shown in mm.

2. Specification of automatic sample changer

2.1 Design specification

Our developed sample changer is a sample stick type to fit our top-loading cryostat with a narrow space of the inner vacuum chamber (IVC) with diameter only of 70 mm as shown in Fig. 1. Overall structure is based on the design of the IRIS sample changer. Characteristics are listed in Table I. The sample changer consists of two parts, namely, a center stick and a Z-stage. The former has a length of approximately 180 cm and a weight of a few kg and allows us to set three samples. The latter weighs approximately 15 kg, has a vacuum tight elevator mechanism actuated by a stepper motor and the vacuum system is connected through a set of bellows. To use this system, at first, the heavy Z-stage is set top of the cryostat, then the long center stick is inserted. When replacing samples, only the long center stick is removed.

To ensure the reproducibility of irradiation position of the three samples, a completely new concept was adopted. Requiring high precision of the sample stick position, instead, we make the sample holder flexible with respect to the stick but fixed always in the same position relative to the IVC. The stick has a pivot between the upper part with a series of baffles and the lower part with three sample holders, which makes the lower part flexibly directed. As shown in Fig. 1a, four guide plates made of polyether ether ketone (PEEK) are attached to the sample holders, which softly touch the inner wall of the IVC and guide the sample into the required position. PEEK—a super engineering plastic—has advantage of small thermal conductivity, low thermal expansion coefficient, good heat and radiation resistance, and good mechanical properties, although it is not suitable for high temperature experiments.

Our sample changer stick was termed “PEACE,” i.e., position encoded automatic cell elevator. The sample position can be controlled locally or remotely through commands from PC. The latter allows us to involve the sample position command to a measurement sequence. When the exposure time for one sample is 3–4 h, a user can obtain three data sets.

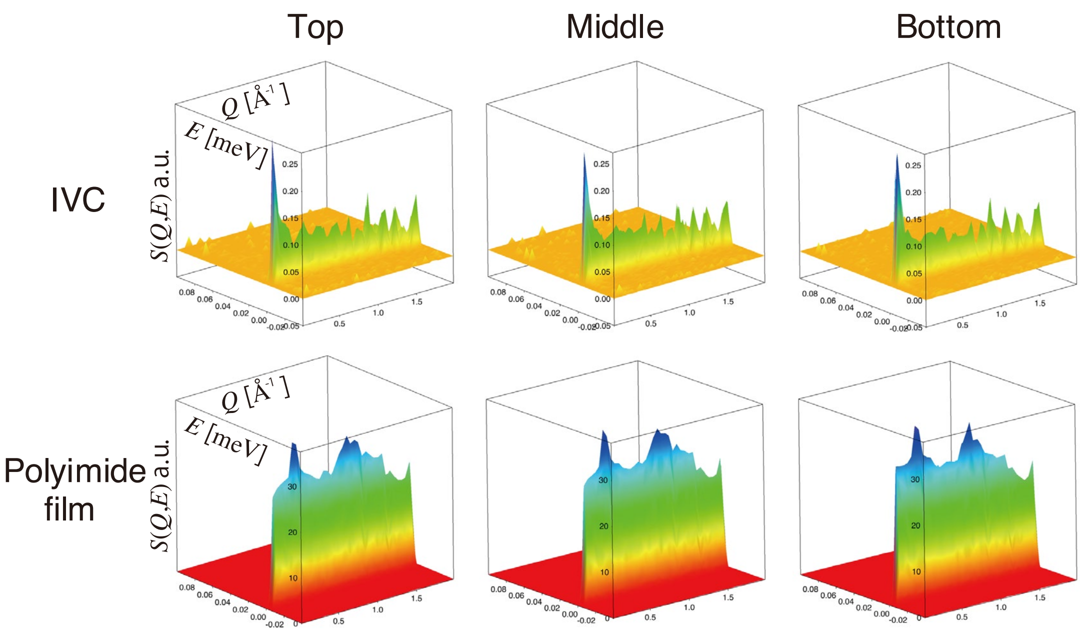

Fig. 2. $S(Q, E)$ profiles of no sample scattering (only inner vacuum chamber (IVC) scattering caused by aluminum) (top panels) and polyimide film with a thickness of 0.2 mm (lower panels), respectively. From left to right panels, top sample position, middle sample position, and bottom sample position of PEACE are shown.

2.2 Performance evaluation

The scattering profiles at room temperature of the no-sample condition, that is, the scattering from PEACE and the IVC in empty state, are shown in the upper panels of Fig. 2. The observed weak intensities of the scattering from the IVC and the PEACE (PEACE background) can be neglected when samples have strong intensities as seen with the intensities in the lower panel which were 2-orders of magnitude larger. Measurement for polyimide films (Kapton film) with a thickness of 0.2 mm show that the scattering profiles of the three positions were identical to each other as shown in the lower panels of Fig. 2. $S(Q, E)$ of vanadium (resolution) obtained by conventional stick and PEACE were also almost identical (Fig. 3). It should be noted that w background of the PEACE has to be treated carefi

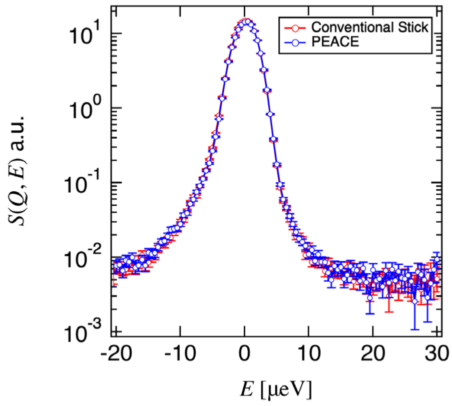

Fig. 3. $S(Q, E)$ of vanadium (resolution) obtained by a conventional stick and the PEACE under $-20 < E$ [μeV] < 30 which profiles were plotted an average at $0.125 < Q$ [Å^{-1}] < 1.875.

The deviation of the sample position from the stick center axis was measured as ±0.3 mm by using an eccentricity measuring apparatus. It is estimated from this value that the irradiated area at the sample can fluctuates ±2.1%. In fact, the difference in the observed scattering intensity among three sample positions was an acceptable value of ±1.6% at the elastic scattering position.

For QENS studies, we often obtain the temperature dependence of the QENS profile of samples and determine physical properties. To cover wide temperature range efficiently, we frequently change way to control temperature from heat exchange by He conduction gas to heating by a cartridge-heater equipped on the center stick in vacuum state. Therefore we also developed another automation tool which is an automatic IVC atmosphere controlling device. This device can control the amount of gas inside the IVC by remotely regulating electromagnetic valves (Fig. 4). Flashing of IVC, setting helium gas pressure, and setting high vacuum, can be performed sequentially. The combination of the PEACE and the BL02 IVC gas controlling equipment can be allowed further automation of measurements.

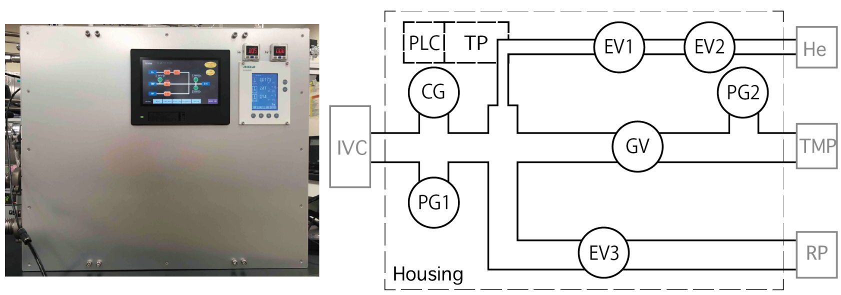

Fig. 4. BL02 (DNA) IVC gas controlling equipment. This system can control the gas pressure electronically. PG: Pirani gauge, CG: Capacitance gauge, EV: Electromagnetic valve, GV: Gate valve, IVC: Inner vacuum chamber, He: He gas, TMP: Turbo molecular pump, RP: Roots pump. TP: Touch panel, and PLC: Programmable logic controller.

3. Summary

A sample changer, termed as "PEACE," was developed and is currently available for use at room temperature. Very good reproducibility of the sample position has been achieved through a new concept of positioning the sample holder, however the use of PEEK for this restricts its use at high temperature. The scattering profile difference between the three positions was found to be reasonably small. Currently, we are developing a control software to operate the data acquisition system. The sample exchange frequency is substantially reduced using this automatic sample changer, then users can have a peaceful night sleeping. We are now developing a method to control and monitor the temperature at each sample position and soon start commissioning of PEACE for use at low temperature circumstance to extend available temperature range to 4K–300 K.

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