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Quartz Cell for a Backscattering Spectrometer

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We have developed a quartz double cylindrical sample cell optimized for a backscattering neutron spectrometer, especially for BL02 (DNA), MLF in J-PARC. A quartz glass tube, with one end closed, is shaved to obtain a wall thickness of 0.55 mm. The inner tube is properly centered onto a protrusion in the outer tube such that the distance between the outer and inner tubes remains constant. This quartz cell can be used for samples that should not be in contact with the aluminum surface. We verified the cell's background effect between the quartz cell and Al cell by employing QENS measurements using D_2O buffer. The elastic intensity profiles of the buffer at $Q < 1$ Å^{-1} while using both quartz cell and Al cell (A1070) were identical. When $Q > 1$ Å^{-1}, however, the profiles were different, which can be attributed to the first sharp diffraction peak of quartz glass. The data should thus be analyzed by consideration of adsorption correction and differences in the individual thickness of the quartz cell for this region.

KEYWORDS: Backscattering spectrometer, Quartz cell, Glass cell, QENS

1. Introduction

Aluminum (Al) and its alloys have been frequently used as sample cell materials for quasielastic neutron scattering (QENS) experiments [1]. It is known that Al is corroded by strong acids and bases [2]. In addition, Al ions are easily formed in aqueous solutions, thereby forming chelate compounds. Thus, the development of a new type of anticorrosive cell is necessary. Therefore, we have developed a quartz cell for backscattering studies.

For QENS measurements, a cylindrical sample shape is selected to ensure annular detector arrangement and achieve a large scattering volume with transmission above 90%. Thus, establishing a cell design is required. Halle et al. performed QENS measurements using quartz capillaries [3]. The capillaries were distributed annularly along the circle perpendicular to the neutron beam. According to the literature, 20 capillaries were prepared for each sample.

For optical measurements, quartz cells are mostly utilized for solution systems containing solutes, such as polymer solutions and protein solutions. In the case of neutron scattering experiments, quartz sample containers are used for small-angle neutron scattering or neutron spin echo. To effectively analyze the dynamics of the contained solute, the scattering profiles of cells and solvents must be subtracted from those of the solutions. These experiments are usually performed individually. Therefore, the sample positioning is essential. The cells with samples should be located at the same position as the empty cell to the maximum extent possible. Quartz tubes with an accurate shape are commercially available, such as a probe insert tube for NMR. However, the currently available tubes cannot be easily adopted, as it is difficult to obtain a desired thickness of the samples from the combination of the inner and outer tubes. We designed a double cylindrical cell consisting of two tubes having different diameters, which were obtained by grinding quartz rods to realize a proper annular shape of the sample.

2. Designing the quartz cell

2.1 Design specification

A quartz glass tube with one end closed is ground to have a wall thickness of 0.55 ± 0.01 mm. The inner tube is properly centered through a protrusion into the outer tube such that the interstice between the outer and the inner tubes maintains a constant distance, as shown in Fig. 1. The total quartz thickness becomes 2.2 mm. The combined quartz tubes are inserted into an Al container made of engineering-grade Al (A1070), which is vacuum-sealed by an indium (In) wire. The Al container also helps to avoid sample leakage in case of breakage of the brittle quartz cell. The centering of the combined quartz tubes is performed by using a lower spacer made of boron nitride (BN) at the bottom and an upper spacer made of polyether ether ketone (PEEK) at the top. The image and the schematic are shown in Fig. 1. The In wire used for sealing limits the temperature range of this cell. We normally use this cell at temperatures below 320 K.

Although the cell manufacturing is costly, all the materials can be reused after proper cleaning. Currently, quartz cells with sample thicknesses of 0.23 mm and 0.48 mm are available for the spectrometer at BL02 (DNA), MLF, J-PARC [4].

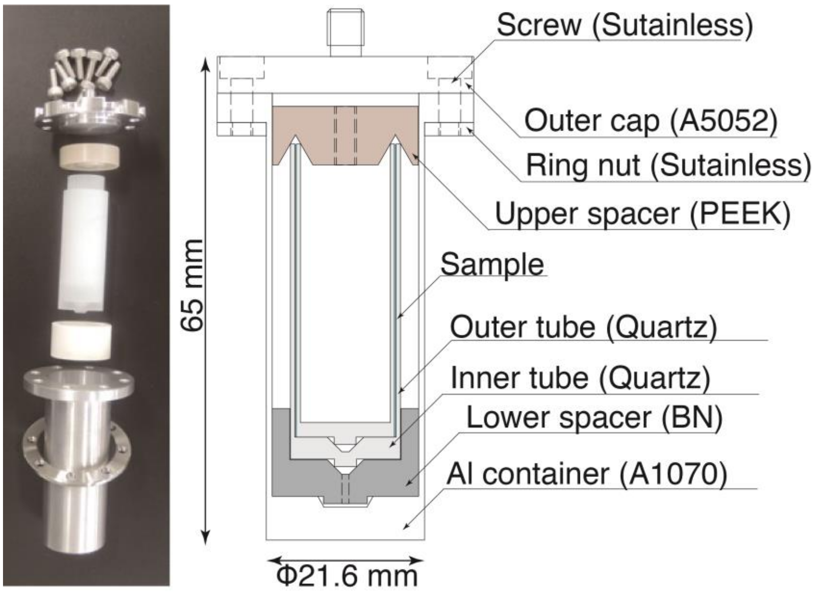

Fig. 1. Design of double cylindrical quartz cell for back-scattering spectroscopy.

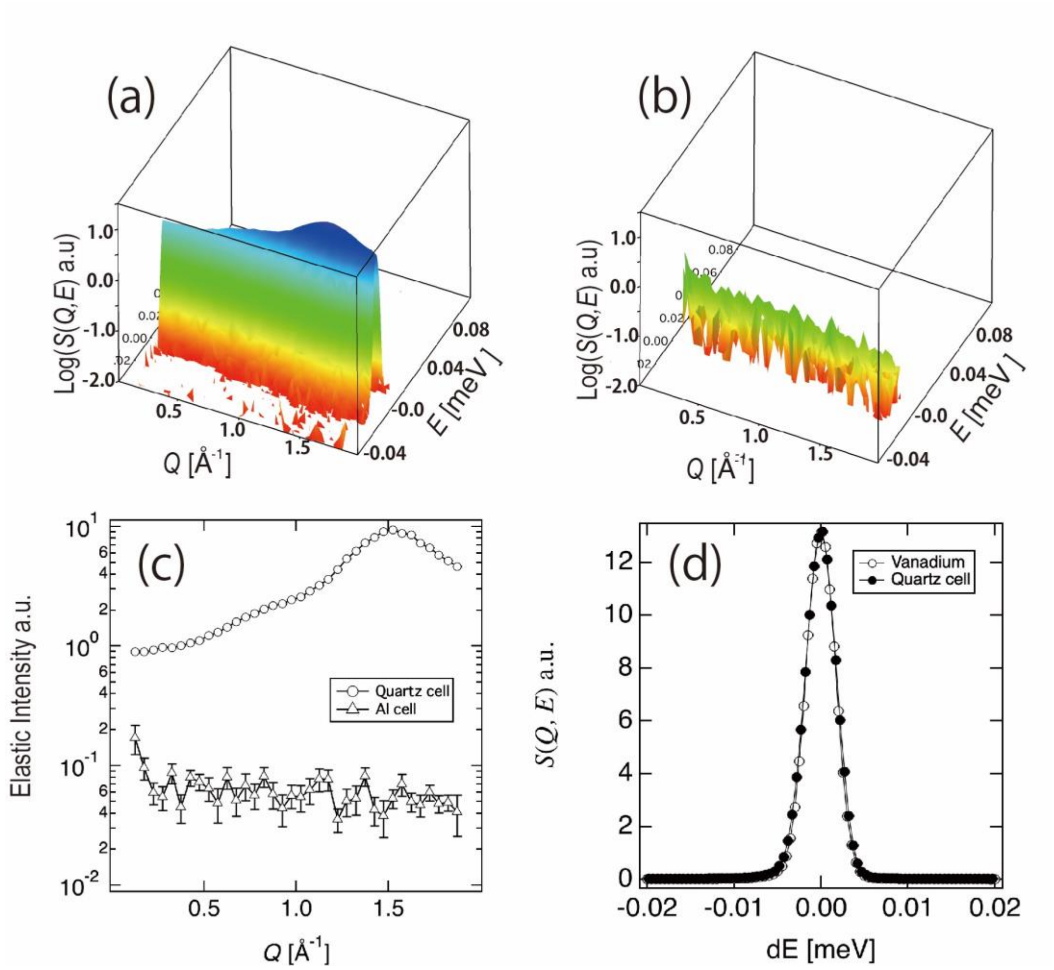

Fig. 2. $S(Q, E)$ map of (a) quartz cell (total thickness: 2.2 mm) and (b) Al cell (A1070) (total thickness: 0.5 mm), and (c) their elastic intensities integrated within ±20 μeV. $S(Q, E)$ profiles with $0.125 < Q$ [Å^{-1}] < 0.185 of a vanadium standard sample, and the quartz cell are compared in (d).

2.2 Performance evaluation

The diameter deviation of the quartz cell was achieved at ±0.01 mm in 20 sets. The deviation of the sample volume was ±2.2 % due to the spacers. The sizes of the Al container, PEEK spacer, and BN spacer were also within ±0.01 mm. The size variation of the quartz sample cell can be considered to be very small. Sample loading into this quartz cell, even in a fume hood, was not a difficult operation.

The profile of the dynamic structure factor, $S(Q, E)$, of the quartz cell in the Al container (QC), obtained under the measurement condition with the energy resolution of 3.6 μeV was compared with its Al cell counterpart in Figs. 2(a) and 2(b). The elastic intensity integrated over $-20 < E$ [μeV] < 20 is shown in Fig. 2(c). The scattering intensity from the QC is more than an order of magnitude higher than that of the Al cell. Quartz glass is a disordered material, which exhibits a strong first sharp diffraction peak

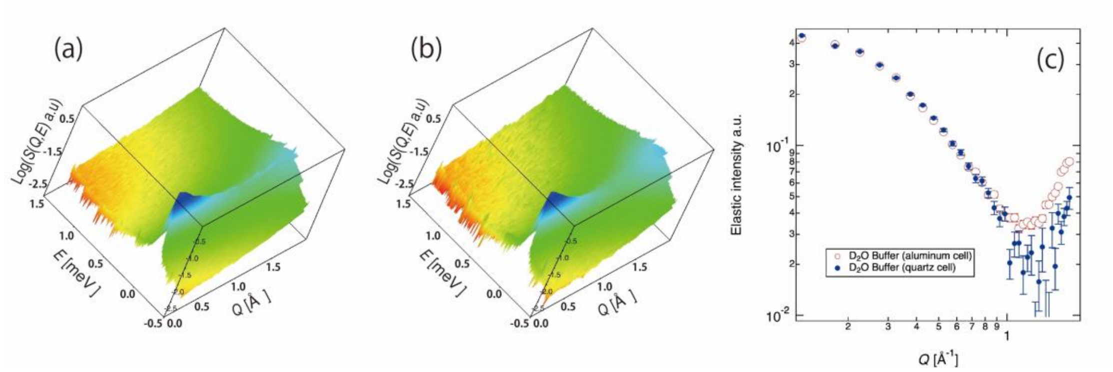

Fig. 3. $S(Q, E)$ map of D_2O buffer using (a) quartz cell and (b) aluminum cell, and (c) their elastic intensity of ±2 μeV.

(FSDP) of coherent elastic scattering at approximately 1.5 $Å^{-1}$. The strong intensity of the quartz glass compared to that of the Al cell is due to the coherent structure factor of the glass as well as the larger scattering volume and the larger neutron scattering cross section. It is known that quartz glass shows boson peaks at 3–5 meV out of the energy range of the BL02 (DNA). The $S(Q, E)$ profiles integrated in the Q region of $0.125 < Q$ [$Å^{-1}$] < 0.185 show no obvious difference in the E range of $-20 < E$ [μeV] < 20 between the QC and the standard sample of a cylindrical-shaped vanadium cell, which is generally used to measure the instrument energy resolution, as shown in Fig. 2(d).

We measured the D_2O buffer using different cells as a case of weak scattering. The D_2O buffer has a low incoherent scattering cross section compared to the H_2O buffer. Figure 3 shows the $S(Q, E)$ map of the D_2O buffer (20 mM Tris-HCl pD = 7.2, 200 mM NaCl, 10 mM dithiothreitol) using QC (Fig. 3(a)) and an Al cell (Fig. 3(b)) under the measurement condition with the energy resolution of 12 μeV as well as their elastic intensity integrated over $-20 < E$ [μeV] < 20 (Fig. 3(c)). These profiles were produced by subtracting a profile of the empty cell multiplied with a constant to account for the individual differences of cells with the zeroth approximation of the neutron absorption. The intensity profiles of the two configurations are similar at $Q < 1$ $Å^{-1}$, and the QC could be utilized even for a sample with weak scattering such as the D_2O buffers. However, when $Q > 1$ $Å^{-1}$, the profiles are different, which can be attributed to the FSDP; thus, the data must be analyzed carefully. For this region adsorption correction taking into account of wavelength dependence has to be considered as well as the difference in individual thickness of the QC.

Other low incoherent metals such as molybdenum [5], niobium [6], and platinum [7] are potential candidates as they are anticorrosive materials. Nevertheless, these materials involve machinability and base material cost. Dr. T. Seydel introduced some experiences on a cell holder for biological samples [8]. An Al container coated with gold, anodization of the Al surface, a thin Teflon coating of the Al container, and a quartz cell are introduced to provide alternate options to avoid the unfavorable corrosion of the Al cell. We are currently determining the corrosivity between the sample and container from the viewpoint of neutron applications. The experimental results will be summarized in the near future.

3. Summary

We have developed a double cylindrical quartz cell for the backscattering spectrometer BL02. The quartz cell is suited for solution samples that can corrode Al, and for samples which the Al ions easily contaminate. The double cylindrical cell consists of two tubes having different diameters which were realized by grinding quartz rods with one end closed and the inner tube was properly centered using a protrusion into the outer tube such that the interstice between the outer and the inner tubes maintains a constant distance where the liquid sample is introduced. The combined quartz tubes are inserted into an Al container which can be vacuum-sealed by the In wire.

We confirmed that although quartz glass exhibits coherent elastic scattering over the entire Q region, a correction factor of the cell scattering multiplied by a constant is subtracted from the spectrum to provide a reasonably easy way for backscattering when $Q < 1$ Å^{-1} for the sample contained in the cell. However, a careful correction will be needed for $Q > 1$ Å^{-1}, where the strong FSDP of the quartz glass is located.

The QC is available for use. A report on using this QC for a protein solution system has already been published [9], and the details of the QC were also described.

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