Sample Container Materials for High Temperature Neutron Scattering Experiment

Takashi Kamiyama[1], Hirokatsu Aoki[2], Tadahiko Masaki[2], Shuji Munejiri[2], Shin'ichi Takeda[3], Yoshinobu Ishii[4] and Toshio Itami[5]

[1]Graduate School of Engineering, Hokkaido University, Kita 13 Nishi 8, Kita-ku, Sapporo 060-8628, JAPAN

[2]National Space Development Agency of Japan (NASDA), 2-1-1 Sengen, Tsukuba 305-8505, Japan

[3]Graduate School of Science, Kyushu University, 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581, Japan

[4]Advanced Science Research Center, Japan Atomic Research Institute, Tokai-mura, Naka-gun, Ibaraki 319-1195, Japan

[5]Graduate School of Science, Hokkaido University, Kita 10 Nishi 8, Kita-ku, Sapporo 060-0810, JAPAN

Abstract

Sample container materials for high temperature neutron scattering experiments are discussed. We have chosen as container materials, sapphire and amorphous carbon. These materials can be used around 2000 K in vacuum. The sapphire container makes strong Bragg peaks on its scattering pattern, so the data correction becomes difficult. On the other hand the amorphous carbon container makes the broad peaks on the spectrum, which can be easily removed on the data correction. It has the incoherent scattering back ground at room temperature, but this contribution in the spectrum disappears at higher temperature. As a whole, the amorphous carbon is better as the container material of high temperature neutron scattering experiment than the sapphire.

1. Introduction

The properties of liquids are important in a wide range of scientific and technical fields by its role as a transport medium, a solvent, a material for processing and so on. However, the study of the dynamical properties of liquids has still remaining problems even in the simple liquids. This is caused by the difficulty of experiments, especially under high temperatures. In the scattering experiments, such as X-ray diffraction or neutron scattering, the experimental setup is difficult because there should exist no obstractions around the sample for taking wider scattering angle and keeping the minimum noise. This condition is particularly difficult for the high temperature scattering experiments. It is not always easy to keep the stable high temperature under this limitted condition. Recently the ultra high temperature furnace can be available by using the methods of infrared heating, metal foil heating or laser heating. Now the high performance furnace reaches up to 2000 K, but there still remains a problem, that is materials of the sample container for the scattering experiments.

The neutron scattering experiment is useful for the investigation of transport characters of liquids. The wavelength at thermal or cold neutrons makes neutron a very suitable probe for studies of the static structures and dynamic structures of liquids with atomic scale. X-ray diffraction method gives us such static structural information, but the X-ray has very high energy compared with the thermal atomic motions. The thermal neutron velocity has the same order as the thermal motions of atoms in the condensed matter, so it can be used like infrared techniques. The X-ray interacts with electrons in the matter. On the other hand, the characteristic of a neutron is absence of charge. By this nature the neutron can penetrate into bulk samples, their containers, cryostats, furnaces and so on. The neutron interacts with atomic nuclei and magnetic moments; it gives us the information about the position of nuclei and magnetic properties at the same time. The most important point to use the neutron is that we can get the space-time information of atomic structures and motions in the condensed matter at the same time. But this characteristic feature makes the interpretation of the neutron spectrum difficult, because all the scattering effects from the sample, container or furnace are included. We have to choose the container material that gives the weak scattering to the spectrum.

2. Container Material for Neutron Scattering

The normal container material in the neutron scattering experiment is aluminum. Aluminum has the small neutron scattering cross section, so it is very useful. But in the high temperature experiments we cannot use the aluminum container, because the melting point of aluminum is 660 K. SiO_2 glass may be available near 1000 K, but it cannot work at higher temperatures. Then we seek for other materials in our experiments; one is sapphire and the other is amorphous carbon.

Figure 1 shows the neutron scattering spectra of containers. The graphite container size is 8 mm inner diameter, 0.3 mm wall thickness and 30 mm height. The sapphire container size is 7 mm inner diameter, 5 mm wall thickness and 20 mm height. The amorphous carbon container size is 10 mm inner diameter, 0.3 mm wall thickness and 30 mm height. We can use the sapphire container up to 1873 K, and the amorphous carbon container up to 2000 K. The spectrum of each material shows respective characteristic features. The sapphire container has some Bragg peaks at 1.9 $Å^{-1}$, 3.1 $Å^{-1}$, 3.6 $Å^{-1}$, 5.2 $Å^{-1}$, 5.5 $Å^{-1}$, etc. Especially, 1.9 $Å^{-1}$ peak is very sharp and strong.

When this sapphire is used as containers, it becomes very difficult to subtract the container contribution from the spectrum of the combination of the sample and the container. In contrast to them, the amorphous carbon spectra show the broad peak

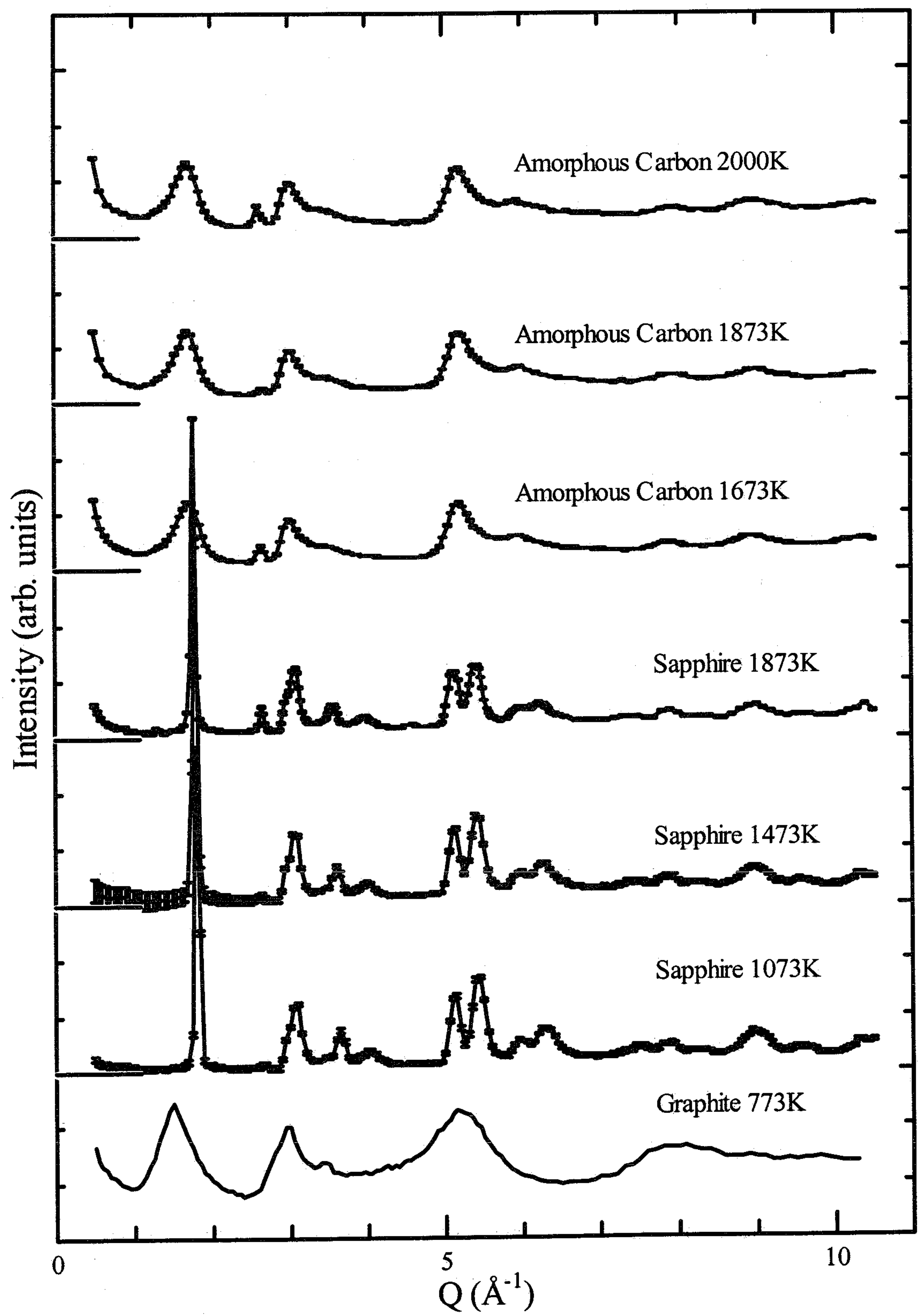

Fig. 1. Neutron total scattering spectra of the containers. The measurements have been done by TAS-1 spectrometer at JAERI, Japan.

pattern. There are mainly three broad peaks around 1.7 Å^{-1}, 3 Å^{-1} and 5.1 Å^{-1}. The small peak at 2.7 Å^{-1} is considered to be derived from the furnace. Similar peak is shown in the graphite spectra. The broad peak characteristics of amorphous carbon resemble also those of the graphite spectrum, shown in the figure 1. However, the graphite spectrum has stronger peaks than the case of the amorphous carbon. The broad and weak peaks of amorphous carbon indicate the easier data analysis. Thus, the amorphous carbon container may be more suitable for the high temperature neutron experiments. The use of the sapphire is limited to the single crystal. We have not yet try, but it seems to be hard to make a container of a sapphire single crystal. It is also not easy to prepare a container from an amorphous carbon. In the case of amorphous carbon the inelastic scattering effect may be weak because of its amorphous structure. The microscopic structure of amorphous carbon is considered that it consists of the superfine graphite crystals whose grain size is around 50 Å. This feature appears under 1 Å^{-1} on the spectra as small angle scattering. This means that it is difficult to use the amorphous carbon container for the small angle scattering experiment.

Figure 2 shows the temperature dependence of the amorphous carbon spectrum. The dotted line indicates the spectrum at the room temperature and the broken line at 1273 K. There is remarkable back ground in the room temperature spectrum. In this figure the difference between the 1273 K spectrum and the room temperature spectrum is also shown (the solid line). This difference shows the monotonously decreasing line. This may be considered as the effect of incoherent scattering by hydrogen atom [1]. By comparing the 1273 K spectrum with the higher temperature spectra of amorphous carbon in figure 1, we note that the back ground levels are almost same. Therefore, we

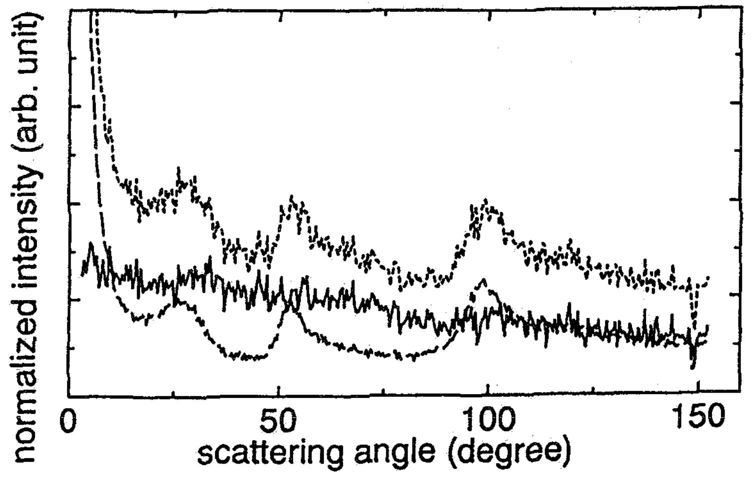

Fig. 2. Temperature dependence of the neutron total scattering spectrum of the amorphous carbon cell. The dotted line correspond to the spectrum at room temperature and the broken line is at 1273 K. The solid line corresponds to the difference between them.

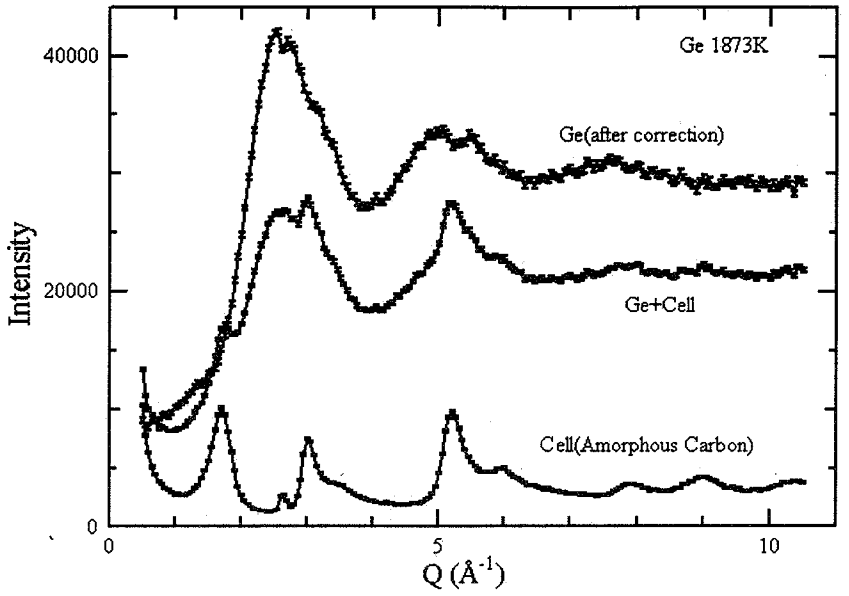

Fig. 3. The neutron scattering experiment of liquid germanium at 1873 K with the use of an amorphous carbon container.

can conclude that there is no effect of the incoherent scattering of hydrogen atoms in the amorphous carbon container on the higher temperature experiment.

In figure 3 is shown the neutron scattering experiment of liquid germanium at 1873 K with the use of an amorphous carbon container. The container scattering is sufficiently small and the scattering peaks of the amorphous carbon are so broad. Therefore, we can extract the sample spectrum easily. This is an advantage of the amorphous carbon container against the sapphire container. We will try to use the amorphous carbon container in other measurements, in particular for the inelastic scattering experiment, and confirm the usefulness of this material.

3. Conclusion

We have investigated the applicability of some materials to containers for high temperature liquid metals on neutron scattering experiments. The sapphire container and the amorphous carbon container can be used around 2000 K. The sapphire container shows some strong Bragg peaks, so the data correction becomes difficult. The amorphous carbon container shows the broad peak pattern that can be removed easily on the data correction. However, the amorphous carbon shows the incoherent scattering at the room temperature. Fortunately, this scattering disappears at higher temperatures. As a whole, the amorphous carbon is better as the container material for high temperature neutron scattering experiments than the sapphire. We will try to check amorphous carbon still more, and confirm the usefulness as the container material for neutron scattering of liquids with high temperatures.

Reference

[1] C. G. Windsor, *Pulsed Neutron Scattering* (Taylor&Francis LTD; London, 1981)