High Pressure Apparatus for Angle Dispersive Neutron Diffraction

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Synopsis

A piston-cylinder type high pressure apparatus was designed for the angle dispersive neutron diffraction. A Ti-53wt% Zr alloy was used for the cylinder. The performance was tested by observing the structural transformation under pressure in RbBr from an NaCl-type to a CsCl-type.

I. Introduction

In the previous reports[1,2], the present authors presented a high pressure apparatus for the neutron diffraction of time-of-flight (TOF) method. The high pressure cell was made of incoherent Ti-Zr alloy. The cell was reinforced by steel binding rings to make full use of the geometrical preference of the TOF method. The performance of the apparatus was checked by using the pulsed neutron source and favorable results were obtained.

In this report, the design and performance of the high pressure apparatus for the angle dispersive neutron diffraction is presented.

II. High pressure cell

The Ti-53wt% Zr alloy was used as a material of the high pressure cell, because the alloy has a special characteristic for thermal neutrons: In this composition, the coherent scattering amplitude of thermal neutron is zero, and no coherent peak is observed in the elastic scattering pattern. The alloy was prepared by an arc-furnace, then remelted and casted to a large cylinder: 3 - 4 cm diameter and 8 - 10 cm long.

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The arrangement of the apparatus is illustrated in Fig. 1: The high pressure cell is a piston-cylinder type, diameter of the center hole is 6 - 8 mm. Both ends of the cylinder are inserted into steel binding rings. The pistons are made of alumina, hardened steel or tungsten carbide. Powdered specimen is put into a thin-walled capsule of teflon, CS_2 is used as the pressure medium. The mechanical strength of the alloy is moderate; a cylinder was blowed out at about 12 kb.

The neutron diffraction experiment was carried out using the TOG diffractometer installed in the JRR-3 reactor, at the Tokai Establishment of JAERI.

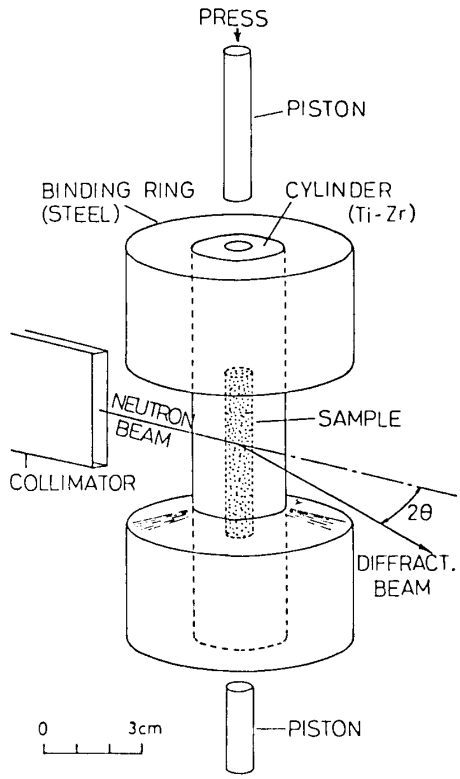

Fig. 1 Arrangement of the apparatus.

III. Neutron diffraction

The structural transformation under pressure in RbBr was observed at room temperature for the test of the performance. Powdered KCl was mixed to RbBr as a reference of pressure. A pattern obtained for RbBr of an NaCl structure at normal pressure is shown in Fig.2. In the figure, the background riginated from the incoherent scattering by the Ti-Zr cell is considerably high. However, peaks of elastically scattered neutrons by RbBr and KCl are clearly identified, and no extra peaks are observed. With increase of pressure, peaks shift to high angle inaccordance with a reduction of the lattice spacing. Then, at higher pressures RbBr transformed to a CsCl-type structure as shown in Fig. 3, the pattern for KCl remains in the NaCl-type.

The variation of volume with pressure in RbBr is shown in Fig. 4 by open circles, the value of pressure was determined by the volume change in KCl. In the figure, the compression curves of RbBr and KCl measured by Vaidya and Kennedy by X-ray diffraction[3] are shown by solid circles and connected with solid lines. The transformation in RbBr occurs at about 4.5 kb and the compression of follows the line obtained by X-rays.

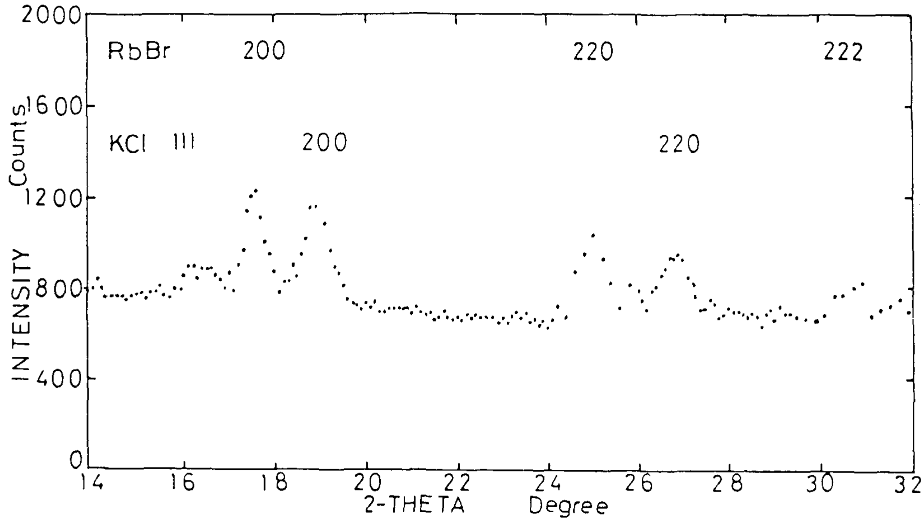

Fig. 2 Diffraction pattern of RbBr-KCl mixture at normal pressure.

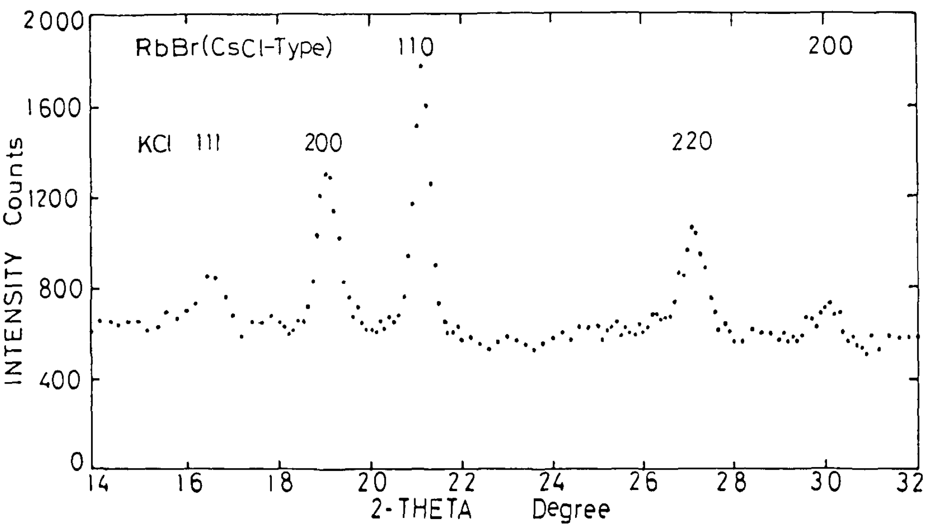

Fig. 3 Diffraction pattern of RbBr-KCl mixture at high pressure.

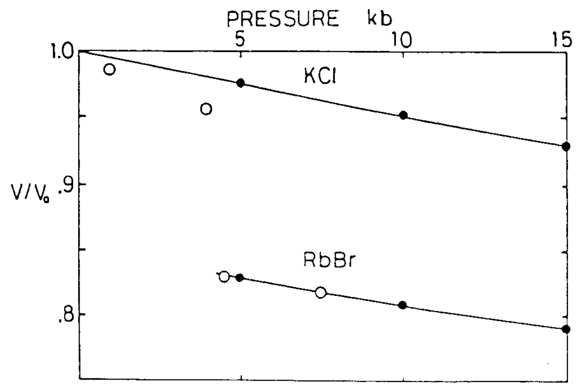

Fig. 4 Volume compression of RbBr obtained by the present experiment O, and those for KCl and RbBr by Vaidya and Kennedy (3) —●—.

IV. Discussion

Many materials are rather transparent for thermal neutrons in contrast with X-rays. Hence, the neutron diffraction is powerful for high pressure works, where specimens are surrounded by massive medium to generate and keep the pressure. In the present case of Ti-Zr alloy cylinder, no diffraction peak of the cylinder is superimposed and the original pattern of the specimen is not disturbed. The absorption of thermal neutron by Ti-53wt% Zr alloy is 0.1 cm^{-1}, the value is lower middle in ordinary substances. The patterns in Figs. 2 and 3 were obtained in about 6 - 12 hours.

The difference of performance of the apparatus between TOF method and angle dispersive method is not so serious. Because, the similar results were obtained for the structural transformation in RbBr. A definitive preference of the TOF method is the applicability for the analysis of dynamic phenomena[4)]. For the diffraction of single crystal specimen[5)], the present apparatus is preferable because the diffraction spots can be obtained in a wide area. In the latter, however, the reinforcement of the Ti-Zr cell is not sufficient. But, if the fine and intense neutron beam is obtained, the space between the binding rings could be made narrow. In this way, the limit of the highest pressure will be improved, and the field of of application can be extended.

Acknowledgment

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