Development of shear cell for high vapor pressure melts

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ABSTRACT

In order to establish a shear cell system for high vapor pressure liquids, a sealing mechanism was investigated. A boron nitride (BN), a pyrolytic boron nitride (PBN) and a glassy carbon were examined for a gas permeability and reactivity to B_2O_3. It was found that the PBN and the glassy carbon were suitable for shear cell disk materials. Simplified shear cells were designed and tested by using PBN and glassy carbon as disk materials. A high vapor pressure liquid, InAs, could be successfully sealed in the simplified shear cells, and the sealing effect was confirmed.

1. INTRODUCTION

As described in the previous section (B1), the shear cell method is useful for liquid diffusion measurements. However, since it consists of a pile of disks, it is hard to use it for high vapor pressure liquids like liquid compound semiconductors, such as liquids GaAs and InAs. In such cases, the vapor of liquids comes out through the disks, and, as a result, the concentration and the volume becomes different from those planned. In order to develop a shear cell with a seal mechanism for high vapor pressure liquids, some experiments were carried out. First, the absence of InAs vapor permeability was examined for a sealing material, B_2O_3. Second, prospective crucible materials were investigated for the nitrogen gas permeability. Third, sealing experiments for InAs vapor were conducted with the combinations of these crucibles and B_2O_3. Finally, a simplified shear cell was designed and was tested for liquid InAs.

2. SEALING MATERIAL TEST

2.1 EXPERIMENTAL

A quartz ampoule was employed for the container of InAs sample, and its top face was covered with a sandwich of boron nitride (BN) lid - B_2O_3 sheet of 1 or 2 mm thickness – BN lid, as shown in Fig. 1. The sample was heated at 773 K for 10 minutes in vacuum to soften the B_2O_3 sheet, then heated up to 1323 K in an Ar atmosphere of 360, 560, 760 and 1140 Torr for 2 hours. After it was furnace-cooled, the weight change was measured. Since a quartz has no gas permeability, the weight loss, if present, should be derived from the permeability of sealing material, B_2O_3 .

2.2 RESULTS

Figure 2 shows the ampoules after the experiments with 2 mm thickness B_2O_3 sheet. The BN lid was lifted at 360 Torr atmosphere and the quartz tube blackened due to As vapor, as shown in Fig. 3. Although the weight differences between before and after experiments were almost 0 % at 560, 760 and 1140 Torr, it was over 9 % at 360 Torr (Fig.4). Therefore the sealed shear cell should be used at more than 560 Torr atmosphere.

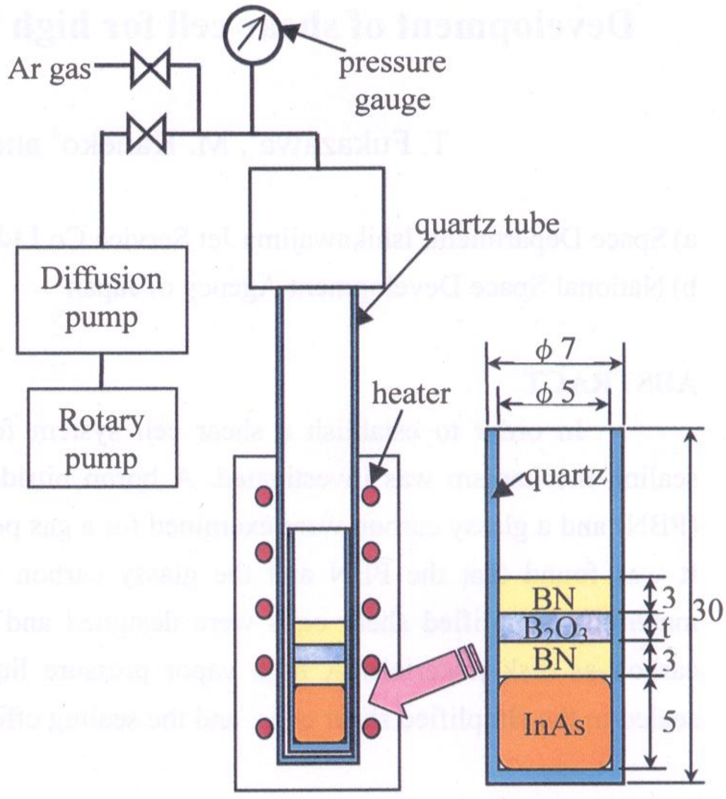


Figure 1 Sample configuration of sealing material (B_2O_3) test

(a) (b) (c) (d) (e)

Figure 2 Ampoule after seal material test: (a) Before experiment, (b) 360 Torr, (c) 560 Torr, (d) 760 Torr, (e) 1140 Torr.

Figure 3 Quartz tube after experiment at 360 Torr

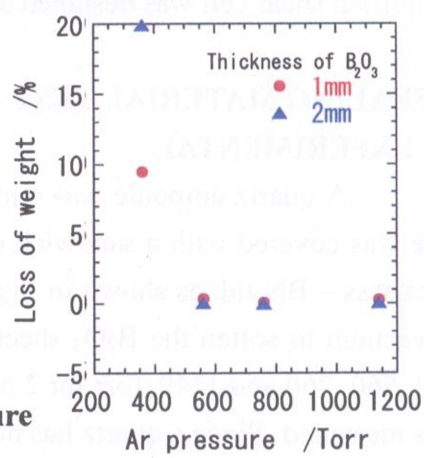


Figure 4 Loss of weight vs. Ar pressure

3. GAS PERMEABILITY TEST FOR CRUCIBLE MATERIALS

3.1 EXPERIMENTAL

Gas permeability tests were carried out for BN and glassy carbon (GC). The BN materials tested here were two kinds of BN, high purity UHS-FL (99.5 % BN) and high density UHS-D (95 % BN including B_2O_3-CaO binder). The oxidized BN of these two kinds (1243 K for 1, 3 and 10 hours) were also supplied to the experiments. Sample thickness was 1.48 mm for oxidized BN, 2mm for untreated BN and 0.64 mm for GC.

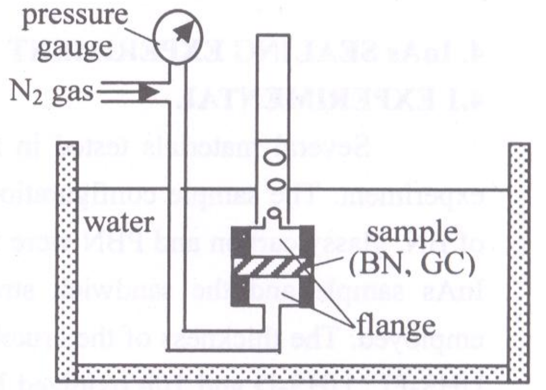


Figure 5 Experimental configuration of gas permeability test

Figure 5 shows the experimental configuration. A sample plate (BN and GC) was put between a pair of flanges. This set was sunk in water and nitrogen gas was passed through from the bottom with the pressure of 0.5 – 3.0 kgf/cm^2. The amount of gas, passed through the sample, was measured.

3.2 RESULTS

Figure 6 shows the gas permeability at each gas pressure. The rate of gas coming through the samples was almost 0 for all materials at 0.5 kgf/cm^2, but it increased with the increase of gas pressure for BN. The rate of the permeability for the oxidized BN decreased if the oxidization time became large, and the gas didn't come through for 10 hour oxidized BN even at 4 kgf/cm^2. It was confirmed that there was no gas permeability for GC until 3.0 kgf/cm^2.

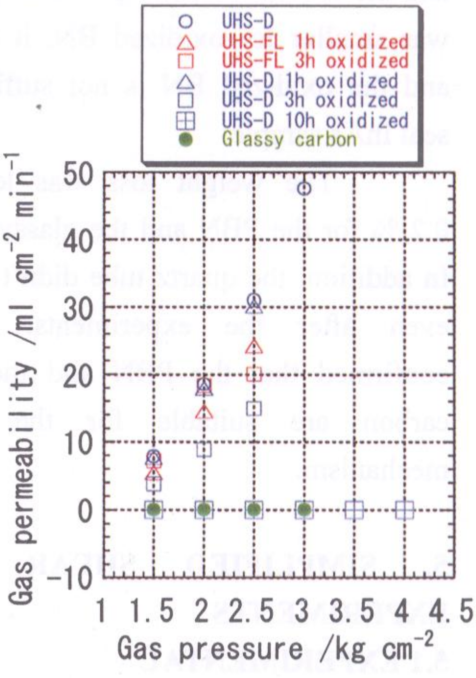


Figure 6 Gas permeability for BN and glassy carbon

A pyrolytic BN (PBN) maker (Advanced Ceramics International Corporation) examined the gas permeability for coated PBN on graphite. They found that there was no gas permeability for more than 10 μm coated

PBN at gas pressure of 3 kgf/cm^2.

4. InAs SEALING EXPERIMENT

4.1 EXPERIMENTAL

Several materials tested in Section 3 were used as a crucible in this sealing experiment. The sample configuration was similar to one in Section 2, and crucibles of BN, glassy carbon and PBN were used instead of the quartz ampoule in Fig. 1. Also, InAs sample and the sandwich structure (BN-B_2O_3 of 1 mm thickness-BN) were employed. The thickness of the crucible was 1.5 mm and 3 mm for BN (3 types of BN, UHS-FL, UHS-D and 10h oxidized UHS-D), 0.5 mm for PBN, and 1.1 mm for glassy carbon.

The sample was held at 773 K for 10 minutes in vacuum to soften the B_2O_3 sheet, then heated up to 1323 K in 760 and 1140 Torr Ar for 2 hours. After it was furnace-cooled, the weight change was measured.

4.2 RESULTS

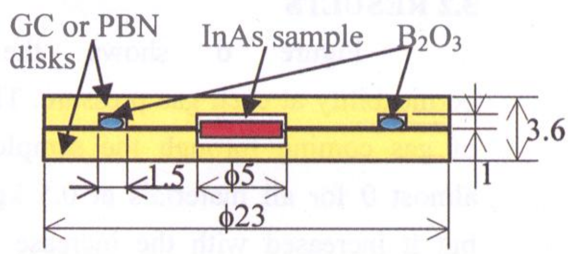
The loss of weight amounted to about 14 – 19 % for both UHS-FL BN and UHS-D BN of 1.5 and 3 mm thickness. The BN lid was lifted to the top of the crucible and the quartz tube blackened at the upper part. Most of the InAs vapor was thought to come out through the gap between the lid and the crucible. Although the weight loss was smaller for oxidized BN, it was 8% and the oxidized BN is not sufficient to seal InAs sample.

The weight loss was less than 0.2 % for the PBN and the glassy carbon. In addition, the quartz tube didn't blacken even after the experiments. It was confirmed that the PBN and the glassy carbon are suitable for the sealing mechanism.

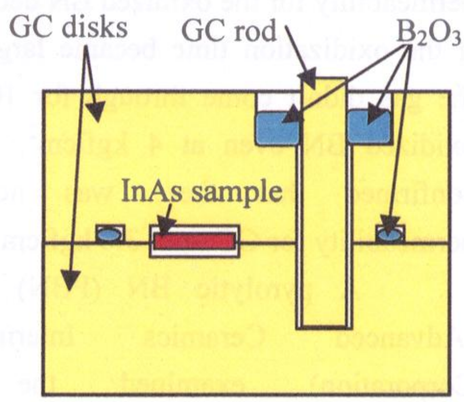
5. SIMPLIFIED SHEAR CELL EXPERIMENTS

5.1 EXPERIMENTAL

Two types of simplified shear cells were designed and tested. Simplified shear cell "A" consisted of two pieces of



(a) Simplified shear cell "A"



(b) Simplified shear cell "B"

Figure 7 Sample configuration of simplified shear cell experiments

cell, which had a groove to set B_2O_3 and a dent for InAs sample, as shown in Fig. 7 (a). Simplified shear cell "B" had one more hole to set a rod, which was considered as a model of a rotation shaft in a shear cell. B_2O_3 surrounded the rod to confirm the sealing around the shaft (Fig. 7 (b)).

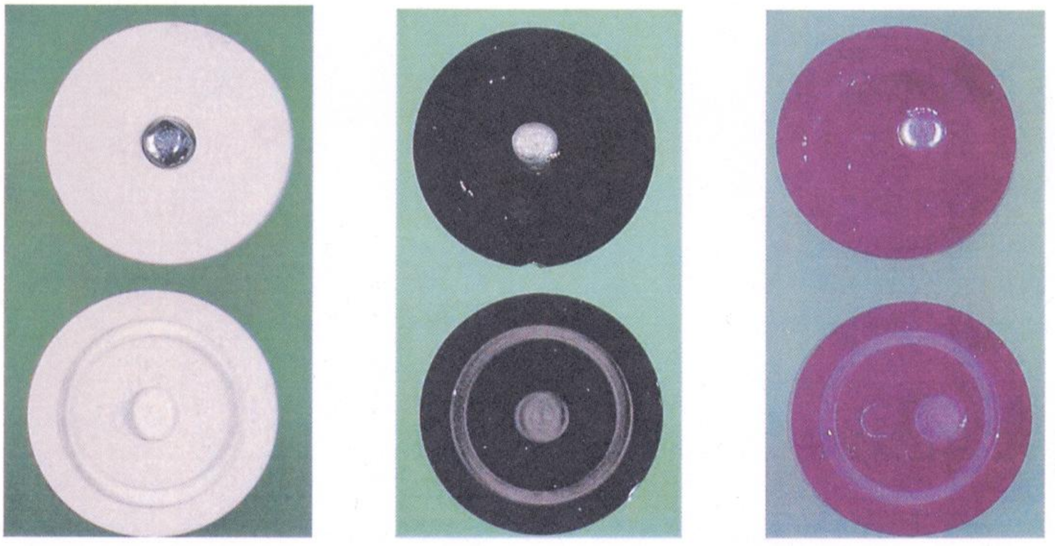
PBN and glassy carbon disks were used for simplified shear cell "A", and glassy carbon disk was used for "B". The simplified shear cells "A" and "B" were respectively contained in the quartz tube in Fig. 1. The experiments were carried out in 1140 Torr Ar with the same temperature change in Section 4.

5.2 RESULTS

Figure 8 shows the simplified shear cells and samples after the experiments. The PBN and the glassy carbon disks didn't react to B_2O_3 or InAs sample and wasn't damaged. The glass tube was clean and InAs was not observed in it. The weight loss was less than 2 % (0.025 g), as shown in Table 1. The validity of the sealing system, which consists of B_2O_3 and glassy carbon or PBN disks, was confirmed.

6. CONCLUSIONS

The sealing system of a shear cell for high vapor pressure melts was studied by using InAs sample. It was confirmed that a PBN and a glassy carbon are suitable materials for shear cell disks and the vapor of InAs can be sealed by using B_2O_3.



(a) Simplified shear cell "A" : PBN

(b) Simplified shear cell "A" : glassy carbon

(c) Simplified shear cell "B" : glassy carbon

Figure 8 Sample after InAs sealing tests with simplified shear cell "A" and "B"

Table 1 Weight change after InAs sealing tests using simplified shear cells

Shear cell type	Disk material	InAs weight (g)			Rate of weight loss (%)
		before	after	difference	
A	PBN	0.1400	0.1386	0.0014	1.00
		0.1470	0.1460	0.001	0.67
	Glassy carbon	0.1483	0.1462	0.0021	1.42
B	Glassy carbon	0.1825	0.1825	0.0000	0.00
		0.1826	0.1802	0.0024	1.31