HIGH PRESSURE CLAMP CELL FOR DILUTION REFRIGERATOR

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Two small high pressure clamp cells using at about thirty millikelvin region are devised. These consist of one couple of Bridgman-anvils and two flanges. One is used for a d.c. electric resistance measurement and another is used for an a.c. magnetic susceptibility measurement under high pressure. The sample is placed between two small anvils and a hydraulic press pressurizes it at room temperature. To maintain the pressure applied to the sample, the anvils are clamped by three bolts. The highest pressure attained by these clamp cells is fifty kbar. The clamped high pressure cell is attached to the underside of the mixing chamber of a helium 3/ helium 4 dilution refrigerator and is cooled down to thirty millikelvin. Several design points of the clamp cell and its cooling process are described.

1. Introduction

High pressure experiments at the liqid helium temperature region have been conducted and several pressure-induced superconductors have been discovered(1) in recent years. As there are the interests that whether there is still any other pressure-induced superconductor or not, whether the superconducting properties are destroyed by the application of very high pressure or not(2) and what is the pressure dependence of the superconducting transition temperature of low Tc superconductors, high pressure experiments at further low temperature region are required.

In order to cool the high pressure apparatus down to the several tens millikelvin region, the following technical problems have to be solved.

First: The heat leakage from the outside have to be reduced.

Second: The small size apparatus must be designed.

Third: Whether hydrostaticity of the pressure transmitting medium exists at low temperature in the case of the small apparatus.

In recent years, several papers of high pressure experiments at temperatures lower than 1 kelvin are reported. Those are as follows. Levy and Olsen(3) cooled a clamped apparatus of 21 kbars using a helium 3 cryostat. Brandt and Ginzburg(4) tried to cool a small ice bomb cell down to 80 millikelvin by an adiabatic demagnetization method and measured the superconducting transition temperature of Cd under 27 kbars. Benoit et al.(5) tried to cool a small clamp cell of 10 kbars down to 16 millikelvin also by the adiabatic demagnetization method and studied a nuclear orientation experiment. Stritzker et al.(6) reported to cool a high pressure cell using a dilution refrigerator down to 70 millikelvin for studying the pressure effects on the superconducting materials.

In order to study the pressure effects on low transition temperature superconductors, two small high pressure clamp cells are devised. These are originally designed for the experiments at the liquid helium temperature(7) and are redesigned for the experiments at temperatures of the several tens millikelvin region.

In this paper, the details of two clamp cells, the cooling method, the pressure calibrations and the temperature distribution of the cell during the cooling process are described.

2. Small high pressure clamp cell

Among several types of high pressure apparatus, it is best to use the clamp cell for attaining temperatures lower than 100 millikelvin, because of its small heat capacity and its small heat leakage during the experiment. In this method, the sample is placed between two small anvils and a hydraulic press pressurizes it at the room temperature. To maintain the pressure applied to the sample, the anvils are clamped by the screw mechanism. In Figs. 1 and 2, two clamp cells are shown which are originally designed at liquid helium temperatures(7), and are redesigned for this purpose. The sample is placed between the anvils and these anvils are held between two flanges and these flanges are tightened by three bolts. As two flanges are used in this type of clamp apparatus, it is very easy to replace the sample, to avoid the twisting of the sample and the breaking of lead wires, and it is convenient to fix the measuring coils for an a.c. magnetic susceptibility which is shown in Fig. 2. For attaining the accurate and reproducible pressure, several torque wrenches are used.

The sample assembly for a D-I type cell is shown in Fig. 3. The pyrophyllite ring is heated to 650°C for 30 minutes in order to increase its hardness, and then is fixed with an insulating cement to the face of the anvil. A sample is placed in the talc disc which is a pressure

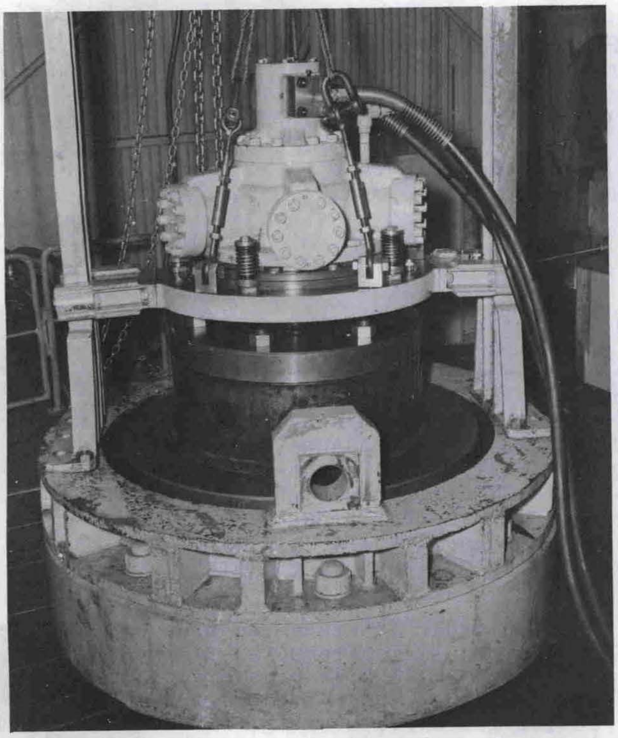

Photo. 2 High Pressure Oil Vessel

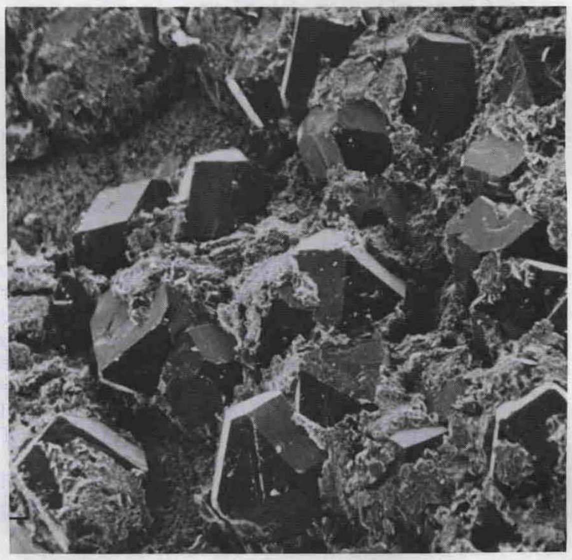

Photo.3 A Scanning Electron Micrograph of the As-grown Diamond Crystals

Table 1 Practically Available Pressure and Volume Relations for These Apparatus

	HO-type				OH-type	SO-type			
edge length a cm	0.5	10	15	25	15	35	45	55	65
anvil enveloped space Vo cm^3	0.059	0.47	1.59	7.366	3.375	20.21	42.96	78.43	129.5
pressure medium volume $(1.3)^3Vo\ cm^3$	0.129	0.53	1.04	16.18	7.425	44.40	94.38	172.31	284.4
achieved practical pressure P_1 kb	250	180	130	100	100	90	75	65	55