# *Clamp Type High Pressure Apparatus Using Small Bridgman Anvil at Low Temperature*

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High pressure apparatus at liquid helium temperature is built. It is a high pressure cell of clamp type using small Bridgman anvil (4 mm face) geometry. The pressure is applied to a sample using a standard hydraulic press and clamped by means of three bolts at room temperature, then the cell is cooled down to low temperature. The pressure is calibrated by means of the phase transition of Bi, Tl and Sn at room temperature, and pressure dependence of superconductive transition temperature of tin at low temperature which is measured by means of the electrical resistance and a.c. mutual inductance bridge.

## § 1. Introduction

In the field of solid state physics, investigations at high pressure and low temperature have been made and these investigations are mainly P. V. T. relations of solidified gases and the measurement of the effect of pressure on the superconductive transition. For researching high pressure effects at low temperature, two directions chiefly exist. One is a study of initial slope of pressure dependence in physical properties under hydrostatic pressure condition below 10 kbar, and the other is a study under very high pressure. In the latter case the hydrostatic pressure condition is poor than the former. For the former study, a direct piston displacement apparatus[1] or a helium gas pressure apparatus[2] have been used. The direct piston displacement apparatus is a simple device of generating pressure less than 20 kbar at low temperature, which has an advantage for applying a continuously variable force to the experimental piston. Then pressure can be controlled by a hydraulic press at room temperature. Because of this reason, the apparatus becomes large and consumes a lot of liquid helium (about 1 $l$/h). Moreover, it cannot cool down to temperature lower than 2K because of its large heat capacity and large heat conduction from the outside.[3]

Pressure in the gas system can be also varied continuously up to the freezing pressure of the gas, but this is hazardous to work with. The pressure range of the gas system is quite limited less than 10 kbar at low temperature.

The latter very high pressure device was originally made by a so-called 'fixed clamp' technique of Chester and Jones[4] and has been developed by Buckel and Wittig.[5] A thin sample was squeezed between two anvils together at room temperature and subsequently clamped. The clamped anvils were detached from the hydraulic press system, and cooled to low temperature. This fixed clamp method of producing very high pressure suffers from the defect that the pressure cannot be varied continuously at low temperature. To change the pressure in the sample, the clamp must be reset at room temperature. However, the absence of heavy external connections means that the heat leaks into the experimental cryostat can be kept very small. By this method, Buckel and Wittig found the new modification of superconductor of Si and Ge which are semiconductors at normal pressure. These discoveries seem to suggest that the other non-superconductive materials may become superconductors at high pressure. It is very interest whether or not, for example, all alkali metals become superconductors under the condition of high pressure and low temperature.

In order to study properties of solid under the extreme condition of high pressure and low temperature, especially higher than 100 kbar and lower than 0.1 K, we have built a high pressure clamp type apparatus at low temperature which accepts a small Bridgman anvil. This cell is improved on the Wittig's type. At the same time we developed a technique of the measurement of a.c. magnetic susceptibility. The pressure in this high pressure cell is calibrated by the phase transition of Bi I–II, III–V, Tl I–II and Sn I–II at room temperature, and by the pressure dependence of superconductive transition temperature of tin at low temperature. In this paper, the design of the pressure apparatus, its pressure calibration and the methods in measuring of the resistance and susceptibility are discussed.

## § 2. Clamp Type High Pressure Apparatus

Clamp type high pressure apparatus which accepts a small Bridgman anvil is most convenient to obtain the extreme conditions of lower temperature and higher pressure. Furthermore, this apparatus can avoid excessive helium consumption.

Thus we have built a clamp type cell used a small Bridgman anvil (4.0 mm face) geometry which is made from tungsten carbide. This has mainly two advantages as follows;

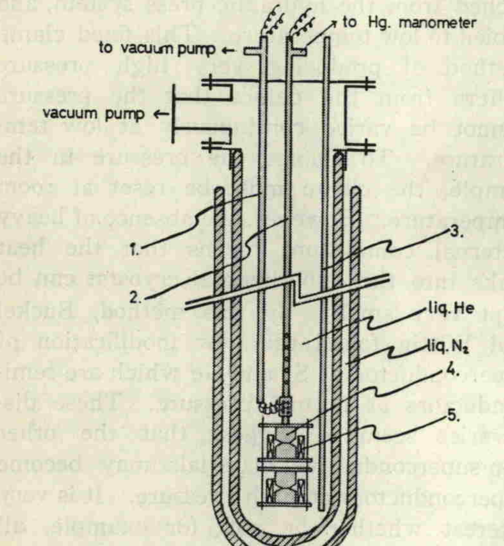


Fig. 1. High pressure cryostat.
1. Vacuum stainless steel tube. Electrical lead wires for measuring the a.c. mutual inductance pass through this tube.
2. Stainless steel tube which supports the high pressure clamp apparatus. Electrical lead wires for the d.c. measurement pass through this tube.
3. Inlet for the liquid helium.
4. Tungsten carbide anvil.
5. Measuring coil.

the first is to have used the flange type for clamping mechanism and the second is to have developed an a.c. mutual inductance method for measuring the superconductive transition temperature using weakly ferromagnetic tungsten carbide anvil.

Wittig[6] had used a mechanism which clamped a Bridgman anvil tightly each other with an attached screw nut.

At first, we had used Wittig's mechanism. But in that way, we had often troubled to break of lead wires during the clamping process because the lead wires are twisted when the screw nut is tightened to clamp. Therefore, we have improved Wittig's cell in several points. The high pressure cryostat is shown in Fig. 1. As shown in Figure 2, to clamp a sample, two flanges and three bolts are used. This type is convenient to exchange the sample and is free from the twist of lead wires. Moreover, using this cell, one can adopt an a.c. method of a measurement which means a no-lead wire method so it is convenient to avoid the trouble of the lead wire discussed above. The material of the high pressure apparatus at low temperature is one of serious problem. Most steels become brittle at low temperature. In general, steels with low carbon and high nickel content (austenitic stainless steel) are sufficiently ductile at low temper-

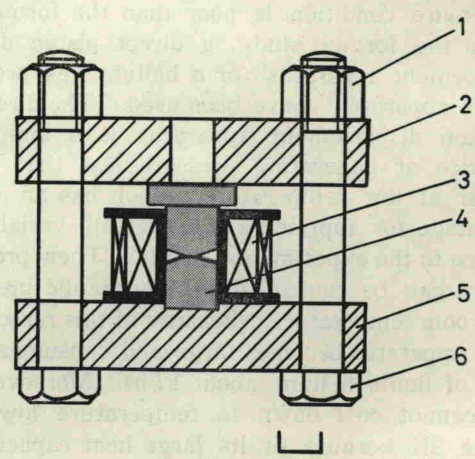


Fig. 2. High pressure clamp apparatus.
1. Fixing nut.
2. Upper flange.
3. Measuring coil.
4. Tungsten carbide anvil.
5. Lower flange.
6. Clamping nut.