Simple clamp pressure cell up to 30 kbar

Hiroshi Fujiwara, Hideoki Kadomatsu, and Kiyokazu Tohma

Faculty of Science, Hiroshima University, Higashisenda 1-1-89, Hiroshima 730, Japan

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A design of simple clamp type pressure apparatus utilized for measurements of magnetic susceptibility and electrical resistivity at low temperatures is presented. The cell consists of WC piston and Be–Cu cylinder which was autofrettage processed, and sample cavity consists of a teflon bucket and an electrode plug. In a temperature range from 300 K down to 77 K, pressure was determined by manganin gauge which was calibrated by Bi I→II transition pressure at room temperature and also the temperature dependence of pressure coefficient of manganin resistance was taken into account. As a result, the cell was capable of generating hydrostatic pressures up to 30 kbar at room temperature and at least up to 25 kbar at 4.2 K.

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INTRODUCTION

The investigation of the magnetic properties at high pressure and low temperature is undoubtedly one of the important projects for the basic understanding of magnetism. Along the lines of this adjudication, we reported the effect of pressure on the crystallographic as well as magnetic phase transitions of some ferromagnetic alloys and compounds in a temperature range down to 4.2 K under hydrostatic pressures up to 8 kbar.[1,2] There, both the piston and cylinder in a clamp type pressure cell were made of Be–Cu, and the cylinder was used within an elastic limit.

However, there should remain many phenomena, which will be uncovered and understood magnetically when the measurements are made under much higher hydrostatic pressures. Recently, we have designed a simple clamp cell which consists of the WC piston and the autofrettage processed Be–Cu cylinder. A teflon bucket, which has been generally used in the high pressure and low temperature measurements,[3,4,5] was employed for a sample cavity in the cell. In the present paper, the details of the design of the cell and the determination of pressure are presented. The apparatus has been practically utilized for the measurements of magnetic susceptibility and of electrical resistivity at low temperatures.

I. PRESSURE CELL, SAMPLE CAVITY, AND MEASURING SYSTEM

Pressure cell. The pressure generating system, the pressure cell, consists of an assembly of shaft, piston and cylinder. They are made of hardened (HR_C 57) alloy steel (JIS SUJ-2), WC (GTi 10, MITSUBISHI METAL Ltd.), and hardened (HR_C 44) Be–Cu (BeA-25, NGK INSULATORS Co., Ltd.), respectively. The hardening was made in our laboratory in accordance with prescription. Figure 1 shows a cross section of the pressure cell. The dimensions of cylinder are 35, 27 and 6 mm, in length, o.d. and i.d., respectively. The assembly is inserted inside the holder (JIS SCM-4) together with the WC backing plate bound with a Be-Cu binding ring. The holder in our device also serves as a protector against the pressure cell. The cylinder was not press fitted through the holder, so that it could be removed. Instead, it was tightened with brass supporting nut and polyester tape was tightly wound over both the bottom part of the cylinder and the binding ring. A thermocouple was attached to the cylinder installed through the hole in the holder as indicated in the left-hand wall of the holder in Fig. 1.

For the purpose of elevating the working pressure, we tried to perform hydraulic autofrettage process to the cylinder during actual pressurizations. When the cylinder was subjected to maximum pressure of 30 kbar the expansion inside the cylinder hole, over which the pressure is actually exerted, was measured. The amount of expansion was about 3% of i.d. Then it was confirmed that further expansion was not found in subsequent pressurizations, indicating the accomplishment of the autofrettage. As a result, it has been found that a maximum working pressure could be elevated constantly up to 30 kbar without any trouble. On the other hand, when the Be–Cu cylinder has been used as an elastic cylinder without autofrettage process, the maximum working pressure would be reduced to about 10 kbar. For the measurements at lower temperatures, the whole assembly has been transferred and placed in the thermal insulating chamber.

Sample cavity. In Fig. 2(a) are shown the details of a sample cavity, which consists of a teflon bucket, 1 mm in wall thickness, and a Be–Cu electrode plug. The seal of the bucket against the piston or the plug is provided by unhardened Be–Cu sealing rings with triangle cross section. Varnish (GE 7031) was applied to the part of bucket in Fig. 2(a) to which the plug touches. The cylinder was completed with fine finishing so as to fit the piston inside the cylinder hole without any play. The seal of the electrode plug was provided as follows by means of single cone sealing. A cone is of stainless steel and the cone angle is 60°. The plug has a hole, 1.4 mm in diameter, and two 60° cone openings at both

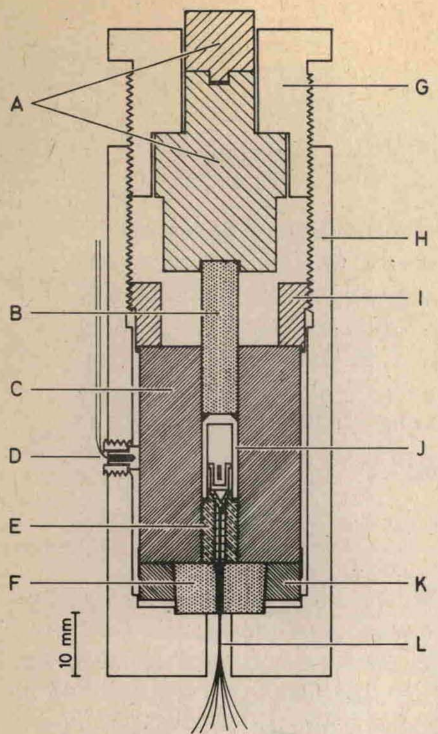

FIG. 1. Cross section of pressure cell. A: Shaft, B: Piston, C: Cylinder, D: Thermocouple, E: Electrode plug, F: Backing plate, G: Lock nut, H: Holder, I: Supporting ring, J: Teflon bucket, K: Binding ring, L: Leads.

the upper and lower ends of the hole, where the upper opening is much deeper. Hereafter, the expression like upper or lower will simply be used concerning the configuration drawn in the figure.

The embedments of the Formvar-insulated copper wires 0.14 mm in diameter, and of the cone into the plug hole were completed with epoxy resin (STYCAST 2850 GT, Emerson & Cuming Inc.), in the following order. (i) The epoxy resin is degassed in vacuum for a minute. (ii) After passing the lead wires through the plug hole, the fused resin is poured into the plug hole from the upper opening. The steel cone is plugged at this step and the resin is cured. (iii) The fused resin is poured from the lower opening, so as not to fill up to the opening. Care must be taken so that the lead wires should not touch both plug and cone. Therefore, the pour was made very carefully by holding the lead wires taut.

Measuring system. The measuring system of the electrical resistance is as follows. A bakelite bobbin was on the top surface of the cone as in Fig. 2(a) and a rectangular or cylindrical sample with lead wires was placed in the bobbin. A manganin gauge wire, referred to as manganin hereafter, for the pressure determination was loosely wound on the bobbin and is connected in series to the current leads of the sample. The resistance has been measured by a conventional dc four-leads method and the total number of lead wires embedded through the plug hole, therefore, was six. The configuration of the coil system for the measurement of the susceptibility is illustrated in Fig. 2(b). The primary coil, 40 turns, was made by winding the Formvar copper wire, 0.06 mm in diameter, on a bobbin. The bobbin in Fig. 2(a) has the same size. A search coil consists of two coils, 10 turns each and with Formvar wire of the same diameter, connected in series but in opposite sense. They were wound on another bobbin which is capable of inserting firmly into the primary coil bobbin. The manganin was wound noninductively and placed around the primary bobbin.

II. PRESSURE CALIBRATION

Among standard fluid transmitting media, referred to as medium hereafter, spindle oil, isopropanol[6] and a mixture of 1 : 1 n pentane and isoamyl alcohol[7] were adopted in the first place. Spindle oil was adopted, since it is relatively viscous so that the possible leak in the initial stage of pressurization could be restrained.

For the purpose of examining the solidifying process of the medium under pressure, the manganin resistance was measured at room temperature. Figure 3 shows the relative change in resistance $\Delta R/R$ of the manganin at room temperature as a function of load to the piston, where the reference was taken at normal pressure. The manganin wire used was obtained from AKABANE YAKIN Ltd., and of 0.1 mm in diameter. The data presented in the figure are those in up-load stroke and the load was applied using a hydraulic press. In Fig. 3, the circles and squares are the experimental points of isopropanol and a mixture of 1 : 1 n pentane and isoamyl alcohol, respectively, and triangles are of spindle oil. As might be expected, the result for the spindle oil shows the indication of solidification at 13 kbar and the oil was not used in practice. Since the results for different runs

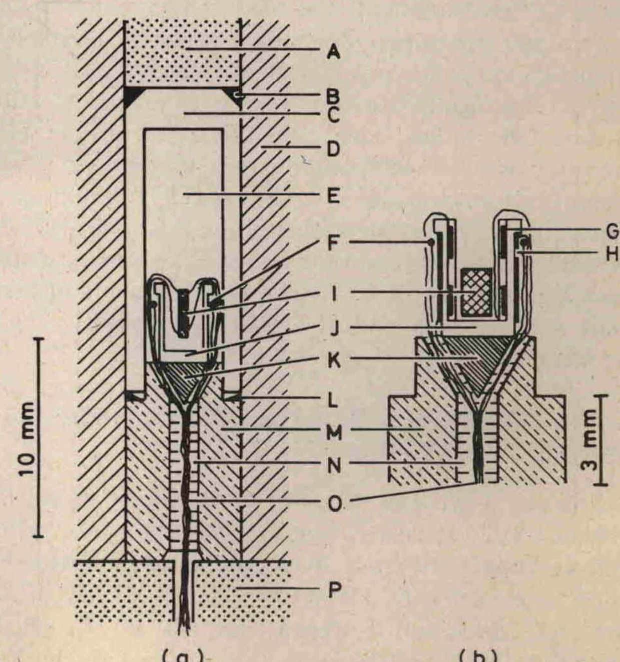

FIG. 2. (a) Cross section of sample cavity for electrical measurement. A: Piston, B: Sealing ring, C: Teflon bucket, D: Cylinder, E: Pressure transmitting liquid, F: Manganin gauge, I: Specimen, J: Coil bobbin, K: Cone, L: Sealing ring, M: Plug shaft, N: Plug hole, O: Lead wires, P: Backing plate. (b) Coil assembly for magnetic measurement. G: Secondary coil, H: Primary coil.