

Fig. 8 Inductance of a coil on a threaded (80 threads/in.) Bi core as a function of ram pressure at room temperature. The inductance was corrected for the leads by subtracting a constant value of 0.6850 μh . A "volume rule" collapse was followed, and the volume change is readily deduced from the data

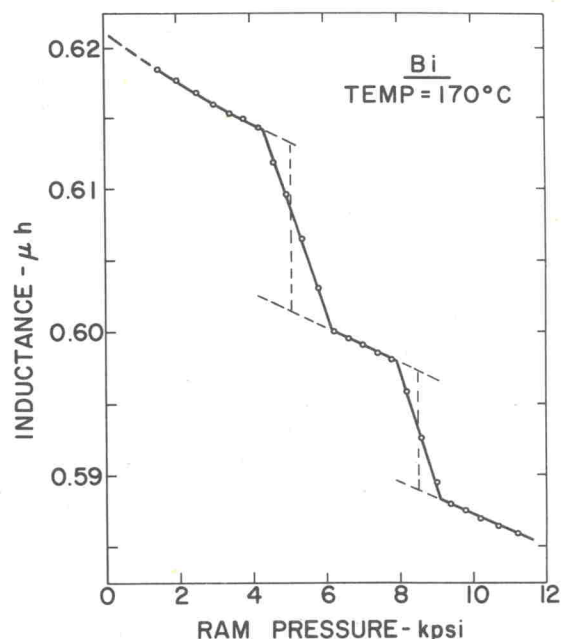


Fig. 9 Inductance of a coil on a threaded (44 threads/in.) Bi core as function of ram pressure at 170 C. Lead inductance of 0.6630 μh was subtracted from the total inductance. A "volume rule" collapse was followed, and the volume change is readily deduced from the data

as Bi, standard cores (0.250 in. dia x 0.250 in. to 0.400 in. long) are recovered with less than 2 percent change in dimensions after being squeezed to 60 kbars. With hard samples such as tungsten there is no measurable change in dimensions after compression. In all room-temperature measurements the coil is enclosed in a sleeve of AgCl.

In the work at elevated temperatures thin-wall (0.015 in.) metal heater tubes (5/8 to 3/4 in. dia) are used. The arrangement is similar to that described previously (1). These tubes are also helpful in reducing sample deformation. Initially, we used heaters made of nonmagnetic "304" stainless steel. We later found out that these tubes become slightly magnetic under pressure. Close examination of this effect revealed that the magnetism is strongest near the ends of the tube where bulging and considerable deformation take place, [see reference (1) for illustrations]. The center of the tube, which is much less strained, barely exhibits the effect. Standard size cores of the same type stainless placed in the center of the pyrophyllite block, where the pressure is uniform and where there is no flow, did not become magnetic. The phenomenon thus appears to be

caused by excessive strains which yield the precipitation of a magnetically ordered phase in the structure. A similar effect is caused by cold-working stainless steel. It has been suggested that in this latter case the magnetic ordering is associated with a dispersion of ferrite particles in the nonmagnetic matrix (11).

We now use tantalum heater tubes for most applications. Temperature is measured by as many as four thermocouples placed at different locations adjacent to the coil (one TC generally touching one end of the core) and monitored simultaneously. With the large size heaters employed, the temperature gradient across the sample is negligible. The thermocouples are introduced through the gasket and ice junctions are used. Small diameter TC wires always break due to the large shear stresses and tension encountered during gasket formation. Therefore, the wires are protected by placing them in ceramic "spaghetti" which slip-fits into a stainless-steel tube.⁴ Pt-Pt13Rh TC wire is used. The pressure effect on the emf of this couple has been investigated (12), and temperature corrections can thus be made. It has

⁴ For some of our purposes we find this more desirable than commercially available sheathed TC wire.

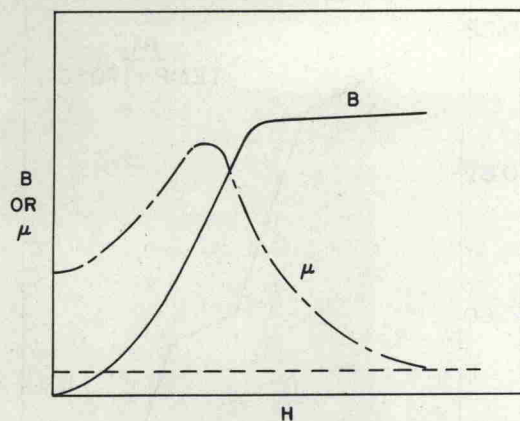


Fig. 10 Typical magnetization curves showing change in magnetic induction and permeability with field strength

been a practice by some to bring TC circuits through the anvils and use the latter as junctions. The anvil faces usually get fairly warm, depending on the sample temperature reached and the length of heating, and TC readings can be seriously off (low).

Typical results on the compressibility of bismuth at 20 C and 170 C are shown in Figs. 8 and 9. The coils were such that a "volume-rule" collapse was followed (10). The data shown were corrected for the inductance of the leads which amounted to subtracting a constant value from the total inductance which was measured at 1 kc. For the 170 C run the coil had much fewer turns than for the 20 C run, hence the lower inductance. Also no AgCl sleeve was used at high temperature, and that is the reason why the transitions are spread over a much wider range than for the 20 C isotherm. The compressibilities in the low and high-pressure phases at both temperatures and the shifts of the transition pressures with temperature are in fair agreement with Bridgman's values (13). Volume discontinuities at the transitions are calculated from the vertical drop in inductance as indicated by the dashed lines in the figures. Use of the terminal points in this calculation would lead to high values as contributions from the compressibilities of both phases would be included. Our value for the total volume decrease across the two transitions is 10 percent which is greater than the 8.6 and 9 percent obtained by Bridgman (13) and LaMori (14), respectively, using the piston-displacement technique.

The resolution of the present technique, particularly in separating the two transitions in Bi, Figs. 8 and 9, is superior to any technique for compressibility measurement presently in use. In over 50 runs, we never failed to see the sharp

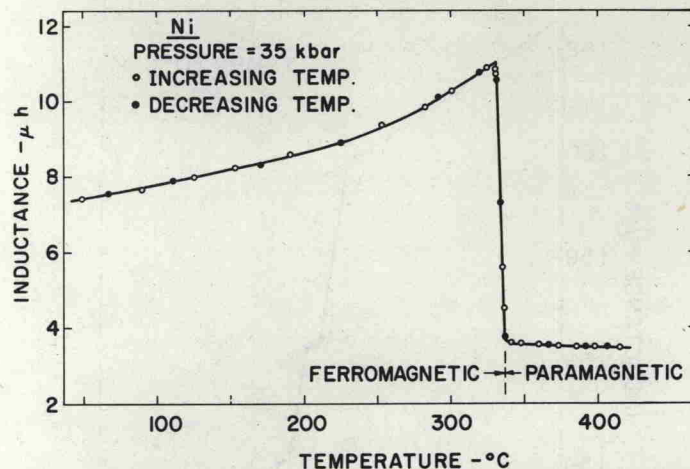


Fig. 11 Determination of the Curie point of Ni from the inductance of a coil enclosing a Ni core. The data were taken at 35 Kbars. No correction for pressure effect on the emf of the Pt-Pt 13 percent Rh thermocouple was made

step separating the Bi transitions. We have not yet established the absolute accuracy of the technique. Our results so far show higher compressibilities than those in the literature. We are looking into possible causes for this discrepancy. In principle, compressibility data to better than ± 1 percent should be obtained when the coil collapses isotropically. Some difficulties may be encountered in studying crystalline samples where large anisotropies in the compressibility exist, for then the relationship $L \sim V^{1/3}$ may not be perfectly valid. We are presently undertaking a study of a large number of materials with varying degrees of compressibilities and anisotropies. By comparing our data with Bridgman's data and recent x-ray results we hope to be able to establish the accuracy of the technique and to what extent anisotropies influence the results.

MAGNETIC MEASUREMENTS

Curie Points

The inductive-coil technique is well suited for studying the compressibility and resistivity of materials whose permeability, μ , is essentially unity and independent of pressure; i.e., diamagnetic and paramagnetic materials. In the case of ferromagnetic materials, volume and resistance data are masked by changes in μ which appear as changes in the impedance of the coil. This suggests the use of the coil for investigating the effects of pressure on Curie points, and, indeed, it turns out to be an excellent technique.

There is no simple and exact formula for the self-inductance of a solenoidal coil of practical dimensions. We generally deal with coils whose