A layered composite material composed of magnetic metal foil and ZrO$_2$ was fabricated by hot-pressing. The interfacial strength between the metal and the ceramic increased with hot-pressing temperature up to 1250°C, and decreased thereafter because of excessive reactions at the interface. The maximum interfacial fracture strength obtained was 420 MPa. Reasonably good magnetic properties, i.e., permeabilities greater than 2000 and ac core losses less than 100 W/kg, were obtained from these specimens. Magnetic core materials fabricated in this process can thus be used under severe conditions such as high temperature and high speed rotation with little dimensional tolerance.

II. Experimental Procedure

The laminated composite was fabricated by hot-pressing the magnetic alloy foil and ZrO$_2$ powder stacked alternately. Commercially available Fe–Co–V alloy (Hiperco 50A, Carpenter Technology Corp., Reading, PA) was used as a magnetic foil and ZrO$_2$ powder was used as an insulating layer. The magnetic alloy had a composition of 48.7% Co, 1.9 V, and 49.4% Fe by weight and a thickness of 0.25 mm. The ZrO$_2$ powder (TZ-3Y, Tosoh Corp., Tokyo, Japan) was in the tetragonal phase and contained 3 mol% Y$_2$O$_3$ as a stabilizer. The average particle size of the powder was 0.3 μm.

To determine the interfacial strength between the ZrO$_2$ and the magnetic foil, flexure bars were made. Billets with a diameter of 3.8 cm and a height of 4 cm were hot-pressed after placing the metal foil perpendicular to the pressing direction at the center of the ZrO$_2$ powder, as schematically shown in Fig. 1(A). Hot-pressings were done at temperatures between 1100°C and 1350°C for 30 min under vacuum. After the billets were

Fig. 1. Schematic diagrams of (A) hot-pressing procedure, (B) specimen for flexural test, and (C) laminated ring specimen for magnetic test.
cut into coupon shape, they were ground with a 220-grit diamond abrasive wheel and subsequently polished with diamond paste down to 1 μm. The polished coupons were cut into 2.5 mm × 3.4 mm × 40 mm bars for flexural testing, as shown in Fig. 1(B). The long edges of the tensile face of each bar were lightly beveled on a 6 μm grit diamond lap. Four-point flexural tests were conducted at room temperature with a crosshead speed of 0.008 cm/s, and inner and outer spans of 6.35 and 19.05 mm, respectively. To observe the effect of magnetic heat treatment, machined flexure bars were heat-treated according to a schedule presented by the supplier of the material in a vacuum (heating to 866°C by 7°C/min, hold at 866°C for 2 h, and cooling to 316°C by 1.8°C/min). The fracture surfaces of the selected samples were examined by scanning electron microscopy (SEM) and X-ray diffraction (XRD).

The magnetic properties of the composite were determined by hot-pressing three layers of alloy foils with ZrO₂ layers between them. The hot-pressed composites were machined into a ring shape with inner and outer diameters of 2.03 and 2.54 cm, respectively, as shown in Fig. 1(C). After the heat treatment, magnetic properties were measured by a commercial vendor (LDJ Electronics, Inc., Troy, MI) with driving and sensing windings of 75 and 50 turns, respectively. A maximum drive field of 20 Oe was employed to saturate the material. Permeability (μ) was determined by the initial curve and the ac core loss was measured at a frequency of 400 Hz with a peak induction of 18 KG.

### III. Results and Discussion

The interfacial fracture strength between the magnetic foil and the ZrO₂ (thickness ~150 μm) was strongly influenced by the hot-pressing temperature. In a 4-point bending test as shown in Fig. 1(B), fracture occurred at the interface of the metal and the ceramic. The strengths of the specimens hot-pressed for 30 min with an applied pressure of 20 MPa at various temperatures are shown in Fig. 2. As shown in Fig. 2, the strength increased with temperature up to 1250°C and decreased thereafter. The increase in strength was mainly due to the increase in the density of the ZrO₂ compact itself with pressing temperature. With an increase in density, the contact area between the metal foil and the ZrO₂ increased, resulting in a stronger bond. The decrease in strength above 1250°C was believed to be due to the excessive reaction between the ZrO₂ and the metal foil. Even though not detectable by XRD analyses, chemical reaction involving partial reduction of the ZrO₂ and oxidation of magnetic alloy was believed to have occurred, resulting in the formation of reaction product, such as iron oxide, at the interface.6 With an increase in the pressing temperature, the reaction product became thicker. Therefore, the strength is determined by the interfacial strength between the reaction product and the metal foil. In the case of Fe, for example, the interfacial strength between the Fe and the iron oxide is relatively low.7 The interfacial strength decreased by about 100 MPa after the magnetic heat treatments, as seen in Fig. 2. These reductions in strength were also due to the formation of thicker reaction product at the interface during the heat treatment.

Scanning electron micrographs support this premise. Figures 3(A), (B), and (C) are fracture surfaces of metal foil, ceramic, and interface, respectively. The grain size of the metal foil was about 20 μm, as shown in Fig. 3(A), while the grain size of the ZrO₂ was about 0.3 μm (Fig. 3(B)), which is about the same as that of the starting powder. The grain size and morphology of the interface phase were quite different from those of the metal and the ceramic, as shown in Fig. 3(C). These micrographs show that a thin layer of reaction product was formed at the interface and the fracture occurred along that interface.

The magnetic properties of the laminated composites were
determined by making the specimen composed of three layers of metal foil and ZrO$_2$ layers between them. The composites were machined into ring shape, Fig. 1(C), and heat-treated under vacuum following the schedule presented in the previous section. The permeability and the ac core loss of the materials depending on the hot-pressing temperature are shown in Fig. 4. Magnetic permeabilities greater than 2000 and ac core losses less than 100 W/kg were obtained from all of the specimens. Interestingly, the magnetic properties were inversely proportional to the interfacial strength of the material; the permeability decreased and the ac loss increased as the interfacial strength increased. Residual stress developed in the magnetic foil is deemed to be related to these variations. The observed magnetic and mechanical properties suggest that this process is suitable for the fabrication of magnetic core materials for applications under severe conditions such as high temperature and high speed rotation with little dimensional tolerance.

IV. Summary and Conclusions

Layered composite materials composed of Fe–Co–V magnetic foil and ZrO$_2$ were fabricated by hot-pressing between 1100° and 1350°C. The interfacial fracture strength between the metal foil and the ceramic was strongly dependent on the hot-pressing temperature. The highest strength of 420 MPa was observed from the specimen pressed at 1250°C for 30 min. Excessive reaction at the interface reduced the interfacial strength. Reasonably good magnetic properties, such as permeabilities greater than 2000 and ac core losses less than 100 W/kg, were obtained from these specimens. This process is applicable to the fabrication of magnetic core materials for use under severe conditions such as high temperature and high speed rotation with little dimensional tolerance.

References