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Dynamic Compaction of Amorphous $Co_{70.3}Fe_{4.7}Si_{15}B_{10}$ Alloy Powders Obtained by a Cavitation Method*

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Synopsis

Amorphous $Co_{70.3}Fe_{4.7}Si_{15}B_{10}$ alloy powders prepared by a cavitation method were dynamically compacted by using a propellant gun. The compacts retained amorphism when they were formed under shock pressures below 8GPa. The highest degree of compaction gave a density of 7.67 Mg/m^3 or 99.6% of that of the amorphous ribbons. After proper heat-treatment, the compact of 0.13mm thick gave a coercive force of 10mOe and a permeability at 100Hz of 12000. Further annealing in rotating magnetic field was found to make the permeability higher. An evident effect of powder size on the soft magnetic properties of compacted powders was also found.

I. Introduction

Amorphous metals obtained by liquid quenching methods have excellent mechanical, chemical, and/or electro-magnetic properties, and their industrial fabrication has already started for several practical uses[1]. It is pointed out, however, that liquid-quenched amorphous metals take only limited shapes; thin ribbon, wire, and powder. This limitation restricts the spread of amorphous metals over a wider industrial field; if bulk amorphous metals are available, they may be useful, for example, as magnetic cores having desired shapes and high strength structural materials, etc..

Techniques so far developed for fabricating amorphous bulk metals are high-rate sputter deposition[2], warm pressing[3], ultrasonic welding[4], plasma-jet and flame-spraying[5], and dynamic compaction[6].

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Among these methods the most promising one for industrial applications may be the dynamic compaction of amorphous metal powders. The dynamic compaction has been made by using explosive[7)8)] or gun method[9)10)]. Mechanical, corrosion resistant and abrasive wear properties of dynamically compacted powders have been reported for $Ni_{40}Fe_{40}B_{20}$[7)], $Ni_{55.8}Mo_{25.7}Cr_{9.7}B_{8.8}$[9)] and $Ni_{40}Fe_{40}P_{14}B_{6}$[10)] amorphous alloys. Negishi et al.[11)] used a propellant gun to consolidate $Ni_{75}Si_{8}B_{17}$ and $Pd_{78}Cu_{6}Si_{16}$ powders and examined the process of dynamic compaction and compaction-induced crystallization.

In spite of these elaborate studies, very little is known about the magnetic properties of dynamically compacted amorphous metals. In the present study, trials were made to compact dynamically amorphous $Co_{70.3}Fe_{4.7}Si_{15}B_{10}$ powders having excellent soft magnetic properties with zero-magnetostriction. This paper states the density, hardness, compressive strength and soft magnetic properties of the compacted powders.

II. Experimental Methods

Amorphous $Co_{70.3}Fe_{4.7}Si_{15}B_{10}$ alloy powders, having a flaky shape and sizes in the range of 149 - 297μm, were prepared by using a cavitation(roller atomization) method developed by the present research group[12)]. The powders were filled by tapping into a steel container of 25mm inner diameter and 20mm depth, and dynamically compacted by the impact of plastic or aluminium flyer launched by a propellant gun[13)].

The intensity of shock pressure loaded onto the powders was controlled by changing the speed of flyer. The initial shock pressure produced by the impact was calculated from the Hugoniot of the flyer and of the powders filled into the container by using the impedance-matching technique. The Hugoniots of the flyers, 2024 Al and polycarbonate, can be found in the literature[14)]. The Hugoniot of the powders was estimated from shock wave data obtained for porous iron[14)] of which density is similar to that of the powders filled into the container.

Disk-shaped compacts having dimensions of 25mm in diameter and 4mm in thickness were removed from the container after dynamic compaction. The view of the compacts removed from the container is shown in Fig. 1(a). The surface of the as-removed compacts had metallic luster. The degree of compaction and the mechanical strength of the compacts were examined by optical microscopy and measurement of density, hardness and compressive strength. The density measurement was made by applying the Archimedian method. Square piller specimens (1mmx1mmx2mm)

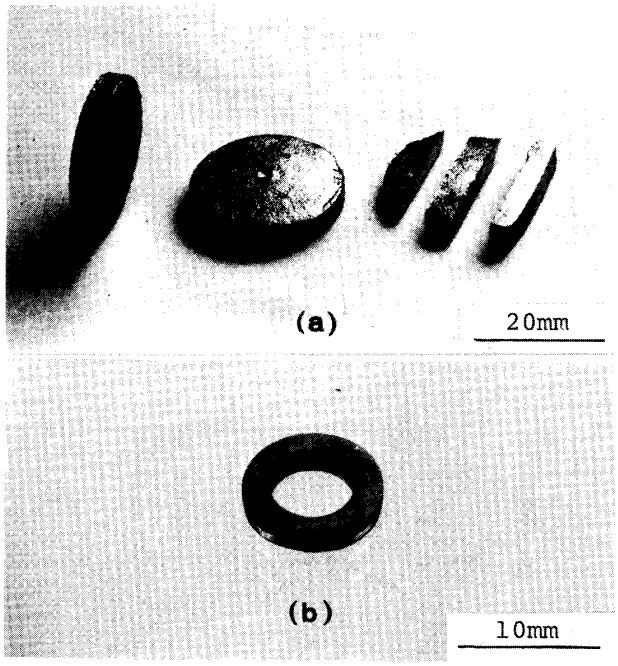

Fig. 1 View of (a) disk-shaped compacts as-removed from the container and (b) a ring-shaped core machined from the disk for measuring magnetic properties. Shock pressure: 5.7GPa.

for the compression test were cut from the compacts by using a diamond wire saw. Crystallization of compacts due to heat generation during dynamic compaction was examined by using both an X-ray diffractometer (CuKα, 35kV, 15mA) and a differential scanning calorimeter(DSC).

To measure magnetic properties, ring-shaped cores with 6mm inner diameter and 10mm outer diameter as shown in Fig. 1(b) were spark-machined from compacts and mechanically polished to remove their heat-affected surface layer. To avoid straining, the core samples were set in plastic cases and then coiled.

III. Results and Discussion

1. Degree of compaction

An optical micrograph of the mechanically polished cross-section of a compact obtained under a shock pressure of 5.7GPa is shown in Fig. 2. Under shock pressures around 5.5GPa, the degree of compaction was quite good. It has been confirmed that compaction at shock pressures below 8GPa keeps the compacts in the amorphous state. However, compacts obtained under shock pressures larger than 6GPa contained many microcracks.

X-ray diffraction patterns taken from the compacted powders

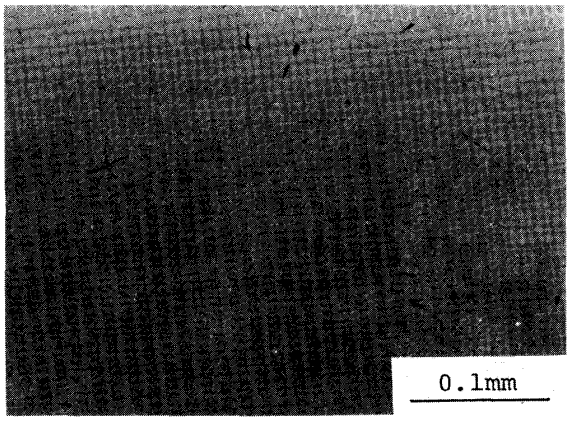

Fig. 2 An optical micrograph of the cross-section of a compact obtained under a shock pressure of 5.7GPa.

exhibited a broad diffraction peak which is characteristic of amorphous metals. In the DSC analysis of the compacts, the transition from amorphous to crystalline state took place in two steps, the first step being at 789K and the second step at 819K. The Curie temperature was 661K. The crystallization energy measured from the peak area was 91kJ/kg. These transition temperatures, the Curie temperature and the crystallization energy are all in reasonable agreement with those of the uncompacted amorphous powder, suggesting that the atomistic structure of the compacts is essentially the same as that of the uncompacted amorphous powder.

Density, ρ, Vickers hardness under a load of 1kg, Hv(1), and compressive strength, σ, of the compacts are plotted in Fig. 3 against shock pressure. The packing density before compaction was 3.10Mg/m^3 or 40% of the density of amorphous ribbon (7.70Mg/m^3). The density of the compacts increased with increase in shock pressure, and the average value reached 7.67Mg/m^3 at shock pressures larger than 5.5 GPa. This density value is 99.6% of the density of amorphous ribbons obtained by a single roller quenching method and is as high as those of dynamically compacted amorphous $Ni_{40}Fe_{40}P_{14}B_6$powders[10].

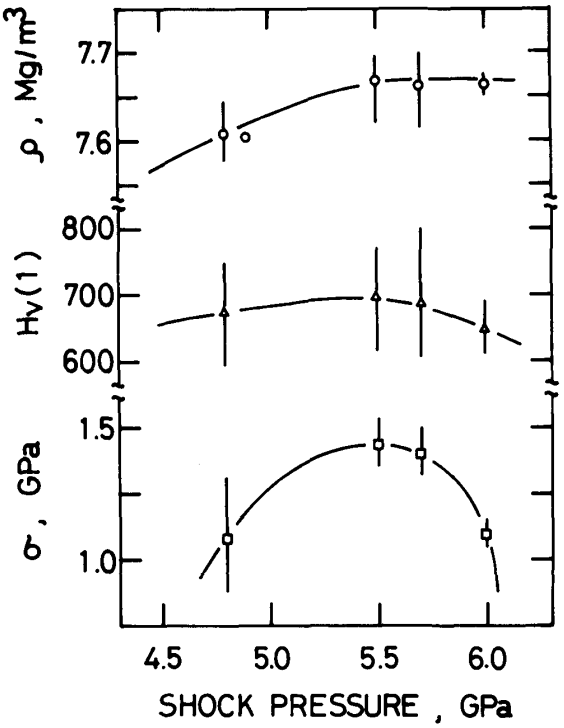

Fig. 3 Change in density, ρ, Vickers hardness, Hv(1), and compressive strength, σ, with shock pressure.

The hardness of the compacts was in a range of 600-800 irrespective of shock pressure. This hardness value is comparable to that of the amorphous ribbons. The compressive strength increased with increase in shock pressure and reached the maximum value of 1.5MPa at a shock pressure of 5.7GPa. This level is, however, only a half of that of the amorphous ribbons, suggesting that poorly bonded interfaces between powder particles still exist in the compacts as seen in Fig. 2. The decrease in compressive strength at shock pressures above 5.7GPa is supposed to be due to the microcracks introduced in the compacts. The formation of the microcracks may be due to the passage of rarefaction waves through

compacted powders. Such deterioration in mechanical properties of compacted powders has been reported also on $Fe_{18}Si_7B_{15}$ alloy[15] and a rapidly solidified AISI9310 steel[16].

2. Magnetic properties

Samples for measuring magnetic properties, i.e., ring-shaped cores, were cut from the compacts obtained under a shock pressure of 5.7GPa which gave the best degree of compaction under the present compacting conditions.

(1) Effect of heat-treatment

To find the heat-treatment conditions which give the best magnetic properties, a sample of 1.73mm thickness was annealed isochronally (10min.). The heating temperature was raised stepwise from 403K to 703K with 100K stepheight. After the anneal at each temperature, the sample was quenched into water. The measurements of magnetic properties were then made at room temperature.

Fig. 4 shows change in coercive force, Hc, and permeability at a driving field of 3mOe, μ(3mOe), with annealing temperature. Soft magnetic properties, i.e., high permeability and low coercive force, deteriorate a little in a range of lower annealing temperatures, but improve markedly above the Curie temperature(661K). It was thus confirmed that the heat-treatment at temperature between the Curie and crystallization temperature improves substantially the soft magnetic properties of dynamically compacted Fe-Co-Si-B powders. Such an improving effect of heat-treatment has been reported for amorphous ribbons having the same chemical composition[17]. A small amount of Hc-increase below the Curie temperature may be attribut-

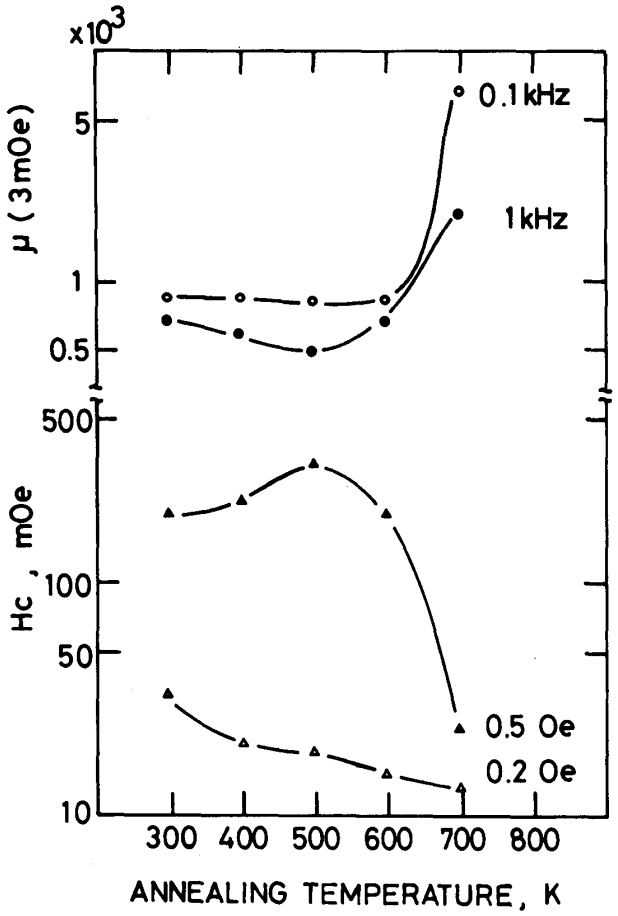

Fig. 4 Change in coercive force, Hc, and permeability, μ(3mOe), with annealing temperature.

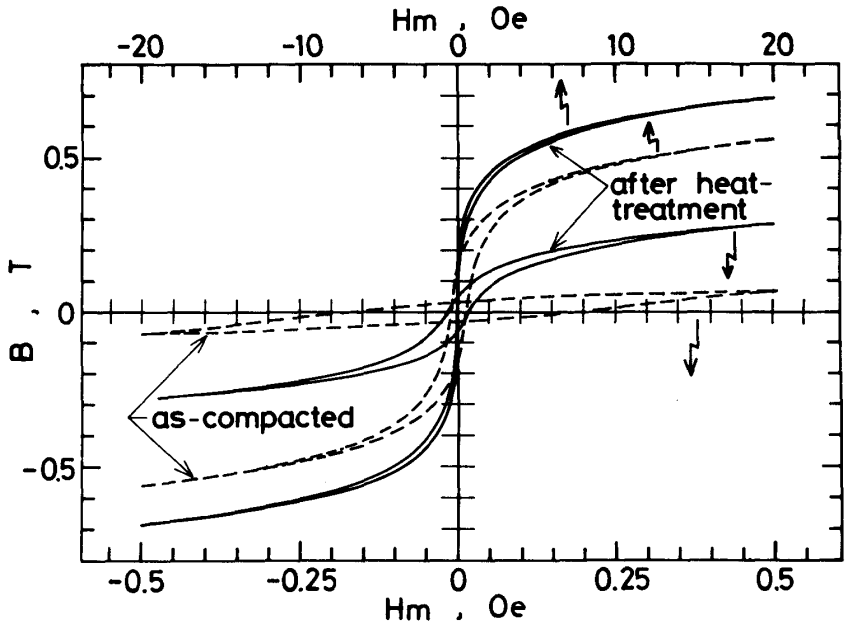

Fig. 5 DC hysteresis loops obtained with a ring-shaped core in the as-compacted state (solid marks) and after heat-treatment (vacant marks). Core thickness: 0.63mm.

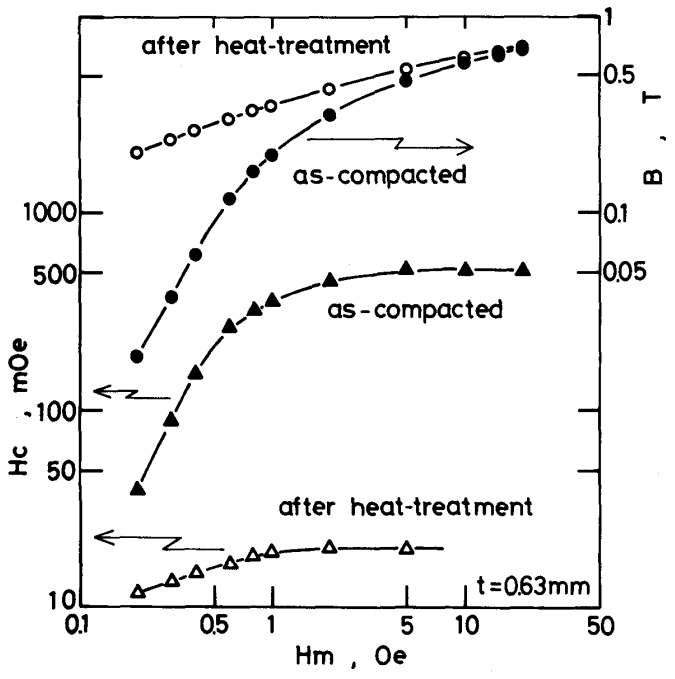

Fig. 6 Plots of coercive force, Hc, and magnetic induction, B, of the same core as in Fig. 5 against the applied maximum field, Hm.

ed to the magnetic anisotropy induced by the heat-treatment[18)19)].

Fig. 5 shows B-H loops of the compact, and Fig. 6 shows plots of coercive force, Hc, and magnetic induction, B, of the same compact against the applied maximum field, Hm. The measurements were made both in an as-compacted state and in a state after heat-treatment (703K x 10min. and water quenching). A large improving effect of the heat-treatment on the soft magnetic properties of the compacted powders

is evident in these figures. However, judging from the shape of the B-H loop, a lack of the saturation in magnetization even at 20e still exists in the heat-treated compact. It has been found with this sample that the permeability increases from a value of the order of 1000 before the heat-treatment to the level of 10000 after the heat-treatment.

(2) Effect of sample thickness

Magnetic properties are affected by thickness of samples. In the present experiment, examination was made with cores having thickness in a range of 0.13-1.73mm and water-quenched after annealing at 703K for 10 minutes. It has been found that a higher field strength is needed for getting the saturated magnetization in a thicker core.

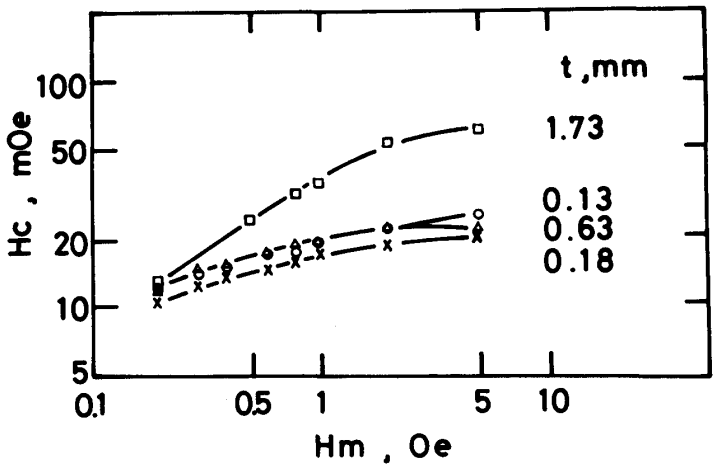

Fig. 7 Plots of coercive force, Hc, against the applied maximum field, Hm, for samples having various thickness.

Fig. 7 shows coercive force, Hc, as a function of the applied maximum field. In the thickness range, 0.13-0.63mm, Hc is almost independent of thickness. Increase in thickness from 0.63 to 1.73 causes large increase in Hc, especially at higher applied maximum field.

The effect of the thickness on the frequency dependency of permeability has so far been examined for thin amorphous ribbons (11-37μm)[20]. Fig. 8 shows the frequency dependency of permeability at a driving field of 3mOe, μ(3mOe). The frequency dependency reported for amorphous ribbons of 25μm thickness having the same chemical composition

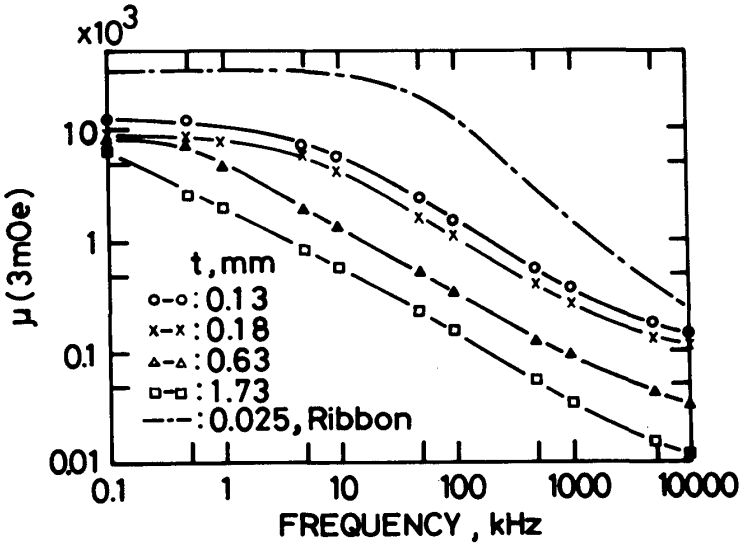

Fig. 8 Plots of permeability at a driving field of 3mOe, μ(3mOe), against frequency for samples having various thickness.

is also shown for the comparison. As seen in the figure, the permeability takes the value of 12000 for a thickness of 0.13mm and decreases with increase in frequency. Thicker cores have lower permeability at any fixed frequency. The thickness effect at around 0.1kHz is mostly due to the fact that the heat-treatment for thicker cores is less effective in removing magnetic anisotropy completely. The thickness effect becomes large in a range of higher frequencies. This is attributed to eddy current loss. Thus, the permeability of compacted powders is less than that of amorphous ribbons because of residual magnetic anisotropy and eddy current loss which is enevitable to thick magnetic cores placed in a high-frequency magnetic field.

(3) Effect of powder size

To examine the effect of powder size on soft magnetic properties, three kind of powders having different sizes, A(44-74μm), B(74-149μm), and C(149-297μm), were prepared. The powders having an initial packing density of 4.3Mg/m^3(powder A and B) or 3.1Mg/m^3(powder C) were dynamically compacted under a shock pressure of 8.2GPa(powder A and B) or 5.7GPa(powder C) and gave compacts having essentially the same hardness and density.

Fig. 9 shows the frequency dependency of permeability measured with the compacts obtained from the powder A, B and C. The measurements were made after conventional heat-treatment(703Kx10min. and water

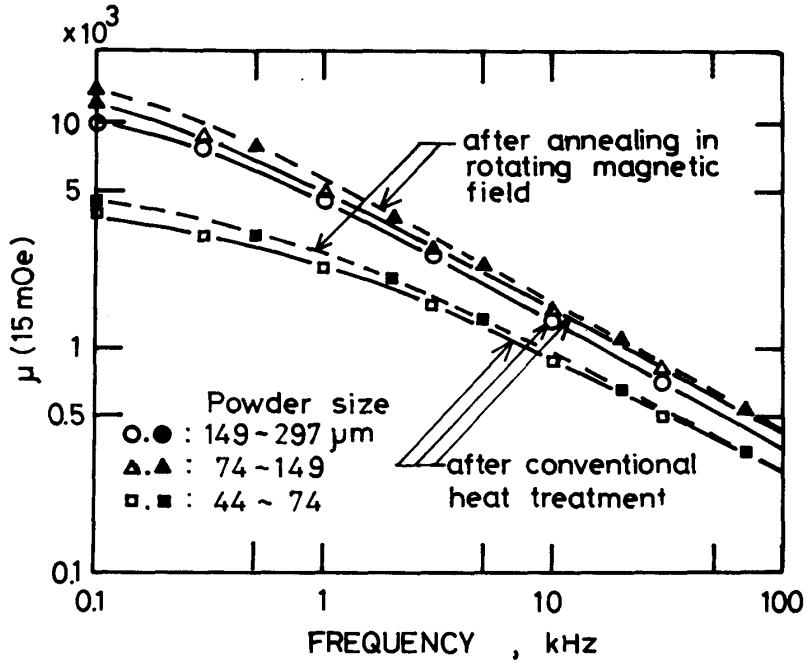

Fig. 9 Effect of powder size on the frequency dependency of permeability of the compacts. Solid and broken lines represent the measurements after conventional heat-treatment and after annealing in rotating magnetic field, respectively.

quenching) or after annealing in rotating magnetic field(703K, 0.8T, 400rpm). Larger size powders, B and C, gave compacts having higher permeability than smaller size powders, A, suggesting that, between compacts made from larger size powders and those from smaller size powders, there exists microstructural difference which may not be detected by density or hardness measurements. The annealing in the rotating magnetic field has a small improving effect on the permeability of the compacts irrespective of powder size.

IV. Conclusions

Amorphous $Co_{70.3}Fe_{4.7}Si_{15}B_{10}$ powders prepared by a cavitation method were dynamically compacted by using a propellant gun. The results are summarized as follows.

(1) The compacts retained amorphism when they were formed under shock pressures below 8GPa. The temperature of two-step crystallization, the Curie temperature and the crystallization energy of the compacts were all in reasonable agreement with those of uncompacted amorphous powder.

(2) The degree of compaction became higher with increase in shock pressure. The highest degree of compaction under a shock pressure of 5.7GPa gave a density of $7.67Mg/m^3$ or 99.6% of the density of the amorphous ribbon.

(3) The soft magnetic properties of the compacts were improved by heat-treatment at temperatures between the Curie and crystallization temperature. The coercive force and the permeability at 100Hz obtained with a core of 0.13mm thickness were 10mOe and 12000, respectively. A small amount of additional improvement was achieved by annealing the compact in rotating magnetic field.

(4) The compacts made from larger size, 74-149μm or 149-297μm, powders gave better soft magnetic properties compared with those from smaller size, 44-74μm, powders.

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