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# Influence of Static Pressure on the Coercive Force of Barium Ferrite Powder

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showed increasing density of a second-phase precipitate as the aging temperature was increased above 250°C. X-ray diffraction analysis confirmed that the precipitate was $\alpha$-Ti. Preliminary electron microscopy indicates that the precipitate particle diameter is about 2000 Å, which is about seven times the size of a flux vortex at 30 kG.

$H_{c2}$ for the material aged at 400°C was determined on a 0.635-mm-diam wire of the same nominal composition (actual: Ti–22.5 at. % Nb), using a pulsed-field technique. Based on the criterion of the first appearance of any measurable resistance, the upper critical field is about 128 kG at 1.2°K. The transition broadened out considerably for low values of current density. At 5 A/cm$^2$ the transition was about 20 kG wide.

## DISCUSSION

The aging of solid-solution Ti–Nb alloys produces an $\alpha$ phase and an enriched $\beta$ phase, before proceeding to the equilibrium $\beta$ and $\alpha$-Ti phases.[11] These various transformations occur quite rapidly, for alloys containing less than about 30 at. % Nb, but tend to be sluggish for the higher Nb alloys.[10] Consequently, it is only with the lower Nb alloys that it is possible to precipitate flux-pinning $\alpha$-Ti particles with reasonable aging times. The results of this study show that, not only is the $\alpha$-Ti that is precipitated an excellent flux pinner (leading to order of magnitude increases in $J_c$), but its precipitation enriches the matrix in Nb, and consequently raises $H_{c2}$ appreciably over the value for $\beta$-quenched, unaged material of the same over-all composition. In the unaged condition, the expected $H_{c2}$ for this alloy is only 110 kG at 1.2°K, while the measured value of the aged alloy was 128 kG.

All of the metallurgical structures produced in this study represent only a point in time during a complex kinetic process which involves not only compositional changes, but particle and grain size changes as well. This preliminary study is now being extended in an effort to arrive at a more quantitative expression between metallurgical structure and superconducting properties.

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# Influence of Static Pressure on the Coercive Force of Barium Ferrite Powder

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In recent studies of ferrite powders, changes of the intrinsic coercive force ($H_{ci}$) have been ascribed to: (1) lattice defects, and (2) particle size reduction which occurs during milling. In order to test the two interpretations, the $H_{ci}$ of barium ferrite powder (ball milled to particle sizes of about 0.02–0.5 $\mu$) was measured under static pressure. The coercive force increased with increasing pressure, reached a maximum at 8000 psi, and then decreased.

To interpret this result on the basis of dislocations would require that the dislocation density be lowered as the pressure is increased, which is unlikely. On the other hand, this behavior is easily understood on the basis of an increase of the effective particle size due to more contact between the particles at higher pressure. According to the latter interpretation, increasing pressure reduces the effect of thermal fluctuations, thus increasing the coercive force. At pressures above 8000 psi, multidomain behavior becomes a predominant factor in lowering the $H_{ci}$. This interpretation is supported by the following experimental facts: below 8000 psi the coercive force decreased when the pressure was released. Above 8000 psi, however, the coercive force increased after the pressure was released.

These experiments show that particle size and exchange interactions between the particles are major factors in determining the coercive force of barium ferrite powders.

## INTRODUCTION

THE effects of ball milling and annealing on the magnetic properties of powders have been studied by several investigators in recent years.[1–10] The interpretations of the observed phenomena differ widely. Torkar and co-workers[2,3] ascribe the changes of magnetic properties in magnetite and barium ferrite powders to the existence of a disturbed surface layer. Van Oosterhout and Klomp[5] interpret their observations on magnetite and zinc ferrite through strains introduced by dislocations. Heimke[6,7,10] considers the introduction of dislocations by milling and the "healing" of dislocations by annealing as the decisive factors for the changes of magnetic properties in barium ferrite powders. Velge and De Vos[8] are also inclined to explain their observations on Mn–Al–Ge powders by structural imperfections. The author[9] is of the opinion that thermal fluctuations and, thus, particle size have a predominant influence on the intrinsic coercive force of barium ferrite powders.

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The present investigation was designed to test whether dislocations or particle size are the dominant influence on the intrinsic coercive force of barium ferrite powder after extended milling.

## SAMPLE PREPARATION

The powder under investigation was prepared by wet mixing 1 part barium carbonate with 4.65 parts of ferric oxide, calcining this mix at 1300°C for about 1 h, and wet milling for 120 h with steel balls about 0.25 in. in diameter. After drying, this material had an $H_{ci}$ of 620 Oe.

## MEASUREMENTS

The coercive force was measured in a brass fixture with a Hall probe. The fixture was constructed so that a Hall probe could slide in a groove under the powder sample which was held in a cylindrical hole by a nonmagnetic punch. The sample was magnetized with about 10 000 Oe. Thereafter it was transferred into the air gap of a small yoke which was powered by a rotatable permanent magnet.[11,12] A demagnetizing force was applied and increased until the reading of the Hall probe did not change when the probe was moved in the uniform field away from under the sample. (The pole pieces were shaped to give a uniform field in an area three times the diameter of the powder sample). Pressure was applied to the powder sample by a motor-driven hydraulic press. The $H_{ci}$ measurements were duplicated within $\pm 3\%$ on the same sample and within $\pm 5\%$ on different samples.

## RESULTS

The results of the experiments are plotted in Fig. 1. It shows that the $H_{ci}$ first increases from 620 Oe to reach a maximum of 890 Oe at about 8000 psi. Then the $H_{ci}$ decreases to 790 Oe while the pressure is increased to 12 000 psi.

The arrows at 5200 and 12 000 psi indicate reversible changes of the $H_{ci}$ which were observed after removal and re-application of the respective pressures. At 5200 psi, the $H_{ci}$ changed between 845 Oe with load and 795 Oe without load. At 12 000 psi, the $H_{ci}$ changed between 790 Oe with load and 850 Oe without load. Attention is drawn to the fact that the direction of the $H_{ci}$ changes is opposite at 5200 and 12 000 psi.

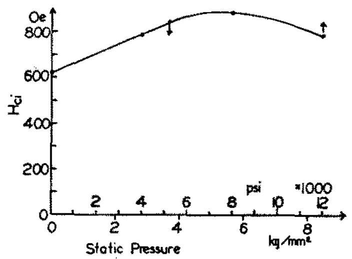


FIG. 1. Effect of static pressure on the coercive force of barium ferrite powder.

## CONCLUSIONS

According to Heimke[6] an increase of the dislocation density lowers the anisotropy constant and thus the intrinsic coercive force. This interpretation does not fit our experimental results because it is unlikely that the dislocation density would decrease with increasing pressure and change reversibly with pressure.

On the other hand, the observed phenomena are easily understood on the basis of an increase of effective particle size due to more contact between particles at higher pressure. The increase of the effective particle size reduces thermal fluctuations, thereby increasing the $H_{ci}$. At pressures above 8000 psi, the contact between particles increases so much that multidomain behavior begins to lower the coercive force. The different sign of the reversible changes of $H_{ci}$ at 5200 and at 12 000 psi strongly supports this interpretation.

This explanation corresponds to the interpretation of Meiklejohn[13] and Kneller and Luborsky[14] on iron, cobalt, and cobalt–iron particles. Their experiments and theoretical considerations show the detrimental influence of superparamagnetic particles on the coercive force of mixtures. These studies also demonstrated that the range of particle sizes for optimum $H_{ci}$ is very narrow.

Ball-milled barium ferrite powders always consist of a wide range of particle sizes reaching from multidomain to nearly superparamagnetic. Electron micrographs[9] of such powders with 120-h ball milling showed particles larger than 0.5 and smaller than 0.02 $\mu$. This explains why coercive forces of ball-milled powders are much below the theoretical values. Annealing of these powders at about 850°–1050°C makes the size range narrower because the small particles are more sinter active than the larger ones. Consequently, annealing raises the coercive force of milled powders more drastically than the application of static pressure.

## ACKNOWLEDGMENT

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