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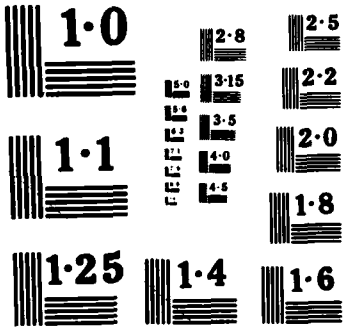

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HOT ISOSTATICALLY PRESSED $Sm_2(TM)_{17}$ MAGNETS

FINAL REPORT
FOR THE PERIOD
14 FEBRUARY 1983 - 13 FEBRUARY 1985

by
K. KUMAR and H. NEWBORN

APRIL 1985

Prepared for the U.S. Army Research Office,
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The Charles Stark Draper Laboratory, Inc.
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High energy product TDK-type, 2-17, Sm (Cu, Fe, Zr, Co) z magnets were produced successfully using hot isostatic pressing (HIP). The best combination of properties was obtained in magnets containing about 26.5 wt%Sm, 4 wt%Cu, 20 wt%Fe, 2 wt%Zr, and 0.4 wt%Co. The maximum value of $(BH)_{max}$ measured was 24.8 MGOe for a magnet with B_R = 10.7 kG and H_{ci} = 15.5 kOe. With higher levels of Cu (OVER)

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19. (up to 8 wt%) much higher H_{ci} values (up to 28.5 kOe) were measured. A minimum amount of Zr was found to be essential to H_{ci} development. HIPing was successfully performed at 1100°C. This resulted in near-complete densification with little grain growth. Post-HIP homogenization was found to lead to enhanced properties after solution treatment and aging. 825°C was determined as the optimum peak aging temperature. A protracted stepped aging treatment resulted in the best properties. Magnets produced from coarse (15 min grind time) alloy powders (with low oxygen contamination) had the properties indicated above. These were substantially superior to magnets produced from conventionally made much finer (1 hour grind time) sinter grade powder, which contained a higher level of oxygen and showed lower H_{ci} even after excess Sm was added to compensate for the higher oxygen. However, H_{ci} values of up to 8 kOe were found to develop in magnets made from fine (1 hour grind time) powders which were processed for lower oxygen contamination in a glove box. An alternate fabrication procedure was investigated. This consisted of fabricating magnets from prehomogenized alloy powders. Initial experiments did not produce a good magnet. Further experimentation is warranted because the approach appears promising.

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Publication of this report does not constitute approval by the U.S. Army of the findings or conclusions contained herein. It is published for the exchange and stimulation of ideas.

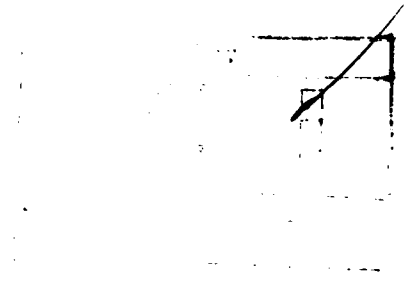

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

CSDL-R-1774

HOT ISOSTATICALLY PRESSED $Sm_2(TM)_{17}$ MAGNETS

1. INTRODUCTION

Ever since researchers from the TDK Electronics Co., Japan, reported the fabrication of a high H_{ci}, 33-MGOe maximum energy product magnet from Sm (Cu, Fe, Zr, Co)$_z$ (z = 6.8 to 7.7) type compositions[1]*, a large amount of effort has been devoted to these materials at several laboratories. These subsequent efforts, however, have not been able to duplicate, let alone exceed, the record energy product value. Consequently, some recent efforts have also been devoted to lower Sm-containing compositions (z ~ 8.35) in hopes of producing still higher energy product magnets.[2] A considerable level of attention is in addition, being given to development of Nd-Fe-B type compositions which seem to have the potential of producing magnets with energy product values of about 50 MGOe.[3] The high, temperature-coefficient values of B_R and H_{ci} of the Nd-Fe-B type materials, however, have precluded their use in several critical applications such as in microwave devices and in inertial instruments. Interest in the TDK-type compositions has, therefore, remained high. Recently, temperature compensated 2-17 TDK-type compositions have also been investigated.[4]

All previously published work on "TDK-type" compositions has dealt with observations on conventionally sintered materials. The conventional procedure has entailed sintering of selected aligned powder compositions at high (1180-1200°C) temperatures.[4] A high-temperature (1150°C) solutionizing treatment is then performed and this is followed by rapid cooling to room temperature. The solutionized sample is aged (for developing high H_{ci}) at a temperature of about 800 to 850°C and this is followed by controlled slow cooling to 400°C at the low rate of

* Superscript numerals refer to similarily numbered items in the List of References.

about 1°C/min. After the desired exposure to 400°C, the sample is cooled to room temperature and magnetized. While this procedure has successfully produced high $(BH)_{max}$ magnets, this has been achieved at some sacrifice to the microstructure. The principal limitation imposed on the material microstructure results from the need to expose the powdered compacts to very high temperatures to achieve densification using sintering. This leads to a considerable amount of normal and secondary grain growth, which by virtue of the Hall-Petch relationship[5] is suspected to reduce the mechanical strength.

Earlier work reported from this laboratory had shown that the hot isostatic pressing (HIP) technology could be successfully used to fabricate high quality $SmCo_5$ permanent magnets.[6] $SmCo_5$ magnets were produced with good grain size control using this process. Work recently reported elsewhere has again confirmed the superior nature of the HIP process over conventional sintering procedures.[7] The HIP process, as developed at this laboratory, has even permitted the fabrication of full-circle radial ring magnets, both bonded metallurgically to selected substrates, and as free-standing structures. Radial ring magnets are desired for a variety of novel designs but cannot be produced using the sintering procedure that is widely employed for fabricating rare earth permanent magnets. This paper now reports upon the successful fabrication of high $(BH)_{max}$ TDK-type $Sm(Cu,Fe,Zr,Co)_z$ magnets with the HIP process.

2. EXPERIMENTAL METHODS

Experiments were performed with alloys procured from three sources. These included two commercial suppliers and the University of Dayton, courtesy of Dr. Alden E. Ray. High $(BH)_{max}$ magnets were fabricated with materials from all three sources. However, differences were observed in the amount of samarium required to generate good properties in magnets fabricated from among the three material sources.

The alloys were crushed, pulverized, ground, and blended to yield powders of desired compositions. The blended powders were placed in rubber boots and aligned in applied dc magnetic fields of 90 and 140 kOe. The loose aligned powders were cold isostatically pressed (CIPed) into green compacts. The compacts were subsequently HIPed to near-maximum density at 1100°C using procedures described elsewhere.[6]

The HIPed materials were heat treated in a purified argon atmosphere within $\pm$5°C of the indicated value of temperature. $4\pi M$ vs. H loops were traced at room temperature with applied dc magnetic fields of up to 140 kOe. The oxygen analyses reported in this paper were obtained using the inert gas fusion technique. Chemical analyses were reported on both alloy ingots and HIPed magnets using x-ray fluorescence. Optical microscopy was performed on polished samples after etching with a 3% nital solution.

3. RESULTS AND DISCUSSION

A large number of compositions were evaluated. Composition variations with respect to Sm, Cu and Zr were studied. The 33-MGOe maximum energy product TDK composition of 25 wt % Sm, 20 wt % Fe, 4 wt % Cu, 2 wt % Zr, and balance Co was used as a baseline. Thermal optimization studies were performed to generate maximum properties.

Nearly complete densification, with very little grain growth, was achieved from HIPing of the green compacts at 1100°C. However, because the magnets did not homogenize adequately at the low 1100°C HIP temperature it was found necessary to expose the HIPed materials to temperatures of about 1180 to 1200°C before good properties could be developed with the optimized solution treatment and aging procedures. The 1200°C exposure resulted in a significant amount of grain growth. (The HIPed magnets produced in this study, however, were, nevertheless, superior to sintered product. By HIPing, near-maximum density was achieved without the limitation imposed by argon gas sintering where a

small amount of argon is expected to be trapped in the magnet when densification to a closed pore structure is attempted in efforts to attain high density).

The homogenized samples were solution treated and this was followed by aging treatments for developing maximum coercivity. For the compositions that were investigated, maximum properties were generated in samples that were solution treated at 1150°C. Samples were quenched from the solutionizing temperature. The quenched samples were initially aged isothermally for different time at a variety of intermediate temperatures in the range of 800 to 875°C and subjected to controlled 1 to 2°C/min cooling to 400°C per the earlier TDK recipe for developing maximum coercivity.[1,4] These studies showed that a long-term (40 hours) exposure to 825°C produced the best properties. Subsequent work showed that additional, substantial improvements could be effected by use of a stepped aging treatment following the 825°C/40 hours isothermal exposure. Of the treatments attempted, the one found most effective consisted of a gradual (1 to 2°C/min) lowering of the temperature of exposure in increments of about 60°C. The samples were held at each of the reduced temperatures for 3 hours until the 400°C temperature was reached where the samples were held for about 5 hours. The samples were then rapidly cooled from 400°C to room temperature. The effect of time of exposure at the 825°C peak isothermal aging temperature on properties using the standard TDK-type procedure and the additional improvements that were obtained with the stepped aging procedures used in this study are shown in Table 1. Also shown in Table 1 are high H_{ci} values that were measured for some samples in which significant homogeneity was achieved with very long term exposure to 1150°C. (These samples were not exposed to the 1200°C temperature prior to the 1150°C treatment.)

The highest value of maximum energy product was measured for a magnet containing 26.5 wt % Sm, 20 wt % Fe, 4 wt % Cu, 2 wt % Zr, and balance Co. This magnet was produced using commercial starting material. It contained about 0.4 wt % oxygen and showed the following

Table 1. Effect of heat treatment on coercivity (in kOe) of selected samples.

Measured Composition Sm/Cu/Fe/Zr/O	HT1 1200°C 4 hr QC*, 1150°C 4 hr QC, 825°C 16 hr, Controlled age**	HT2 HT 1† 1150°C 3 hr QC, 825°C 40 hr, Controlled age	HT 3 HT 2† 1150°C 12 hr QC, 825°C 40 hr, Controlled age	HT 4 HT 3† 1150°C 2 hr QC, 825°C 40 hr, Stepped age †	HT 5 1150°C 24 hr QC, 825°C 40 hr, Controlled age	HT 6 HT 5† 1150°C 62 hr QC, 825°C 40 hr, Controlled age	HT 7 HT 6† 1150°C 6 hr QC, 825°C 40 hr, Stepped age
26.3/4.4/20.2/2.2/0.38	8.0	10.0	11.75	15.5	4.5	6.0 ⧖	9.0 ⧖
26.3/4.4/19.7/2.2/0.44	7.25	9.0	9.5	13.0	7.5	10.0	13.0
28.2/4.4/19.5/2.3/0.38	7.25	8.5	8.0	8.0	-	-	-

* - QC - quick cooled

** - controlled age per TDK recipe

† - stepped as described in the text

⧖ - "constricted" 4πM vs. H loop

properties: B_R = 10.7 kG, H_{ci} = 15.5 kOe, and $(BH)_{max}$ = 24.8 MGOe. These best properties were obtained after homogenizing the as-HIPed material for 4 hours at 1200°C followed by a total of 22 hours solutionizing treatment at 1150°C after which it was quenched. The solutionized sample was step-aged (following a 40-hour soak at 825°C) for developing maximum H_{ci} and improved second quadrant behavior. This same magnet had shown H_{ci} = 11.8 kOe and $(BH)_{max}$ = 23.4 MGOe when it was cooled slowly after the 825°C exposure, per the TDK procedure. Figure 1 reveals the microstructure of this sample after the optimized step-aging treatment developed in this study. The as-HIPed microstructure is included for comparison.

The optimized microstructure in Figure 1A is characterized by very large grains (which resulted from the 1200°C homogenizing exposure) and what appears as a reasonably large scale precipitation within the interiors of the majority of the grains. The aligned nature of the precipitation observed within the grain interiors appeared discontinuous for most samples. In a few samples these appeared as striations [as reported earlier[8] and observed for the sample in Figure 3 of this paper]. The aligned precipitation was interpreted as indicative of a preference for precipitation to occur on certain crystallographic planes. Previous work[8-11] had mostly concentrated on detailed electron microscopic evaluations of the grain interiors and had suggested a correlation between H_{ci} development and the formation of a cellular microstructure with typical cell sizes of about 1000 A. Therefore, it is clear that if H_{ci} increases are related to microscopic differences in composition between the cell boundaries and the cell interiors then the relatively macroscopic (observed) precipitation in Figure 1A must be indicative of "excess" elemental composition of certain species in the starting alloys. The demonstrated need for the "excess" elements (which was inferred from the detailed experimental variations in composition that were performed for optimization purposes) must, additionally mean that either: (1) the macroscopic (observed) precipitation must also contribute to development of H_{ci}, and is, therefore, not "excess" but is required, or (2) that the alloy is

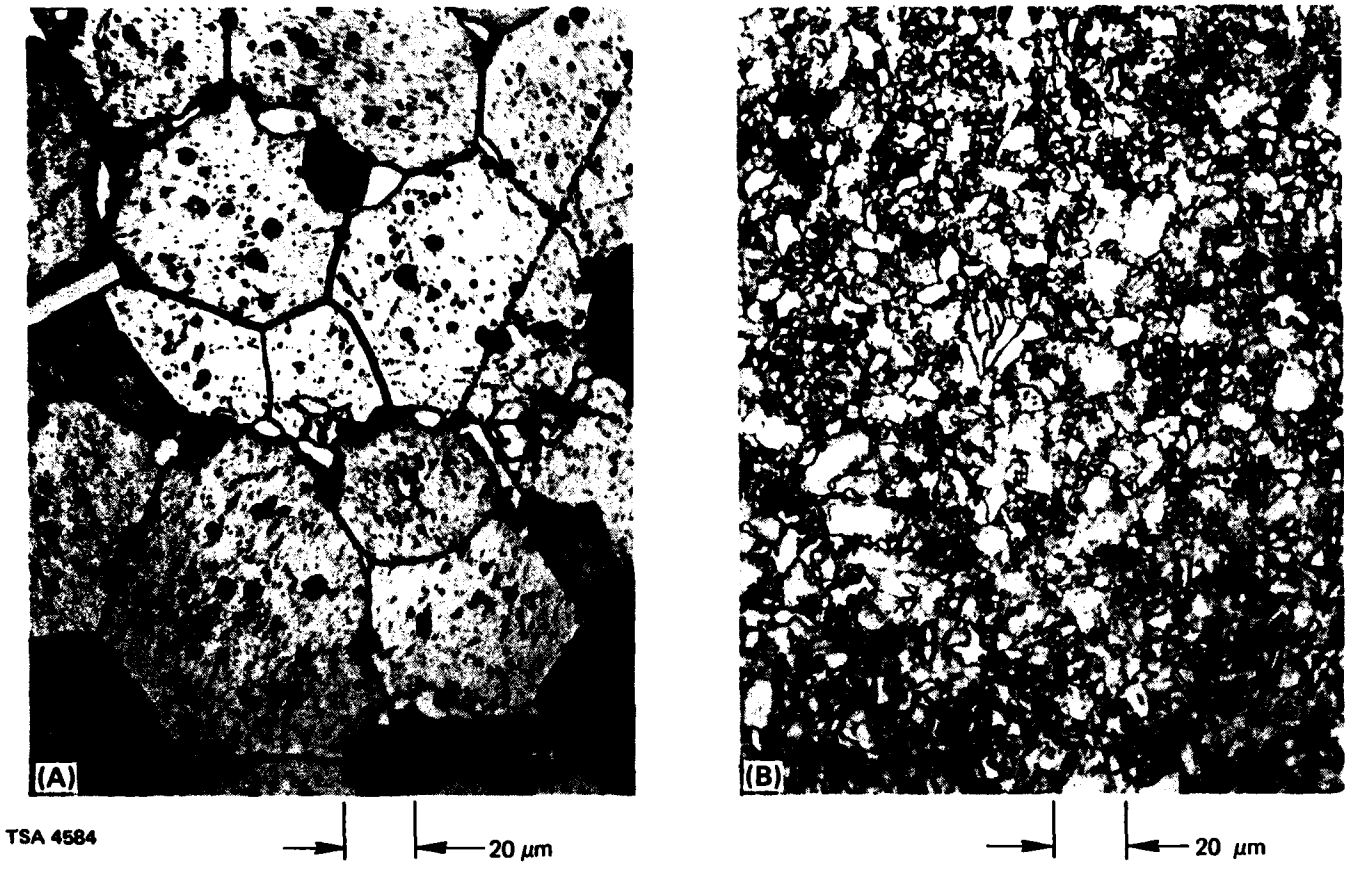

Figure 1. Micrographs obtained on the 24.8 MGOe sample. (A) After optimized heat treatment; (B) As-HIPed. (A) shows a fine (aligned) discontinuous precipitation and large darkly etched regions within the grain interiors suggesting chemical inhomogeneity. A few lightly etched areas were also observed located at the grain boundaries. 3% Nital etch.

substantially inhomogeneous both at the relatively macro- as well as the more microscopic level.

For magnets fabricated from powders processed using conventional procedures, which included handling of the powders in air, it was found (during early experimentation) that meaningful values of H_{ci} were developed only in magnets produced from relatively coarse powders. The powder particle size was controlled through selection of the grinding time in a laboratory-sized attritor ball mill. The coarse powders that produced good properties were obtained using a 15-minute grind time. When the powders were ground for about 1 hour, considerably finer sizes were obtained. Figure 2 shows that much finer particle sizes were associated with powders that were ground for 1 hour compared to those that were ground for much shorter time [10 minutes in Figure 2(A)]. Typically, the 15-minute powder contained about 0.30 to 0.45 wt % oxygen and the 1-hour powder over 0.6 wt % oxygen. Even when additional Sm was added to the 1-hour powders to compensate for the increased level of oxygen, the H_{ci} value continued to be lower than that obtained from the coarse powders (see Table 2). This suggested that the effects of oxygen were more subtle than could be offset by the required (calculated) adjustment of the composition.

Table 2. Effect of initial particle size on coercivity (for conventionally prepared powder).

NOMINAL COMP. (wt %) Sm/Cu/Fe/Zr/O	BALL MILL TIME (hours)	MAX H_{ci} GENERATED (kOe)
26/3.9/19.4/1.9/0.373	0.25	17
26/3.9/19.4/1.9/>0.6*	1.0	0
28/4/20/2.0/>0.6*	1.0	4

*Typical measurements indicate expected values >0.6 wt % O.

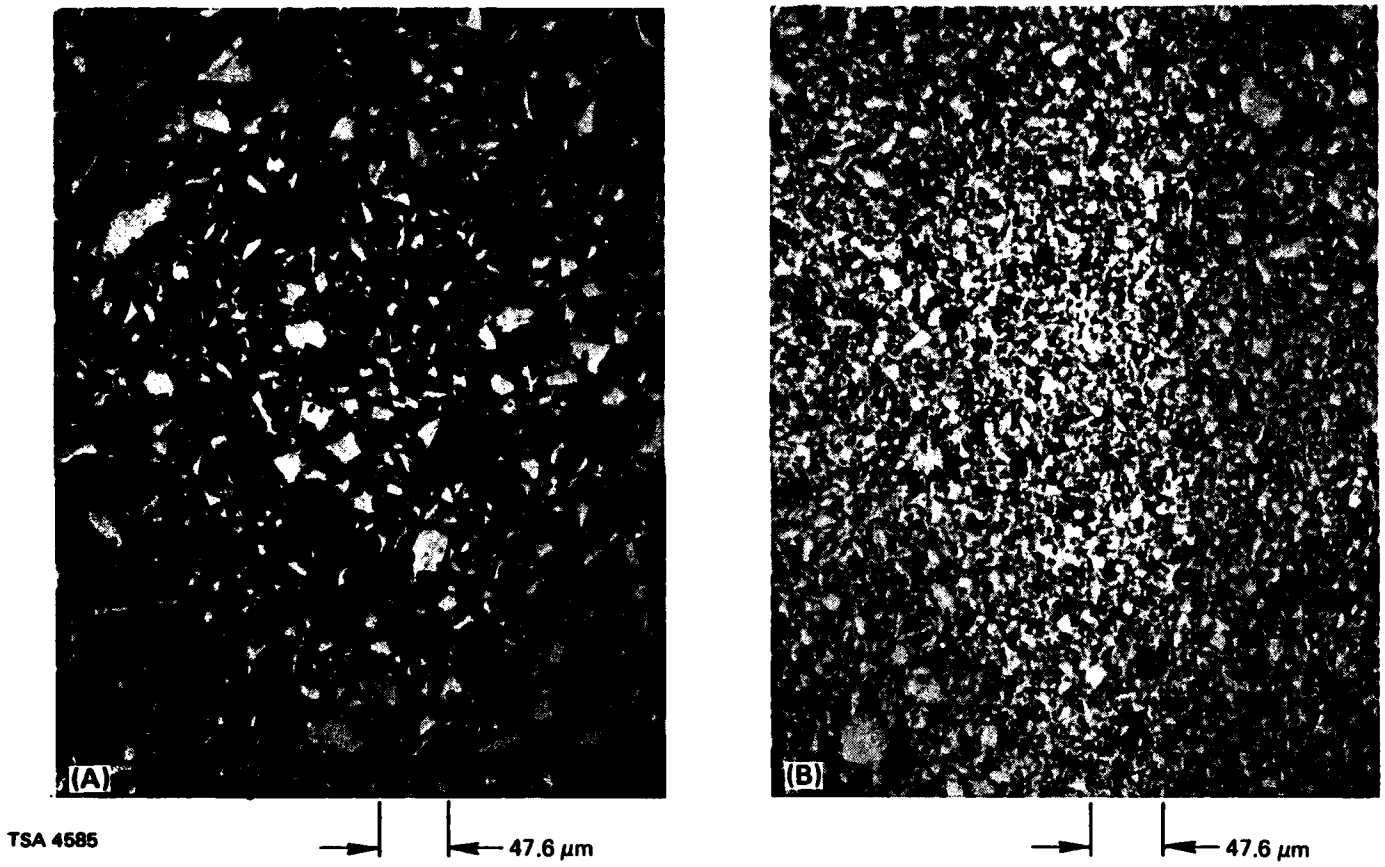

Figure 2. Effect of ball mill time on powder particle size. (A) 10 minutes; (B) 60 minutes.

Powders were (additionally) prepared for reduced oxygen pick-up inside an argon glove-box. Alloy compositions procured from one specific source were used for these studies. Since these powders were removed during processing a few times from the glove-box, prior to HIP densification, some oxygen pick-up was observed. However, analysis revealed that magnets produced from powders ground in the glove-box for 40 minutes contained a level of oxygen which was similar to that of magnets produced from powders which were processed for 15 minutes in air. The best magnet produced with powders prepared in the glove box showed: B_R = 10.2 kG, H_{ci} = 8.5 kOe, and $(BH)_{max}$ = 18.3 MGOe. The microstructure of this sample, shown in Figure 3, revealed a finer grain structure than previously observed for samples produced from conventionally prepared coarse powders (see Figure 1). The nominal intended composition of this magnet was 27 wt % Sm, 20 wt % Fe, 4 wt % Cu, 3 wt % Zr, and balance Co. The 27 wt % Sm optimum observed for magnets produced from glove-box processed powders was consistent with observations made on magnets produced from conventionally processed coarse powders of these alloys. Those studies showed that peak magnetic properties (using these alloys) were obtained at the same 27 wt % Sm composition, unlike the case for magnets produced from alloys procured from the other two sources that showed 26.5 wt % Sm to be the optimum. This magnet was homogenized at 1180°C for 4 hours prior to the solutionizing and stepped aging treatments. Use of the lower 1180°C homogenizing temperature (compared to the 1200°C temperature used previously with the coarse conventionally processed powders) proved sufficient to chemically homogenize the material for developing an acceptable value of H_{ci}. The increased homogenization obtained at the lower (1180°C) temperature for the glove-box sample (over that typically observed for magnets produced from coarse powders) was attributed to decreased diffusion distances associated with the use of the much finer 40 min grind time powder. These experiments suggested that high H_{ci} could be generated in fine grained TDK-type magnets if the magnet material could be sufficiently homogenized without having to resort to procedures that also lead to large scale grain growth.

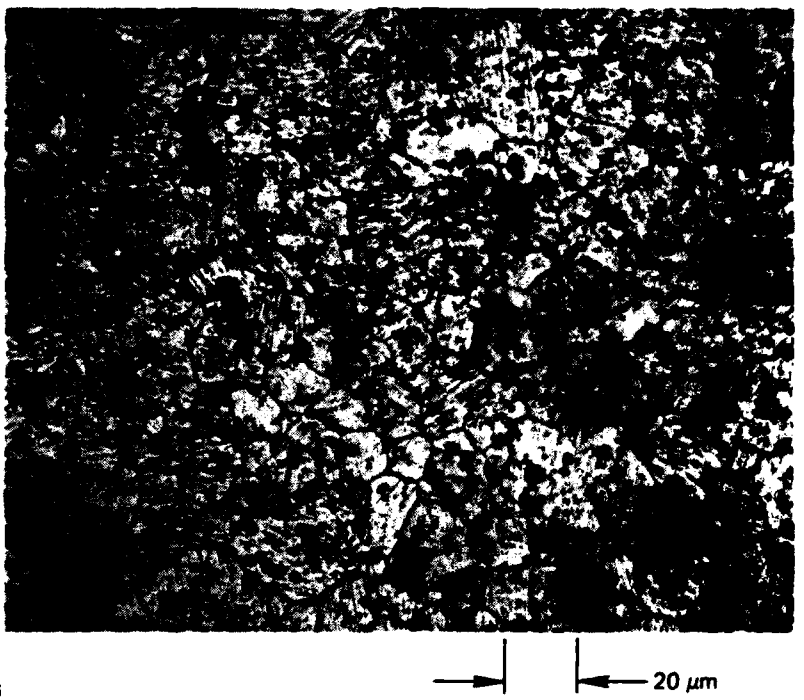

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Figure 3. 18.3 MGOe magnet produced from glove-box processed powders. 3% Nital etch.

The foregoing showed that the microstructure based objective for these magnets is the fabrication of fine grained as-HIPed materials which are sufficiently homogenized, after consolidation, such that the maximum post-HIP temperature of exposure is limited to 1150°C (which is required for the solutionizing treatment). Experiments performed in this study showed very little grain growth in the TDK-type compositions (over the as-HIPed 1100°C microstructure) from exposure to 1150°C. To achieve the desired microstructure, an alternate fabrication approach was investigated. This consisted of procuring the alloy of the desired composition and subjecting it to an extensive (4 hours) homogenization treatment at a high (1180-1200°C) temperature prior to reducing the alloy ingots into fine powder particle form. The composition selected for this initial experiment nominally consisted of 26.5 wt % Sm. 20 wt % Fe, 4 wt % Cu, and 2 wt % Zr. Powders produced from the homogenized alloy with a 15-minute grind time were, as before, HIPed at 1100°C. The densified magnet was then directly exposed to 1150°C for 12 hours for solution treatment after which it was quenched and aged using the extended 825°C stepped aging procedure described above. The usual pre-solution-treatment homogenizing step was thus avoided. Initial measurements made on magnets produced this way were disappointing. It is possible that the poor second quadrant behavior measured for this magnet was related to an incorrect bulk composition of the procured alloy. It is also possible that the homogenization required for the alloy ingots needed much longer time at the elevated temperature because of the greater diffusion distances. Additional experiments will be performed to study these possibilities in a follow-on phase of this activity.

Variations with respect to Zr and Cu were also investigated under this study. Work performed with glove box processed powders suggested a dependence of H_{ci} development on the level of Zr that is present. The 18.3 MGOe sample discussed earlier was one of three prepared in identical fashion, nominally containing 2.0, 2.5, and 3.0 wt % Zr. The measured H_{ci} values for these three samples were 0,5.5, and 8.5 kOe respectively. These results confirmed earlier findings that showed a

need for a minimum level of Zr in the magnet for generating an acceptable level of H_{ci}.[11] More quantitative conclusions, however, could not be reached in this study because of uncertainties in chemical analysis results, pertaining to Zr.

Large increases in H_{ci} were observed with additions of Cu (as a partial replacement for cobalt) to the optimized composition. However, decreases in B_R and $4\pi M$ were also observed, partly accounting for lowered values of $(BH)_{max}$. Of greater significance to $(BH)_{max}$, with increasing amount of Cu, was a rapid initial decay of $4\pi M$ with increasing reverse field in the second quadrant. The $4\pi M$ value was then found to stabilize at a lowered value. Thereafter elements of a reasonably square hysteresis loop were observed. The hysteresis loop obtained on a sample containing 26.5 wt % Sm, 20 wt % Fe, 8 wt % Cu, 2 wt % Zr, balance Co, that was homogenized at 1180°C for 4 hours and solutionized at 1150°C for 12 hours followed by the stepped aging treatment is shown in Figure 4. The hysteresis loop is characteristic of what would be expected from a sample composed of two phases; the minor constituent (~ 20 vol %) consisting of low H_{ci} material and a substantially larger constituent (~ 80 vol %) consisting of high H_{ci} material. The value of 28.5 kOe shown for H_{ci} on this sample was the largest measured in this effort. The microstructure of this sample in the optimum aged condition showed a considerable amount of a second phase in the grain boundary regions (see Figure 5). This grain boundary phase possibly accounted for the lower H_{ci} material that was indicated in the demagnetization curves and may have also partly contributed to the lowered B_R (9.8 kG) measured for this sample compared to the >10.5-kG values measured previously for compositions that had contained the lower (4 wt %) amount of Cu. The roughly 20 vol % calculated for the lower coercivity material from the $4\pi M$ vs. H plots and the ~ 20 vol % observed for the boundary phase using optical microscopy appeared to correlate and support this explanation. No such dominant grain boundary phase was observed in either the earlier 4 wt % Cu samples or even in samples containing 6 wt % Cu. The pronounced knee in the second quadrant (indicative of a two-phase material) was also absent in the

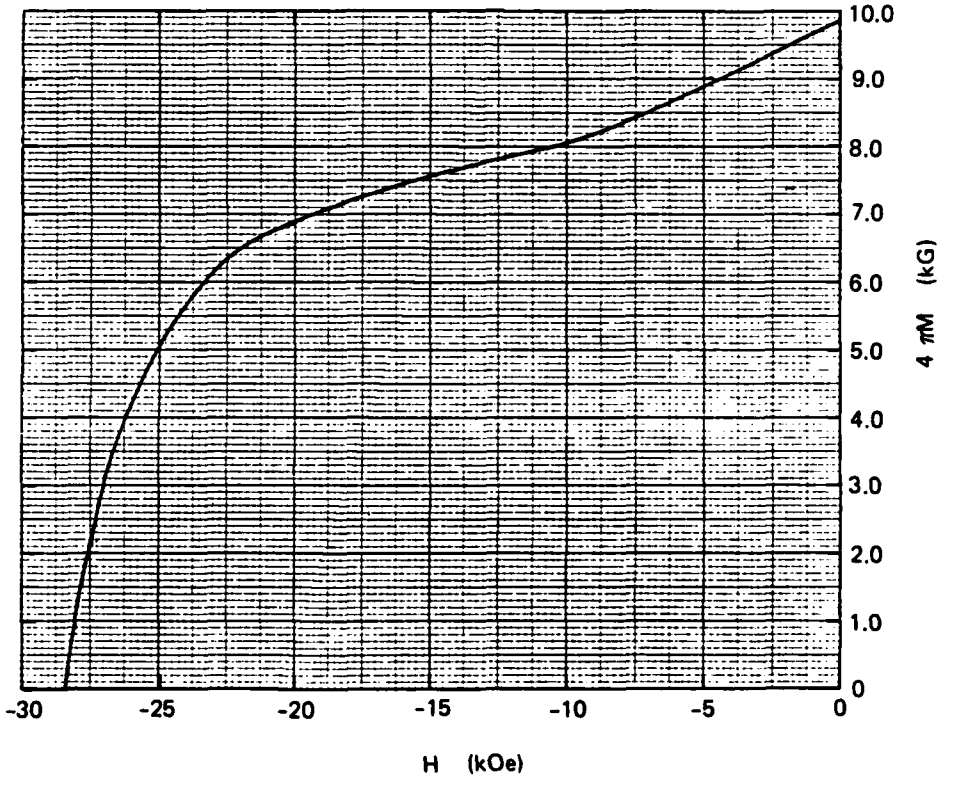

Figure 4. Second quadrant behavior observed for the 8 wt % Cu sample.

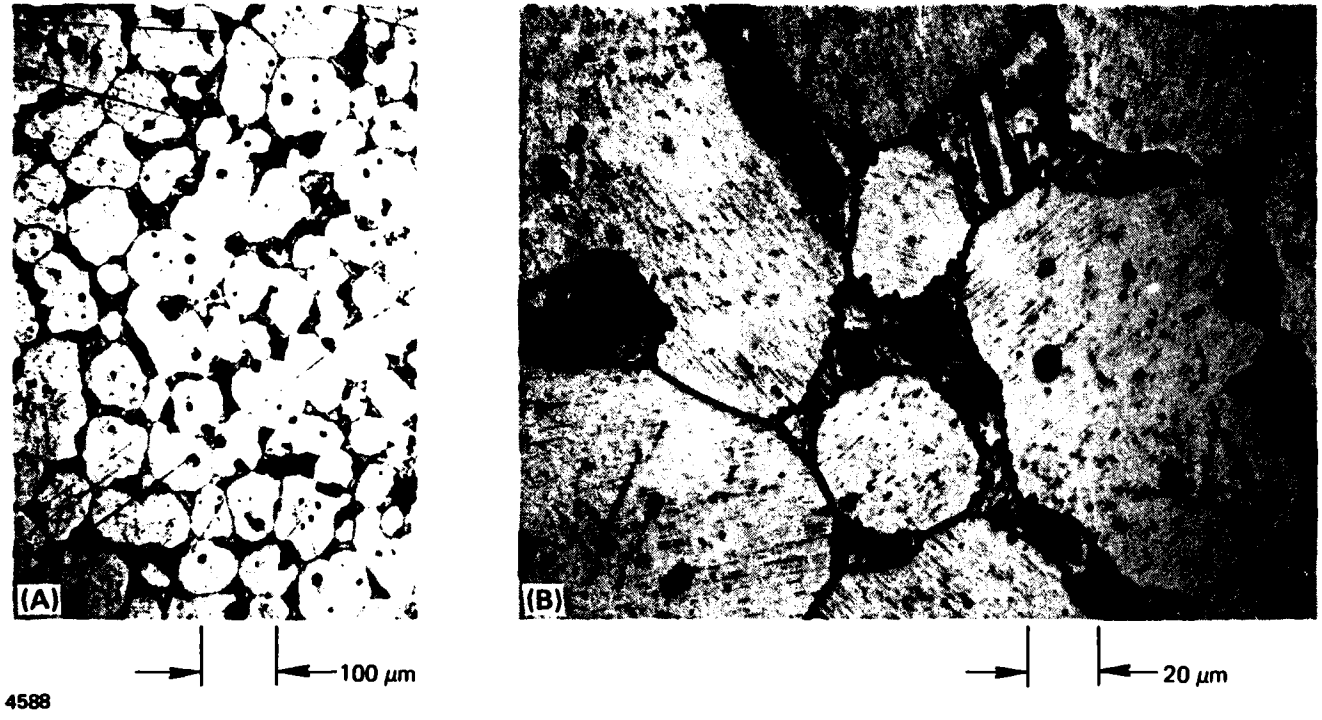

Figure 5. Micrographs obtained on the 8 wt % Cu sample showing a large amount of second phase at the grain boundary. Different magnifications.

4 wt % Cu sample and was only minimally observed for the 6 wt % Cu sample. It appears possible that 6 wt % Cu is near the upper bound on Cu content desired in these magnets. The 6 wt % Cu sample showed a B_R value of 10.0 kG.

4. SUMMARY AND CONCLUSIONS

(1) HIP has been demonstrated as a feasible process for producing high $(BH)_{max}$, high H_{ci} TDK-type 2-17 permanent magnets. These magnets are expected to be more structurally sound than those produced with the conventional sintering procedure.

(2) These 2-17 compositions can be HIPed to nearly maximum density at 1100°C without appreciable grain growth. Additional higher temperature homogenizing and solutionizing treatments, followed by lower temperature aging, are required for developing H_{ci} in magnets produced from conventionally processed alloys and powders.

(3) The best isothermal aging temperature was determined to be 825°C. The optimized aging treatment consisted of a 40-hour exposure to 825°C following which the sample was step-aged in steps of 60°C to 400°C. The sample was held at each intermediate temperature for 3 hours and exposed for 5 hours to 400°C.

(4) Contamination from oxygen appeared important in influencing H_{ci} generation in these magnets. For powders processed in conventional manner, coarse powders (with lowered oxygen contamination) were deemed more desirable than finer powders, which are typical of materials required for sintering. HIP permitted the fabrication of dense magnets from the coarse powders as well, thereby minimizing oxygen contamination.

(5) Work performed with glove box processed fine powders (that contained oxygen levels similar to the conventionally produced coarse powders) showed that it is possible to produce high H_{ci} magnets (8.5 kOe was achieved) which also possess a finer (than presently obtained) grain structure.

(6) Optical microscopic observations showed the existence of substantial precipitation within the interiors of most of the grains in the optimized step-aged condition. This was interpreted to mean that either: (1) this precipitation contributed to H_{ci} development, in addition to contributions arising from previously reported cellular structures within the grain interiors, or (2) was indicative of substantial microscopic and macroscopic inhomogenity associated with a required "excess" elemental composition.

(7) This work confirmed the need for a minimum amount of Zr to produce an acceptable H_{ci}. Large increases in H_{ci} were observed from Cu additions up to 8 wt % Cu. However, a large amount of a boundary phase was observed in the 8 wt % Cu sample which also contained a pronounced knee in the second quadrant of the demagnetization curves. The rapid reduction in magnetization at low reverse fields (giving rise to the observed knee) appeared to correlate well with the amount of the boundary phase. This knee and the boundary phase were substantially minimized in a 6 wt % Cu containing sample indicating a possible (desired) upper bound for Cu.

(8) An alternative fabrication procedure for producing fine-grained TDK-type 2-17 magnets with good properties was investigated. This consisted of HIPing powders of pre-homogenized alloy ingots. Initial attempts were unsuccessful. This was attributed to possible discrepancies in composition. The approach appears worthy of additional experimentation.

5. RECOMMENDATIONS FOR FUTURE WORK

(1) Continue to investigate fabrication of fine-grained TDK-type magnets using pre-homogenized alloy powder.

(2) Perform feasibility studies to HIP fabricate internally temperature compensated TDK-type 2-17 magnets.

(3) Investigate the HIP fabrication feasibility of higher z (lower Sm) containing compositions.

(4) Perform flux stability evaluations on uncompensated and temperature compensated TDK-type 2-17 HIP magnets.

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