

APPLICATION OF HIGH PERFORMANCE MATERIAL PROCESSING - ELECTROMAGNETIC PRODUCTS

Francis G. Hanejko, George W. Ellis, Timothy J. Hale

Hoeganaes Corporation
Cinnaminson, NJ 08077

Presented at PM2TEC'98
International Conference on Powder Metallurgy & Particulate Materials
May 31-June 4, 1998 - Las Vegas, NV USA

Abstract

The use of powder metallurgy (P/M) parts in AC applications is limited by the inability to reduce the total core loss of the P/M components to levels achieved with conventional laminations. The use of iron powder polymer composites has proven successful in high frequency applications such as ignition coils and high-speed motors. Broader application of P/M in AC applications will require a material that will provide superior magnetic performance at low frequencies. This paper will discuss the use of high performance materials for DC applications (in particular, alloys of iron and silicon) as well as the use of insulated powders for both high and Low frequency applications.

Introduction

P/M magnetic materials continue to find an increasing variety of applications ranging from tone wheels in anti-lock braking systems to high frequency ignition coils (Ref. 1). The choice of material system and processing conditions is determined by the requirements of the applications and the compacting and sintering equipment available (if sintering is required): For example, P/M iron 3 weight percent (w/o) silicon alloys are produced by premixing high purity iron with a silicon-iron master alloy. However, these alloys require high temperature sintering to homogenize the silicon addition. When properly processed, these alloys achieve high permeability, Low coercive forces, and high material resistivity. The high permeability of the iron silicon alloys can be achieved with alloys of iron and phosphorus; however, these iron phosphorus alloys lack the Low coercivity and high material resistivity of the iron silicon alloys (Ref. 2).

Recently, a new type of P/M magnetic material was introduced. These new materials referred to as insulated iron particles are a composite of iron powder and a high strength polymer. Magnetic properties of the iron powder polymer composites (IPPC) are influenced by the amount and type of polymer and the particle size distribution of the iron powder (Ref. 3). Iron powder polymer composites are used in the as compacted condition and exhibit Low eddy current losses. Applications for these materials are AC magnetic devices that require the minimization of eddy current losses. One drawback of the iron powder polymer composites is the high coercive force of the as pressed component. This high coercive force increases the hysteresis losses dramatically, resulting in reduced magnetic performance at Low frequencies (less than 200 Hz). Consequently, these materials have found usage in higher frequency applications but are not well suited for the Low frequency, high volume applications at 60 Hz.

The intention of this paper is to present the magnetic characteristics of the high performance iron silicon sintered magnetic materials. The DC magnetic performance of these alloys will be presented along with limited AC testing on the sintered alloys to demonstrate the reduced magnetic performance under AC conditions. A new class of insulated irons will be discussed; these new materials have Low coercive force and Low total core loss at operating frequencies of 60 Hz. These new materials utilize a new type of insulating material that allows for a Low temperature annealing of the as compacted component, thus eliminating the deleterious effects of the cold working of the iron powder during compaction.

Sintered Properties of Iron Silicon P/M Magnetic Alloys

Iron and iron phosphorus alloys are the most commonly used P/M magnetic material; their advantages are Low material cost, ease of fabrication, and high saturation induction (Ref. 4). Inherent limitations of these materials are Low material resistivity and relatively high coercive force. In wrought steel, additions of silicon increase the material resistivity and lower the coercive force, both effects resulting in improved magnetic performance. In ferrous P/M alloys, the use of silicon as a prealloy addition is not practical because silicon dramatically decreases the compressibility of iron powders. Silicon containing P/M magnetic materials are produced via additions of a Ferro-silicon master alloy to a high purity iron powder. Following compaction, the iron silicon compact requires sintering at 2300°F (1260°C) to homogenize the silicon addition. Although requiring elevated temperature sintering, the use of master alloys gives the P/M parts fabricators the flexibility to customize the alloy addition to meet the component's specific magnetic requirements.

Table I and Table 2 summarize the effect of silicon additions of 1 w/o, 2 w/o and 3 w/o on the magnetic properties of P/M compacts compacted at 35 tsi and 50 tsi (480 MPa and 685 MPa). Sintering was done in a 100% hydrogen atmosphere at 2300°F (1260°C) or 2400°F (1315°C); delubrication was accomplished in a separate furnace at 1550°F (845°C) in a wet hydrogen atmosphere. Increasing the silicon addition from 1 w/o to 3 w/o raised the permeability from 3100 to 5600 at the 2300°F (1260°C) sintering temperature with a corresponding decrease in the coercive force from 1.6 Oersteds to 1.0 Oersteds (all testing done at 25°C). Sintering at 2400°F (1315°C) increased the permeability from 3500 to 5950 with a corresponding decrease in the coercive force from 1.5 Oersteds to 0.9 Oersteds.

The addition of Ferro-phosphorus to the iron silicon system is reported to activate the sintering thus providing higher densities and improved magnetic performance (Ref. 5). To investigate this potential, a premix was prepared containing high purity iron with the addition of 3 w/o silicon and 0.45 w/o phosphorus. Unlike the previous experimental work (Ref. 5), no substantial increase in sintered density was observed; however, the combination of the silicon and phosphorus improved the permeability approximately 10% while lowering the coercive force approximately 5% relative to the iron - 3 w/o silicon alloy. The iron silicon phosphorus system has the benefit of increased resistivity (up to 80 μΩ cm at a 4 w/o silicon and 0.45 w/o phosphorus addition) with corresponding increased magnetic performance. These materials give the P/M process added flexibility in meeting the diverse magnetic applications imposed upon P/M parts.

The abundance of DC magnetic data for P/M materials can be utilized to optimize the material system and processing to meet the specific magnetic requirements of most applications. Recently, requests have been received to investigate sintered materials in Low frequency AC (less than 60 Hz) applications. The use of sintered P/M materials is not recommended for AC applications because of the excessive core losses inherent in the relatively thick P/M sintered components.

The total core loss of a component under AC magnetic Conditions can be expressed mathematically as follows:

$$\text{Total Core Loss} = a * LA * f + b * (B^2 * f^2 * t^2) / \rho \quad \text{Equation 1}$$

in which:

a = Hysteresis loss constant

LA = Loop Area of the hysteresis curve

f = frequency

B = Induction in Tesla

t = thickness of the component or lamination in mm

b = Eddy current loss constant

ρ = material resistivity

The expression a*LA*f relates to the hysteresis loss portion of the total core loss and is a function of the induction and frequency to the first power. The expression b*B²*f²*t² gives the losses associated with the eddy currents and is proportional to the frequency, thickness, and magnetic induction to the second power (Ref. 6). Thus, the greatest contribution to the total losses is from the eddy current losses; minimizing these will lower the overall losses of the component at AC frequencies.

To investigate the effects of AC frequencies on the total losses of a sintered part, a magnetic toroid (0.25 inches [6.3 mm] thick) of high purity

iron with 3 w/o silicon was tested at AC frequencies of 30 Hz, 60 Hz and 100 Hz. The measured core loss as a function of induction level and frequency of this sintered material is presented in Figure 1. The total core loss for the 0.25 inch thick part at 60 Hz and 10,000 Gauss (1 Tesla) is approximately 10 watts/pound; more than double a Low quality electrical steel lamination. In addition to the high total core loss, the coercive force of the sintered material at 60 Hz was approximately 70e; this is compared with approximately 1 Oe when tested under DC conditions.

Iron Powder Polymer Composites

The development of iron powder polymer composites was expected to broaden the application base for P/M into high volume AC magnetic applications. Powder metallurgy because of its inherent shape making capability and high material utilization offers the potential to be an ideal part making technique to produce high volume AC electrical devices. Iron powder polymer composites utilize a high strength polymer to both electrically insulate the powder particles and add mechanical strength to the as compacted part. The iron powder polymer composites utilize warm compaction techniques to facilitate the plastic flow of the polymer; thus increasing the green density and giving a significant increase to the green strength of the component.

Iron powder polymer composites are designed to be used in either the as pressed or the as pressed and cured condition. Curing dramatically increases the green strength of the component and is done at temperatures less than 600°F (315°C). Typical mechanical properties of the iron powder polymer composites in the as compacted condition are ~ 12,000 psi transverse rupture strength (TRS), while curing increases the TRS to ~ 35,000 psi. Components made from the iron powder polymer composites are brittle and exhibit no elongation during tensile testing. The measured modulus of elasticity of these materials is approximately 5×10^5 psi.

Magnetic properties of the iron powder polymer composites are dependent upon the amount of polymer, the particle size distribution of the iron powder, and any pretreatment of the iron powder prior to polymer coating. Table 3 presents the magnetic data of three iron powder polymer composites. Ancorsteel SC 120 utilizes a coarser particle size distribution with the lowest polymer content and yields the highest compacted density. Consequently, it exhibits the best DC magnetic properties and has the highest AC magnetic performance at Low frequencies. Ancorsteel TC 80 utilizes a smaller particle distribution combined with an initial oxide treatment of the powder followed by the polymer coating. This increased level of insulation gives the lowest DC magnetic performance but yields the best high frequency performance, exhibiting constant permeability up to 50,000 Hz.

Figure 2 presents the core loss of the Ancorsteel SC 120 compared with M50 and M19 steel laminations. The core loss of the iron powder polymer composites are approximately equivalent to the M50 laminations at 60 Hz and slightly lower at 200 Hz. Compared with the M19 lamination material, the Ancorsteel SC 120 exhibits higher core loss at both 60 Hz and 200 Hz. Previous experimental work showed the iron powder polymer composite materials exhibit lower core loss compared with the M 19 type steels at frequencies in excess of 500 Hz (Ref. 3). This high frequency performance creates market opportunities for this material in this higher frequency range (above 200 Hz); however, the bulk of the opportunities for AC magnetic components are at 60 Hz. At this frequency, the iron powder polymer composites do not offer any significant material or performance benefits to displace the current lamination steels.

The Low frequency performance of the iron powder polymer composites results from the high hysteresis losses of these materials. In Table 3, the coercive force of these materials is approximately 4.7 Oersteds. This high level of coercive force broadens the hysteresis loop thus reducing the magnetic performance at the Low frequencies. The presence of the polymer insulating coating insulates adjacent powder particles thus minimizing the eddy current losses producing a component that has enhanced high frequency performance. Ideally, an insulated powder that has both Low hysteresis losses and Low eddy current losses could compete with the laminated steels in the high volume Low frequency applications.

Low Core Loss Insulated Powders

The widespread usage of iron powder polymer composites remains limited because of the high hysteresis loss effecting high core losses in the lower frequencies (< 200 Hz). High hysteresis loss is a result of the cold work imparted to the iron lattice during compaction. Recalling Equation 1, hysteresis losses can be expressed as the product of the hysteresis loop area multiplied by the frequency and the hysteresis loss constant. The hysteresis loop area is a function of the induction level, the permeability, and the coercive force. Iron powder polymer composites are used in the as compacted condition; the cold working during compaction effects the structure sensitive magnetic properties (in particular the permeability and coercive force). For pure iron, the coercive force of a fully annealed material toroid at an induction level of 12,000 Gauss is approximately 2.0 Oersteds (Ref. 7). Even minor amounts of cold work raise the coercive force to approximately 4.5 Oersteds. This increase in coercive force raises the hysteresis losses, thus increasing the overall losses at Low frequencies.

Thus for the non-sintered iron powder composites to gain greater acceptance, this Low frequency deficiency must be overcome. It has been suggested that redesigning of the AC electric device to minimize the air gap can overcome the shortcoming; however, practical component design precludes this. As stated earlier, the cold working during compaction raises the coercive force. To investigate the effects of powder compaction on the magnetic properties, a pure iron powder was surface coated with 0.035-w/o phosphoric acid and then premixed with 0.75 w/o zinc stearate. Toroids for magnetic property testing were compacted over a range of pressures from 10 tsi (137 MPa) to 50 tsi (685 MPa). The as compacted toroids were then cured at 350°F (177°C) and subsequently tested for their magnetic properties.

Table 4 presents the effects of compaction pressure on the DC permeability and coercive force of the pressed and cured iron toroids. Compaction pressures as low as 10 tsi (135 MPa) increase the coercive force to approximately 3.5 Oersteds while compaction at 50 tsi (685 MPa) raises the coercive force to ~ 4.5 Oersteds. Magnetic data for pure iron compacts sintered at 2050°F (1120°C) gave a coercive force of approximately 2 Oersteds. Thus even minor amounts of cold working result in a significant increase in the coercive force; consequently, a significant increase in the hysteresis loss.

Additional testing was done to investigate the effects of Low temperature annealing on the magnetic characteristics of pressed iron powder toroids. In this effort, toroids were pressed to a nominal density of 7.0 g/cm³, the powder was prepared using a proprietary surface insulation on a high purity iron powder. Following compaction, toroids were annealed at temperatures ranging from 300°F (150°C) to 1200°F (650°C). The toroids were tested under DC magnetic conditions and at 60 Hz to evaluate the effect of the annealing temperatures on the DC and AC magnetic properties. Figure 3 shows the permeability and DC coercive force of the toroids over the temperature range evaluated. At 1200°F (650°C) the permeability increased -30% over the permeability measured at 300°F (from -90 to -125); while, the DC coercive force decreased from 4.7 Oersteds to 3.0 Oersteds.

Figure 4 presents the AC core loss at 60 Hz as a function of induction level and annealing temperature. Annealing at 1200°F (650°C) lowered the total core loss of the magnetic material approximately 30%. Although 1200°F (650°C) does not seem adequate to fully anneal the iron powder, the reduction in the coercive force and corresponding reduction in the total core loss can be explained by the process of recovery that occurs during annealing (Ref. 8). Recovery is the first stage of annealing in which the strained metal lattice recovers the physical and mechanical properties of the strain free lattice without any recrystallization or grain growth. The temperature at which recovery begins is a function of the amount and type of cold work imparted to the metal. Thus, the high compaction pressures used facilitate the onset of recovery at this Low temperature; eliminating the deleterious effects of cold work on the magnetic properties of the pressed iron powder.

The experimental testing described above demonstrated that a 1200°F (650°C) annealing cycle is sufficient to raise the DC permeability and lower the DC coercive force while also giving reduced AC core losses. However, conventional iron powder polymer composites' materials can not withstand this temperature without degradation of the polymeric material. Phosphoric acid treatment of the iron powder is also ineffective at this temperature, giving reduced AC magnetic properties. This effort suggests that a new type of insulating material is needed that is compatible with conventional P/M techniques and allows for a modified magnetic annealing practice at a minimum temperature of 1200°F (650°C).

A proprietary Organo-metallic compound was developed that met the above criteria. This coating material is compatible with the iron powder and

1.

completely wets the powder surface. (Specific details of the coating are proprietary and are currently patent pending.) The coating material is applied to the iron powder utilizing a fluid bed technique. Once coated, the powder is compacted at 400°F (205°C) to 500 °F (260°C) using lubricated dies. The amount of insulating material utilized is approximately 2 w/o of the pore-free density of the resulting iron powder coating mixture. Pore-free density of the composite material is approximately 7.5 g/cm³. Compressibility of this material is shown in Figure 5, at 55 tsi the resulting annealed density is approximately 7.22 g/cm³ or approximately 95% of the pore-free density. Annealing of the compacts is accomplished at 1200°F (650°C) in a nitrogen atmosphere for a minimum time of one (1) hour.

2

Figure 6 presents the DC and 60 Hz AC hysteresis curves of this new material. Both the AC and DC curves exhibit Low coercive force and a Low hysteresis loop area. The non-traditional look of the B-H curves of Figure 6 is a result of the insulating material acting as a distributed air gap within the part thus shearing the B-H curve. Note the similarity of the DC and AC curves; the overall shape remains constant with minimal broadening of the curve at 60 Hz.

To measure the magnetic properties of this new insulated material, toroids were compacted at 50 tsi from this material and subsequently annealed at 1200°F (650°C). The toroids were tested at DC, 60 Hz and 200 Hz. For comparison M50 and M19 laminated steel toroids were also tested. Shown as Figure 7 are the DC hysteresis curves of the annealable iron powder composite and the M50 steel lamination material. As anticipated the wrought laminated materials exhibit higher DC permeability's and DC saturation. The reason for the reduced DC performance of the insulated powder is the presence of the powder coating.

Figure 8 presents the core loss as a function of the induction at 60 Hz for the annealable IP material compared with M50 and M 19 steel laminations. The annealable IP material exhibits a total core loss at 60 Hz and 10,000 Gauss of approximately 3 watts/pound. This value is considerably below the value of the M50 lamination steel and higher than the M19 lamination steel. The total core loss at 200 Hz for the annealable IP material is presented in Figure 9. At this higher frequency, the annealable IP has lower total core loss relative to the M50 but is still higher than the M19 material. This data indicates that the annealable IP material can successfully replace components made from the M50 type materials but is not a replacement for the M19 at the two frequencies examined thus far. Additional work is necessary to document the magnetic performance of this material at higher frequencies relative to steel laminations.

Limitations of this annealable material are the Low green strength of the annealable IP (~ 10,000 psi TRS) and the Low DC permeability. The Low permeability is a direct consequence of the highly insulating Organo-metallic coating. Figure 10 is a photomicrograph of the material that was pressed at 50 tsi and annealed at 1200°F (650°C). Note the continuous nature of the insulating film between the powder particles. This insulating film produces Low total core loss at operating frequencies; however, it creates a non-conducting highly insulating boundary thus preventing transfer of magnetic flux through adjacent powder particles.

CONCLUSIONS

- P/M sintered iron with 3 w/o silicon exhibits DC permeability up to -6000, with a DC coercive force of -1.00ersted. Additions of 0.45 w/o phosphorus to 3 w/o silicon material have the beneficial effect of raising the permeability and lowering the coercive force. The choice of alloy system and processing can be optimized to meet the specific component magnetic requirements.
- Iron powder polymer composites utilize a high temperature polymer to both electrically insulate the iron powder and impart high green strength to the as compacted component. Incorporating a curing cycle will improve the strength of the part without any detrimental effect on the AC magnetic properties. The limitation of the iron powder polymer composites is the high coercive force resulting in high hysteresis losses thus limiting their widespread usage at Low frequencies.
- A new powder coating has been developed that completely wets the iron powder and is compactable via P/M techniques. This new coating can be annealed resulting in reduced hysteresis and eddy current losses at Low frequencies. Total core loss for this new material at 60 Hz and 10,000 Gauss is approximately 3.0 watts/pound.

- Additional experimental work is required to optimize this new insulating material to improve the permeability and thus improve the overall Low frequency magnetic performance.

References

1. H. Rutz, F. Hanejko, G. Ellis, "The Manufacture of Electronic Components by the Powder Metallurgy Process", *Advances in Powder Metallurgy and Particulate Materials -1997*, Vol. 1, Part 1, compiled by Robert McKotch and Richard Webb, Metal Powder Industries Federation, Princeton NJ, 1997, pp 47-61.
2. C. Lall, *Soft Magnetism Fundamental for Powder Metallurgy and Metal Injection Molding*, Monographs in P/M Series No. 2, 1992, Metal Powder Industries Federation, Princeton NJ.
3. C. Oliver, H. Rutz, "Powder Metallurgy in Electronic Applications", *Advances in Powder Metallurgy and Particulate Materials - 1995*, Vol. 3, Part 11, compiled by Marcia Phillips and John Porter, Metal Powder Industries Federation, Princeton, NJ, 1995, pp 87-102.
4. L. Frayman, D. Ryan, J. Ryan, "Modified *PIM Soft* Magnetic Materials for Automotive Applications", SAE Technical Paper 980333.
5. Technical Report, "Powders for Soft Magnetic Sintered Parts", Published by HOGANAS AB, Brochure PM 92-01.
6. R. Bozorth, *Ferromagnetism*, D. Van Nostrend Co., Inc., 1959.
7. F. Hanejko, C. Oliver, H. Rutz, "Effects of Processing and Materials on Soft Magnetic performance of Powder Metallurgy Parts", *Advances in Powder Metallurgy and particulate Materials - 1992*, Vol. 6, compiled by Joseph M. Capus and Randall M. German, Metal Powder Industries Federation, Princeton, NJ, 1992, pp 375-404.
8. R. E. Read-Hill, *Physical Metallurgy*, Van Nostrend Reinhold Company, 1964.

Table 1
Magnetic Properties of Iron Silicon Alloys Sintered at
2300°F (1260°C) at a Field Strength of 25 Oersteds

Composition	Compacting Pressure tsi (MPa)	Density (g/cm³)	Maximum Perm	Coercive Force (Oe)	Bmax, Gauss
1 w/o Si	35 (480)	7.03	2900	1.67	12,800
	50 (685)	7.23	3100	1.65	13,700
2 w/o Si	35 (480)	7.13	4150	1.27	13,300
	50 (685)	7.34	5000	1.17	14,300
3 w/o Si	35 (480)	7.17	4900	1.10	13,600
	50 (685)	7.32	5600	1.03	14,300
3 w/o Si + 0.45 w/o P	35 (480)	7.13	5200	1.01	13,800
	50 (685)	7.26	5650	0.98	14,000

Table 2
Magnetic Properties of Iron Silicon Alloys Sintered
at 2400°F (1315°C) at a Field Strength of 25 Oersteds

Composition	Compacting Pressure tsi (MPa)	Density, (g/cm ³)	Maximum Perm	Coercive Force (Oe)	Bmax, Gauss
1 w/o Si	35 (480)	7.08	3500	1.45	13,200
	50 (685)	7.30	3550	1.45	14,100
2 w/o Si	35 (480)	7.25	5150	1.09	14,000
	50 (685)	7.40	5650	1.04	14,600
3 w/o Si	35 (480)	7.27	5700	0.96	14,200
	50 (685)	7.36	5950	0.92	14,400
3 w/o Si + 0.45 w/o P	35 (480)	7.25	6100	0.91	13,900
	50 (685)	7.35	6500	0.88	14,400

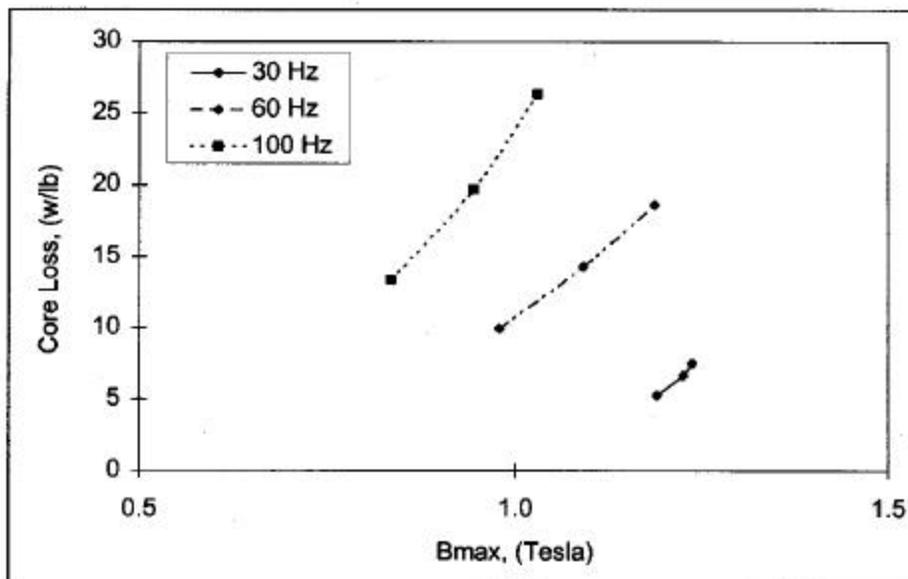


Figure 1: Core loss of Iron 3 w/o Silicon sintered test ring (0.25 Inch thick) as a function of induction and frequency.

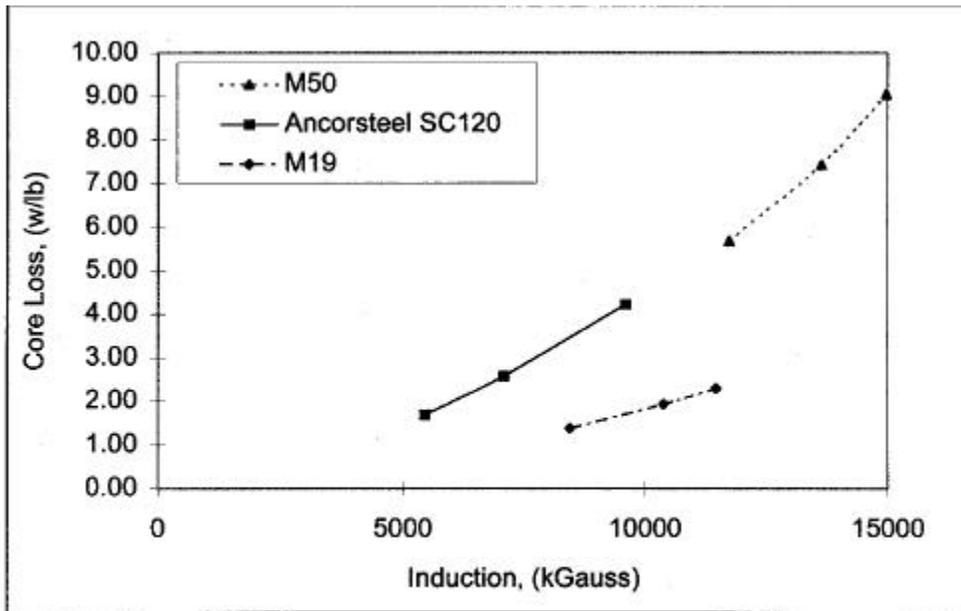


Figure 2: Core loss of Iron Powder Composites compared with M50 and M19 steel laminations.

Table 4
Effects of Compaction Pressure on the Magnetic Properties
of Iron Powder @ 40 Oersteds

Compacting Pressure tsi (MPa)	Density, (g/cm³)	Coercive Force (Oe)	Maximum Perm	Bmax @ (Gauss)
10 (135)	5.70	3.3	97	3,300
20 (270)	6.47	4.1	179	5,900
30 (410)	6.92	4.3	225	7,400
40 (540)	7.14	4.4	245	8,200
50 (685)	7.26	4.4	245	8,300

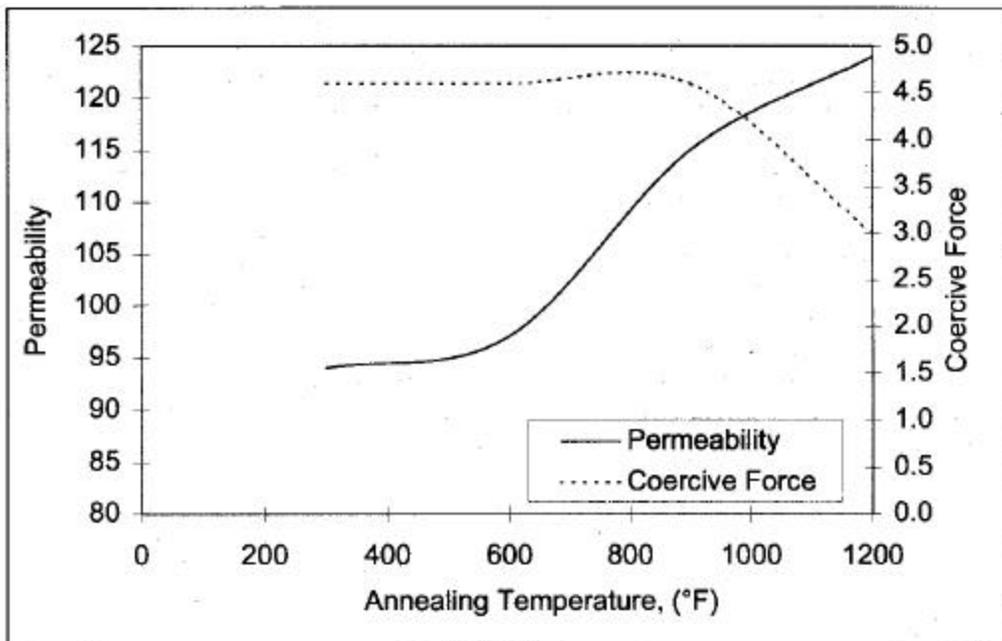


Figure 3: Effect of Annealing on the Permeability and Coercive Force of Iron Compacts.

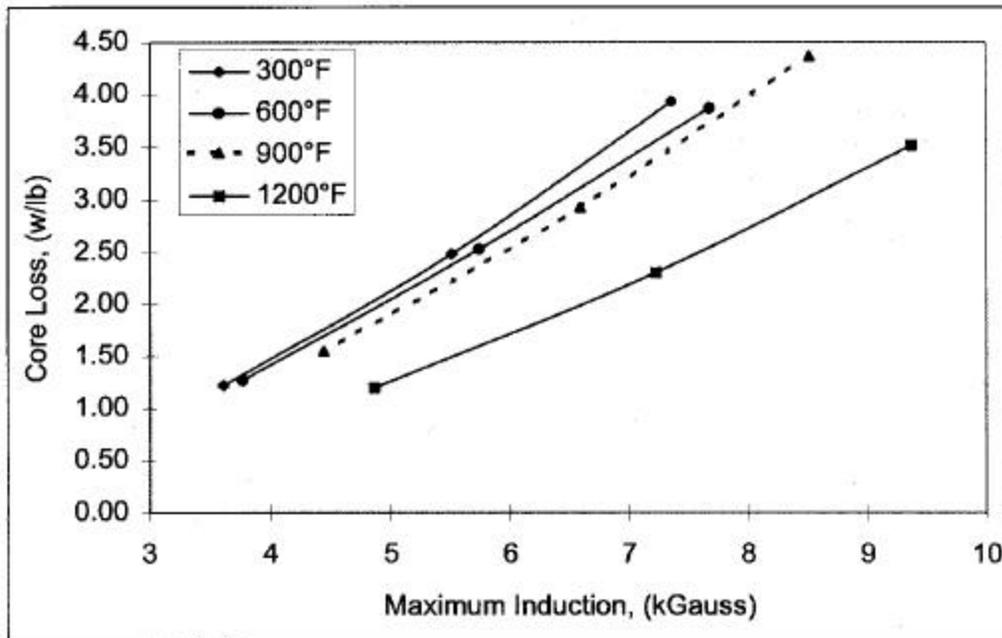


Figure 4: Core loss at 60 Hz of pressed iron cores annealed at various temperatures.

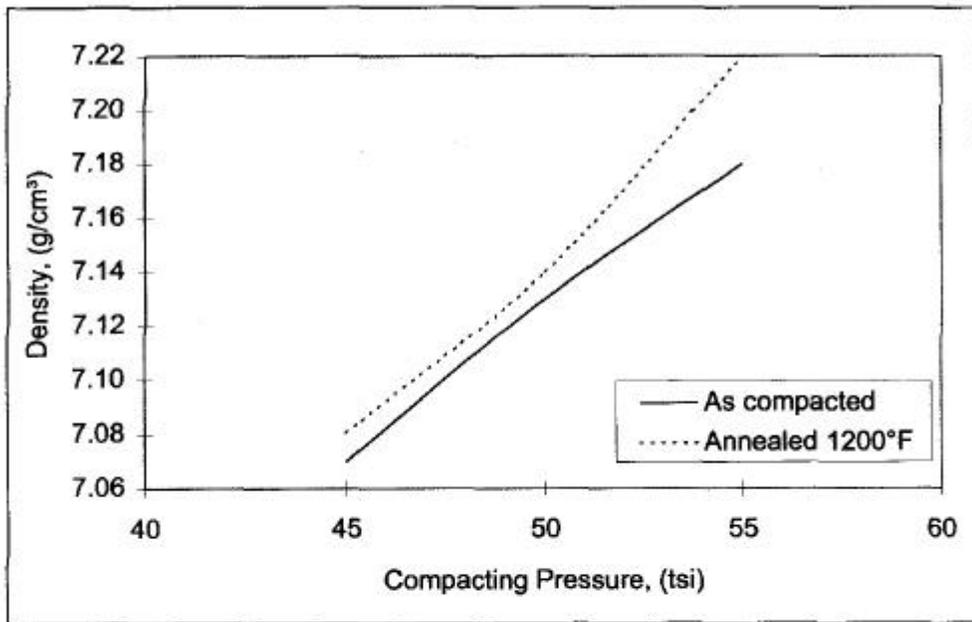


Figure 5: Compressibility of Proprietary iron organo-metallic compacts.

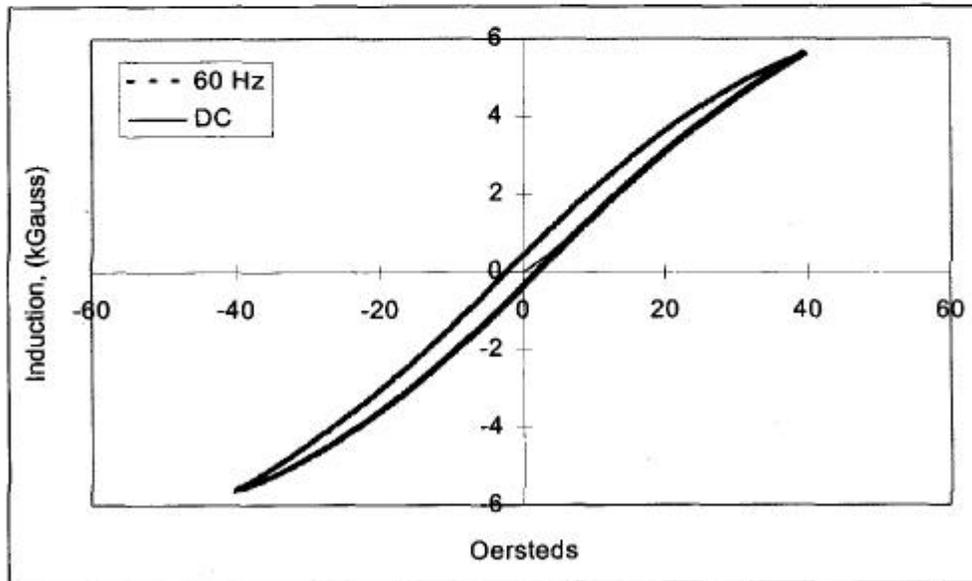


Figure 6: DC and AC hysteresis curves of newly developed insulated iron powder.

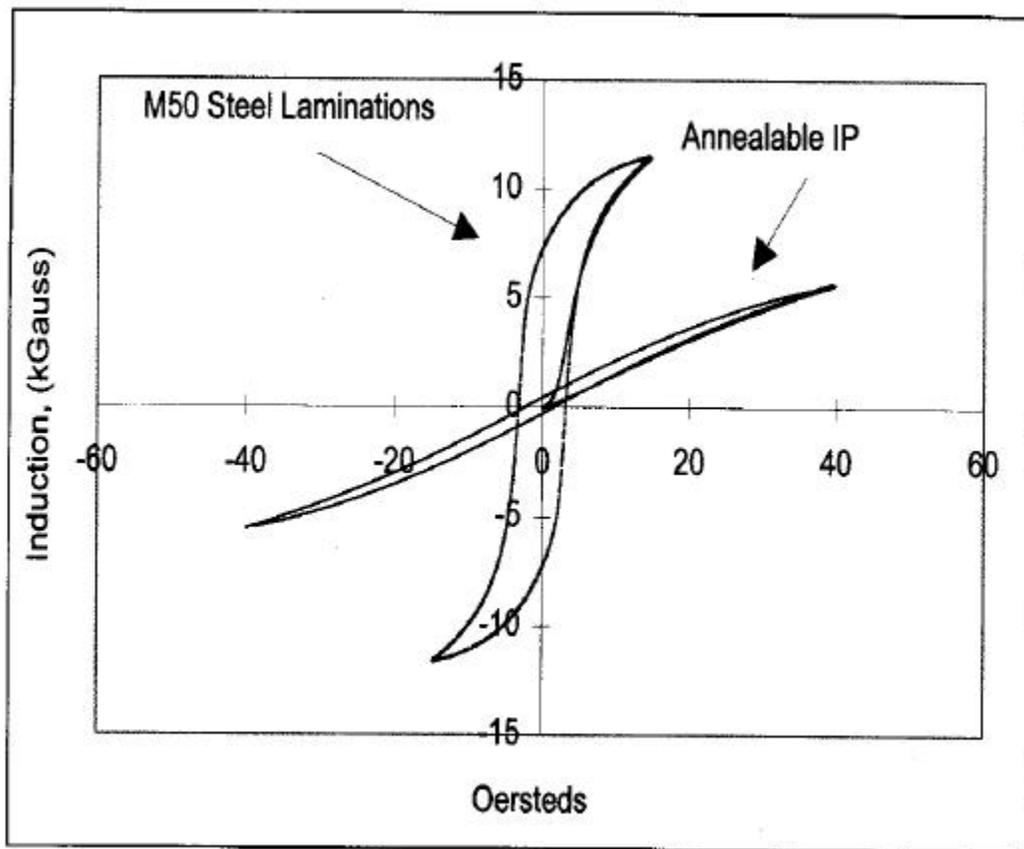


Figure 7: DC hysteresis curves of M50 and new IP material.

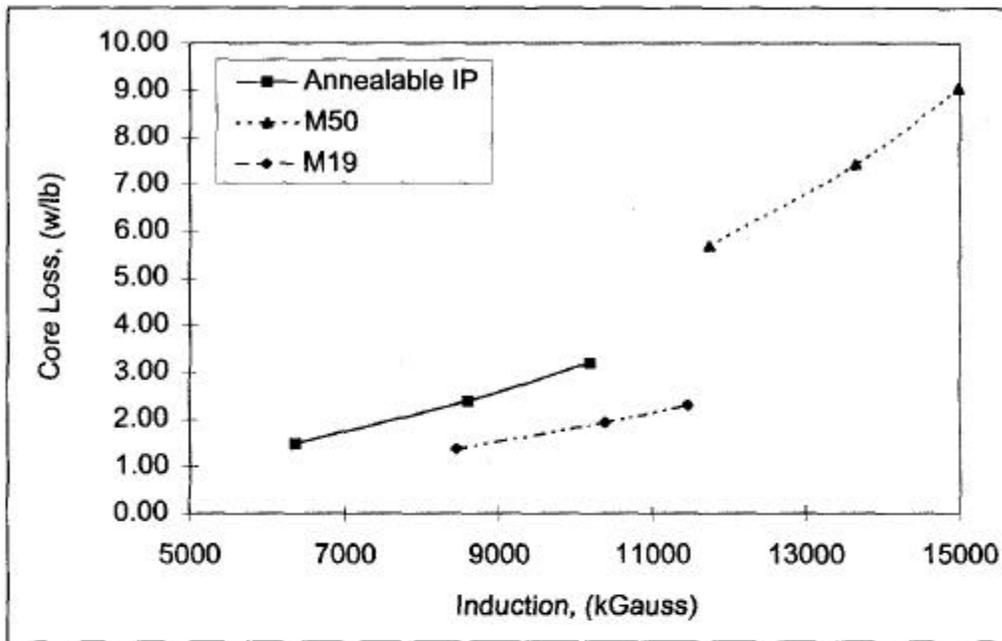


Figure 8: Core loss at 60 Hz of the new IP material and M50 and M19 steel laminations.

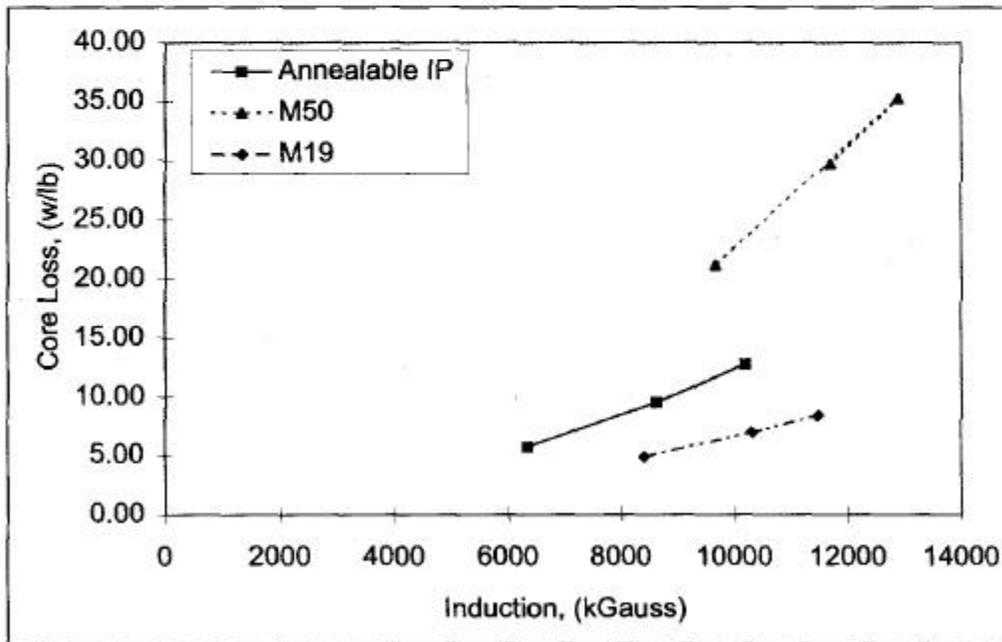


Figure 9: Core loss at 200 Hz of the new IP material and M50 and M19 steel laminations.

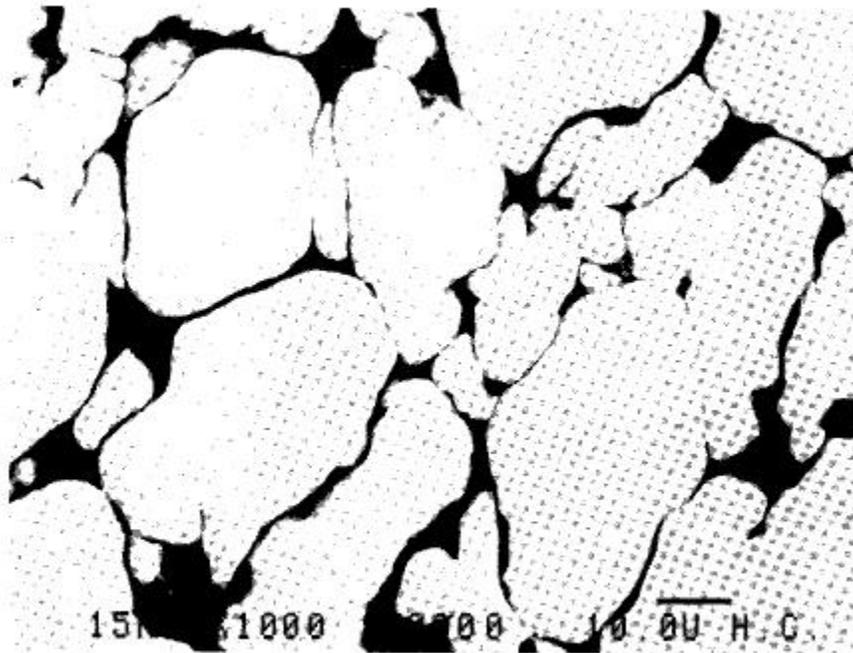


Figure 10: Photomicrograph of pressed and annealed IP core material.