Iron-Silicon Water Atomized Powders for Electromagnetic Applications

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ABSTRACT

Iron powder coated with polymers and/or phosphate coatings have been in the market since 1990. A major issue with insulated particle (IP) or soft-magnetic composites (SMC) powders is the inability to withstand heat treat temperatures required for relieving the residual stresses induced during the compaction process. Induced stresses during compaction result in high hysteresis losses. Typically the induced stresses can be relieved by stress relieving at temperatures above 700 ^{o}C, although full recovery does not take place until temperatures reach above 1000 ^{o}C. The phosphate or polymer coatings used to insulate the individual iron powder particles cannot withstand curing temperature greater than 700 ^{o}C.

A new water atomized iron powder, alloyed with silicon, with an oxide coating on the particle surface has been developed. This coating can withstand very high heat treat temperatures.

Key Words: Soft magnetic powders, Fe-Si powders, reactor core, electromagnetics

INTRODUCTION

Powder metallurgy (PM) offers an excellent choice for lower cost solutions for designers of automotive structural and electronic components. [1] It has been the desire of powder metallurgists to replace lamination steel with powder metal designs. Lamination steels are stamped to shape and are stacked and welded. PM components can be made directly to the final shape in one single operation. The development of the PM process for replacing lamination steel was inspired by the fact that eddy current core loss can be confined to an individual particle in a compact as long as a coating electrically isolates the particle. Major applications for these types of materials are in devices, such as motors, induction coil cores, actuators etc.

Work at Hoeganaes in the late 1980's on a similar technology resulted in the development in polymer coated iron powders.[2-4] This technology required heating of the powder to 150 ^{o}C and the die to 250 ^{o}C to achieve plastic softening and achieve densities as high as 7.45 g/cm^{3}. Powder heating and compaction at elevated temperatures posed number of concerns to parts makers and hence an alternate coating technology was developed in the early 2000's. This new powder does not require heating of the powder for producing parts and also use moderate (93 ^{o}C) die temperatures. This powder is referred to as AncorLam. This insulated iron powder is compacted with heated dies at 93°C. Densities as high as 7.5 g/cm^3 can be achieved at compaction pressures of 800 MPa, with resistivity exceeding 5,000 micro-Ohm/cm. The green strength of these compacts after curing at 450^{o}C for one hour in nitrogen atmosphere is dependent on compaction pressure. At 800 MPa a green strength of as high as 90 MPa is achieved. These products are very useful for a wide range of applications, from DC motors and AC motors operating at above 400 Hertz and other inductor applications. While these achievements helped promote the growth of PM in electromagnetic products [4-19], the permeabilities are too high for new applications such as inductors used in hybrid vehicles. These applications require permeabilities in the range of 100. Hence a pre-alloyed powder of iron with silicon was investigated. Gas atomized Fe-Si powders having low permeabilities have been reported in the literature for use at this permeability level. [20]. However, spherical powders obtained by gas atomization are difficult to process by conventional compaction process.

A new process, reactive atomization, utilizing water, has been developed which is able to produce powders that are coated with thin layer of oxide and are easily compacted. Fe-Si alloy powders were the first to be developed by this process. These reactive atomized magnetic (RAM) powders offer interesting properties useful in hybrid vehicles.

EXPERIMENTAL

The production of silicon irons for magnetic applications is accomplished by utilizing reactive water atomizing (RAM) followed by high temperature annealing. The atomizing process is different from conventional atomizing in that it utilizes very low water volumes but high water pressure. The low water volume allows for a slow cooling rate similar to what is achieved by gas atomization, leading to large as atomized grain size. This large grain size is critical to achieve the necessary magnetic properties- higher permeability and lower coercive force. The higher water pressure allows a particle size distribution and shape that is conducive to press and sinter powder metallurgy and gives a part with acceptable green strength.

If these powders were produced by gas atomization the powders would be spherical in shape and compaction would be very difficult. Utilization of water atomizing also leads to a significant cost savings over gas atomizing. This oxide layer will not be reduced during high temperature annealing (1050 ^{o}C) and the oxide layer is thick enough to provide an insulating layer which aids in the magnetic properties. The high temperature annealing allows for the further growth in grains improving the magnetic properties such as permeability and lower coercive force, reducing core losses. The typical impurity levels of the powders are shown in Table I.

Table I: Impurity levels and physical characteristics of RAM silicon iron powders.

Material	Carbon	Sulfur	Oxygen	Nitrogen	AD	Flow Rate
	w/o	w/o	w/o	w/o	g/cm^3	secs (50 g)
RAM 3% Si/Fe	.002	.004	.34	.006	3.51	25.3
RAM 6% Si/Fe	.018	.003	.67	.007	3.44	24.8

Material	+80 Mesh	+100 Mesh	+140 Mesh	+200 Mesh	+230 Mesh	+325 mesh	-325 mesh
RAM 3% Si/Fe	5.1	7.5	20.1	18.1	11.9	16.8	20.5
RAM 6% Si/Fe	5.6	10.2	21.6	16.8	11.7	15.3	18.8

The RAM powders were premixed with lubricants and compacted at 800 MPa to 1200 MPa to produce green torroids. The ratio of inner diameter to outer diameter is 0.67.

The torroids produced were cured at different temperatures in a nitrogen atmosphere. The core properties were measured using a commercial magnetic Hysteresis graph. The DC portion complies with ASTM A773/773-M1 and AC portion to ASTM A927/927-M99.

Resistivity of the compacts was measured using a four probe technique. Resistivity gives a measure of insulation between the particles.

RESULTS AND DISCUSSION

Microstructure of as atomized RAM Fe-3Si powder is shown in Figure 1(a). As can be seen, individual particles are coated on the surface with a thin layer of oxide.

(a) (b) (c)

Figure 1: (a) Optical micrograph of oxide coating on RAM Fe-3Si powder in as atomized and annealed condition (b) grain structure of annealed RAM Fe-3Si and (c) SEM Image of RAM Fe-3Si.

The powder can be compacted and cured at fairly high temperatures without loss of resistivity. Microstructure of a compact of this powder after curing at 1000 ^{o}C is shown in Figure 2. The oxide layer can stays in tack even after compaction at 800 MPa and curing at 1000 ^{o}C. The thin oxide layer formed by the RAM process behaves like a dielectric layer and improves resistivity.

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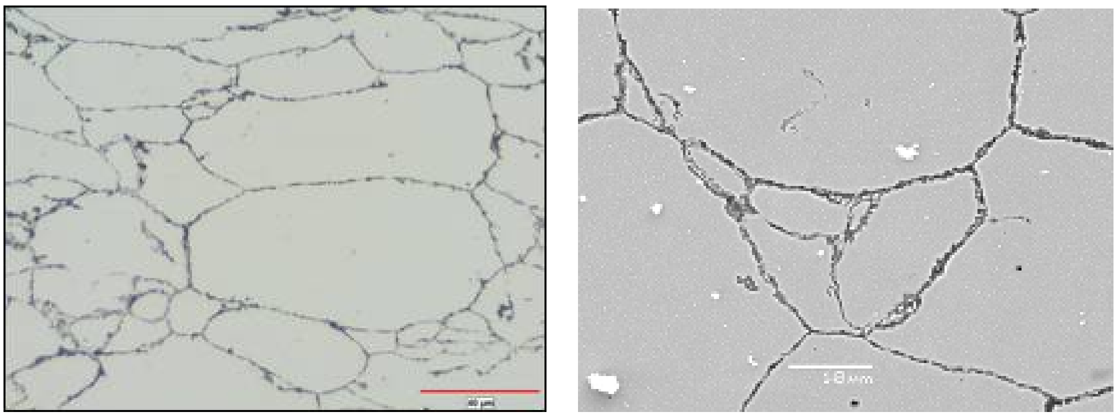

(a) (b)

Figure 2: Compacted and cured Fe-3Si powder produced by the RAM process. (a) Optical Micrograph, (b) SEM Image.

A comparison was made on the effect of curing temperature on the resistivity of compacts made from conventional water atomized iron powder and RAM Fe-Si powders. Resistivity is a good indicator of insulation between individual particles. Ancorsteel 1000C coated with phosphate coating was compacted into rectangular bars and resistivity measured and compared with a Fe-Si RAM powder coated in the same fashion. Ancorsteel A1000C was chosen for comparison because the residual level in the iron is very low and is used for electromagnetic applications. As can be seen in Figure 3 the RAM powder maintains high resistivity at much higher curing temperatures. The resistivity in the Ancorsteel A1000C drops significantly at curing temperatures around 450 ^{o}C, while the resistivity of the RAM Fe-3Si and the RAM Fe-6Si are not impacted by curing temperature until 600 ^{o}C and 1000 ^{o}C respectively. Even at 800C the resistivity of Fe-3Si and Fe-6Si powders are higher than 100000 micro-Ohm-cm, required target for many inductor applications operating at frequencies higher than 1 kHz. The higher curing temperature of the RAM Fe-Si powders allows for elimination of the residual stresses during compaction and reduces core losses.

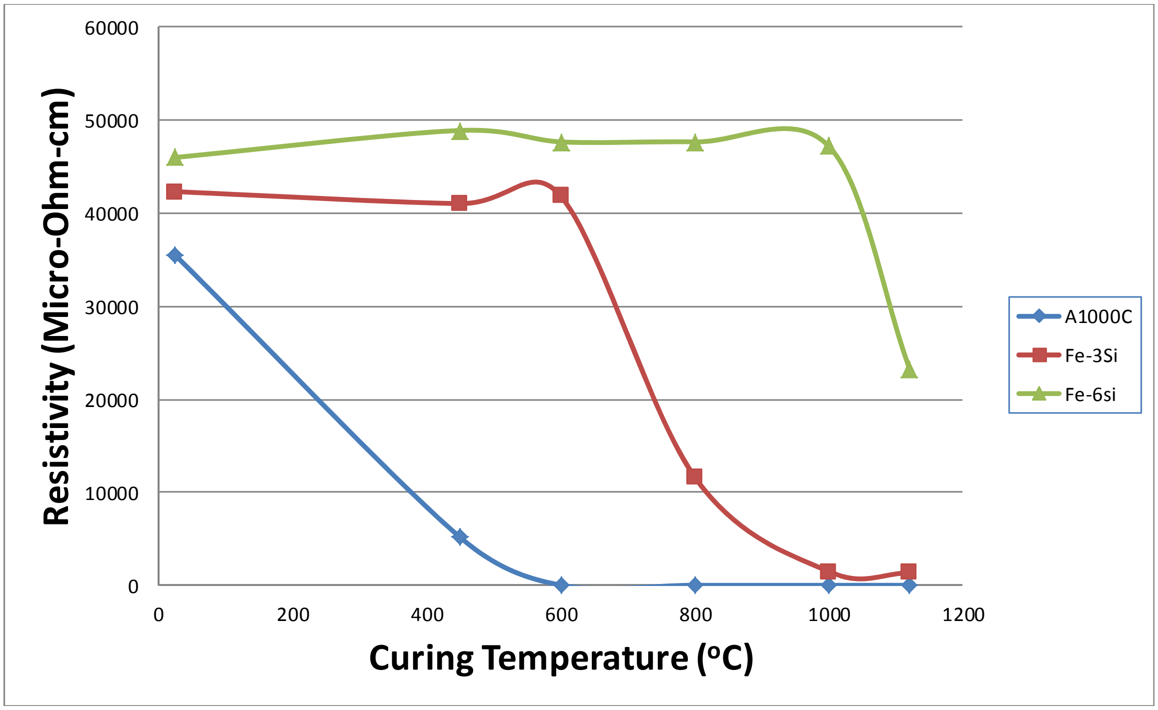

Figure 3: Variation of resistivity as a function of curing temperature for Ancorsteel 1000C powder, RAM Fe-3Si and RAM Fe-6Si all with phosphate coating.

An iron silicon alloy with 3 w/o silicon (Fe-3Si) was produced using the RAM process and after annealing was screened in to -105 micron powder. The screened powders were then coated with silicone. Torroids were pressed at 1176MPa and cured up to 800 ^{o}C in nitrogen. The hysteresis loop was measured and the coercive force is shown in the Table 2.

Table 2: Coercive force as a function of density in RAM Fe-3Si.

Density	Curing Temperature	Coercivity	Maximum permeability
g/cm^3	^{o}C	(Oe)	
6.63	750	3.67	129
6.70	775	3.70	134
6.76	800	3.63	143

The effect of curing temperature on the core loss of compressed Fe-3Si powder core material was also studied and is shown in Figures 4 and 5.

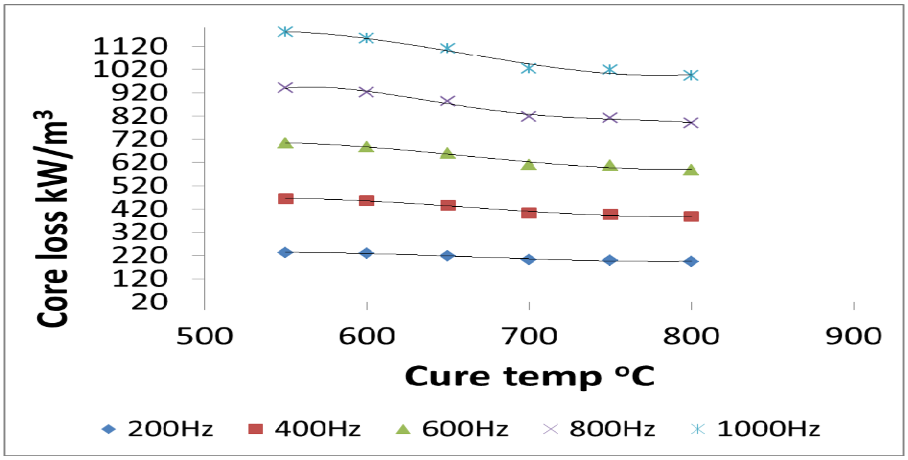

Figure 4: Core loss versus Curing Temperature for RAM Fe-3Si at different frequencies.

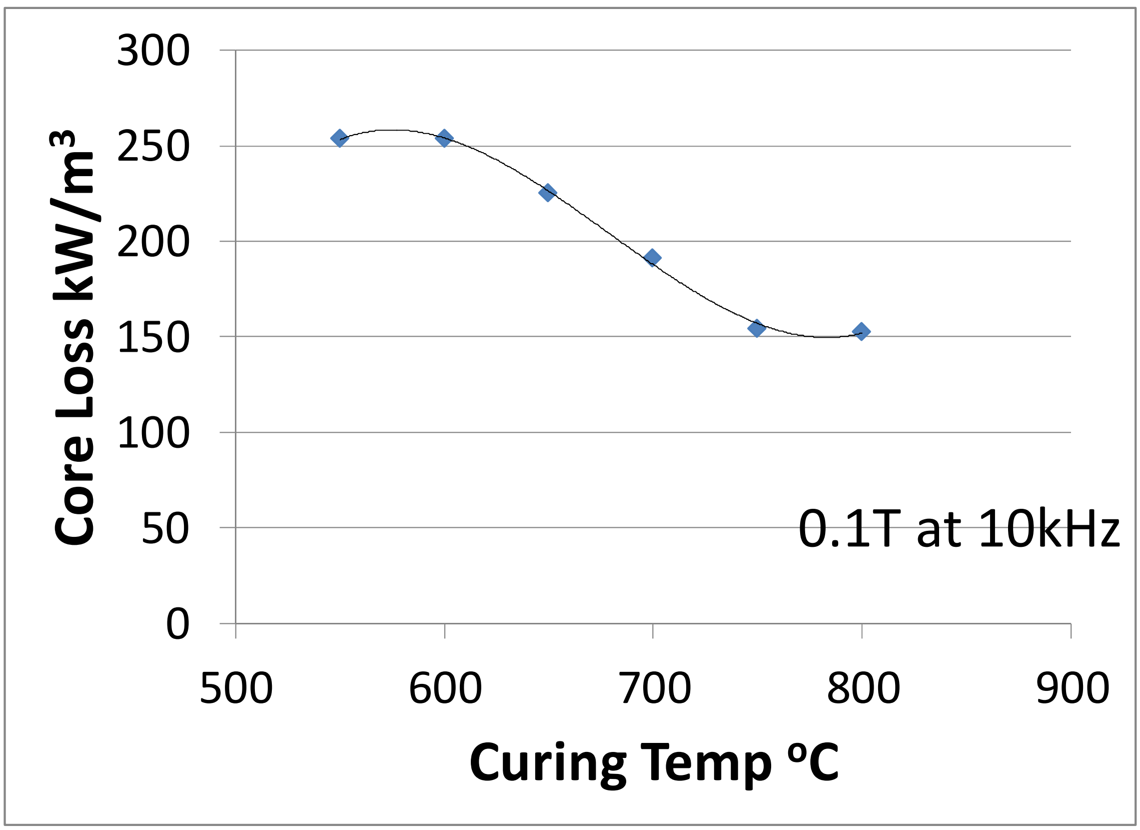

Figure 5.Core loss at 0.1T and 10 kHz as a function of curing temperature for RAM Fe-3Si coated powder compacts.

There is a distinct drop in core loss above 650 ^{o}C and the coreless continues to decrease. Resistivity of the compact after curing at 800 ^{o}C was over 10,000 micro-Ohm meter. Saturation flux density was 1.63T.Induction measured at B100Oe is 0.82T. Table 3 shows the core losses measured at 0.1T at 20 kHz and 10 kHz at various curing temperatures. Further work is in progress to cure at higher temperatures to improve the core loss. Higher density will improve induction further. The target core loss at 0.1T and 10 kHz is less than 100 kW/m^3.

Table 3: Effect of curing temperature on core losses for RAM Fe-3Si at a density of 6.67 g/cm^3.

Curing Temperature (^{o}C)	0.1T ,10kHz,(kW/m3)	0.2T,10kHz,(kW/m3)
550	253	934
600	253	920
650	213	813
700	187	734
750	166	680
800	153	667

Results on Fe-6Si atomized by the RAM process showed more promising results. The powder was coated and pressed at 830 MPa at room temperature and cured at 1050 ^{o}C in nitrogen atmosphere for 30 minutes. Magnetic data on this compressed core is shown in Figure 5 below. Core loss improved over Fe-3Si, however induction measured was 0.45T at B100Oe. Satuartion flux density was 1.11T. Further improvement in Induction by compacting to higher densities is currently in progress.

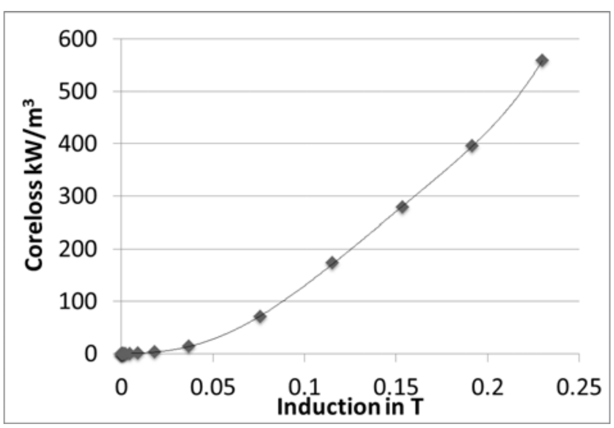

Figure 5: Core loss at 0.1T and 10 kHz as a function of induction for RAM Fe-6Si.powder compact

CONCLUSIONS

Low cost Reactively Atomized Magnetic (RAM) Fe-Si alloyed powders were developed so the powder could be cured at higher temperatures (>500 ^oC) than conventional powders leading to improved magnetic properties. Fe-3Si powders cured at 800 ^oC showed core loss of 150kW/m^3. The major component of the core loss is hysteresis losses and work is in progress to reduce the coercive force to below 3.0 Oe. Results on Fe-6Si powders were more promising. These powders could be cured up to 1050 ^oC without degrading the insulating coatings. This is the highest temperature reported thus far on coated iron based powders. Improvements on density are needed to achieve the necessary induction.

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