Newly Developed Soft Magnetic Composites for AC Applications with Reduced Iron Losses.

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ABSTRACT.
A broad variety of AC applications may be considered from iron powders due to attractive possibility to utilize 3D magnetic flux path when a reliable insulating coating, being sustainable at elevated temperatures, can be applied towards each individual particle within soft magnetic composite (SMC). With this regard, recently developed SMC that utilizes novel inorganic insulating coating formulation is presented while its magnetic performance and some technological characteristics are discussed with relation to applied processing parameters such as compaction and annealing operational steps. Obtained results indicate that the novel SMC exhibits high electrical resistivity that resulted in low Iron losses at elevated temperatures.

INTRODUCTION.

For more than two decades Soft Magnetic Composites (SMC) represent a constantly growing segment of advanced particulate materials which are utilized for high performance electromagnetic components to be applicable as a viable alternative to laminations for lowering iron losses at alternating current (AC) conditions. While alternating current mode is undoubtedly more efficient at transformation of high density power, it also offers simplified electromagnetic application design. For that reason, utilization of ferromagnetic powdered materials (PM), being coupled with composite technology, gives opportunity to form 3-D flux patterns, herewith leading to miniaturization of electromagnetic devices while permitting to achieve superior power output of fabricated AC components. [1-4] Among many suitable applications for soft magnetic composites (SMC) it worth to mention transverse flux machines, claw pole motors and BLDC motors, high frequency transformers, fast switching solenoids, various sensors , inductor cores for converters and diesel fuel injector stator cores, alternators and inverters for electric vehicles, etc. [2, 5-7]

In particular, for small electrical motors and high-speed electromagnetic actuators among other apparent advantages of AC vs. DC approach, there are tangibly lower copper losses, no necessity for commutators' use , efficient control of actuators' acceleration, adjustable torque capability, and simplicity in installation and maintenance. [3-4, 8-10] While particulate technology and PM materials have received considerable recognition in direct current (DC) applications for automotive, electronic, and other industries [11-16], the advent of SMC materials brought tangible benefits for both design and manufacturing practice. Among referenced gains, there is more efficient output per electromagnetic devices' size/weight/cost while enabling desired flux concentration. [2, 5, 9] Hereto, the SMC approach provides relative simplicity in production of net shape components, being combined with supplementary recycling capabilities. [9] On the top of that, the PM approach for manufacturing of SMC applications results in obtaining of higher iron fill factor with a simultaneous substantial decrease of iron losses within a broad range of frequencies [4, 8, 10]

According to established SMC fabrication routes, iron-based particles are insulated either by polymer coatings [17-19], and/or phosphate coatings [20-21], or hybrid organo-metallic oxide multi-layer coatings [22-27] as well as by various types of inorganic coatings [28-31]. And the last but not the least, SMC fabrication techniques expand flexibility in ferromagnetic substrates selection while introducing a specially engineered alloying material compositions like Fe-Si, Fe-Ni, Fe-Si-Al, etc. material systems. [30-32] However, there are certain practical limitations in a broad usage of metal compositions, e.g. nanocrystalline soft magnetic materials and /or produced by metal spinning process amorphous powders can be quite costly and technologically challenging for parts in a mass production. [3, 33]

Through already customary PM way of SMC materials fabrication, the surface of applied iron powder particles is quite frequently treated by phosphoric acid and its derivatives while is creating an electrically insulating layer. [20, 22, 28] Referenced phosphorous-based compounds significantly enhance the resistivity of the compact but are limited to a maximum curing temperature of approximately 425°C to 500°C (797 to 932ºF). [21] In general, both phosphate and polymer coatings used to isolate the individual iron particles cannot withstand curing temperatures higher than 700ºC (1290ºF). [28, 31]

 The iron loss comprises both hysteresis loss as well as eddy current loss, cumulative effect of which is exhibited on Figure 1, and depicted by equations {1} and {2}. [7,15,34]
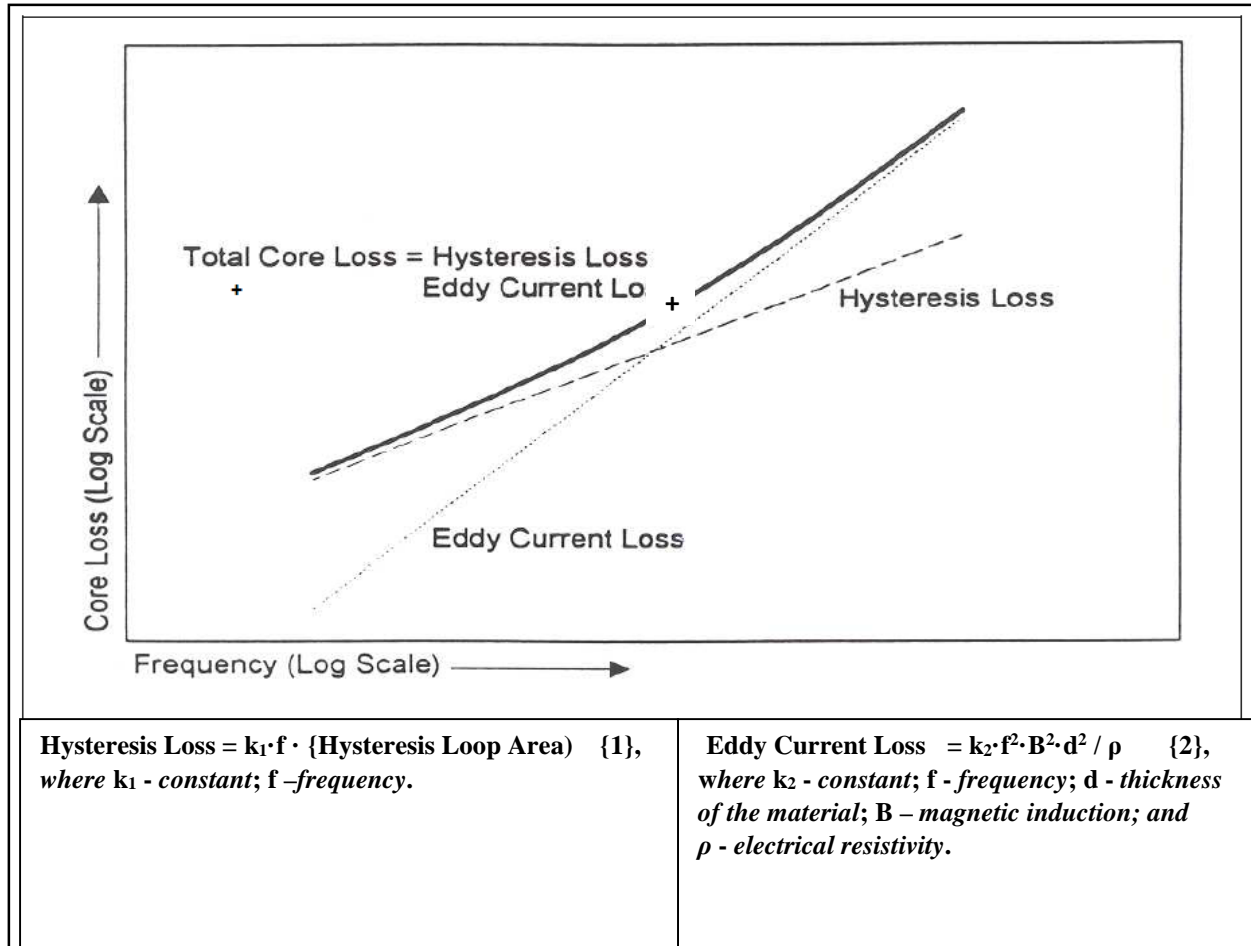
Figure 1 content labels:

Total Core Loss = Hysteresis Loss
+
Eddy Current Lo

Hysteresis Loss

Eddy Current Loss

Core Loss (Log Scale)

Frequency (Log Scale)

Hysteresis Loss = $k_1 \cdot f \cdot$ {Hysteresis Loop Area} {1}, where k_1 - *constant*; f –*frequency*.	Eddy Current Loss = $k_2 \cdot f^2 \cdot B^2 \cdot d^2 / \rho$ {2}, where k_2 - *constant*; f - *frequency*; d - *thickness of the material*; B – *magnetic induction; and* ρ - *electrical resistivity*.

Figure 1. Total Iron Core Losses at Various Frequencies. (Ref. [7, 15, 34])

To diminish eddy current losses, each iron or iron-based ferromagnetic particle within SMC compositions should be ideally covered by isolative coating; the latter one must possess a high level of electrical resistivity. [28, 31-33] Due to SMC powder design concept, the presence of a none-metallic insulation coating creates certain issues specifically a lower permeability, being linked to generation of a distributed air gap. [35-36] For this reason, the presence of such distributed air gap has to be taken in account for proper design of certain electromagnetic applications with SMC utilization. For instance, presence of the distributed air gap, being associated with SMC insulation coatings, may provide certain benefits like allowing higher DC- current level to be applied before magnetic saturation occurrence, i. e. helping to avoid rapid or so-named "sharp" saturation, which is quite inherent to discrete air gap. [35, 37] Also, distributed air gap, being caused by insulating coating layer around iron particles within SMC, can also diminish fringe losses in comparison to presence of discrete air gap, hereby minimizing localized heating (which is pretty natural for discrete air gap designs), and correspondingly boosting efficiency of electromagnetic applications, especially with growth of applied AC frequencies. [38] Hereto, in applications like BLDC motors, in which permanent magnets are utilized in the magnetic circuits with relatively sizable air gap, the lower permeability of SMC (vs. steel laminations) material is not significant. The rational for that are those circuits by design are run mainly by very low permeability values within air gap, where inherent for SMC isotropic magnetic properties allow designer to create shape of the motor to

be conformant to the shape of the particular application, hereby offering more compact space for the whole assembly. [5, 8-10]

Because of inherent lower magnetic permeability, would SMC components be chosen to compete on magnetic performance at either adequate level or to be better than steel lamination assemblies, the higher densities of fabricated SMC components should be improved; then, correspondingly, magnetic induction is going to be increased as well as permeability. [2, 5, 15, 36] With this regard, in order to obtain higher density level for SMC application, an insulation coating advised to be quite thin [28, 36] In the same time, high compaction pressures utilized for attaining of high components' density level lead to significant cold working of the iron powder core material, which in its turn negatively affects SMC structurally-sensitive magnetic properties like coercive force, and associated with that permeability. [32, 39] Based on results of the study [40] dedicated to earlier developed oxide-coated SMC, Figure 2 presents the effects applied processing parameters on resulting magnetic properties. Figure 2a reflects effect of raising compaction pressure towards density increase, and in parallel to that also demonstrates growth of the DC coercive force. Per referenced work as well as findings from some other studies, appearance of cold work stresses caused by compaction even at relatively low molding pressures result in sizable increase of coercive force. [38-41] For that reason, following compaction thermal treatment (which is often mentioned as either "annealing" or "curing") has been applied in order to eliminate away the deleterious effects of cold work, deriving from the compaction.

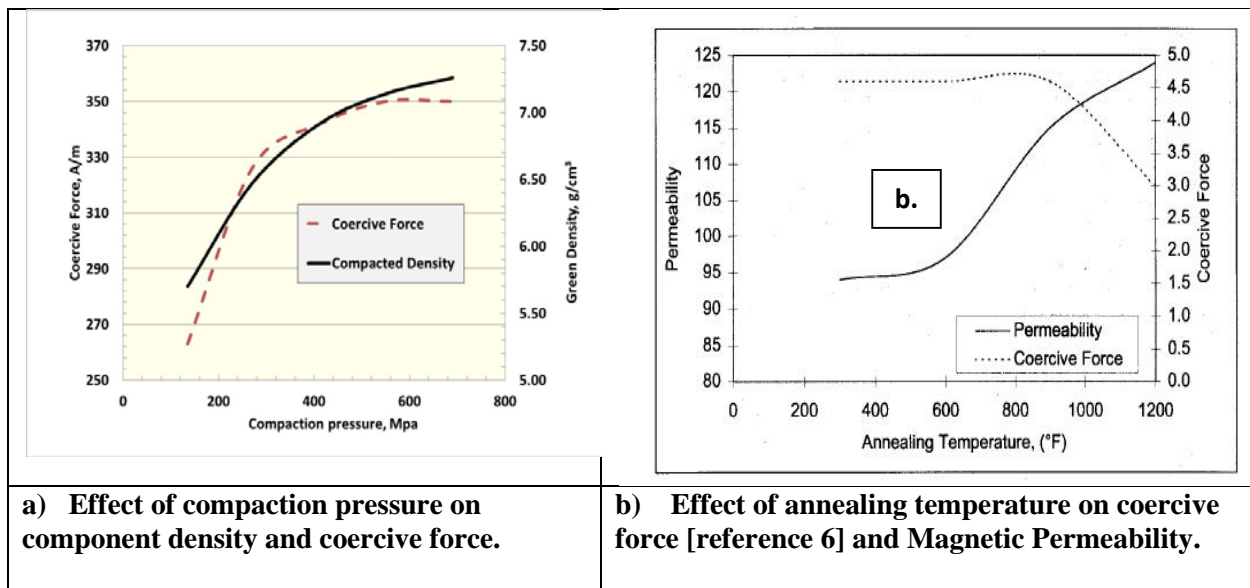| a) Effect of compaction pressure on component density and coercive force.	b) Effect of annealing temperature on coercive force [reference 6] and Magnetic Permeability.

Figure 2. Effect of Processing Parameters (Compaction Pressure and Annealing Temperature) on Magnetic Properties of SMC with Organo-Metallic Coatings (Ref. [40])

Thus, curing within the range of temperatures from   450°C (840°F) to 650°C (1200°F) showed a rather significant decrease in coercive force and corresponding growth of permeability related to escalation of annealing temperature as depicted at the Figure 2b. [40] Although aforementioned temperature is well below the industrial norms for annealing, of which for conventional laminated steels typically over 700°C. [38] Apparently, limitations to "anneal" the SMC compact *at* desirably higher temperatures associated with potential risk of insulation coating destruction may result in bigger hysteresis losses, herewith increasing the total core loss of the electromagnetic devices. In order to increase the "curing" temperature

of SMC materials, several endeavors have been exercised including developed techniques to utilize Magnesium Oxide based coatings as well as Magnetite type oxides created during the atomization of the iron powder and silicon based coating. [26-27, 31] Each of these approaches, although being good effort by itself, expose an inherent drawbacks such as increased compaction pressures to achieve a minimum density or high cost to produce SMC applications in mass production by using such coatings. For that reason, cost-efficient sustainable annealing thermal treatment is of a critical importance for stresses relief and reduction of other processing distortions associated with fabrication of electromagnetic AC applications. [28, 38, 42] Thus, in the study [38] it is suggested that the higher temperature of annealing thermal treatment can be applied, the more efficient is processing stresses removal happens. However in the case of SMC fabrication approach, would excessive temperatures be applied onto SMC artifacts at referenced annealing thermal treatment, it may cause either degradation or even disintegration of the coating insulation layer around iron particles. With this regard, increase of the thermal resistance of insulating coating should lead to enhanced "survivability" of SMC at higher annealing temperature. [28, 32, 41-42] Therefore, enhancement of referenced coating's characteristic is of a great importance towards next generation of SMC materials and its processing techniques. Besides that, optimal insulation coating for SMC applications mass manufacturing should provide a relative ease of fabrication, the capability of uniform deposition of the coating on the powder, but in the same time it should expose good resilience and adequate strength in order to sustain shaping compaction pressure, plus thermal expansion compatibility of insulating coating towards the SMC's iron powder substrate.

With this regard, recently developed SMC materials that utilize novel inorganic insulating coating formulations are presented in this study and reported below for potential consideration of designers and fabricators of soft magnetic applications. Obtained SMC magnetic performance data are discussed with relation to processing parameters of certain applied operational steps like compaction and annealing.

EXPERIMENTAL PROCEDURE.

Recently designed and developed novel formulations (designated as "H" and "I") have been used as inorganic electrically insulating coating, by applying them onto iron powdered particles via direct deposition. As a pertinent pre-operational step, the high purity iron powder was pre-treated with a dilute etching solution in order to remove surface oxidation and other impurities. The particulate mass was then heated to facilitate the dispersion of the liquid novel formulations onto the surface of the iron powder particles. For that purpose, either "H" or "I" novel coating formulation and iron powder have been mixed up in order together until reaching uniform dispersion, and then obtained powder mix while thoroughly prepared was dried creating an insulation coating around iron particles.

Once coated, the iron powder was premixed together with newly developed powder lubricant, AncorMax225® [42] for subsequent compaction, then shaped into standard transverse rupture strength (12.5 mm x 12.5 mm x 32 mm) specimens, and magnetic toroids (36 mm OD x 25 mm ID x 6 mm tall) at 93°C (199ºF) die temperature, and compaction pressures of 830 MPa . Finally, the green specimens were annealed /cured over a range of temperatures to investigate the potential magnetic performance enhancements from the applied novel coating formulations relative to SMC type materials, targeting   low to medium frequency AC applications. Within referenced magnetic studies, evaluated curing/annealing temperatures were correspondingly set to be 450°C (842°F), 540°C (1004°F), 650°C (1202°F), and 760 °C (1400°F) with utilization of 100% nitrogen, whereas also a specially formulated gaseous atmosphere

have been used within the annealing furnace. In addition, transverse rupture strength of fabricated SMC compositions have been measured done in conformity to MPIF Standard 41. [43] The transverse rupture strengths (TRS) bars were also used to evaluate both green and cured density as well as resistivity of the obtained compositions after the various curing conditions. As it known, resistivity can characterize the quality of inter-particle insulation within SMC compositions. [28, 31] The resistivity of fabricated TRS specimens was measured via the four-point probe technique utilizing a Keithley AC Resistance Bridge in line with ASTM Test Method A712-75. For the performed magnetic studies, toroidal-shape specimens once cured were wrapped with 40 secondary turns of 28 gauge enamel coated copper wire. Then, a second layer of insulating tape was wrapped atop the secondary windings, and lastly 200 primary winding turns of 22 gauge enamel coated copper wire were wrapped onto tested toroids. Magnetic testing (both AC and DC) of the toroids was accomplished using an automatic hysteresisgraph (model SMT 700, Magnetic Instrumentation, Inc.). The DC portion of the measurements has been performed in accordance to ASTM A773/773-M1, and AC measurements have been done in compliance to ASTM A927/927-M99 at frequencies up to 1000 Hz and induction level of 1 Tesla. In addition, a dilatometeric study has been done in line with ASTM Standard E228 for comparative evaluation of coefficients of thermal expansion (CTE) of the novel insulating coatings vs. CTE of the pure iron.

RESULTS AND DISCUSSION.

As it was noted in earlier performed study [44], the newly developed coating formulations "H" and "I" are capable of withstanding processing temperatures up to 1800-1950ºF (980-1065ºC) when used as a porosity sealant in PM parts. This high temperature capability is linked to its chemical content and fabrication processing technique (Figure 3).

Fig. 3. Appearance of the Insulation Film "H" (a) and the Insulation Film "I" (b) After Heat Treatment, being performed at 870ºC (1600ºF) in Ambient Air Atmosphere (Batch Furnace) Conditions.

In addition to their ability to withstand high annealing temperatures, a key for their use as an insulative powder coating is the capacity to have coefficient of thermal expansion (CTE) matching that of the base iron material. Based on the performed preliminary dilatometer study, Figure 4 exhibits the thermal expansion data for   the referenced above novel coatings, namely, "H" and "I" coatings films in

comparison to CTE of a 7.2 g/cm³ green density high purity iron powder compacted with admixed pressing lubricants, being measured in the same conditions. The thermal expansion of the experimental coatings is linear from room temperature to 750 °C (1400ºF] as it exposed at Figure 4, whereas the CTE graph of the compacted iron bars shows a non-linear behavior at 400°C (752ºF]; such non-linearity is a result of lubricant burn-off. [45] It is apparent that without the admixed lubricant the pure iron would also be linear except temperatures of polymorphic transformations. Thus, the significance for the coating and for the iron powder compact to bear practically the similar CTE is to eliminate potential cracking of that coating during heating or cooling cycles while annealing/curing process is applied, because cracks occurrence in the coating may result in iron particle-to-particle contacts, hereby, inducing eddy current losses and correspondingly leading to high AC magnetic losses.
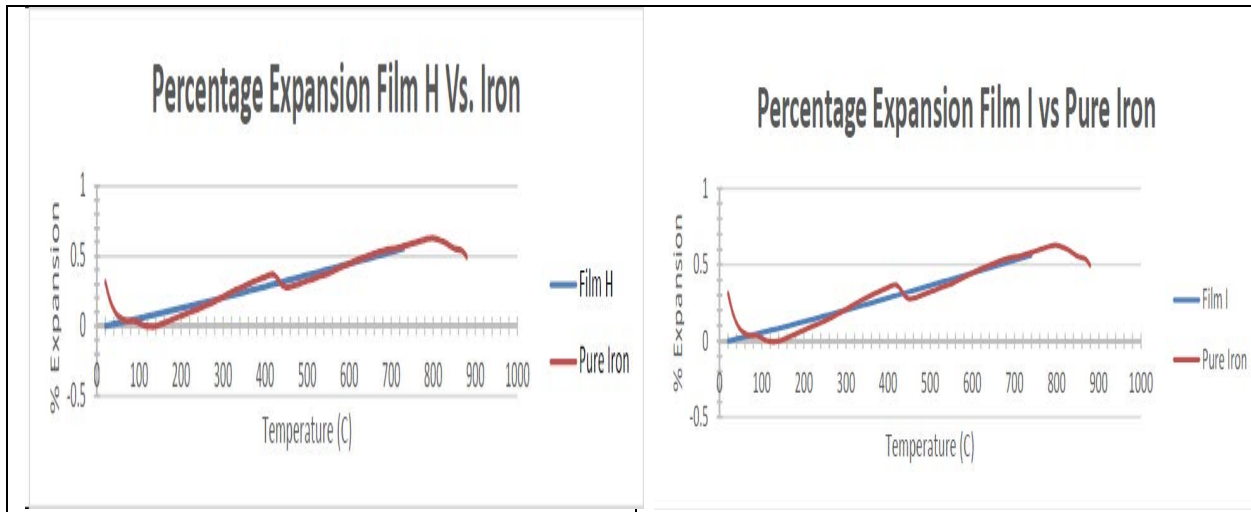

Figure 4. CTE of Insulation Films "H" (a) and "I" vs. CTE of Cold Compacted Iron Powder.

Resistivity of the new SMC materials have been evaluated as a function of annealing/curing temperature, and obtained results are presented in Figure 5. It has been suggested that maintaining resistivity at least at 10,000 μΩ·cm level should minimize eddy current losses at AC test conditions. [28]
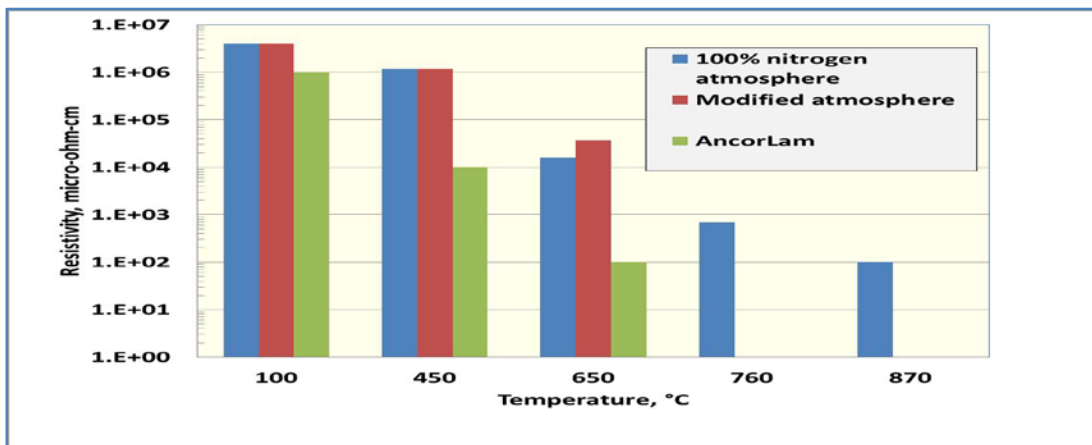

Figure 5. Resistivity of SMC with unique coating "H" as a function of curing temperature.

As it shown at Figure 5, the SMC with developed coating "H" exhibited resistivity of 15,000-17,000 μΩ·cm when cured at 650°C, and correspondingly 21,000 μΩ·cm that was measured for the SMC composition with applied coating "I". Those values are significantly higher than the standard AncorLam material at the same temperature, and superior to AncorLam at an even lower curing temperature, such as at 450°C. [46-48] However, by an increase of annealing/curing temperature of the experimental novel coatings up to 760°C resulted in a lower resistivity of approximately 1000 μΩ·cm, suggesting that annealing at 760°C causes excessive AC magnetic losses. These initial results were developed by application of annealing/curing operational steps at protective furnace atmosphere, containing 100% nitrogen. Additional experimentation was also performed at 650 °C using a modified furnace atmosphere; the results of this modified atmosphere showed a resistivity growth up to approximately 40,000 μΩ·cm.

Magnetic toroids were also compacted and annealed using the two atmospheres and a temperature range up to 870 °C. The coercivity data at 1 Tesla for both DC and AC conditions (at 50 Hz) are shown in Figure 6. DC coercivity is an approximate measure of the hysteresis loss; thus, lower coecivity values for PM soft magnetic applicationl are preferred because, as it known, provide increase of permeability. [3, 5,7, 36] However, it is also important to keep the AC coercive force as low as possible due to the fact that growth of Hc values in AC indicate an increase of eddy current losses. [28, 48-49]. Ideally, the DC coercive force would be identical to AC coercive force measurement. In this study, as it shown at Figure 6, the SMC materials that employed novel coating "H" showed a DC coercivity decreases from 350 A/m at 450C to 200 A/m at 650°C. With the application of a 650°C-curing cycle, the coercivity (H_c) remained approximately equal for both DC and AC testing hereby implying the beneficial effect of annealing at 650 °C and the fact that the novel insulation has not degraded at that applied annealing temperature. Hereto, annealing at 650°C shows a significant decrease in Hc value from the "as pressed" condition. An increase of the annealing temperature above 650 °C produced only minor decreases in the DC coercive force vs.the 650 °C condition. However, the AC coercivity has been increased at 760°C and even more at 870°C, implying greater eddy current losses.
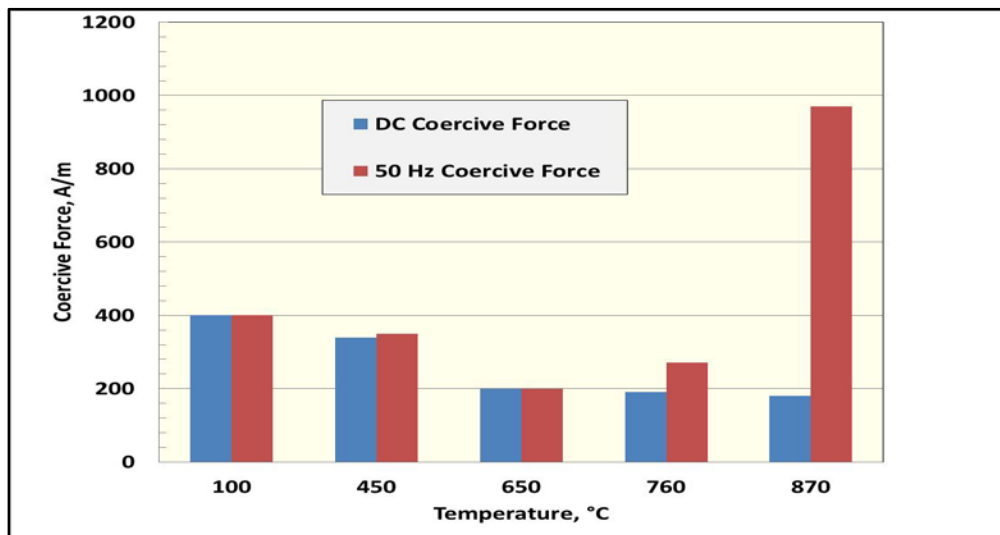

Figure 6.   Coercive force of unique coating "H", being measured via DC and AC (50 Hz).

Table 1 presents the magnetic data for both the standard AncorLam and for SMC, being coated by novel coating formulations "H" and "I".

Table 1.   Magnetic Data for Various Materials

Properties & Characteristics	AncorLam*		SMC Specimens with Novel Coating (Pamlico 1092H and Pamlico 1092I] Compositions at 100% nitrogen protective atmosphere.				At Modified Furnace Protective Atmosphere
			Pamlico 1092H			Pamlico 1092I	Pamlico 1092H
	Annealing/ Curing Temperature, °C						
	450	650	450	650	760	650	650
Density, g/cc	7.45	7.45	7.2	7.2	7.25	7.15	7.26
Permeability	500	800	140	185	200	155	190
Hc (DC), A/m	340	190	340	190	190	160	165
Hc (50 Hz), A/m	370	>1000	350	210	272	200	170
Core loss, @ 50 Hz and 1 T, w/kg	6	30	9	5	8	5.5	4.5
Hc (400 Hz), A/m	400	>1000	390	330	720	365	190
Core Loss @ 400 Hz and 1T, w/kg	59	>1000	86	60	160	74	40
Core Loss at 1 kHz and 1 Tesla, w/kg	170	>1000	225	180	NA	274	110
Resistivity, $\mu\Omega \cdot cm$	8000	<100		11000		21000	15000
TRS, MPa (psi)	124 (18000)	>172 (>25,000)	29 (4250)	33 (4750)	44 (6330)	72 (10500)	28 (4000)

*AncorLam – Trademark SMC product of Hoeganaes Corporation (Cinnaminson, NJ. USA)

At the present stage of development, these novel coatings seems to be somehow thicker than the coating used in the standard AncorLam SMC type materials; thus, resulting in lower compacted density. Correspondingly, such lower density causes reduced permeability, leading to the need of using higher applied fields in order to achieve the same level of magnetic induction. The strength of the new coatings is lower than the conventional AncorLam SMC material. At the same time, it has been noticed that the higher annealing temperature shows only minor increases in strength suggesting that the coating is mostly

intact but regions of particle-to-particle contacts may emerge herewith creating the higher eddy current losses. In consequence of this fact, strength improvement is going to be as one of the focused areas for future development efforts. Also presented in Table 1 are data exhibiting the magnetic performance of SMC with the novel coatings but at modified gaseous atmosphere conditions within the annealing furnace. Referenced modification of furnace's gaseous atmosphere has exhibited the lowest core loss properties of evaluated novel SMC materials. Referenced modified atmosphere combined with a 650°C annealing cycle showed a total core loss of 4.5 w/kg at 50 Hz and 1 Tesla conditions, and core loss of 40 w/kg at 400 Hz and 1 Tesla testing parameters. There was noted a slight increase in the coercive force from the value 170 A/m up to 190 A/m as the frequency has been growing from 50 to 400 Hz. However, this minor increase implies that hysteresis losses are the major component of the total losses at 400 Hz and 1000 Hz.

Shown in Figure 7 is the metallographic analysis of this the novel SMC coating after annealing at 650 °C. A well-defined particle coating is evidently surrounding each particle.
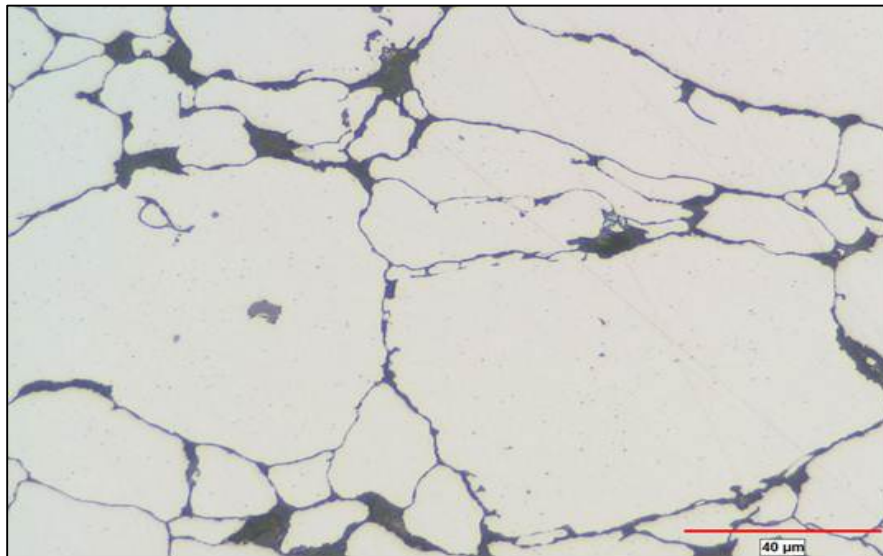

Figure 7. Unetched microstructure of insulated material annealed at 650°C.

CONCLUSIONS AND RECOMMENDATIONS.

From the performed study the following conclusions can be made:

1. Novel insulating coatings as the integral part of SMC material, have been investigated with regard to its potential utilization for AC applications within range of frequencies of 1 KHz and below. Results demonstrate high electrical resistivity of newly developed novel coatings at expanded range of post-compaction annealing temperatures up to 650°C that resulted to sizably diminished iron losses of SMC specimens.

2. Curing at 650°C substantially reduces the DC coercive force. Higher temperatures up to 800 °C show minor improvements above the 650°C temperature in mechanical strength. However, with the current level of development of the novel coatings curing at 760°C results in excessive eddy current losses and subsequently higher total losses.

3. A modified curing atmosphere showed significant improvement in the magnetic losses. The losses measured were the lowest throughout this evaluation and the losses measured were primarily hysteresis losses.

4. Maintaining a bulk resistivity greater than 10,000 micro-ohm-cm is crucial to maintain reduced total magnetic core losses.

FUTURE WORK.

The test results reported in current study present the initial development of this new magnetic coating. Future work to be done includes

1. Optimization of the coating physical characteristics, the coating amount and coating process.

2. Further optimization of the annealing temperature and furnace conditions for optimal magnetic and mechanical property performance.

3. Improvement of the as annealed strength of the compacted iron powder to levels achieved with commercial SMC products.

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