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Role of Ceramic Coating on Electrical and Magnetic Properties of Iron Powder

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Soft magnetic composite is a promising second generation magnetic material. It is widely used in both DC and AC applications. In the present work, magnesium compound coated iron powder (M-SMC) was used to fabricate the toroid cores of size Ø 30 × Ø 20 × 10 mm by powder metallurgy route. All these toroid cores were cured at different temperatures ranging from 600°C to 1000°C for 30 min in argon atmosphere controlled furnace. The electrical and magnetic properties of toroid cores were analyzed by Impedance Analyzer and B-H Analyzer respectively. M-SMC core cured at 800°C showed improved electrical properties for operating frequency up to 12000 kHz whereas magnetic properties were limited to applied magnetic field of 800 A/m.

Keywords: toroid core, M-SMC, curing, powder metallurgy

1. INTRODUCTION

Soft magnetic composites (SMCs) can be described as ferromagnetic powder particles surrounded by an electrical insulating film and these SMC find use in electromagnetic applications. It is normally manufactured by conventional powder metallurgy (PM) route. These composite materials offer several advantages over the traditional laminated steel cores in most of the applications. New developments in powder composites make SMC material interesting for application in electrical machines. These composites have several advantages, such as reduction in weight and size.[1] SMC materials are magnetically isotropic and hence ideal for the construction of electrical machines with complex structure and three-dimensional (3-D) magnetic flux path. This removes the well-known restrains on conventional laminated machines, e.g., the magnetic flux must flow within the lamination plane to avoid excessive eddy current loss. Therefore, SMC materials open up great opportunities to develop electrical machines with innovative structures.[2] Improvements in bonding agents, pressing techniques and heat treatments have helped in the development of SMC materials to give a good combination of magnetic properties (relative permeability and saturation induction) but with high electrical resistivity.[3] Heat treatment at higher temperatures results in partial stress relief of the components and this lowers the hysteresis loss. However the temperature, time and atmosphere must not exceed the tolerance limits of the surface insulation on the particles. Degradation of surface insulation layer results in particle to particle contact and thereby higher eddy current losses in the component.[4]

Magnesium coated iron powder is emerging as an attractive material for soft magnetic applications due to its metallurgical bonding with iron core. In this paper, an attempt has been made to improve the understanding of the effect of temperature on electrical and magnetic properties of magnesium compound coated SMC (M-SMC).

2. EXPERIMENTAL PROCEDURE

2.1 Materials

Pure electrolytic iron powder (99.8% pure) with an average particle size 105 μm supplied by Industrial Metal Powder (I) Pune was used for preparation of M-SMC powder. This powder was then used to fabricate M-SMC toroid cores.

2.1 Preparation of magnesium compound coated powder

The hydrogen loss of pure iron powder was carried out at 900°C for 60 minutes according to ASTM standard E159-10. Using Fe-O phase diagram, the stability of FeO phase was identified in terms of temperature (700°C) at corresponding oxygen content of the oxidation environment so as to form FeO oxide layer. The oxidized iron powder was fed into a specially designed fabricated system to produce desired magnesium compound coating as shown in Fig. 1. Fabricated apparatus consist of 3 chambers, horizontal vaporization chamber, vertical coating chamber and collection chamber. Ultra-high purified argon gas was passed through purifier consisting of pyrogallol acid and silica gel. Temperature of horizontal vaporization chamber of tubular

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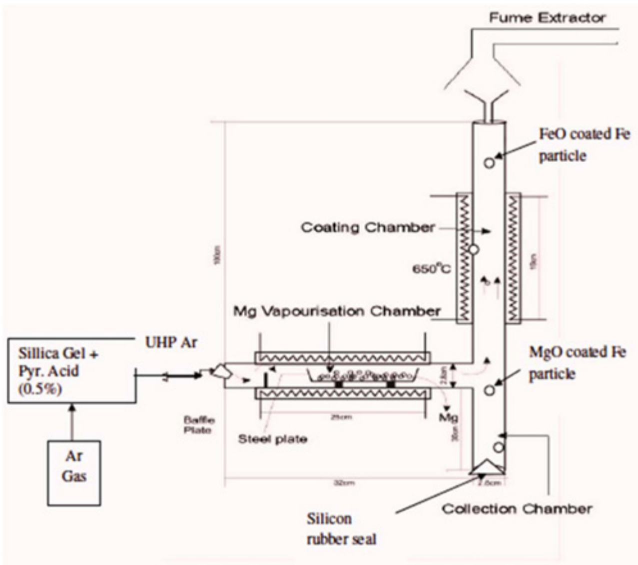

Fig. 1. Operating principle for preparing magnesium compound coated iron powder.

furnace was maintained at 700°C at which magnesium begins to vaporize i.e. below boiling point (B.P) of 1107°C. In vertical chamber iron oxide particles were fed under gravity and these particles while coming downward reacts with magnesium vapour carried away by argon gas in countercurrent direction. The iron oxide layer of the particle got reduced by magnesium vapour leading to the formation of thin magnesium compound coating on the surface of iron particle in vaporization chamber.[5]

2.2 Fabrication of toroid cores

The die was designed for the fabrication of toroid core having OD 30 mm, ID 20 mm and H 10 mm for analysis of electrical and magnetic properties. Green toroids were made at 900 MPa pressure with 0.5% (by wt) Kenolube as the lubricant. These green compacts were then cured for 30 min at 600°C, 800°C and 1000°C in argon atmosphere in controlled tubular furnace which was heated slowly with an average heating rate of 5°C/min. These cured compacts were then cooled in the furnace itself.

3. CHARACTERIZATION OF TOROID CORES

3.1 Physical properties

Archimedes' principle was used to measure the cured density of toroid cores. Resistivity was calculated by measuring resistance of toroid core using 7½ Digital Multimeter (KETIHLEY 2001).

3.2 Electrical properties

Electrical properties of the toroid cores were measured by Impedance Analyzer (Model -4162A, Make-HP) by varying frequency from 1 kHz to 13000 kHz. Core Factor (CF) for each toroid core was calculated by Eq. (1). Toroid core was wound with 15 numbers of turns (N) by Teflon coated copper-tin wire. Inductance (L) and Quality Factor (Q) at different frequencies were noted. Initial Permeability (μ_i), Relative Permeability (μ_r), and Power Losses (Loss Factor) were derived using equations (2), (3) and (4) respectively, where, μ_0 is the permeability of free space i.e. $4\pi \times 10^{-7}$ H/m.

$$CF = \left(\frac{OD+ID}{OD-ID}\right) \cdot \left(\frac{1}{4 \cdot H \cdot N^2}\right) \tag{1}$$

$$\mu_i = L \cdot CF \tag{2}$$

$$\mu_r = \frac{\mu_i}{\mu_o} \tag{3}$$

$$Loss\ Factor = \frac{1}{\mu_i \cdot Q} \tag{4}$$

3.3 Magnetic properties

Magnetic properties of the toroid cores were measured by B-H analyzer (Model-BHU60, Make-Rikendenshi) which works on the principle of applying magnetic field (H) against magnetic flux density (B). All results were carried out at 50 Hz frequency (F) and 10 Oersted (800 A/m) magnetic field. Toroid core was wound with 25 numbers of turns by Teflon coated copper tin wire. Magnetic properties like Saturation Induction (Bs), Coercivity (Hc) and Retentivity (Br) were calculated manually by plotting the B-H graph. Mean magnetic area (A) was calculated from size of the core. Hysteresis Loss (P_h) and Eddy Current Loss (P_{ed}) at different frequencies were calculated by equations (5) and (6) respectively, where ρ is the theoretical density in g/cc.

$$P_h = \frac{A \cdot F}{\rho} \tag{5}$$

$$P_{ed} = \frac{d^2 \cdot Bs^2 \cdot F^2}{\rho} \tag{6}$$

3.4 Metallographic analysis

The Mg compound coated compact was subjected to polishing on emery paper and final polishing was carried out on velvet cloth lapping machine with intermittent application of fine suspensions of alumina to get better finish on polished surface. A freshly prepare etchant i.e. 4% Nital was used for revealing micro structure by optical microscope (Axiovert 40Mat, CARL ZEISS).

3.5 X-ray diffraction analysis

The samples of M-SMC toroid cores cured at different temperatures were exposed to XRD analysis using copper target. X-ray analysis was done with low scanning rate, with 2θ values ranging from 10° to 100°. Analysis for phase identification was carried out using X'Pert high score plus software.

4. RESULTS AND DISCUSSIONS

4.1 Analysis of physical properties

Table 1 shows physical properties of M-SMC cores that were cured at 600°C, 800°C and 1000°C. There is marginal change in density. It is found that the resistivity of M-SMC core cured at 800°C is higher than other cores cured at 600°C and 1000°C. High resistivity is due to the ceramic nature of magnesium oxide coated on iron particles which can lower the eddy current loss due to its inherent insulating property. The reason for improvement at 800°C is discussed in the later part of this paper.

4.2 Sensitivity to electrical properties

As the frequency increases, inductance and initial permeability decreases with increase in the loss factor.[6,7] It is found from the Figs. 2, 3 and 4 that at frequency of 12000 kHz, Inductance, Initial Permeability and Relative Permeability of M-SMC 600 core are high by 47.7%, 47% and 48.9% with reference to M-SMC 800 and by 54.8%, 58.8%, 57.8% with reference to M-SMC 1000 respectively. Figure 5 shows the behavior of toroid cores in terms of Loss Factor (i.e. power losses). Table 2 shows the comparison of electrical properties of toroid cores at 12000 kHz frequency.

Table 1. Physical properties of Mg compound coated cores.

Toroid Core (s)	Cured Density (g/cc)	Resistivity ($\mu\Omega$-m)
M-SMC 600	7.19	22434
M-SMC 800	6.95	23793
M-SMC 1000	7.01	23420

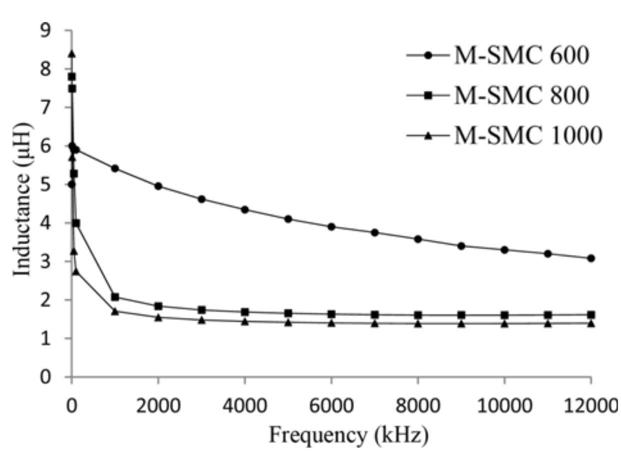

Fig. 2. Effect of frequency on Inductance of toroid cores.

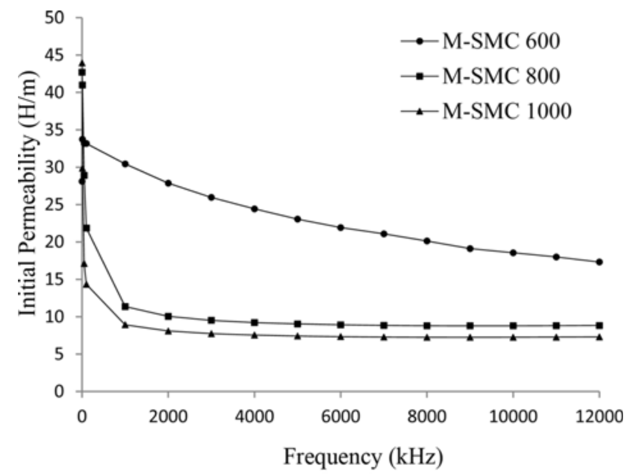

Fig. 3. Effect of frequency on Initial Permeability of toroid cores.

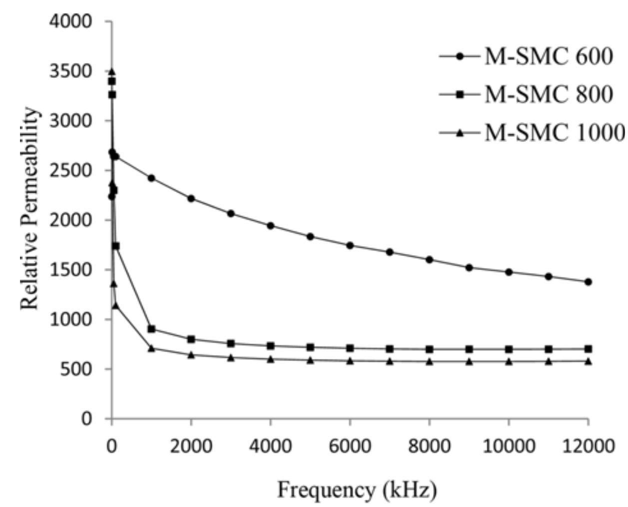

Fig. 4. Effect of frequency on Relative Permeability of toroid cores.

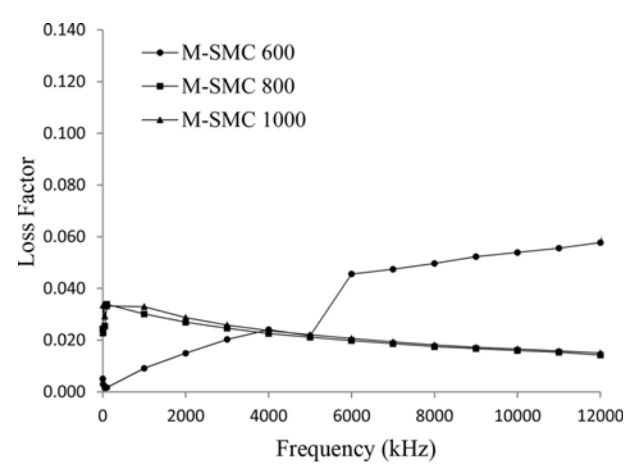

Fig. 5. Effect of frequency on Loss Factor of toroid cores.

Table 2. Summary of electrical properties of toroid cores.

Property (s) @ 12000 kHz	M-SMC-600	M-SMC-800	M-SMC-1000
Inductance, μH	3.08	1.61	1.39
Initial Permeability, μH/cm	17	9	7
Relative Permeability	1378	703	581
Loss Factor	0.058	0.015	0.015

At 12000 kHz frequency, the Loss Factor of M-SMC 600 core is reduced by 74.13% with reference to M-SMC 800 and M-SMC 1000. It can be attributed to the stress relieving of the matrix thereby reduction in the dislocation density. The dislocations are the atomistic defects wherefrom the atoms are missing, which act as hindrance to the electron flow. Hence at higher curing temperature, say 800°C and 1000°C, the dislocations can additionally generate due to vibrations of atoms. Therefore, it is observed that as the curing temperature increases, electrical properties starts deteriorating (Table 2).

4.3 Sensitivity to magnetic properties

The AC magnetic properties of toroid cores were measured using B-H Analyzer (Model- BHU60, Make-Rikendenshi) by applying 800 A/m (10 Oe) magnetic fields at 50 Hz frequency. Saturation Induction, Coercivity, and Retentivity were calculated from the B-H loop. It is evident from Table 3 that the Coercivity of M-SMC core does not show variation up to 800°C curing temperature while it increases by 88.89% at 1000°C. The Retentivity of M-SMC 800 is reduced by 83.33% and 91.66% with reference to M-SMC 600 and M-SMC 1000 respectively. Hysteresis Loss of M-SMC 800 core is reduced by 74.89% and 83.05% with reference to M-SMC 600 and M-SMC 1000 respectively as can be seen from Fig. 6. Similarly, Eddy Current Loss of M-SMC 800 core is reduced by 82.23% and 53.33% with reference to M-SMC 600 and M-SMC 1000 respectively as seen from Fig. 7. From this analysis, it is revealed that temperature affects electrical and magnetic properties of magnesium compound coating as seen from Tables 1, 2 and 3.

4.4 Effect of temperature on structure property correlation

The curing temperature can be varied without damage to coating. Higher density may increase the induction at low field and also the DC permeability. The heat treatment provides low volume fraction of defects, reduces the distortion within particles, lowers dislocation density and hence the magnetic permeability increases.[7] Microstructure of cured M-SMC consists of purely ferrite phase and every particle of iron made up of fewer number of ferrite grains. The shape of ferrite grains and its number per iron particle may vary with prior cold working, that is, deformation during compaction and curing temperature, which is normally above the recrystallization temperature of electrolytic iron (395°C).[8]

Table 3. Summary of magnetic properties of toroid cores.

Property (s) @ 10 Oe, 50 Hz	M-SMC-600	M-SMC-800	M-SMC-1000
Saturation Induction, T	0.05	0.02	0.036
Coercivity, A/m	7.95	7.95	71.62
Retentivity, T	0.006	0.001	0.012
Hysteresis Loss, W/kg	0.00243	0.00061	0.0036
Eddy Current Loss, J/kg	0.394×10^{-5}	0.07×10^{-5}	0.15×10^{-5}

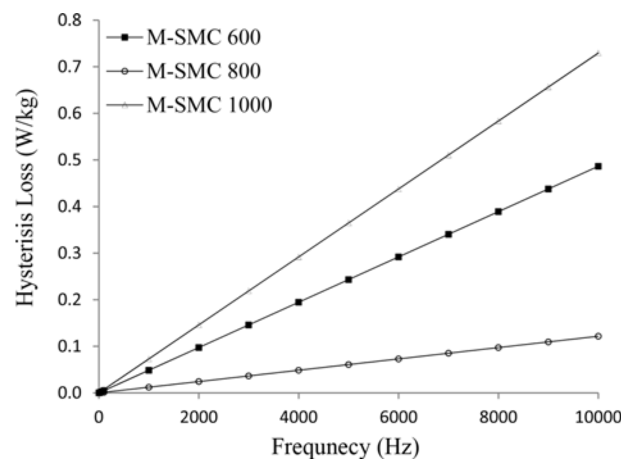

Fig. 6. Effect of frequency on Hysteresis Loss of toroid cores.

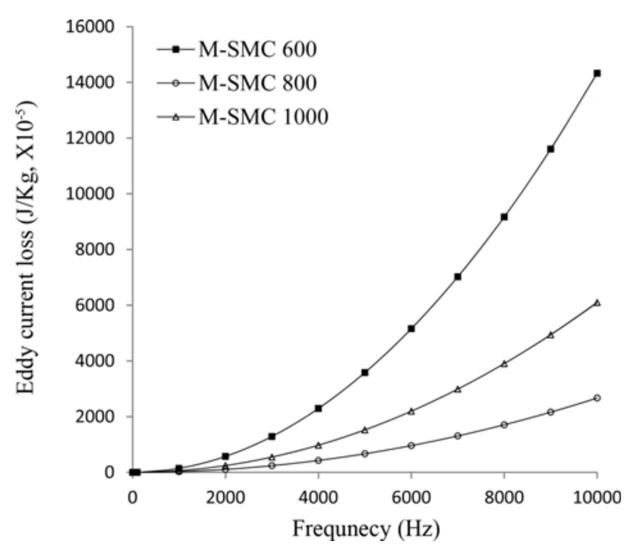

Fig. 7. Effect of frequency on Eddy Current Loss of toroid cores.

4.4.1 Electrical properties of M-SMC

It is observed that electrical properties such as inductance and permeability are relatively high at 600°C than other temperatures of curing. It implies that these properties are least affected by the dislocation density and number of

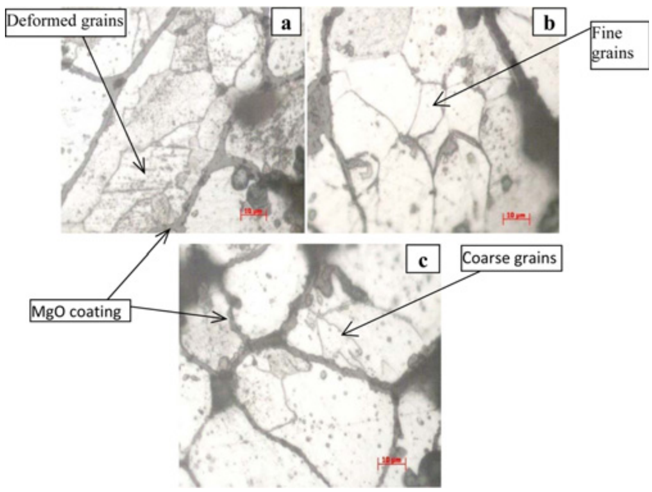

Fig. 8. Effect of curing temperature on coating thickness; Optical Micrograph (a) M-SMC cured at 600°C for 30 min showing crystalline grain; (b) M-SMC cured at 800°C for 30 min showing fine grains; (c) M-SMC cured at 1000°C for 30 min showing coarse grains.

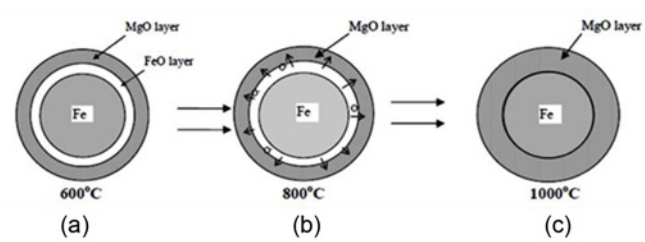

Fig. 9. Schematic model illustrating oxygen ion transfer from inner core to outer core (A) particle cured at 600°C, (B) intermediate stage at 800°C, (C) particle cured at 1000°C.

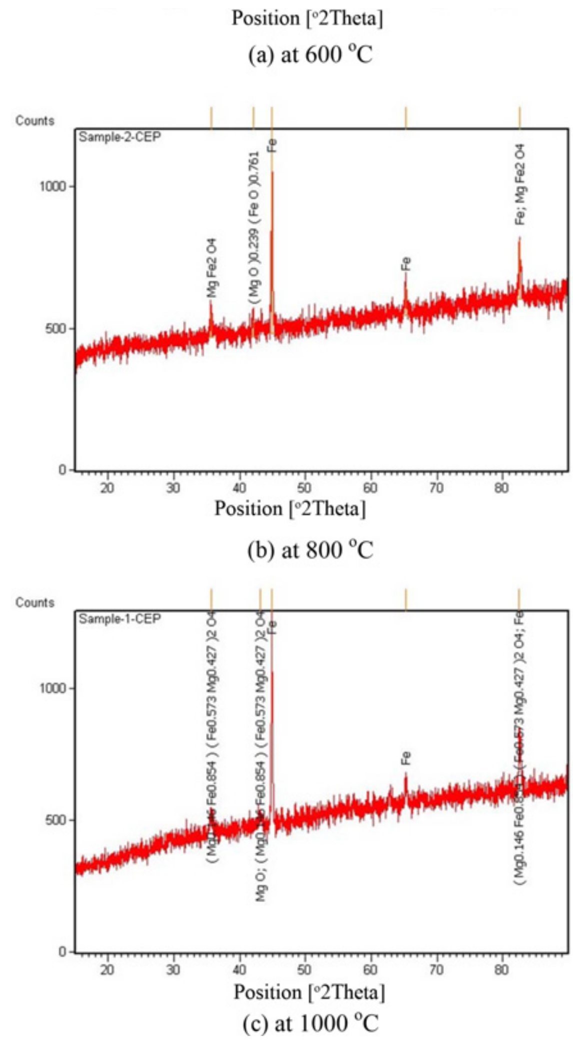

Fig. 10. XRD analysis of toroid core cured at (a) 600°C, (b) 800°C, (c) 1000°C.

ferrite grains irrespective of their size and shape. The deterioration in properties is evident at 800°C and 1000°C which is believed to occur due to thinning down of the coating thickness as a result of variation in molecular concentration with increasing curing temperature as evident in Fig. 8. It may be noted that the decrease in coating thickness might be due to oxygen iron transfer from inner subsurface to outer surface in accordance to the Arrhenius law,[9] which is schematically explained in Fig. 9. Figures 10(a), 10(b) and 10(c) show the XRD analysis of M-SMC toroid core cured at 600°C, 800°C and 1000°C respectively. It was found that Mg fraction in MgO-FeO compound decreased from 0.64 to 0.239 (i.e. percentage of cations in ionic bonding) with increase in temperature. Thus at 600°C, MgO-FeO compound becomes nonstochiometric showing dominance of covalent bond which in turn helps in more conduction.[10] It was found that at 800°C, MgO-FeO compound become stoichiometric showing dominance of ionic bond and hence electrical properties are decreasing with increasing temperature. Therefore, M-SMC core cured at 600°C shows the improved electrical properties than the other cores.

4.4.2 Magnetic properties of M-SMC

M-SMC core cured at 800°C shows decrease in Coercivity but it increases beyond 800°C. M-SMC core cured at 1000°C has impaired the magnetic properties. It is well studied that at higher temperature, ferrite grain starts growing at the expense of small sub grains such that domain wall motion gets disturbed and results in increasing Coercivity and also Retentivity which in turn increases the hysteresis loss. The possible microstructural development behind variation in magnetic properties is explained schematically in Fig. 11. It is observed that mechanical deformation gives remarkable influence on the microstructure. It means if cold work structure is heated above the recrystallization temperature, new strain free grains are formed. There are four stages proposed to explain the phenomena. Firstly coated particles

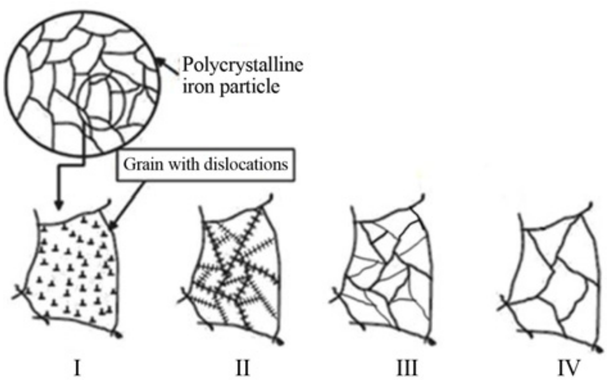

Fig. 11. Schematic model explaining morphological changes in ferrite grains.

are cold compacted which induces internal stresses that results in generation of dislocations (stage I). Stage II shows the rearrangement of the dislocations so as to form sub grains/low angle boundaries. Thus increase in curing temperature changes sub grain boundaries to fine grain structure (stage III) which may be closure to structure that has obtained in M-SMC cured at 800°C. If such structure is held at high temperature, say 1000°C, the subgrain structure may turn to coarse grain structure (stage IV) that can offer more resistance to domain wall motion and hence it may cause increase in Coercivity and loss in other properties. It was observed that eddy current loss is minimal for M-SMC cured at 800°C than that cured at 600°C and 1000°C (Table 3).

5. CONCLUSIONS

Magnesium compound coated iron powder was successfully compacted and cured at varied temperatures in the form of toroid cores. From the foregoing analysis, the following conclusions could be drawn –

(a) Inductance, Initial Permeability and Relative Permeability of M-SMC 600 core is high by 47.7%, 47% and 48.9% respectively with reference to M-SMC 800 and by 54.8%, 58.8%, 57.8% with reference to M-SMC 1000.

(b) Significant reduction in Coercivity, Retentivity, Hysteresis loss and Eddy current loss were found to be at 800°C curing temperature. At this temperature, polygonal fine ferrite grain structure was observed (Fig. 8b). It helps in reducing the resistance to domain wall motion and hence the magnetic properties were improved.

(c) Electrical properties of M-SMC toroid cores improved at 600°C than at other curing temperatures whereas magnetic properties exhibit improvement at 800°C in M-SMC. This variation in response may be attributed to magnetic domain wall motion in individual grains. It means that magnetic properties depend on microstructural features of individual iron particles and the coating thickness and electrical properties depend on nature of bonding.

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