Iron-based powder solutions for soft magnetic composite applications

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Abstract

The recent acceleration in interest and market demand in hybrid and fully electric vehicle systems has brought new opportunities to the PM market for use of Soft Magnetic Composite (SMC) materials. As greater acceptance of these materials in both the industrial and automotive market continues, products which serve specific industry needs are being developed and standardized for use. The success of SMC powders for these specific applications is based on three important inputs – base iron, insulating coating and lubricant. Thermal processing of the SMC compact also plays a critical role, as material deformation behavior has a direct impact on magnetic properties. These factors and their influence on final properties will be discussed.

Main text

Introduction

Soft magnetic composite materials consist of ferromagnetic material and insulation layers between the ferromagnetic material. For powder metallurgy SMC, the material also contains a lubricant to aid in compaction and ejection. For the purpose of this paper, the ferromagnetic material is commercially pure iron and the insulation layer is applied to the surface of each iron powder particle. Compacting iron powder premixes into a component involves high pressure and deformation of the material. Deformation has a negative effect on the magnetic performance. As an example, the permeability of cured SMC compacts is about 5 to 7 times smaller than a fully sintered, fully recrystallized sample at the same density. How the material compacts and deforms plays an important role in the resulting magnetic properties.

As density has an outsized role in the magnetic properties, compaction conditions to boost green density must be considered. Elevated temperature compaction has been used for decades to increase density of PM components [1,2]. The benefit of warm or warm-die compaction is based on improved lubricant behavior at the operating temperature as well as changes in the flow stress of iron. Tanaka teaches that the yield stress of pure iron is strongly dependent on temperature from 0° to 350° K, more so than Fe-Ni and Fe-Si alloys [3]. The yield strength of iron is roughly half at 340° K (67 °C) as compared with 300 °K (27 °C).



Figure 1: The effect of temperature on the yield stress of Fe, Fe-Ni and Fe-Si [3].

When it comes to magnetic properties, saturation (B_{max}) is a strong function of density. Additionally, maximum permeability often increases with density but is dependent on process conditions. It has been shown that increases in compaction temperature, resulting in higher density, has increased permeability [4]. Interestingly, permeability is not simply an effect of density. In a study where samples of the same composition were compacted to a constant density, samples compacted at lower pressure and higher die temperature resulted in better permeability than the higher pressure, room temperature condition [5]. An important input is the lubricant, as some lubricants work better at different temperatures. Lefebvre showed that lubricant type affected density, which in turn affected permeability [6], whereas Jansson surprisingly showed a 50% improvement in permeability with one lubricant compared with 2 others at nominally the same density [7]. The effect of compaction temperature and lubricant is clearly not fully understood in SMC materials, and the objective of this work is to add further knowledge within this topic.

Experimental Procedure

Commercially pure iron powder Ancorsteel 1000C was used as the base powder for the study. Four different coating/lubricant variants were tested, Table 1. The coating is a common insulation coating used in the industry, the die wall spray was zinc stearate and the 0.4% admixed lubricant was a combination of lubricants capable of spanning the compaction temperature range.

Designation	Coating	Lubricant
DW	No	Die wall only
Coat + DW	Yes	Die wall only
Lubricant	No	Admixed lubricant
Coat+Lube	Yes	Admixed lubricant

Table 1. Lubricant and coating conditions

Green strength bars and magnetic toroid samples were pressed to a density of 7.35 ± 0.01 g/cm³ over a range of compaction temperatures, from nominally 0 °C to 175 °C, in 25 °C increments. A select number of bars were tested in the green state while the majority of samples were cured at 500 °C for 1 hour prior to testing. The cured density was measured for all samples and resistivity testing was performed. Toroids were then wrapped in a series of insulating tape, copper wire, insulating tape and copper wire for magnetic testing. The DC properties were measured at a flux density of 1 Tesla. Select bars were metallographically cross-sectioned, polished and etched to reveal the microstructure. Micro-indentation hardness measurements at 25 gf were taken in both areas that appeared heavily deformed and those that appeared undeformed.

Results

The bars pressed to density required different pressures with the different coating / lubricant / temperature combinations, Figure 2. The lowest compaction temperature required the highest compaction pressure in all cases with a rapid decrease in pressure between 4° and 125° C. Admixed lubricant samples exhibited the largest change in compressibility between 4° and 100° C. This is not unexpected as the admixed lubricant chosen for the study had to perform at the highest temperature and therefore was not ideal at the lowest temperature. The impact of admixed lubricant becomes negligible at 100° C and higher, so it is therefore expected that the temperature range of 75 to 100 °C is best for the admixed lubricant. Relative to the admixed lubricant effect, the effect of coating was minor. A slight increase in pressure was required when coating was present, and this is most easily observed at the highest temperatures. In addition to the coating and admixed lubricant effects, there is an underlying improvement in the material compressibility up to 125 °C. With the die wall only sample, the required compaction pressure drops roughly 100 MPa. Beyond 125 °C there is little improvement. This trend appears to align with the yield strength results of Tanaka [3] and suggests that the flow stress or deformation behavior of commercially pure iron continues to change over the industrially relevant temperature range.



Figure 2: Effect of temperature on the compaction pressure required to reach 7.35 g/cm³ green density.

The maximum permeability is strongly affected by two factors: the compaction temperature and the amount and distribution of non-ferrous material in the compact. The permeability is clearly different between the four materials as seen in Figure 3. In the case of iron plus die wall lubricant only (DW), the permeability is much higher than the other materials as non-ferrous material is not present between particles. Non-ferrous material includes porosity and the coating on particles. Application of a thin coating on the particles (Coat + DW) greatly reduces the permeability due to the effective air gap that is uniformly present between particles. The addition of only lubricant (Lubricant) results in a similar drop in permeability. It is interesting that the lubricant only sample still has a substantially lower permeability after curing where all the lubricant has been removed. The cured compact appears to have a memory of where the lubricant was located in between particles, so there remains an air gap that lowers permeability. Not surprisingly, the material with the most amount of non-ferrous material (Coat+Lube) has the lowest permeability.



Figure 3: Maximum permeability at 1 Tesla versus compaction temperature for all samples.

The compaction temperature has a strong effect on relative permeability for all samples, although the effect is somewhat muted for the coated sample with die wall only. There is a steep increase in

permeability up to 100 °C followed by a plateau or slight degradation as temperature increased further. From 4 to 100 °C, the percentage increase in permeability for the DW, Coat + DW, Lubricant, and Coat+Lube samples is 55, 20, 47, and 41, respectively. It is interesting that the DW only sample experienced the greatest increase, whereas the coated sample the least and samples with admixed lubricant were in between. This differs significantly to the required compaction pressure where admixed lubricant had the largest effect.

Toroids that were pressed at 21 °C and 75 °C were also tested in the green state. The permeability was significantly reduced compared with the cured condition. For the 21 °C samples, permeability was reduced to roughly 71% of the cured result. For the 75 °C samples, most of the green samples had a permeability roughly 66% that of the cured material. For the DW sample, this value decreased to 55% that of the cured. In addition to an increase in compact strength, curing is known to improve magnetic properties and these results are consistent with these earlier findings. It is interesting though that the permeability ratio of cured vs green for the DW sample at 75 °C was different than the other samples.

The curves for core loss are somewhat similar to that of permeability in that the order of samples and scale of response is consistent, Figure 4. The die wall (DW) sample had the worst core loss since there was no barrier between particles to prevent electrical current passing from particle to particle. It is not clear why the DW sample exhibits a temperature effect; perhaps the reduced flow stress allows particles to join more intimately together up to 100 °C. Oxygen pickup on the powder prior to compaction could explain the slight drop off at higher temperatures. Better contact between particles would also explain the increasing permeability results in Figure 3. The lubricant only sample shifted higher in core loss as the temperature exceeded 50 °C. This can be attributed to a change in lubricant behavior since the admixed material can move more easily from between iron particles as temperature increases. The coated samples showed little effect of compaction temperature on core loss as the coating remained in place and was an effective electrical insulator.

Core loss was significantly lower for the green samples of DW, Coat+DW and Lubricant compared with the cured samples, with the largest change for the DW samples. Since eddy current loss contributes to core loss and the green samples have less direct electrical connection compared with cured samples, it is logical that the core loss would decrease. This is not true, however, for the Coat+Lube sample. Here there is sufficient insulation in the cured state, so the core loss does not improve in the green state. In fact, at lower frequency, the core loss of the cured sample is superior to that of the green sample due to an improved coercivity.



Figure 4: 400 Hz core loss at 1 Tesla versus compaction temperature for all samples.

The as-polished microstructure of the cured samples was evaluated and the two extremes (DW and Coat+Lube) are shown in Figure 5. Evidence of the coating is clearly visible in Figure 5b, whereas

only a few boundaries are visible in the uncoated DW sample. Upon etching, the particle boundaries become more easily visible in both samples along with grain boundaries within particles, Figure 6. An evaluation of the microstructure showed that the material does not deform uniformly. Certain particles, presumably due to different shapes and grain orientations, deform more heavily than others. Therefore micro-indentation hardness was taken in both regions for the DW and Coat+Lube samples at 4 °C and 100 °C compaction.

It was found that the areas that appeared more deformed had higher hardness, Table 2. It follows that greater deformation would increase work hardening in the material, and since deformation reduces magnetic performance, it is important to understand how the material accommodates the deformation. Secondly, the Coat+Lube sample had lower overall hardness than die wall only sample. Internal lubrication likely caused better particle rearrangement and possibly less work hardening during deformation. Finally, it was observed that the 4 °C samples had surprisingly lower hardness than the 100 °C compaction samples. Given the higher permeability of the 100 °C samples, it was expected that the hardness would be lower in the 100 °C samples. It is unknown why this behavior was observed.



Figure 5: As-polished microstructure of (a) DW (uncoated) and (b) Coat+Lube compacted at 100 °C.



Figure 6: Etched microstructure of (a) DW (uncoated) and (b) Coat+Lube compacted at 100 °C.

Sample	Smooth Area	Visibly Deformed Area
DW, 4 °C	169	195
DW, 100 °C	173	197
Coat+Lube, 4 °C	140	185
Coat+Lube, 100 °C	158	193

Table 2. Average micro-indentation hardness results (HV₂₅ gf) for select samples.

Conclusion

Compaction temperature is an important variable in the production of SMC compacts. Increased temperature reduces the required compaction pressure to achieve a green density for all materials studied. Beyond that, there is a clear influence on the permeability of the material. Materials compacted at lower temperatures have lower permeabilities, with increasing results as temperature rises until roughly 100 °C, after which no further benefit is observed. The change in compressibility and permeability of the sample containing only iron (DW) is a function of the iron yield behavior with changing temperature. Additionally, coating and admixed lubricant reduce the maximum permeability that can be achieved by increasing the effective air gap between particles. Core loss was also found to increase with temperature in the uncoated samples, while the coated samples were largely unaffected. Finally, the deformation in the compacts is not uniform and the amount of work-hardening is reduced with the addition of coating and admixed lubricant.

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