Porosity and Magnetic Losses in Soft Magnetic Composites

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Abstract. The paper investigates the effect of porosity on the magnetic properties of Soft Magnetic Composites produced by constant compacting conditions, using niobium powders was a non-magnetic phase. Losses increased with the total non-magnetic volume fraction. Annealing under a nitrogen-hydrogen atmosphere decreased losses (due to stress relieving) despite a permeability decrease brought by a Nb phase transformation expansion.

Introduction

Soft Magnetic Composites (SMC) are gaining marketshare as higher frequencies are been used in electrical motors. When comparing SMC with electrical steel sheets, SMC show lower Total Magnetic Losses at high frequency due to the iron particle insulation, which lower the eddy current loss component [1]. It should be mentioned that there are some uncertainties about the estimation of classical and excess losses [2]. At lower frequencies, the magnetic behaviour of the Soft Magnetic Composites is severely impaired by their Hysteresis Losses, as they are 5 to 10 times larger than those of non-oriented electrical steel sheets. As a consequence, the frequency break even point is still too high for many applications. Decreasing hysteresis loss is an important objective in SMC development.

The factors that are usually considered to influence the hysteresis behaviour of electrical steels are grain size, inclusion content, texture and residual stresses. In the case of SMCs, the effect of plastic deformation when the parts are pressed has to be taken in consideration. Preliminary results, showing that hysteresis losses of an iron powder pressed to 900 MPa were about 400 J/m^3 (at 0.5T) whereas the same powder, polymer bonded and pressed to 200 MPa (presumably with less plastic deformation), presented 500 J/m^3 , indicated that porosity (or the non-magnetic volume fraction) is also a factor to be investigated.

In powder metallurgy processing of mechanically soft materials, such as iron powders, porosity is normally controlled by the compaction pressure. This process variable is inconvenient for the proposed investigation, as changing the compaction pressure would change the amount of plastic deformation in the particles, which would also affect the hysteresis loss. To avoid such interference, and supposing that pores are just non-magnetic regions inside the material, different volume fraction of “pores” could be produced by adding non-magnetic particles to the iron powder. Fine Niobium powder was used for that purpose. No effort was made to electrically isolate the particles, at the present stage.

The effect of porosity decreasing magnetic permeability is well known [3]. This effect is usually attributed to an interparticle gap effect, as a source of demagnetizing field. The magnetization curve is “sheared” by the demagnetizing field of porosity as it is by gaps in the magnetic circuit. The “gap effect” also shears the hysteresis curve but it should not change the hysteresis area, unless effects other than a demagnetizing field are at work.

As the effect of plastic deformation is superimposed in the intended investigation, it was considered interesting to try to separate those effects by stress relieving. At the same time, the possible effect of heat treatments on recrystallization and grain growth should also be investigated.

Experimental procedures

Iron powder was mixed with different quantities of Niobium powder, as shown in Table 1, to produce samples with different “non-magnetic volume fraction”. The iron powder was classified and the fraction between sieves mesh 70 and 120 were used, so the average iron particle is 150 μm . This rather large particle size was chosen aiming at favouring larger grain size. Niobium powder with average particle size of 2 μm, produced by decrepitating and milling, was used. It is much smaller than the iron particles, so it could be well distributed around them. Aiming at powders with different niobium and iron Volume fractions (X^{Fe}_{V}) , six different mass fractions were mixed (X^{Fe}_{m}) were mixed, from 0 to 25 volume%. They were all compacted at 900MPa, so they should all show high green strength and they were all subjected to the same plastic deformation. Rings of 50 mm external diameter and 42 mm internal diameter were produced.

A set of compacted samples was submitted to a heat treatment at 800° C for 2h, under a 90%nitrogen + 10% hydrogen atmosphere, to eliminate the effect of compaction-induced plastic deformation of iron particles on the magnetic properties.

Ring Density was measured using the geometrical method. Niobium, iron and pore volume fraction was calculated by the equations below.

$$X_V^{Fe} = \frac{\frac{m_{Fe}}{d_{Fe}}}{\frac{m_{Nb}}{d_{Nb}} + \frac{m_{Fe}}{d_{Fe}}} \qquad X_m^{Fe} = \frac{m_{Fe}}{m_{Fe} + m_{Nb}}$$

$$Fe_{volume\%} = \frac{m_{sample} * X_m^{Fe} * 100}{d_{Fe} * V_{sample}}$$

$$Nb_{volume\%} = \frac{m_{sample} * X_m^{Nb} * 100}{d_{Nb} * V_{sample}}$$

$$Poro_{volume\%} = 100 - Fe_{volume\%} - Nb_{volume\%}$$

Nb+porosity (the non-magnetic phases) volume fraction on the heat treated samples was calculated by applying a 64 point grid on photographs taken in scanning electron microscope using secondary electron contrast.

Rings samples were coiled with 270 turns in the primary coil and 280 turns in the secondary coil. Quasi-static magnetic properties were measured using a MF-3D fluxmeter. Due to electric current supply limitations, maximum induction had to be confined to 0.5T. Each measurement is the average of 3 hysteresis curves.

We have chosen the “constant magnetic induction on the iron volume fraction” criterion to compare the hysteresis area of samples with different porosity, or, more precisely, with different “non-magnetic volume fraction”. A “constant magnetic induction on the sample”, which is standard procedure for comparing losses on steel sheets, would mean different magnetic polarization in the iron fraction, as porosity increased.

Results and discussion

Table 1 shows the results of density measurements and calculated volume fraction. Iron volume fraction ranged from 88 to 64%. As a comparison, commercial SMCs have iron volume fraction around 91%. Porosity varied, as Nb volume fraction increased. Annealing induced a slight densification in the 0%Nb sample and expansion on all Nb containing samples. No microstructural evidence of Fe-Nb intermetallic phase formation was found, neither any hardening (Vickers hardness values varied from 86 to 100, for 50g). Nitrogen and hydrogen pickup (respectively 3700 and 900 ppm) indicates that niobium reacted with the atmosphere leading to significant expansion that explains the expansion of the rings.

Microstructure of the heat treated samples was analysed. Figure 1 shows the microstructure of sample 15T, where Nb particle distribution and pores can be seen. Based on such secondary electron images, the “experimental non-magnetic volume fraction” was determined and shown in Table 1. Its values are larger than the calculated Nb volume fractions, but smaller than the Fe+Nb volume fractions, calculated according to equations 2 and 3. The density decrease in annealing indicated that the Nb particles underwent a significant expansion during annealing

Table 1. Phase volume fractions

Sample ID	condition	density	Nb	Pores	Fe	Non-mag (calc)	Non-mag (exp)
		g/cm^3	%	%	%	%	%
0	pressed	6,96	0	11,6	88,4	11,6	-
5		6,77	4,3	14,4	81,4	18,6	-
10		7,07	8,8	11,0	80,1	19,9	-
15		7,18	13,5	10,0	76,5	23,5	-
20		7,20	17,9	10,1	71,9	28,1	-
25		6,93	21,5	13,9	64,6	35,4	-
0T	annealed	7,12	0	9,5	90,5	9,5	-
5T		6,58	4,2	16,8	79,1	20,9	6,3
10T		6,76	8,5	14,9	76,6	23,4	15,7
15T		6,88	13,0	13,7	73,4	26,6	20,6
20T		6,63	16,6	17,3	66,2	33,8	22,8
25T		6,49	20,2	19,3	60,5	39,5	30,3

As exemplified by Figure 1, Nb addition did not create a perfect ring of Nb particles around each Fe particle, but most of the Fe particles are isolated from each other, either by Nb white islands or by dark intergranular strings which must be porosity plus dislodged particle sites. Figure 2 and 3 show that the 800^o C heat treatment did not lead to grain enlargement neither by small deformation recrystallization nor by normal grain growth. The difficulties in getting grain size enlargement in those materials will impair the magnetic properties of SMCs. It was expected that at least some regions of the samples would have got strained on the order of ε = 0.05, which should lead to large grain sizes even with a low annealing temperature. Nevertheless, no sign of such behaviour was found.

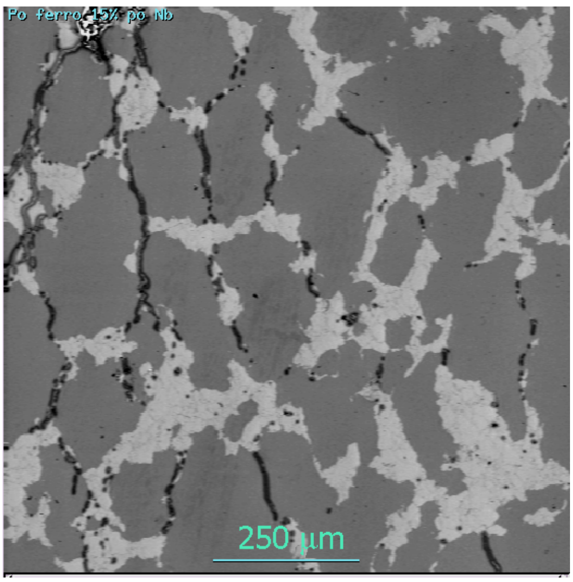

Figure 1. Microstructure of sample 15T. Secondary electrons image in SEM. White areas are Nb (+N). Fe particles are predominantly isolated.

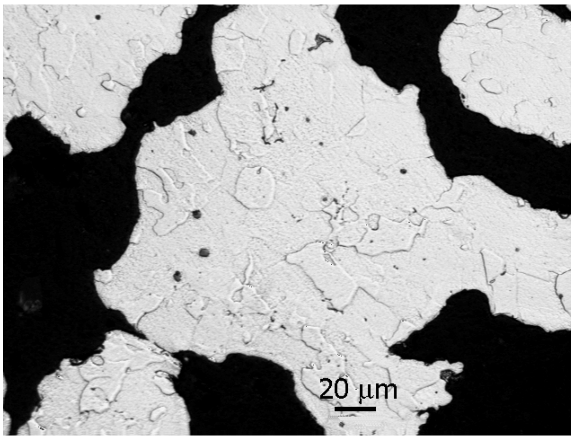

Figure 2: Iron particles etched with nital.

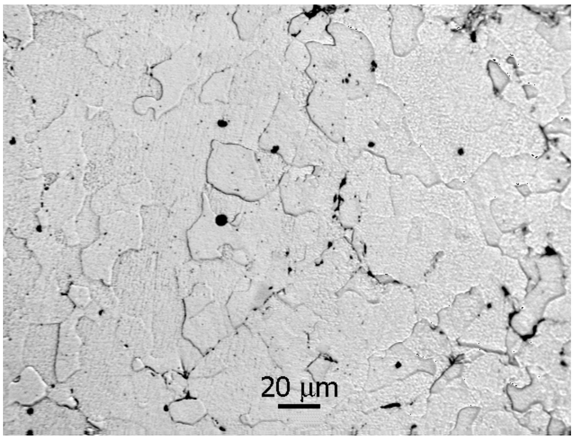

Figure 3: Sample 0T.

Figures 4 and 5 show the hysteresis curves for the as-pressed and for the annealed samples. As expected, larger values of H are necessary to reach 0.5T, as the non-magnetic volume fraction increased. Unexpected was the observation that annealing deteriorated permeability. Figure 6 shows that permeability correlates rather well with the non-magnetic volume fraction, and the deleterious effect of annealing should be related to the Nb phase expansion by reaction with the atmosphere. Figure 7 shows that hysteresis losses of the annealed rings increase almost linearly with the non-magnetic volume fraction and the extrapolation to zero leads to a value that is very close to the value of a decarburized steel sheet with small grain size. The as-pressed rings show a much higher value and smaller density dependence. It was not possible to confirm the observation of Adler et ali [3] that the fraction of coercivity due to pores is a function of specific pore surface, more than pore volume fraction.

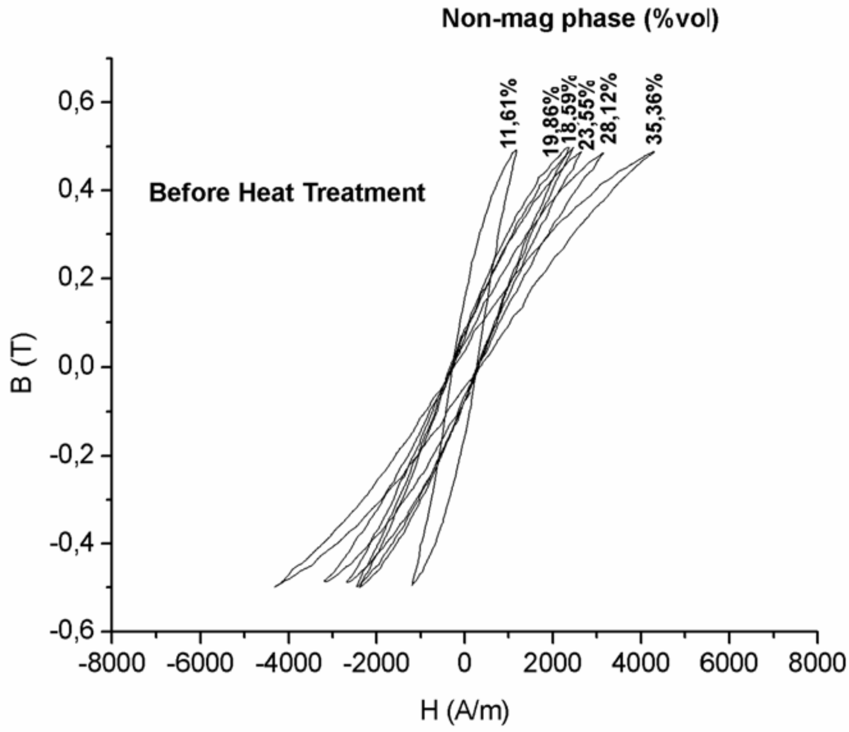

Figure 4. Quasi-static hysteresis curves for as-pressed samples

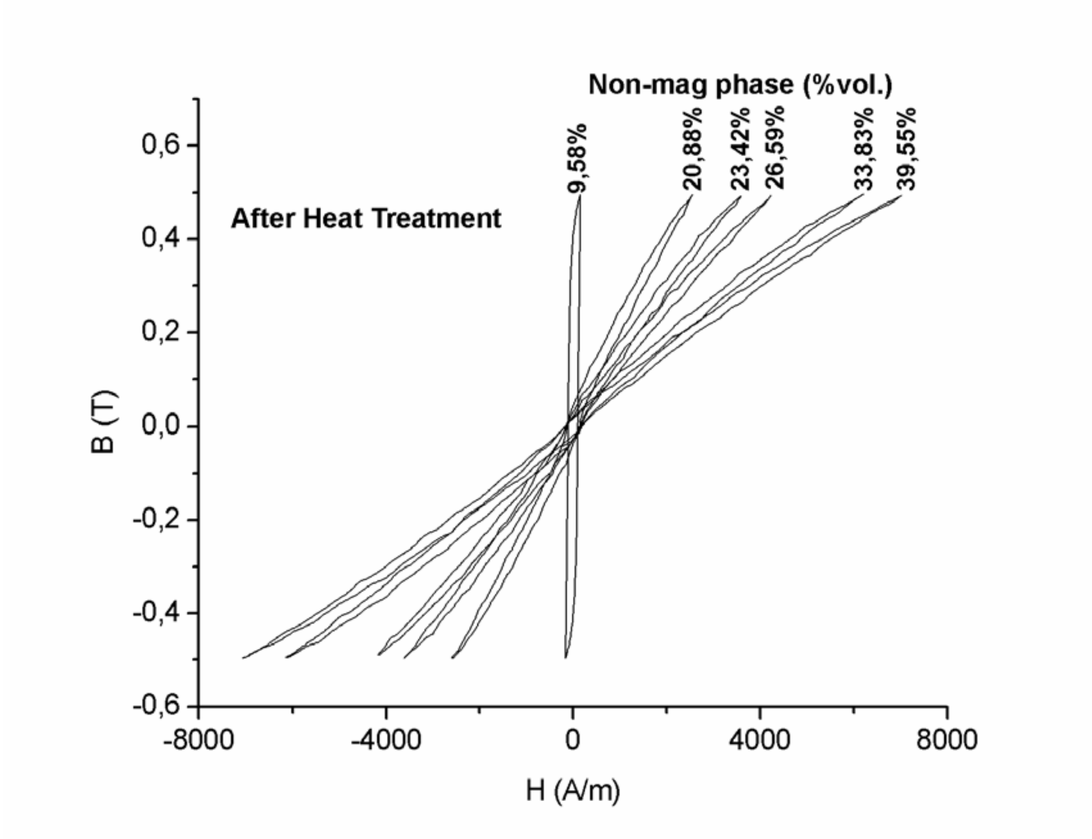

Figure 5. Quasi-static hysteresis curves for annealed samples

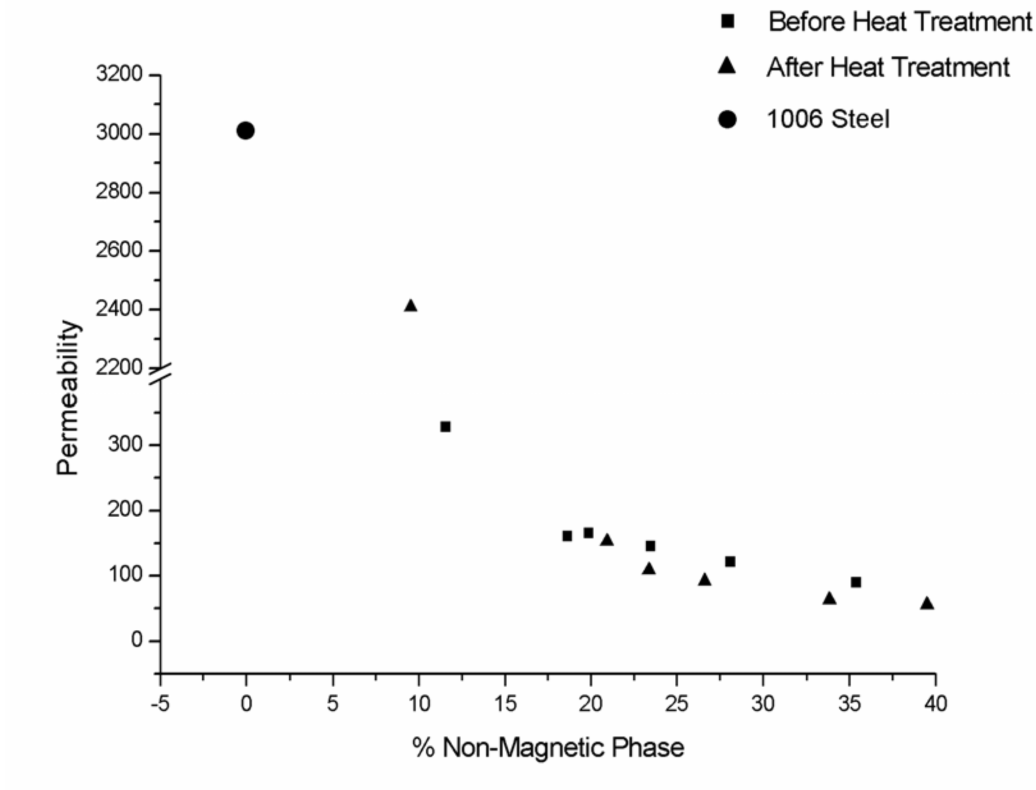

Figure 6. Effect of non-magnetic phase volume fraction on on magnetic permeability at 0.5T

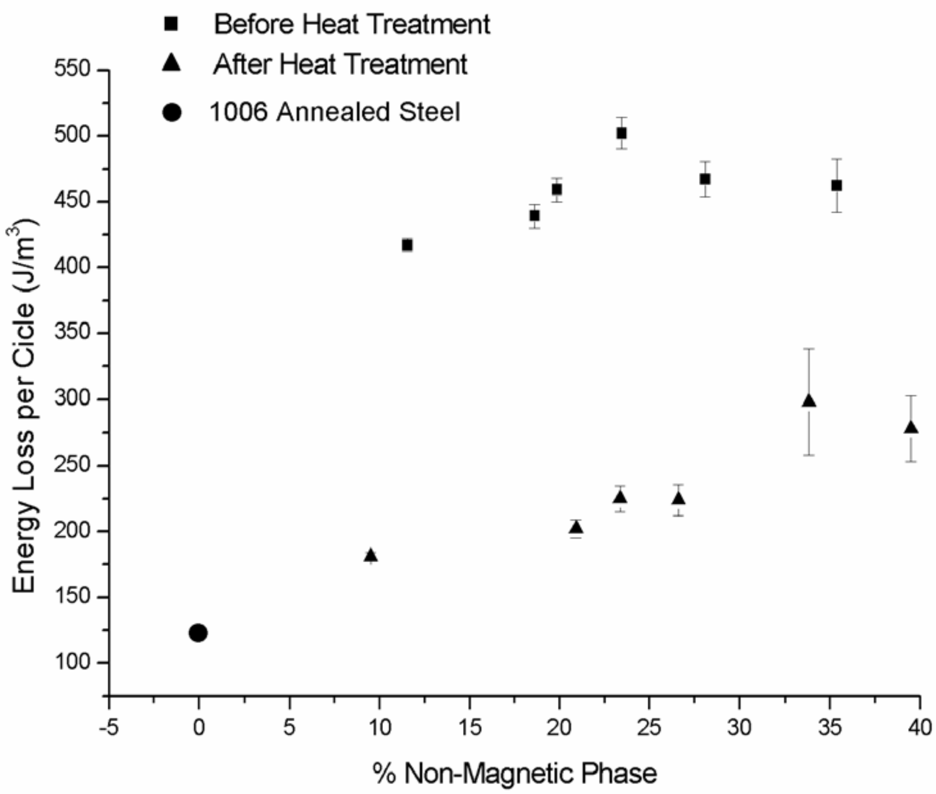

Figure 7. Effect of non-magnetic phase volume fraction energy loss per cycle at 0.5T

Conclusions

In the as-pressed state, increasing the non-magnetic volume fraction from 9 to 35%, for constant compacting pressure, led to small increase in hysteresis losses at 0.5T, using the "constant magnetic induction on the iron volume fraction" criterion. Annealing at 800^o C decreased significantly the hysteresis losses of all samples and showed a more significant trend of increasing losses with increasing non-magnetic volume phase. Its extrapolation to zero pores leads to a value that is close to the one of an annealed decarburized steel with small grain size . No grain growth was noted, so the effect of annealing must have been only stress relieving.

Permeability decreased with increasing non-magnetic volume fraction, but a non predicted effect was the significant decrease of permeability with annealing. A small density decrease and microstructural evidence of an increase in the inter-particle separation suggested that the transformation of Nb particles into hydrides, during annealing, is the main cause for the permeability decrease.

Acknowledgements

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