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Effects of processing upon the properties of soft magnetic composites for low loss applications

I. GILBERT*, S. BULL, T. EVANS
Department of Mechanical, Materials and Manufacturing Engineering, University of Newcastle-upon-Tyne, Newcastle upon Tyne NE1 7RU, UK
E-mail: gilbert_ian@hotmail.com

A. JACK
Department of Electrical and Electronic Engineering, University of Newcastle-upon-Tyne, Newcastle upon Tyne NE1 7RU, UK

D. STEPHENSON, A. DE SA
Department of Mechanical, Materials and Manufacturing Engineering, University of Newcastle-upon-Tyne, Newcastle upon Tyne NE1 7RU, UK

We have investigated the effects of processing variables and methods on the density, structure and magnetic properties of soft magnetic composite (SMC) materials primarily for use in low loss applications. Two commercially available SMC materials from Höganäs AB, Sweden were used throughout the study. The materials differed only in terms of the solid state lubricant incorporated in the powder. This was sufficient to affect the density of pressed samples. Both monotonic and cyclic pressing regimes were used to compact the materials, with pressures sufficiently large to control the bulk mechanical properties of the samples produced (Hardness typically 1.6 GPa). Samples were heat-treated to both relieve any stresses imposed during pressing, and to control the final composition through oxidation or non-oxidation processes. Magnetic properties were observed using a vibrating sample magnetometer and Barkhausen noise analyser and electrical properties determined using a micro-ohmmeter. Both magnetic and electrical properties were observed to be strongly dependent on sample density and heat treatment. © *2004 Kluwer Academic Publishers*

1. Introduction

Soft magnetic composites (SMC) are not new, but until recently their properties have been poor compared to those of laminate materials. 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) but with high electrical resistivity. Unlike conventional laminated materials the resistivity is isotropic and this reduces the constraints on design imposed by the lamination approach [1, 2]. SMC is made from iron powder coated with an organic binder. The distinct advantage of the powder is that it can be pressed to a net shape with tight tolerances. During the pressing process, the iron grains are strain hardened, which has a deleterious effect on the magnetic properties. These can be restored by warm compaction or annealing, but this is often at the expense of the electrical resistivity. As such, low loss applications require the optimisation of both the pressing and heat treating of the SMC materials so as to minimise the contribution to losses from both magnetic and electrical sources.

The object of this study was to investigate the effects of processing variables and methods on the density, structure and magnetic properties of SMC.

2. Experimental

Two commercially available (Höganäs AB, Sweden) raw materials were used: Somaloy 500 + 0.5% Kenolube (Ken); Somaloy 500 + 0.6% LB1 (LB1); differing only in the type and content of lubricant. Morphology and composition of the powders were characterised using scanning electron microscopy (CamScan S4-80DV with LaB_6 source) and EDX (eXL Link analytical) techniques.

Samples were pressed in 10 mm diameter cylindrical steel dies at a range of monotonic pressing pressures from 200 to 800 MPa, and also under cyclic conditions, with the maximum pressure varying from 200 to 600 MPa and stress amplitude such that a minimum stress of 6 MPa was maintained.

Samples were heat treated under different conditions based upon the Höganäs specifications (275°C for 1 h for LB1 and 500°C for 30 min for Ken).

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*Current address: Instituto de Ciencias de Materiales de Aragón, CSIC-Universidad de Zaragoza, 50009 Zaragoza, Spain.

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Density was determined both before and after heat treatment from weight and dimensional measurements.

Mechanical hardness was determined from both microhardness tests (Shimadzu microindenter, 500 gf load) and nanoindentation (Nanoindenter II^{TM}, 5 mN load). The latter traversing several grains during measurement to determine variations in hardness both within the grain and at grain boundaries.

The resistivity of the samples was measured using a 4-point Tinsley Ohmmeter on pressed specimens with cross-sectional area $3.3 \pm 0.1 \times 10^{-5}$ m^2.

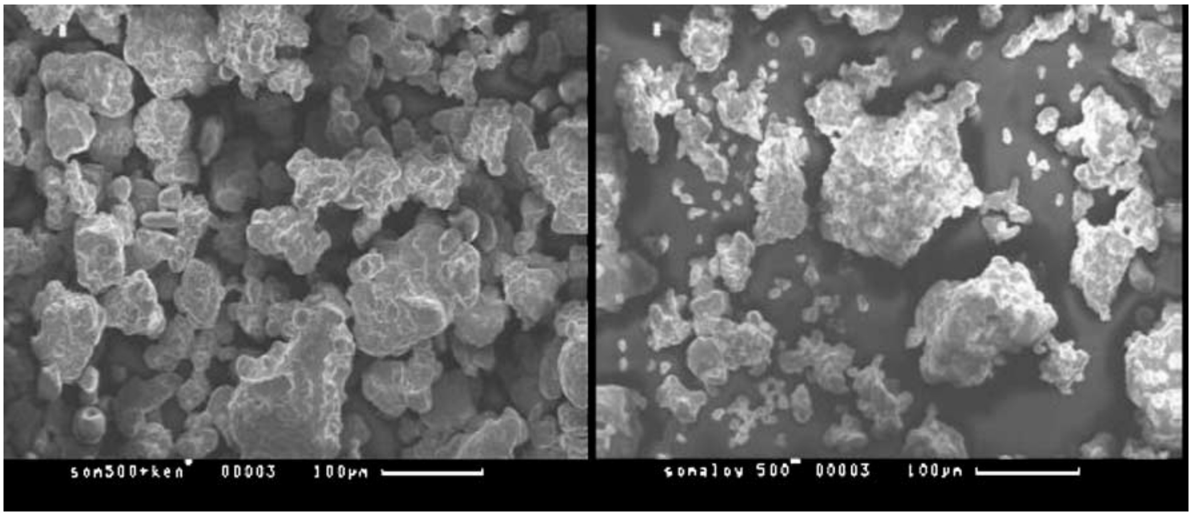
Rectangular sections (5 mm × 5 mm × 3 mm) were cut from the pressed cylinders and etched to remove any damage. These were used to measure magnetisation in an *in-house* built vibrating sample magnetometer (VSM) [3, 4] (Newport Instruments Type A magnet, SEMAS power supply DC 20V 25A max). Barkhausen analysis was carried out using a Stresstech AST μscan 500. An excitation frequency of 4 Hz and maximum field strength of approximately 6 kA/m was used. The Barkhausen noise signal was captured using a pick-up coil and was analysed using the Stresstech AST μ scan software.

X-ray diffraction using a mechanically (Philips PW1050) controlled Philips 1730 with monochromated Cu K$_\alpha$ radiation was performed to determine phases present after heat treatment.

3. Results and discussion

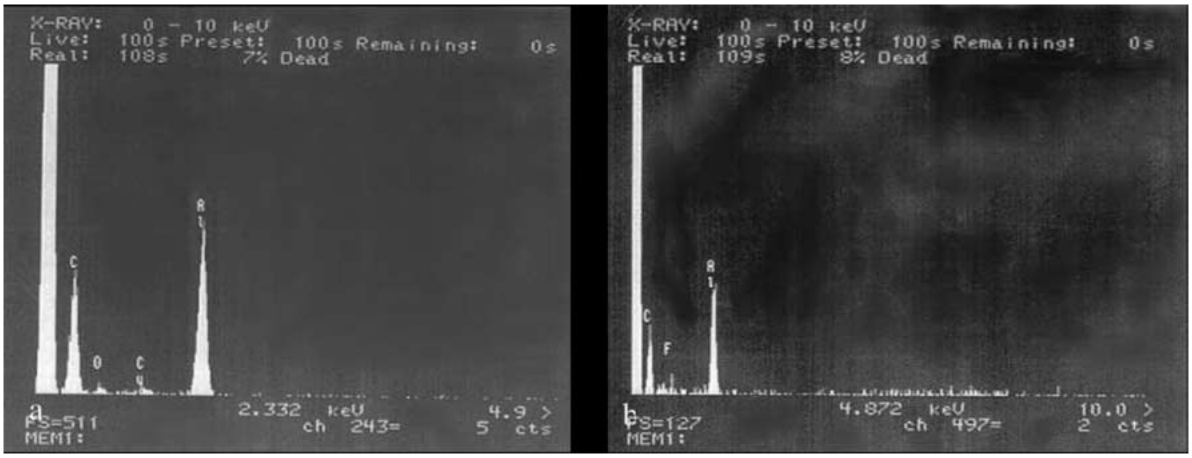
3.1. Raw materials

Powders examined in the SEM revealed a discrete distribution in particles sizes, ranging from 30 to 100 μm. Metallic particles in both powder types were irregularly shaped due to their method of production. In both cases the lubricant was present as discrete particles in the raw powder mix, with the Kenolube particles (15–25 μm) larger than the LB1 particles (5–10 μm) (Fig. 1). Using EDAX the lubricant particles were examined to reveal two different lubricants (Fig. 2). Ken samples exhibited peaks for both carbon and fluorine, inferring the lubricant is PTFE based. LB1 samples exhibited only a carbon peak, and so the lubricant is assumed to exist as a waxy carbonaceous material. The size, shape and different lubricants will effectively control the characteristics of the bulk sample once pressed. The aim with the pressing process is to produce a sample as dense as possible (reducing magnetic losses through areas of demagnetisation, i.e., porosity), while incurring as little damage as possible (again reducing magnetic losses),



(a) (b)

Figure 1 Scanning electron micrographs of Somaloy 500 with the lubricant: (a) Kenolube and (b) LB1.



(a) (b)

Figure 2 EDAX spectra for the two lubricants: (a) LB1 and (b) Kenolube.

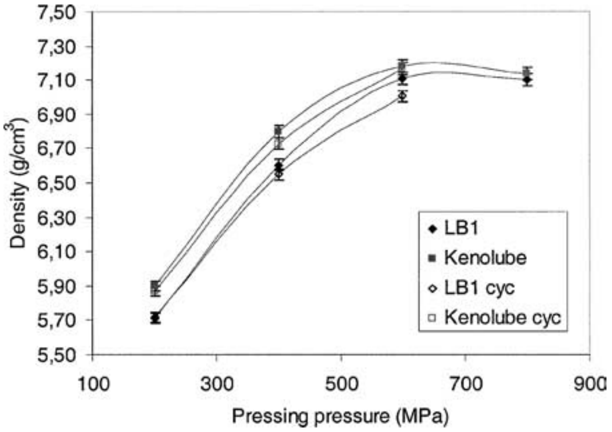


Figure 3 Density as a function of pressing pressure for both materials used, and using the two different pressing regimes of monotonic and cyclic loading.

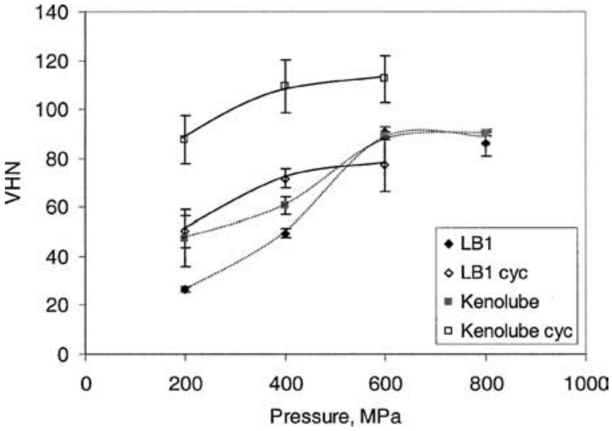


Figure 4 Vickers hardness of bulk specimens as a function of pressing pressure for both materials, using two different pressing regimes of monotonic and cyclic loading (curves are guides for the eye).

but trying to keep an insulating barrier between individual particles (reduce electrical losses).

3.2. Density

Density measurements exhibited a dependence on the type of lubricant used, as observed with differences between the two powder types (Fig. 3). Samples exhibited the expected behaviour [5] with an increase in pressing pressure resulting in an increase in density reaching a plateau between 600 and 800 MPa for the monotonically loaded samples. Samples pressed under cyclic conditions exhibited lower densities than those pressed under monotonic loading. Ken samples exhibited higher densities than LB1 samples under both loading conditions, a function of the better effectiveness of the PTFE-based lubricant. A high density was desirable in these samples, primarily because we are aiming to press to as near a net shape as possible and also because we require the magnetic and electric properties to be close to optimum as possible. A high density specimen will be magnetically superior because it will contain fewer pores. Porosity acts as areas of demagnetisation, reducing the saturation magnetisation. High densities can be achieved by compaction with high pressures. This however, can be detrimental to the final characteristics. The metal grains are work hardened during compaction, introducing dislocations into the sample, thus creating areas that can pin Bloch wall movement and increase energy losses. Subsequent heat treatments could be used to relieve some of these stresses and strains, or as in the present case, the degree of strain hardening might be limited by employing a cyclic pressing technique. There is a fine balance between trying to achieve high enough densities while keeping magnetic energy losses to a minimum.

3.3. Hardness measurements

Microhardness tests, which sample both the iron grains and porosity (Fig. 4), show an increasing hardness with pressing pressure reflecting the increase in density and work hardening in the grains. This was confirmed by nanoindentation testing of individual grains in polished samples where the hardness value is around 1.6 GPa compared to less than 0.7 GPa for the virgin powder. In order to achieve reliable test data, a layer approximately 5 μm thick was removed from the surface by electropolishing in a 2% perchloric acid-butoxyethanol mixture at 10 V dc.

3.4. Electrical characteristics

Resistivity measurements [6] were carried out on monotonically pressed samples as a function of heat treatment temperature and press lubricant (Fig. 5). The Ken samples exhibited a decrease in resistivity with increasing temperature, with the rate of decrease increasing between 350 and 400°C. The LB1 samples show an increase in resistivity up to 350°C followed by a sharp decrease above 350°C. After heat treatment in accordance with the Höganäs recommended schedule, there is a reduction in resistivity for both lubricants. The resistivity of the Kenolube samples is not affected by pressing pressure whereas the LB1 samples show a decrease with increasing pressure (Fig. 6).

3.5. Magnetic characteristics

Owing to the need to machine samples to shape, vibrating sample magnetometry (VSM) was only carried out on heat-treated material as non-heat-treated samples

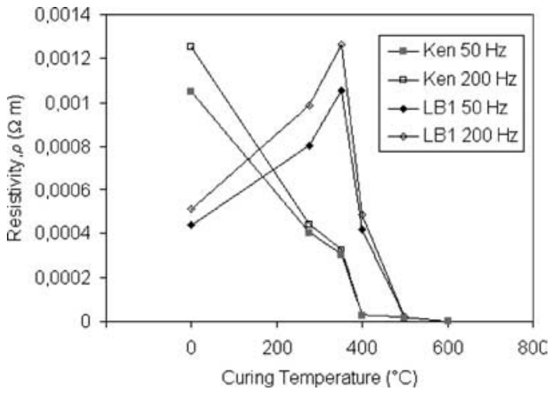


Figure 5 Resistivity of the two materials as a function of heat treatment. Measured at two different frequencies.

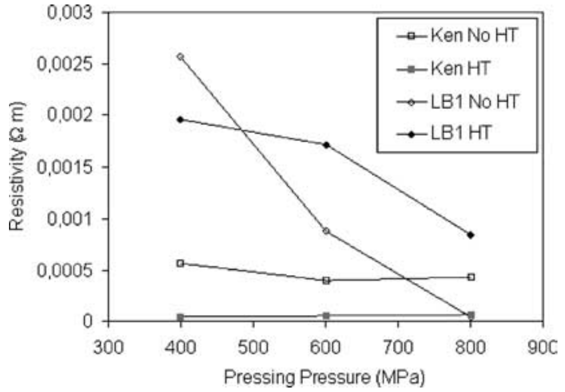


Figure 6 Resistivity of the two materials before and after heat treatment as a function of pressing pressure. Heat treatment was carried out in accordance with that recommended by the material manufacturer.

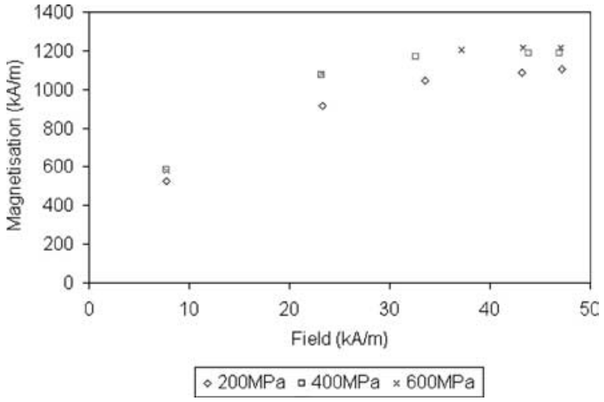


Figure 7 Magnetisation-Applied field measurements for a Somaloy500 + Kenolube material after pressing and heat treatment, showing the effects of pressing pressure.

were too brittle. The VSM technique is most useful in assessing saturation magnetisation (Fig. 7). The results demonstrate that M_s is related to density as anticipated. A maximum value of 1.6 kA/m was obtained. The maximum relative permeability obtained by this technique was 124. The width of the M-H loop measured using VSM was very small, corresponding to a low coercive field, H_c, and remanent magnetisation, M_r, as such it was difficult to discriminate between samples processed in different ways by using this technique.

The most successful method for characterising the magnetic changes caused by the SMC manufacturing process was Magnetic Barkhausen Noise (MBN) analysis [7–9]. The probe used in this test did not produce a high enough magnetic field to saturate the samples but when tested under identical conditions a reliable comparison between them could be made. An increase in pressing pressure gave an increase in the peak MBN signal for both monotonically loaded and cyclically loaded samples and the peak was higher for the cyclic loading case (Fig. 8) [10]. This is consistent with the earlier mechanical results indicating higher hardness for cyclically loaded material. There is thus no advantage in going to cyclically pressed material.

After heat treatment the peak MBN values decreased and the MBN peaks broadened (Fig. 9). The effect of heat treatment of the SMC on its magnetic properties was far more significant than any variations introduced by the pressing process. However, as well as improving the magnetic properties of the SMC it can reduce the electrical resistivity. This is deleterious for low loss applications, so the heat treatment process used will always be a compromise.

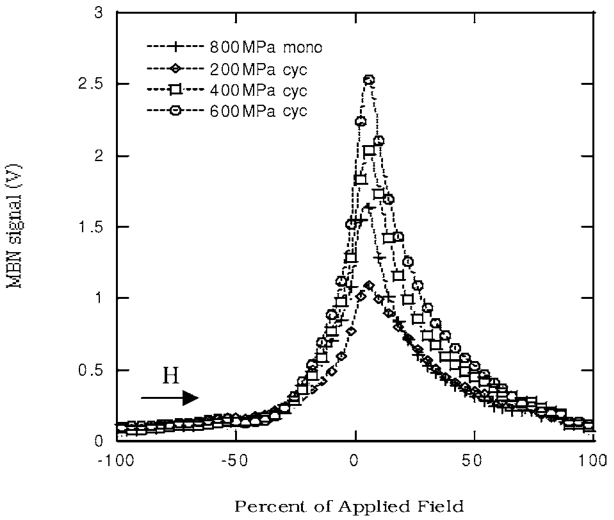


Figure 8 Typical Barkhausen noise measurements for Somaloy500 + Kenolube material after pressing and heat treatment, showing the effects of the various pressing regimes.

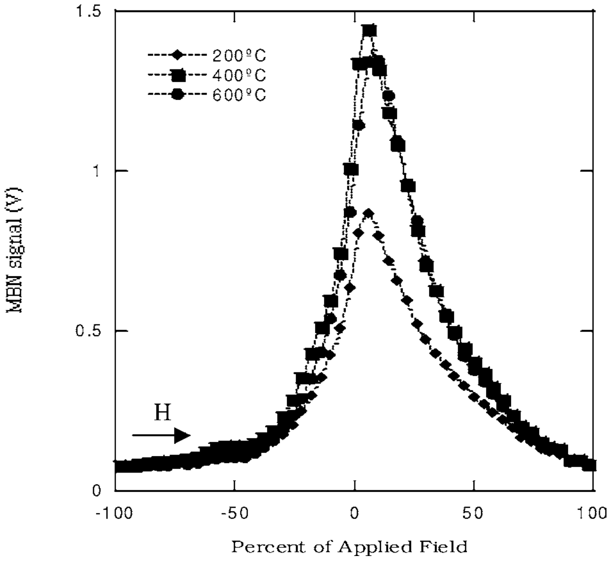


Figure 9 Typical Barkhausen noise measurements for Somaloy500 + Kenolube material after pressing and heat treatment, showing the effects of various heat treatment temperatures.

3.6. "Bluing" treatment

One initially surprising result was the relatively low values of permeability measured on SMC samples produced for this study compared to values claimed by Höganäs. Measurements on a Höganäs motor core revealed a high content of magnetite in the material that must have been introduced at the heat treatment stage. Accordingly, heat treatment trials were carried out under controlled conditions to determine the appropriate conditions for magnetite formation [11–14]. Heat treatment in a steam/N_2 atmosphere for 30 min dramatically increased the magnetite content and the permeability

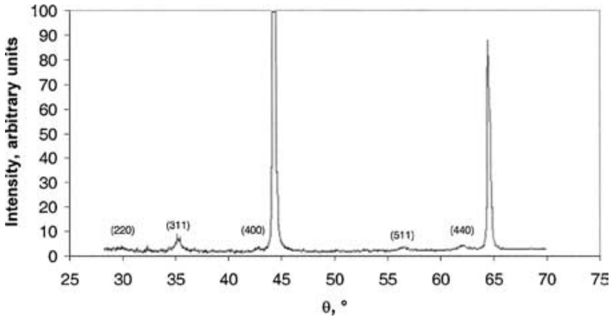


Figure 10 XRD pattern of sample after a 'bluing' treatment verifying the presence of magnetite.

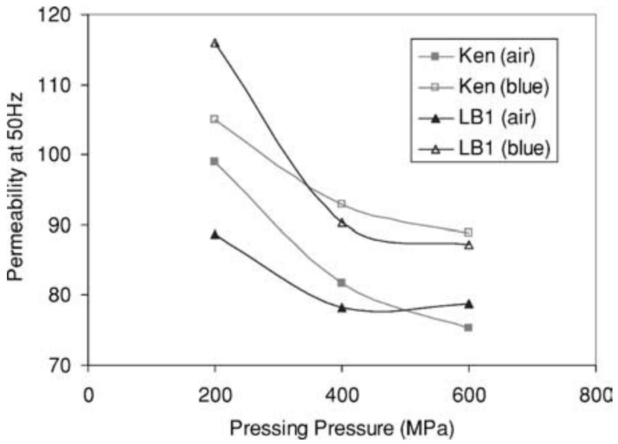


Figure 11 Magnetic permeability determined at 50 Hz as a function of pressing pressure for the two materials contrasting the effects of different heat treatments.

(Fig. 10). The permeability-frequency response is similar for air oxidised and steam/N_2 oxidised materials but the permeability is higher in the latter case (Fig. 11). The decrease in permeability with frequency is more characteristic of a ferrite than a metallic material [15].

4. Conclusions

In summary we have demonstrated that heat treatment is more important than detailed control of the pressing process in the manufacture of SMCs. During compaction, extensive plastic flow occurs and the SMC is work hardened, which increases its electrical resistivity but reduces its magnetic softness. Heat treatment is necessary both to restore magnetic properties and improve binding strength. High electrical resistivity can be reduced by pressing at very high pressures and by heat treatment at high temperature. However, at intermediate temperatures where the resistivity would be expected to fall as the carbonaceous coating on the iron particles is dissolved in the iron, the resistivity is maintained due to oxidation of the particle surfaces. If this oxide is controlled through subsequent heat treatments, the magnetic properties of the SMC can be enhanced.

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