Quantification of the effect of changes in steel microstructural parameters on EM sensor signals

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ABSTRACT

In an accompanying paper being presented in NDESAI, the utility of Multi-frequency electromagnetic sensors has been shown to be able to detect steel microstructure changes, for example the austenite to ferrite transformation and the presence of decarburisation in high carbon steel rods. In the present study, steels with various carbon contents have been used to study the effect of phase balance changes (ferrite, pearlite, un-tempered martensite and tempered martensite) on the EM readings. With an increase in pearlite content in ferrite/pearlite microstructures, the relative permeability and hence inductance value decreases. Changing the microstructural state from pearlite to martensite,in a high carbon steel, decreases the relative permeability and hence inductance value, whilst tempering increases these values. In addition, steel wires, with a fully pearlitic microstructure have been used to determine whether the EM sensor can be used to quantify interlamellar spacing changes. The low frequency inductance value was found to increase approximately linearly with an increase in the interlamellar spacing for the range of values investigated.

Introduction

Steels often show a change in their mechanical and/or electro-magnetic properties due to processing or in-service thermal-mechanical exposure altering the microstructure. In order to obtain accurate quality control, it is important to be able to monitor these changes non-destructively, and preferably in a non-contacting manner. Electromagnetic (EM) sensors are sensitive to changes both in relative permeability and electrical resistivity of the samples, which vary with composition, microstructure and temperature. In the last decades, multi-frequency EM sensors have been shown to be able to monitor the austenite-ferrite phase transformation in steel [1-3], and have been shown to be capable to work on-line during steel processing [4]. They have also been shown to be able to detect decarburisation in steel rod both on-line and off-line [5-7]. However, the influence of key materials variables on the EM signal is not yet fully understood. To extend the potential applications of the sensor, it is important to determine the sensitivity of, and understand the relation to, each factor.

Much research has been carried out on the link between microstructure and electrical and magnetic properties for electrical steels. Littman et al. report that the alloy composition, grain size, texture, impurities, inclusions, precipitates, electrical resistivity, and thickness of low carbon electrical steels can affect their magnetic properties [8]. Hou et al. carried out a study of alloy composition, grain size, process variables and rolling strain on the permeability of low carbon electrical steels and suggested that grain size is the predominant factor [9]. It has been reported that a larger grain size increases the ac and dc permeability.The electrical steels assessed in these studies had very low carbon (0.002-0.004 wt%) and high silicon (up to 2wt%) content with a large ferrite grain size of 50-400?m, whilst the structural steels in this work, typically have a smaller grain size and mixed (ferrite, pearlite, bainite, martensite) microstructure.

Thompson et al. [10] studied the magnetic permeability of ferrite-pearlite steels as a function of carbon content by studying the Barkhausen noise emissions. The initial relative permeability values were predicted to be 288, 86, 63 and 56 - for 0.17, 0.44, 0.67, 0.87 wt% carbon steels respectively. The permeability values were found to decrease with an increase in carbon content due to the effectiveness of cementite lamellae in pinning domain walls at higher pearlite contents. Hao et al. [7] and Yin et al. [11] used multi frequency EM sensors to measure the ferrite fraction varying from 0% to 100% in austenite-ferrite dual phase steel microstructures (produced from stainless steel powders). It was reported that, for the sensor geometry used, the real inductance at low frequency (10Hz) showed a significant change with ferrite fraction (approximately linear) when low ferrite fraction samples (< 40%) were used, but above approximately 40%, little further change in signal occurred, although these samples could be distinguished using a different EM signal parameter, the zero crossing frequency [7].

This paper reports on the effect of phase balance (amount of ferrite, pearlite, martensite, and tempered martensite) in C-Mn steels and the interlamellar spacing in pearlitic steel on the relative permeability and hence the EM sensor reading.

Materials and experiments

Melting grade (pure) iron and C-Mn steels with various carbon contents have been used to study the effect of ferrite/pearlite phase balance on the EM sensor readings. Three blocks of the 0.8C steel were austenitised at 890°C for an hour and quenched in water to form a fully martensitic microstructure. Then, two of the samples were tempered at 280°C or 450°C for one hour to form different tempered martensite microstructures. Tyre grade steel wire samples, supplied by Tata

Steel India, were austenitised at 1100°C followed by salt bath heat treatment at 590°C, 600°C, 610°C, 620°C and 630°C for 6minutes.Three nominally identical samples for each heat treatment condition were provided. The pearlite interlamellar spacings of these isothermally transformed samples were measured by Tata Steel India using the intercept method.The chemical composition of all the steel samples is given in Table 1.

Table 1. Chemical composition for the steel samples used in this work, all in wt%.

	C	Si	Mn	S	P	Cu
Pure iron	-	-	-	-	-	-
0.17C	0.17	0.28	0.80	0.03	0.01	0.09
0.38C	0.38	0.26	0.75	0.03	0.02	0.12
0.53C	0.53	0.29	0.72	0.01	0.02	0.09
0.8C	0.80	0.20	0.96	0.03	0.02	0.02
Steel Wires	0.67	0.20	0.66	0.01	0.02	-

Sectioned samples were mounted in conductive Bakelite, ground and fine-polished to an OPS finish and etched in 2% Nital. The microstructure was characterised by using a Zeiss optical microscope. The ferrite/pearlite phase balance and ferrite grain size of the samples were analysed using "Image J" image analyser software. The hardness was measured on polished samples by Vickers micro hardness measurement with a 500g load. Each hardness value was determined by taking the average of 4 measurements.

Electrical resistivity measurements were performed at room temperature using a conventional four point DC method with a "Cropico microhommeter" which has a resolution of 0.1 microhms on a 30 milli-ohm range. Each resistivity value was determined by taking the average of 10 measurements on the sample machined for EM sensor measurements.

Samples for EM measurements (cylindrical shape with 4.95mm diameter and 50mm length) were machined from the as-received materials or the heat-treated samples.The EM sensor used in this study has exciting and sensing coils that are air-cored. Each coil has an inner diameter of 7.95mm, 0.2mm height, 10.5mm length and 56 turns. The coils were driven by an frequency response analyser (SL1250) at frequencies from 10 Hz to 65000Hz, and the real inductance values were taken. The sensor output was used with a COMSOL FEM model developed for the sensor-sample geometry and the relative permeability was predicted by fitting the modeled results to the experimental measured ones.

Results and discussion

Ferrite-pearlite phase balance:

Optical microstructures of the (as received) pure iron, and C-Mn steels with 0.17C, 0.38C, 0.53C and 0.8C samples at 400X magnification are shown in Figure 1. Table 2 shows a summary of the measurements of average ferrite grain size, pearlite amount, hardness and resistivity for all the steel samples (as-received and heat treated) with standard deviation values. The ferrite grain size and hardness results are as expected from the heat treatments. The difference in resistivity between the C-Mn steels is relative small (minor differences may be caused by differences in the solid solution elements present not specified in the composition details received). From Table 2 it can be seen that it is difficult to distinguish the C-Mn steels by resistivity measurements.

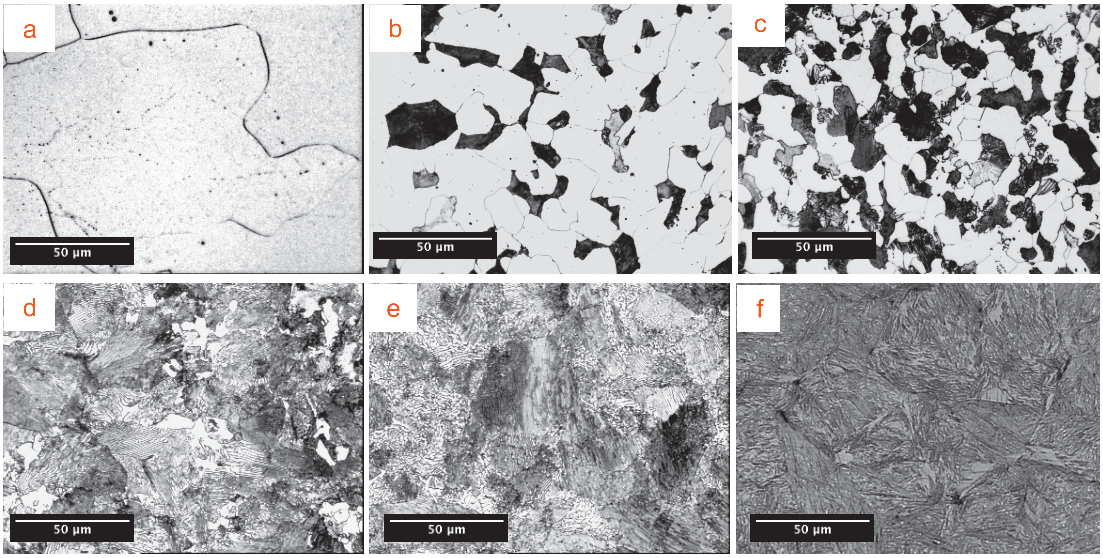

Fig. 1: Optical microstructures of a) pure iron, b) 0.17C, c) 0.38C,d) 0.53C,e) 0.8C as-received and f) 0.8C as-quenched samples at ×400 magnification.

Table 2: Summary of the microstructure, hardness and resistivity values of the ferrite-pearlite microstructures.

Sample	Ferrite grain size (µm)	Pearlite%	Hardness (HV)	Resistivity (nΩm)
Pure iron	155	0	72.8 ± 1.1	104.0 ± 0.3
0.17C	25	27.2	146.8 ± 0.4	210.9 ± 0.1
0.38C	14	44.2	171.3 ± 1.3	218.6 ± 0.2
0.53C	-	90.2	224.7 ± 2.1	243.7 ± 0.3
0.8C	-	100	277.2 ± 3.2	230.0 ± 0.2

The EM field produced by the exciting coil in the sensor acts on the steel samples in two ways [5]. At lower frequencies, it tends to magnetise the sample thus the inductance increases. Here, the relative permeability of the sample dominates the inductance value. Secondly, the change in magnetic field induces eddy currents that oppose the driving current and the inductance decreases.

As the frequency increases, eddy currents become more dominant and eventually the EM signal approaches a very low inductance value, where the samples cannot be easily distinguished. The measured real inductance versus frequency (logarithmic scale) results for the as-received pure iron, 0.16C, 0.53C and 0.8C steel samples, using the EM sensor are shown in Fig.2a.

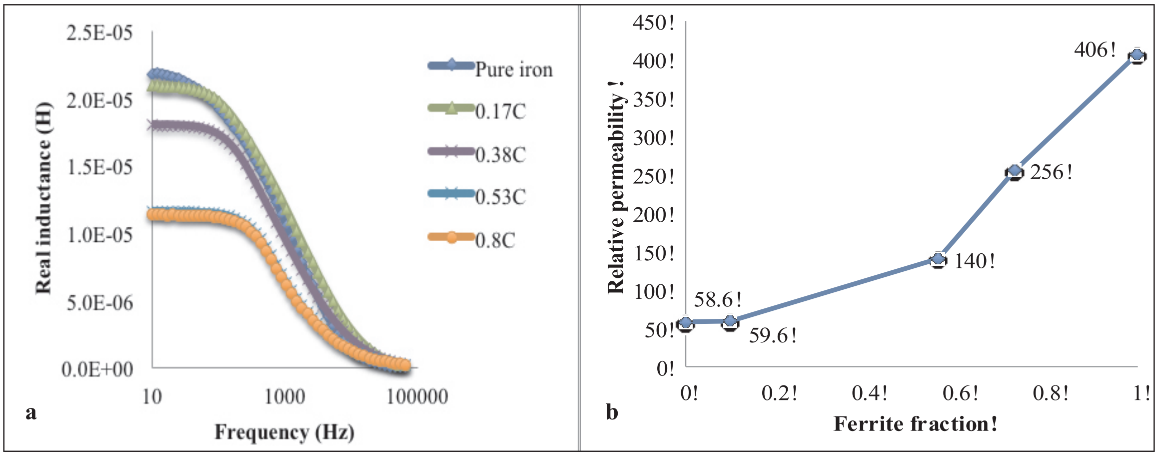

Fig. 2: a) Real inductance changes with frequency b) relative permeability for pure iron, 0.17C, 0.38C, 0.53C and 0.8C as-received (i.e. ferrite + pearlite) steel samples.

At low frequencies (below approximately 100Hz), the real inductance values, whic here dominated by relative permeability, decrease with the increase in carbon content up to 0.53 wt% C due to the formation of pearlite, which has a much lower relative permeability than ferrite [6]. However, the 0.53C and 0.8C steels show little difference in real inductance at low frequency. This is believed to be due to both the 0.53C and 0.8C steels have a high pearlite content (90.2 and 100% respectively) with the ferrite regions in the 0.53C steel being small and not connected and hence not contributing much to the permeability. The pure iron sample shows a different curve shape influenced by the lower resistivity of the pure iron sample compared to the C-Mn samples. The relative permeability values calculated from the modeland the ferrite fraction in the different steel microstructures is plotted in Fig.2b showing an increase in inductance as the ferrite fraction increases - further data is required for the low ferrite fractions (10-30%) to determine when the inductance increases significantly.A similar effect has also been reported by W.Yin et al. who found that in austenite + ferrite steel samples a low fraction of ferrite (<40%), present as isolated regions, due to the powder processing fabricate route, did not result in much increase in permeability in both measured and FEM (finite element method) modelled results[12]. Further increase in the ferrite amount gave a significant increase in relative permeability.

Pearlite interlamellar spacing:

The resistivity values versus pearlite interlamellar spacing, for the salt bath heat treated steel wire samples, are shown in Fig 3a. The resistivity changes, due to the changes in interlamellar spacing, are small and fall within experimental scatter. This means that resistivity measurements are not sensitive enough to determine the interlamellar spacing. The EM sensor results of real inductance at 10Hz versus pearlite interlamellar spacing areshown in Fig 3b. Unlike the resistivity results, the low frequency real inductance gives a very good correlation with interlamellar spacing. This is believed to be due to the reduced pinning effect on the magnetic domain wall movement in the samples with a larger spacing. The reproducibility in EM sensorreadings between the nominally identical samples is quite good.

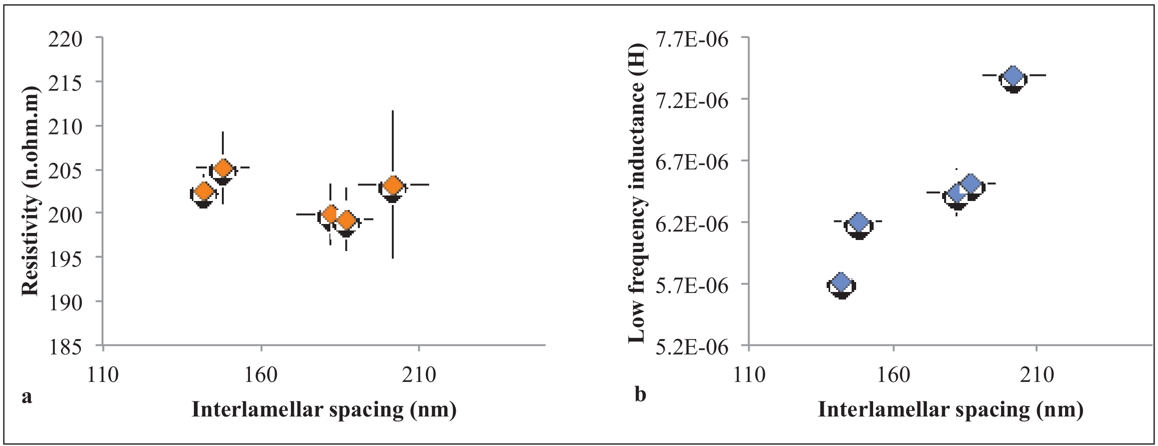

Fig. 3: a) Resistivity and b) real inductance values at 10Hz versus pearlite interlamellar spacingfor the wire steel salt bath heat treated samples (standard error shown).'

Pearlite and martensite:

The EM sensor results of the real inductance versus frequency for the 0.8C steel in the as received, as quenched, tempered at 280°C, and tempered at 450°C conditions are shown in Fig 4. Changing the microstructural state from pearlite to untempered martensite decreases the low frequency inductance value. For the high carbon steel, the retained austenite amount is predicted to be about 16%, using the Koistenen and Marburger equation [13]. Therefore, the reduced inductance value is consistent with the presence of retained austenite (which is paramagnetic with a relative permeability of 1) in the microstructure resulting in a decrease in the relative permeability. The role of carbon in solution (martensite) as opposed to in carbides (e.g. pearlitic cementite or tempered martensite) is not yet known, but is likely to hinder domain movement also contributing to a reduction in permeability and hence inductance value. The decrease in inductance value is also consistent with an increase in the dislocation density, which will affect the magnetic domain wall movements and hence relative permeability. Tempering the as quenched martensitic sample at 280°C results in precipitation of carbides, decomposition of retained austenite and a decrease in dislocation density. The relative permeability and hence the low frequency inductance increased. Tempering of the as quenched martensite at 450°C results a similar effect to that seen for the 280°C tempered sample but with coarser and spheroidised carbides, which will further reduce the domain wall pinning effect and result in an increase in low frequency inductance.

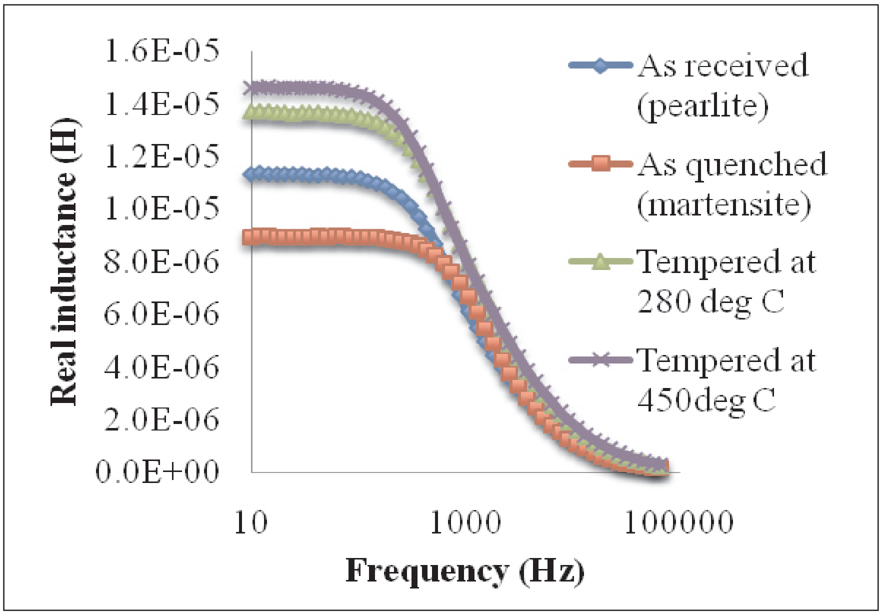

Fig. 4: Real inductance changes with frequency for martensite, tempered at 280°C and tempered at 450°C samples.

Conclusion

A multi-frequency EM sensor has been used to measure the ferrite/pearlite phase balance and pearlite interlamellar spacing. With an increase in pearlite content in ferrite+pearlite microstructures, the relative permeability and hence inductance value decreases up to 90% pearlite formed. For the salt bath heat treated pearlitic wire steels, the low frequency inductance was found to increase with an increase in the interlamellar spacing with an approximately linear relationship for interlamellae spacings between 0.14-0.20µm. Changing the microstructural state from pearlite to untempered martensite decreased the relative permeability and hence the inductance value in a 0.8 wt% C steel which is consistent with the presence of retained austenite and increase in dislocation density. Tempering the martensitic microstructure results in an increase inrelative permeability and hence inductance value due to the precipitation of carbides, decomposition of retained austenite and a decrease in dislocation density.The good reproducibility in the EM measurements indicates that the technique has the potential to non destructively monitor these microstructural parameters.

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