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Magnetic sensing for microstructural assessment of power station steels: Differential permeability and magnetic hysteresis

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Abstract. Failure of power station steel components can have severe economic impacts and also present significant risks to life and the environment. Currently components are inspected during costly shut-downs as no in-situ technique exists to monitor changes in microstructure of in-service steel components. Electromagnetic inspection has the potential to provide information on microstructure changes in power station steels in-situ. In this paper, tests have been carried out on pipe and tube samples in different microstructural conditions, using a lab-based closed magnetic circuit and impedance measurement systems. EM properties have been identified with correlations to material properties, which can quantify degradation in-situ and at elevated temperatures.

1. Introduction
Measurement of the electromagnetic (EM) properties of materials can be vital; as such properties are related to the materials’ microstructure, condition-related parameters and consequently performance. For instance, the permeability and conductivity of porous Cu and Fe foams, manufactured by the lost carbonate sintering process, have proven to be related to their porosity [1]. In steels, ferrite fraction variations [2, 3], creep damage and precipitation [4] cause changes to EM properties. Factors such as residual stress [5], temperature and case hardening [6] also have an affect on the steels’ EM properties. Such EM properties can be obtained through inspection of the material using purpose built EM sensors.

Power generation steel components such as boiler tubes and steam line pipes are exposed to high temperature and pressure during their lifetime, and therefore experience carbide coarsening and occurrence of creep cavitation [7], possibly leading to component failure; an unplanned shutdown period can cost approximately ~$2M per day per power station. These components are currently inspected during costly shut down periods and no in-situ technique exists to directly monitor the changes in microstructure of such in-service components at an elevated temperature.

Magnetic induction techniques have been used extensively in measuring the EM properties of materials at relatively low frequencies (i.e. <10 M Hz). In doing so, various sensor configurations have been used, for example, cylindrical air-cored sensor [8,9], H-shaped ferrite-core coils and printed circuit board (PCB) based planar coils [10]. The ability to monitor any microstructural changes in steel components through their EM property variation has great potential and may become very useful in the future. In this paper, a lab-based closed magnetic circuit measurement system and a cylindrical air-cored sensor were designed to measure the EM properties of 50 mm long cylindrical power generation steel (P9 and T22) samples in different microstructural states. The obtained EM properties from both sets of systems are then compared with each other. Analytical and numerical methods (finite elements (FE)) have been used to calculate the sensor response, through which the conductivity and

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permeability of the samples were inferred. In addition, the relative incremental permeability of P9 and T22 steel samples have been obtained through the measurement of minor loop deviations from the initial magnetisation curve, the results of which are connected to the permeability values obtained from the spectroscopy of these samples. From the two magnetic hysteresis and incremental permeability measurements, correlations have been established between the electromagnetic properties and material properties such as hardness. Such correlations may allow one to quantify degradation in power station steels both in-situ and at elevated temperatures; and could, therefore, lead to considerable savings while allowing frequent and detailed component inspection.

2. Methodology

2.1. Physical Principle

Any microstructural variation in steel may lead to changes in its EM properties, e.g. permeability and conductivity. EM sensors function on the basis of detecting and identifying variations in these quantities measured from samples. By measuring the response of such EM sensors over a range of frequencies, the permeability and conductivity can then be inferred.

The effect of eddy currents in the sensor response is very weak at low frequencies, and any contribution to the inductance change is mainly from the magnetisation of the sample. Therefore the inductance measured at low frequencies is related to the sample permeability. However, the effect of eddy currents in the sample becomes stronger with increasing frequency; and therefore variations in inductance are gradually dominated by the effect of conductivity or polarisation delay of the sample. The electric and magnetic properties of the samples can be determined by complete analysis of the inductance-frequency spectra.

2.2. Experimental Procedure and Sensor FE Simulation

Two groups of power generation steels were studied, the first group being P9 steel (8.40Cr-0.97Mo-0.12C-0.52Si-0.44Mn wt%) and the second group being T22 (1.90-2.60Cr-0.87-1.13Mo-0.05–0.15C-0.50Si-0.30–0.60Mn wt%), both removed from service at 520 °C for approximately 11 years. Selected samples were heat treated to simulate the service entry microstructure by normalising at 950 °C or 940 °C for 1 hour and then tempering at 760 °C for 1 hour or 720 °C for 1.5 hours, for the P9 and T22 steels respectively. In addition to the tempered and ex-service samples, as normalised samples were also examined. Cylindrical rods 50 mm long and 4.95 mm in diameter for each condition were prepared for the EM sensor measurements.

Samples were measured by inserting them into an air-cored cylindrical sensor, which consists of two identical coils arranged with their axes aligned, one exciting and the other receiving. The coils have a length of 9.8 mm, inner diameter of 6.9 mm and outer diameter of 8.3 mm with a separation of 10.4 mm. Measurements of trans-impedance were taken with an impedance analyser (Solartron 1260) at frequencies from 10 Hz to 1 MHz. Analytical and numerical methods have also been employed to calculate the sensor response of these samples and presented in [9]. In addition to the analytical methods, FE models have been built in Maxwell (ANSYS, Inc.) to model the actual measurement arrangement, taking full account of the sensor and sample interaction, in order to determine the EM properties such as relative permeability and conductivity, of these samples by fitting with the experimental data. The geometry, setup and the FE model for the sensor are shown in Fig 1.

**Fig 1.** (a) The air-cored cylindrical sensor and sample setup and (b) Axis symmetrical FE model for the sample and air-cored cylindrical sensor - Note Z is the axis of rotational symmetry.
3. Measurement and Experimental Results

3.1. Metallographic Tests
The microstructure for the P9 and T22 grade samples have been analysed and presented previously [8, 9, 11]. However, for completeness, the microstructure of the P9 and T22 samples with different conditions are shown in Fig 2. Referring to Fig 2(a), the microstructure of the as-normalised P9 consists of predominantly martensite mixed with some bainite. Subsequent tempering produces a simulated service entry microstructure, i.e. tempered martensite / bainite as shown in Fig 2(b). After long service exposure, the microstructure showed equiaxed ferrite with large carbides distributed within ferrite grains or on grain boundaries as shown in Fig 2(c). The as-normalised T22 steel shows a mixed microstructure of bainite and some proeutectoid ferrite, as shown in Fig 2(d). After tempering, many carbides can be observed along prior austenite grain boundaries, on ferrite boundaries or within bainite regions, as shown in Fig 2(e). The microstructure of T22 after the service exposure consists of equiaxed ferrite and a great many carbides outlining the ferrite grain boundaries or finely dispersed within the ferrite grains, as shown in Fig 2(f).

![Fig 2. Microstructure of the P9 samples in different conditions: (a) as normalised (b) as tempered and (c) ex-service and microstructure of T22 samples in different conditions: (d) as normalised (e as tempered and (f) ex-service](image)

3.2. Electromagnetic Sensor Measurements
The measured mutual inductance spectra for P9 and T22 with different conditions are shown in Fig 3.

![Fig 3. (a) Real and (b) Imaginary Inductance spectra for P9 and T22 samples of different conditions (TFS: Taken from Service, NORM: Normalised, TEMP: Tempered) measured using cylindrical air-cored sensor](image)
The real inductance graphs, shown in Fig 3 (a), have a characteristic shape, with a flat section for frequencies below approximately 100 Hz; a roll off over the intermediate frequencies and finally a flat high frequency response. The flat low frequency response is determined by the differential permeability of the sample, suggesting that the permeability is constant up to a frequency of at least 100 Hz. The roll-off over the intermediate frequency response results from eddy currents circulating coaxially in the sample; here the electromagnetic skin depth is reducing with frequency and eventually becomes much smaller than the radius of the sample. At high frequencies, the skin depth effectively approaches zero and the real mutual inductance approaches a constant slightly negative value determined only by the dimensions of the coil / sample geometry. The graphs of the imaginary component in Fig 3(b) show a characteristic curve with maximum responses which coincide with maximum energy transfer eddy current heating from the transmitter coil to the sample.

3.3. B-H Measurements

Another aspect of obtaining the magnetic properties of a sample is to measure its magnetic hysteresis. The B-H curves for the three P9 and T22 steel samples in different conditions have been measured (Fig 4). The B-H curves demonstrate the micro-magnetic properties of steels including coercivity, remanence, saturated magnetisation etc. These properties and the relative permeability can be affected by different microstructural features. Therefore, the B-H measurement and EM sensor can be complementary techniques.

![Fig 4](image)

As shown in Fig 4, the B-H loops for both set of samples (P9 and T22) follow the same trend; the ex-service sample results in the lowest coercivity ($H_C$), with a small increase in $H_C$ for the tempered samples and a large increase in $H_C$ for the normalised samples (Table 1). The coercivity values reflect the magnetic hardness of the samples, which in turn is indicative of material hardness. As the P9 normalised sample is predominantly martensitic with a Vickers hardness value of HV401, it exhibits the greatest $H_C$ value. As bainite typically has a lower hardness than as-quenched martensite, but higher hardness than ferrite, the bainite / proeutectoid ferrite microstructure of T22 normalised sample (HV316) shows a reduction in $H_C$ in comparison to the P9 normalised sample. The difference between the ex-service and tempered samples is less pronounced, with a decrease in hardness after the in-service exposure being reflected by a small decrease in $H_C$ for both sample sets; with HV212 and HV203 for P9 and T22 TEMP and also HV158 and HV129 for P9 and T22 TFS respectively.

Fig 5 shows the evolution of the minor loop as deviations from the initial magnetisation curve. The reason for such measurements is to obtain the relative incremental permeability of these power station steels through their magnetic hysteresis, the results of which are connected to the permeability values obtained from the spectroscopy of these samples. The origin of the first minor loop corresponds to the demagnetised state, where B and H are equal to zero. As the initial magnetisation curve approaches saturation, domain walls are swept away by field pressure. The dominant magnetisation mechanism
can be described as the progressive alignment of the field against anisotropy, as the domains rotate from their magnetic easy axes towards the direction of the applied field [12]. Consequently, only the reversible components remain, resulting in a closed loop, with a smaller variation in Fig 5(b) for a given applied field, as shown in the graph (bottom right). As illustrated in Fig 5, the incremental permeability ($\mu_\Delta$) can be obtained from the minor loops. Such a quantity can be calculated as the ratio of the variation in flux density ($\Delta B$) and the corresponding change in the applied field ($\Delta H$), scaled with respect to the permeability of free space ($\mu_0$): presented in equation 1.

$$\mu_\Delta = \frac{1}{\mu_0} \times \frac{\Delta B}{\Delta H}$$  \hspace{1cm} (1)

**Fig 5.** (a) Initial magnetisation curves and (b) minor loop deviations for P9 Tempered sample - B and H offsets removed from minor loops for comparison

### 3.4. Permeability and Conductivity Evaluation

Finite element models were set up to simulate the sensor response. The simulated results are compared to the measured results to fit the conductivity and permeability in a least squared sense. The comparison of the simulated results with the measured results for the cylindrical sensor has been covered in [9]. It is evident that the measurement and simulation results are in good agreement. Permeability and conductivity values for the P9 and T22 samples can therefore be inferred, a list of which is provided in Table 1. This table presents the permeability values for the power station steels (P9 and T22) for both impedance analyser measurement (using the cylindrical air-cored sensor) and also their corresponding coercivity and remanence values obtained from the magnetic hysteresis loop measurements. It is interesting to see how the values for P9 and T22 normalised, tempered and ex- serviced samples increase from small to large values respectively. It is also evident that when a series of samples all have basically similar metallurgical microstructure, i.e. the tempered P9 and T22 samples, which are all forms of tempered martensite, then the variation in permeability is very small. Thus, the permeability values for P9 and T22 tempered samples (P9-TEMP and T22-TEMP) fall into a narrow range of 52-53.9 for the cylindrical sensor. However, when there is a microstructural change, there is a big change in permeability. As a result, the samples with un-tempered martensitic microstructures (P9-NORM and T22-NORM) have permeability of 28.7 and 45.1 for the cylindrical sensor. On the other hand, samples with a predominantly ferritic structure (P9-TFS and T22-TFS) have permeability of 83 and 60.4 for the cylindrical sensor. Although the permeability values are not precisely the same for the two sets of measurements, similar trends for the samples can be observed.

As presented in Table 1, the samples with ex-service heat treatment have the lowest coercivity ($H_C$) values, with a minor increase in $H_C$ for the tempered samples, followed by a considerable increase in $H_C$ for the normalised samples. As the P9 normalised sample is predominantly martensitic (with an associated high dislocation density), higher fields are required for demagnetisation, and therefore it exhibits the greatest $H_C$ value. The lower (mechanical) hardness bainite / proeutectoid ferrite microstructure of the T22 normalised sample shows a reduction in $H_C$ in comparison to the P9 normalised sample. On the other hand, the difference between the ex-service and tempered samples is
less pronounced, with the decrease in hardness following in-service exposure being reflected by a small decrease in $H_c$ for both sample sets.

Table 1. Summary of relative permeability and conductivity for different P9 and T22 steel samples

<table>
<thead>
<tr>
<th>Samples and conditions</th>
<th>$\mu_R$ obtained from Cylindrical Sensor</th>
<th>Conductivity (MS/m)</th>
<th>$\mu_i$ obtained from BH curves</th>
<th>Coercivity (A/m)</th>
<th>Remanence (T)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P9-(MTG L3101A-1)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>P9-TEMP Normalised and Tempered at 760°C for 1h</td>
<td>52</td>
<td>1.82</td>
<td>55.2</td>
<td>0.778</td>
<td>1.057</td>
</tr>
<tr>
<td>P9-NORM Normalised at 950°C for 1h</td>
<td>28.7</td>
<td>1.69</td>
<td>22.0</td>
<td>3.514</td>
<td>0.559</td>
</tr>
<tr>
<td>P9-TFS Taken from service</td>
<td>83</td>
<td>1.83</td>
<td>90.9</td>
<td>0.512</td>
<td>0.684</td>
</tr>
<tr>
<td><strong>T22</strong> - (MTF C33-3)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>T22-TFS Taken from service</td>
<td>60.4</td>
<td>3.87</td>
<td>57.4</td>
<td>0.644</td>
<td>0.712</td>
</tr>
<tr>
<td>T22-TEMP Tempered at 720°C for 1.5h</td>
<td>53.9</td>
<td>3.53</td>
<td>48.5</td>
<td>0.718</td>
<td>0.988</td>
</tr>
<tr>
<td>T22-NORM Normalised at 940°C for 1h</td>
<td>45.1</td>
<td>2.99</td>
<td>36.9</td>
<td>2.154</td>
<td>0.679</td>
</tr>
</tbody>
</table>

It is also clear from Table 1 that the coercivity has an inverse relationship with permeability (obtained from impedance measurements), as the samples with the lowest coercivity values have the highest permeability values. The coercivity values reflect the magnetic hardness of the samples, which in turn is indicative of material/physical hardness, therefore both the permeability and coercivity values are linked to magnetic and material hardness.

4. Conclusion

In this paper, a cylindrical air-cored sensor has been designed to measure the EM properties of 50 mm long, 4.95 mm diameter power generation steel (P9 and T22) samples taken from pipes and tubes exposed to different heat treatments. Numerical methods (finite elements) were used to calculate the sensor response, and as a result the conductivity and permeability of these samples were inferred. Magnetic hysteresis curves for the power station steel samples were also measured, from which relative incremental permeability values were obtained. The incremental permeability values measured through minor loop deviations from the initial magnetisation curve were compared to the permeability values obtained from the spectroscopy of these samples, and their corresponding EM properties have been identified with strong correlations with material properties such as hardness. Correlations between EM properties and changes in the microstructure (normalised only, normalised and tempered and ex-service) were established and will be used in future to develop a field deployable tool for in-situ inspection.

References