Assessment of Microstructural Changes in Grade 91 Power Station Steel through Magnetic Measurements

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Abstract. The use of Grade 91 steel for high-temperature power plant applications can result in substantial reductions in component thickness compared to using alloys with lower strength, resulting in reduced thermal stresses and improved service life. Although Grade 91 offers superior resistance to thermal fatigue and creep to other grades used in the industry, this is dependent on the creation and maintenance of a specific microstructure, which can be altered through routine component fabrication, installation and maintenance and through thermo-mechanical exposure in service. Electromagnetic inspection has the potential to assess the level of degradation in steel components through in-situ measurements. In this paper, correlations are drawn between microstructural changes in heat treated Grade 91 samples and electromagnetic properties including incremental permeability, magnetic Barkhausen noise and distortion analysis of magnetic excitation voltage. Results are reported from tests carried out on 5mm diameter rod samples machined from a section of Grade 91 tube and after heat treatment to produce the following microstructures; service entry, simulated degeneration due to prolonged service exposure and simulated mis-heat treatment. Test results from a rod sample machined from a section of ex-service Grade 91 tube are also reported. Clear relationships are established between quantifiable magnetic properties such as coercive force and mechanical properties such as Vickers hardness. Finally, an analysis of the magnetic mechanisms linking the microstructural changes that occur with different heat treatments to MBN and incremental permeability signal features is undertaken. The results demonstrate the potential viability of electromagnetic methods for the assessment of microstructure, including changes due to thermal aging in Grade 91 power station tubes and pipes.

1. Introduction

The superior mechanical properties of Grade 91 steel, commonly used in power station applications, is dependent on the creation and maintenance of a tempered martensite microstructure with a fine distribution of carbide precipitates for optimum performance. This microstructure can be altered through component fabrication, installation and maintenance procedures such as hot bending, forging and welding, so it is vital that NDE
procedures are developed which can monitor these changes at different stages of component life. Current procedures for the assessment of microstructural changes in steel components in power stations involve site inspections during shut-down periods and often involve lengthy procedures such as replica metallography [1] or hardness testing. Electromagnetic (EM) inspection [2] has the advantage that it can be performed quickly, in-situ with minimal surface preparation. EM properties are however highly material and microstructure specific, so inspection techniques must be calibrated to particular materials and tailored to monitor known microstructural changes, if these techniques are to be deployed with confidence.

A number of different approaches are available to assess the magnetic properties of a particular material; the most basic of these is the measurement of the major BH loop. Various parameters can be derived from the major loop, including coercive force which can be used to quantify the magnetic hardness of a material, which in turn is indicative of material hardness [3]. Information can also be derived from small minor loop deviations from the major loop. Although the same magnetic parameters are assessed in major and minor loop measurements, the interaction between magnetic domain walls and material microstructure can be different as the microstructural features effectively pinning domain walls vary with the applied field strength [4]. Previous work [5] has shown that minor loop parameters show greater sensitivity to changes in material properties such as hardness; this has been attributed to the technique’s high sensitivity to lattice defects.

Although the link between magnetic Barkhausen noise (MBN) activity and material properties such as microstructure, hardness and residual stress is more complex, using techniques such as analysis of the MBN signal profile [2, 6], information pertaining to the material microstructure can be inferred through the interaction between domain walls and lattice defects such as dislocations, grain boundaries and precipitates at different magnetic field ranges. As these microstructural changes are major causes of degradation for power station steels, MBN could be a useful tool for the assessment of this degradation.

An alternate technique to probe the magnetic properties of a material using very simple test equipment and without the complications associated with BH measurements has recently been developed. Distortion Analysis of Magnetic Excitation (DAME) exploits the non-linear distortion of the excitation voltage and the tangential magnetic field when a ferromagnetic material is placed between the poles of a U-shaped electromagnetic yoke [6]. This non-linear distortion reflects the magnetisation behaviour of the sample placed in the magnetic loop. Since the magnetisation behaviour strongly depends on the microstructure of the ferritic steel DAME has been shown to reflect changes in the microstructure, texture and stresses in ferromagnetic steels [7].

2. Measurement System and Sample Summary

2.1. Measurement system for major and minor BH loops and MBN
A schematic of the measurement system developed for the BH measurement is shown in Figure 1. A low frequency time varying signal is fed to two power amplifiers, which supply current to two excitation coils wrapped around a silicon-steel core. The cylindrical sample to be tested is fitted into a slot in the core, to maximise coupling between core and sample. The surface axial magnetic field (H) is measured using a Quantum Well Hall sensor, developed at the University of Manchester [8]. The flux density of the induced field (B) is measured using a 30-turn encircling coil connected to an instrumentation amplifier. For MBN measurements this is replaced by a 3000-turn encircling coil and the low frequency component of the signal is rejected through the addition of a passive high-pass filter.
For the major loops, 1 Hz sinusoidal excitation is used and 18 cycles are recorded and averaged. Incremental permeability curves are derived from 10Hz minor loop deviations from the major B-H loop. In this case, the sample is taken through several major loop cycles before the DC offset is held at a constant pre-determined value and 90 minor loop cycles are recorded and averaged to reduce noise. Triangle wave excitation at a frequency of 0.1Hz is used to generate MBN profiles, with the signal from the MBN pickup coil and the applied axial field from the Hall sensor being recorded simultaneously. The signal from the coil is rectified and a moving average technique used to generate the MBN profile, which is then plotted against H.

Fig. 1. Schematic of system of for the measurement of major and minor BH loops

2.2. Measurement system for distortion of magnetic excitation voltage (DAME)

Fig. 2. Schematic of the experimental set-up used for acquiring and analysing the DAME signals
The schematic of the experimental set-up used to acquire the DAME measurements is shown in Figure 2. A bi-polar triangular waveform at a quasi-static excitation frequency of 0.4 Hz is fed to a bi-polar power amplifier. The alternating voltage ($V_T$) output (±20V / ±1A) of the power amplifier is used to excite the commercially pure iron electromagnetic (EM) yoke which generates maximum magnetic field strength ($H_{\text{max}}$) of ~ 20 kA/m. The excitation coil around the EM yoke is connected to a current limiting resistor ($R_{\text{CL}}$) in series. The EM yoke has an air gap of 25 mm between its poles. The voltage signal across the excitation ($V_E$) is passed through a 100 Hz low pass filter to remove any high frequency noise. The total applied excitation voltage ($V_T$) is tapped directly from the voltage source output as an independent variable. The total applied excitation voltage ($V_T$) and the voltage across the excitation coil ($V_E$) are acquired over 4 cycles of magnetisation with a total duration of 10s at a sampling frequency of 200 kS/s. The data is smoothed further by averaging over 4 magnetisation cycles. The voltage across the excitation coil ($V_E$) is plotted as a function of the total voltage ($V_T$). The average voltage across the excitation coil ($V_E$) is also differentiated with time to obtain ($dV_E/dt$) and plotted as a function $V_T$ for further analysis. The DAME measurements were made using the laboratory system developed at Newcastle University, UK. Further details are given elsewhere [7].

2.3. Test samples
T91 steel tubes (53 mm outer diameter, 13.5 mm wall thickness and > 900 mm length) were supplied by the Electrical Power Research Institute (EPRI) in the normalised and tempered condition. Cylindrical samples (4.95 mm diameter and 100 mm length) were machined from the tube and heat treated to different conditions in laboratory furnaces to simulate the microstructures expected from prolonged thermal exposure and/or mis-heat treatments / mis-manufacturing. The chemical composition of the steel is given in Table 1.

| Table 1. Chemical composition (weight percent) of the as-received T91 tube material |
|---|---|---|---|---|---|---|---|---|---|---|---|
| C | Si | Mn | P | S | Cr | Mo | Ni | V | Nb | N | Ti |
| 0.11 | 0.271 | 0.469 | 0.015 | 0.003 | 8.9 | 1.00 | 0.135 | 0.169 | 0.055 | 0.046 | 0.003 |

The T91-AR sample refers to the as received (service entry) condition, which has a Vickers hardness value of 237 HV. The microstructure is tempered martensite consisting of martensitic laths with many precipitates (mainly $M_{23}C_6$) within the laths and, predominantly, on the lath boundaries. The T91-T100h sample has been long-term tempered which results in a reduction in hardness level to 195 HV. The microstructure consists of coarsened martensitic laths and precipitates and some equi-axed sub-grains have also developed. There is significant degeneration of the tempered martensite with the presence of coarse precipitates within the laths and grains, comparable with degeneration due to prolonged service exposure. The T91-M sample refers to the simulated mis-heat treatment which gives the lowest hardness value of 157 HV. This sample shows a ferritic microstructure consisting of large equi-axed ferrite grains, with a high number density of coarse precipitates occurring on ferrite grain boundaries and also many coarse and finer precipitates within the grains.

One further cylindrical sample, T91-ES (4.95 mm diameter and 50 mm length), was machined from an ex-service section of tube (44.5 mm outer diameter, 6.3 mm wall thickness and < 70 mm length) that had been taken from service as an antler tube on a super-heater outlet header at 585 °C, under 16.5 MPa pressure (designed) for about 50,000 hours. The tube has a microstructure of slightly degenerated martensite with slightly increased lath size as compared to the T91-AR sample, with some equi-axed subgrains with fine precipitates along the lath / grain boundaries. Few precipitates within the laths or grains were observed. The T91-ES sample has a hardness of 219 HV. Full details of heat treatments and metallurgical details with micrographs can be found here [9].
3. Magnetic Measurements

3.1. Major BH loop and DAME profile

Figure 3a shows the major BH loops for all four samples. The maximum H values for these measurements are >35kA/m, this is to ensure that the material is driven as close to saturation as is possible with the system used for the tests, but only the central sections are shown for clarity. It can be seen from Figure 3a that there is a significant change in the shape of the loops for the different heat treatments. The change in shape of the BH loops for 100 hour tempering (T91-T100h) as compared to the as received sample (T91-AR) follows the same trend observed by Piotrowski et al. [3]. The loop becomes thinner, with a corresponding decrease in coercive force (see Table 2) and an increase in ‘squareness’ leading to an increase in remanence. The mis-heat treated (T91-M) sample exhibits a further decrease in coercive force in-line with the further decrease in hardness.

Figure 3b shows the time derivative of the B measurement coil voltage plotted against the surface magnetic field (H). As the flux density (B) is the integral of the coil voltage, this is proportional to the second derivative of B (d²B/dt²). Only the increasing half cycle (-Hmax to +Hmax) is shown for clarity. The time derivative reflects inflections in the BH curve which are not immediately apparent from the BH loop; for example, the BH curves for T91-T100h and T91-AR look quite similar, but can be clearly distinguished from the time derivatives, with two peaks for T91-T100h and only a single peak for T91-AR.

Figure 4 shows the average voltage across the excitation coil and the DAME profiles for all four samples. The variation in average voltage across the excitation coil around the yoke (VE) as a function of total applied voltage (VT) is shown in Figure 4a for the range -5V to +10V of VT (~ -5kA/m to +10 kA/m applied magnetic field range) for better clarity. The total applied voltage (±20V) is directly proportional to the applied magnetic field (±20kA/m). It can be seen from Figure 4a that the variation in VE is linear without any sample between the poles of the EM yoke, but varies non-linearly when a sample is introduced, resulting in non-linear distortion of VE. It can also be seen from Figure 4a that the shape of the distortion in VE is different for each microstructural condition. This difference is more clearly revealed in Figure 4b in which the time derivative of VE (dVE/dt) is plotted as a function of VT. The other half is symmetrical, but is not shown here for clarity.

The variation in (dVE/dt) is influenced by the rate of change of permeability (magnetisation) of the ferromagnetic sample introduced in the flux path. The distortion of VE is mainly caused by the effects of Faraday’s law and Lenz’s law of magnetic induction in response to the changing magnetisation of a ferromagnetic sample in the flux path. The VE plots with samples present deviate from the plot with no sample present with an increase...
in the gradient of the curve corresponding to the demagnetisation (-H_{max} to Coercive field, H_{C}) of the sample followed by a decrease in the gradient of the curve corresponding to the remagnetisation (H_{C} to +H_{max}) of the sample in a half cycle of magnetisation, as can be observed in Figure 4a. With respect to this, the \((dV_{E}/dt)\) profile shows a peak corresponding to the demagnetisation section and a trough corresponding to the remagnetisation section as can be observed in Figure 4b. This shows the effect of different magnetisation behaviours of ferromagnetic samples with different microstructures. The unique shape of the DAME profiles (Figure 4b), clearly indicates the difference in magnetisation processes and hence the difference in the rate of change of permeability (d\mu/dt) or magnetic flux (d\Phi/dt) in these ferritic steel samples with different microstructural features.

Comparison of Figures 3b and 4b show that although the time derivative of the BH curve and the DAME profile are not identical, there are various similarities between them, suggesting different microstructural influences on the mechanism of magnetisation. All the samples show a peak and trough profile, but with distinct variations for each sample. T91-AR and T91-M both have a single peak just after the zero crossing, with T91-M also exhibiting and a rise in the peak profile much earlier than the other samples for both plots. Examination of the plots for the 100 hour tempered sample (T91-T100h) and the ex-service sample (T91-ES) shows that both measurement techniques result in a two peak profile, with the peaks for the ex-service sample being spread further apart.

3.2. Incremental permeability and MBN profile

Figure 5 illustrates the process used to generate the incremental permeability curves. Several minor loops are superimposed on one half of the major BH loop, as shown in Figure 5a. In this paper only the increasing (-H_{max} to +H_{max}) section of the major loop is used. For each of these minor loops, a calculation of incremental permeability is made using the relationship \(\mu_{\Delta}=\Delta B/\Delta H_{\mu_0}\), where \(\mu_0\) is the magnetic permeability of free space, resulting in the incremental permeability (\(\mu_{\Delta}\)) curve shown in Figure 5b, with the maximum value being given in Table 2.

Table 2. Vickers hardness (HV), coercive force (H_{C}) and maximum incremental permeability (\(\mu_{\Delta MAX}\))

<table>
<thead>
<tr>
<th>Sample</th>
<th>T91-AR</th>
<th>T91-T100h</th>
<th>T91-M</th>
<th>T91-ES</th>
</tr>
</thead>
<tbody>
<tr>
<td>HV</td>
<td>237 ± 2.1</td>
<td>195 ± 2.2</td>
<td>157 ± 3.5</td>
<td>219 ± 3.4</td>
</tr>
<tr>
<td>H_{C}</td>
<td>1.014</td>
<td>0.933</td>
<td>0.670</td>
<td>0.998</td>
</tr>
<tr>
<td>(\mu_{\Delta MAX})</td>
<td>95.41</td>
<td>103.15</td>
<td>190.23</td>
<td>113.09</td>
</tr>
</tbody>
</table>

Fig. 4. a) Variations in average voltage across the EM yoke (V_{E}) and b) Variations in time derivative of the voltage across the EM yoke (dV_{E}/dt) as a function of total applied excitation voltage (V_{T}) measured without any sample and with different T91 steel bar samples for half the magnetisation cycle (-V_{T to} +V_{Tmax}). Only the section corresponding to V_{T} of -5V to +10V is shown for better clarity.
Fig. 5. Generation of the incremental permeability curve for sample T91-AR; a) Major BH loop with minor loops superimposed, b) Incremental permeability curve generated from minor loops for one minor loop amplitude (ΔH). Each point (o) on the incremental permeability curve corresponds to a minor loop.

Figure 6 shows MBN profiles (top) and $\mu_\Delta$ curves (bottom) for all samples. For $\mu_\Delta$ the field amplitude of the minor loops is increased from 170A/m to 850A/m, resulting in a family of incremental permeability curves for each sample. The $\mu_\Delta$ curves increase in amplitude as the minor loop amplitude increases. This is caused by a greater $\Delta B$ for a given $\Delta H$; as minor loop amplitude increases, more energy is applied to domain walls to overcome more pinning sites resulting in a higher $\Delta B: \Delta H$ ratio.

The MBN and $\mu_\Delta$ curves for the T91-AR sample (Figure 6a) show a single peak at around 0.7kA/m and 0.9kA/m respectively. The peaks are at the highest field of all samples, corresponding to the greatest coercive force and the lowest maximum incremental permeability ($\mu_{\Delta MAX}$) value (see Table 2). Thus this sample can be said to be both magnetically and mechanically hardest.

Two peaks appear in the MBN and $\mu_\Delta$ curves for the T91-T100h sample (Figure 6b), although the first peak at around 0.35kA/m only appears for minor loop amplitudes >~0.5 kA/m. The first peak appears only when the minor loop amplitude is large enough for the magnetic field to be reduced past $H=0$, indicating some difference in the distribution of domain wall interaction with pinning sites in this region of magnetisation. The peak at lower fields could be an indication of presence of two distinct ranges of domain wall displacement with different activation energy distribution acting in different field ranges.

The MBN and incremental permeability curves for the T91-M sample (Figure 6c) show a single peak at around 0.5kA/m and 0.6kA/m respectively. The $\mu_\Delta$ peak is at the lowest field of all samples, corresponding to the lowest coercive force and the highest $\mu_{\Delta MAX}$ value. Thus this sample can be said to be both magnetically and mechanically softest (see Table 2).

Fig. 6. MBN profiles (a) and incremental permeability curves (bottom) derived from minor loop deviations from major BH loop for cylindrical samples; a) T91-AR, b) T91-T100h, c) T91-M, d) T91-ES
4. Conclusions

In this paper, four different magnetic measurement techniques have been employed to examine three grade 91 samples, heat treated to simulate different power plant service conditions and one ex-service grade 91 sample. The different measurement techniques show distinct variations in the shape of the curves / profiles reflecting changes in microstructural features such as coarsening of martensitic laths, growth of sub-grains and carbide precipitates caused by different degrees of thermal ageing. Various features such as the two dominant peaks for the T91-T100h sample and the significant activity before the zero field transition for the T91-M sample are reflected in the results for all four magnetic measurement techniques.

Changes in the shape of the major BH loop show systematic variations in relation to the changes in microstructure induced by the thermal ageing process and the coercive force has a direct correlation with material hardness. The variation in incremental permeability derived from minor loops also reflects the effect of microstructural softening; although unlike the coercive force, maximum incremental permeability does not directly reflect material hardness. Further investigation is required to determine the reasons for this, but it does highlight that care is needed when choosing magnetic parameters to correlate to material properties. It is interesting to note that the first peak in the T91-T100h $\mu_0$ curves only appears at higher minor loop amplitudes, this shows that a larger $\Delta H$ will enhance the range of magnetisation in the minor loop resulting in a different sensitivity to microstructure.

The results from the time derivative of the BH loop and DAME measurement system also demonstrate the ability of the DAME technique to evaluate some of the aspects of the major BH loop using a greatly simplified measurement system. In contrast to BH loop measurement, where a separate search coil wrapped around the sample is required, for DAME, only the voltage over the excitation coil needs to be measured. This requires much less complexity in the measurement system and also means that the technique can be deployed for a variety of sample shapes without a special search coil being constructed. However, further assessment of the relationship between the DAME and MBN/BH signals for a range of steel microstructures is required.

References