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Electromagnetic evaluation of the microstructure of Grade 91 tubes/pipes



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ABSTRACT

This paper assesses the feasibility of transferring a laboratory-based electromagnetic (EM) sensor technique, which has already proved sensitive to significant (e.g. phase balance) or subtle (e.g. number density of fine precipitates) microstructural changes in steel, to non-destructive evaluation of the microstructure of power generation components such as tubes/pipes. It has been found that Grade 91 steels, in different conditions representative of service entry, thermally aged or ex-service, can be distinguished using laboratory-based measurement systems on small machined cylindrical samples as well as by an industry deployment EM sensor system on full-diameter tube samples. The measurements for the tube samples follow the same trend as the machined cylindrical samples. The results indicate an industrial deployable sensor system can be used for sorting service-exposed or mis-heat-treated/mis-manufactured Grade 91 steel tubes/pipes from the correctly heat treated service-entry ones.

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1. Introduction

Grade 91 steels are widely used in the power generation industry for high-temperature components such as boiler tubes and steamline pipes. They are usually heat treated or supplied in the as normalized and tempered condition before entering service [1]. However, there is evidence that mis-manufactured, or mis-heat-treated, materials/components have been supplied [2]. Importantly, traditional methods for quality checking have not always identified the presence of the mal-heat treatment [3]. Using a component with an incorrect microstructure in service has resulted in product failure at times below that expected by that of Design Codes [4]. To avoid mis-heat-treated material from entering service a fast, accurate and non-destructive sorting tool is needed.

Commercial sorting tools such as the X-Ray fluorescence analyzer, which can sort steel grades by analyzing the alloy element contents, are not sensitive to microstructure and hence not able to distinguish between steels of the same grade but different microstructures due to different heat treatment conditions or service history. The conventional inspection techniques for microstructure assessment currently used by the power generation industries, such as examination of surface replicas or hardness measurements are only suitable for limited spot checks [3]. Moreover hardness measurement lacks specificity to fine microstructural detail, and similar information can result from steels with a surface decarburisation layer compared to mis-heat-treated steels. Thus, a non-destructive technique, to complement hardness testing, for the assessment of the microstructure in Grade 91 steels would be of significant benefit.

Magnetic hysteresis loop (or BH loop) and Magnetic Barkhausen Emission (MBE) techniques have been used to evaluate microstructural changes in Grade 91 steels during creep tests under 125 MPa stress at 600 °C [5,6] or tempering at different temperatures (ranging from 650 °C to 950 °C) [7]. Changes in the magnetic properties such as coercivity (H_c) and remanence (B_r) or the MBE parameters such as the peak height/position of the root mean square (RMS) of voltage profiles were correlated to microstructural

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Nomenclature

μ_r	relative permeability
ω	angular frequency
B	magnetic induction
B_r	remanence
H	magnetic field strength
H_c^*	coercive force for the non-saturated major loops for the tubes
H_c	coercivity
M	mutual inductance
M_0	inductance at low frequencies
Z	trans-impedance
EM	Electromagnetic
LMP	Larson–Miller parameter
MBE	Magnetic Barkhausen Emission
PIRMP	Peak Interval of Reversible Magnetic Permeability

changes (e.g. precipitation of large carbides or Laves phase [5,6]), mechanical hardness [7], or used to indicate different creep stages [5]. Moorthy reported double peaks in the MBE profiles for a P9 and a T22 steel after tempering for different times and correlated them generally to the domain walls overcoming pinning from precipitates or grain boundaries respectively [8]; however, the role of the precipitate location e.g. on the martensitic lath boundaries (expected to be minor) or within the laths (important role) was not considered. Bong et al. [9] reported evaluating remnant life of Grade 91 steels by looking at the relationship between a parameter called Peak Interval of Reversible Magnetic Permeability (PIRMP \approx twice the coercivity force) measured using a laboratory-based EM sensor system and the Larson–Miller parameter (LMP). It was reported that the PIRMP data were practically insensitive to the Larson–Miller parameter after a LMP of 21965 (i.e. equivalent to a thermal exposure at 610 °C for 75,000 h or about 8.5 years, which would still be in the early stage of the expected service life for a T91 steel tube) and whilst the mechanical properties, such as yield stress or tensile strength, are much more sensitive than the PIRMP to the LMP.

Multi-frequency EM sensors have proved sensitive to changes in ferrite (α)/austenite (γ), shown using model alloys, in-situ analysis and FE based modeling software [10,11]. Prototype EM sensors are being used for in-situ monitoring of the $\gamma \rightarrow \alpha$ transformation during steel processing [12]. EM sensors have also been used to detect decarburisation, shown with high carbon steels for on-line and off-line monitoring [13,14]. The theory as to how the relative permeability and resistivity of a sample affect the multi-frequency EM response, for any sensor geometry, is presented elsewhere [15]. It has been shown that the multi-frequency EM sensors are able to detect the initial relative permeability and resistivity changes, resulting from microstructural changes in P9 and T22 power plant steels during service at high temperatures [16]. The developed laboratory-based EM sensor is also capable of differentiating P91 steels with different N:Al ratios in the short term tempered or long term aged condition, based on the principle that the N:Al ratio significantly affects the number density of intra-lath MX carbonitride precipitates [17]. These precipitates determine the mean free path to magnetic domain wall motion and hence the initial relative permeability of the steels and the EM signals.

Whilst a variety of related EM systems for microstructural changes have been developed for laboratory-based assessment there are no reports of industrial deployable sensors which have been successful in sorting mis-heat treated materials. This paper

assesses the feasibility of transferring the present laboratory-based EM sensor technique that has already proved sensitive to microstructural changes, to non-destructive evaluation of the microstructure of power generation components such as steel tubes/pipes. Potential applications include sorting materials e.g. the mis-heat-treated or mis-manufactured materials from normal service-entry ones, amongst others, such as microstructural changes during service.

2. Materials and experimental details

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) as normalized at 1060 °C for 20 min and tempered at 780 °C for 1 h. The chemical composition of the steel is given in Table 1. Cylindrical samples (4.95 mm diameter and 100 mm length) were machined from the tube for EM sensor measurements using a laboratory-based cylindrical sensor and a laboratory-based BH loop measurement system. Selected cylindrical samples have been heat treated to different conditions in laboratory furnaces to simulate the microstructures expected of prolonged thermal exposure by accelerated tempering at 780 °C for 100 h or mis-heat treatments/mis-manufacturing. The accelerated tempering condition was chosen because it generated significant microstructural degradation and was also achievable for large demonstration tube samples. The mis-heat-treatment was simulated by the following procedure [18] in order to generate a partially ferritic structure rather than a fully martensitic one on air cooling:

- 1) heating up to 950 °C and dwelling for 30 min;
- 2) programmed furnace cooling by 100 °C/h down to 760 °C;
- 3) dwelling for 3 h;
- 4) cooling in still air.

These heat treatments were carried out on two lengths (250 mm length) of full diameter tube samples, for demonstration trials, in industrial furnaces. T91 tubes of approximately 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 superheater outlet header at 585 °C under 16.5 MPa pressure (designed) for about 50,000 h were supplied. The specific chemical composition of the steel is not presently available but the composition did comply with the applicable specification. Cylindrical samples (4.95 mm diameter and 50 mm length) were machined from the tube for EM measurements.

Metallographic samples were polished to a 0.25 μm diamond paste finish and etched in Kallings reagent. A Field Emission Gun Scanning Electron Microscope (FEG–SEM) and an EDS system were used to obtain SEM micrographs and analyze alloy contents at selected points/areas. Additional metallographic samples were polished to a 1 μm diamond paste finish followed by several etching–polishing cycles and a final polishing with OPS (oxide polishing suspension) for 10 min for electron backscattering diffraction (EBSD) analysis using an EBSD in a FEG–SEM. Lath boundaries and grain boundaries in SEM micrographs were reconstructed as trace features and objects respectively using Image–Pro Plus. Average distances between two neighbouring trace features were taken as the lath width.

A four-point probe technique was employed to independently measure the resistivity of the steels with a direct current Cropicco DO5000 micro-ohmmeter at room temperature using machined cylinder specimens of 4.95 mm diameter. The resistivity values were used as input into a Comsol model (described later) to determine the relative permeability values.

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