# Magnetic Detection of Microstructural Change in Power Plant Steels

Victoria Anne Yardley

Emmanuel College

This dissertation is submitted for the degree of Doctor of Philosophy at the University of Cambridge

### PREFACE

This dissertation is submitted for the degree of Doctor of Philosophy at the University of Cambridge. The research described herein was conducted under the supervision of Professor H. K. D. H. Bhadeshia and Dr M. G. Blamire in the Department of Materials Science and Metallurgy, University of Cambridge, between October 1999 and April 2003.

Except where acknowledgement and reference are made to previous work, this work is, to the best of my knowledge, original. This dissertation is the result of my own work and includes nothing which is the outcome of work done in collaboration except where specifically indicated in the text. Neither this, nor any substantially similar dissertation has been, or is being, submitted for any other degree, diploma, or other qualification at any other university. This dissertation does not exceed 60,000 words in length.

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In loving memory of Edward and Mary Yardley

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# ABBREVIATIONS

- b.c.c. Body-centred cubic
- ppm Parts per million
- **ABBM** Alessandro, Beatrice, Bertotti and Montorsi model
- $\mathbf{A}\mathbf{Q}$  As-quenched
- ${\bf BN}\,$ Barkhausen noise
- ${\bf CSL}\,$  Coincidence site lattice
- **EBSD** Electron backscatter diffraction
- FEG Field emission gun
- ${\bf FWHM}\,$  Full width half maximum
- **IQ** Image quality
- MAE Magnetoacoustic Emission
- **NDT** Nondestructive testing
- **ODS** Oxide-dispersion strengthened
- **OIM** Orientation imaging microscopy
- **PHD** Pulse height distribution
- **RMS** Root-mean-square
- **SEM** Scanning electron microscope
- ${\bf TEM}\,$  Transmission electron microscope
- **VSM** Vibrating sample magnetometer

# NOMENCLATURE

**Note:** Two SI systems for magnetics nomenclature exist, but the Sommerfeld system has been used throughout; equations not conforming to this system have been converted. A comparison table including the two SI systems and the cgs system can be found in Jiles (1998).

#### General

- d Grain diameter
- E Efficiency
- M Magnification
- $M_f$  Martensite-finish temperature
- $M_s$  Martensite-start temperature
- ${\cal P}\,$  Larson-Miller parameter
- t Time
- T Absolute temperature
- ${\cal T}_1$  Absolute heat source temperature
- $T_2$  Absolute heat sink temperature
- $T_M$  Absolute melting temperature

#### Magnetics

- ${\bf B}\,$  Magnetic induction
- $B_S$  Saturation induction
- $B_R$  Remanent induction
- $E_a$  Anisotropy energy

 $E_{area}$  Area reduction energy (Kersten model)

 $E_d$  Demagnetising energy

 $E_{demag}$  Inclusion demagnetising energy (Néel model)

- $E_{ex}$  Exchange energy
- $E_m$  Magnetostatic energy
- $E_{pin}$  Energy dissipated against pinning
- $E_{supp}$  Energy supplied
- **H** Magnetic field
- $H_C$  Coercive field
- $H_d$  Demagnetising field
- $H_e$  Weiss mean field
- $H_{max}$  Maximum applied field
- $H_S$  Field at which  $M = M_S$
- $K_1$  Anisotropy constant
- **M** Magnetisation
- $\mathbf{m}$  Magnetic moment
- $M_R$  Remanent magnetisation
- $M_S$  Saturation magnetisation
- $N_d$  Demagnetising constant
- P Barkhausen noise power
- $T_C$  Curie temperature
- V Voltage

- $W_H$  Hysteresis energy loss
- $\alpha\,$  Mean field constant
- $\beta$  Term characterising nearest-neighbour interactions
- $\gamma$  Domain wall energy
- $\delta\,$  Domain wall thickness
- $\lambda_{UVW}$  Magnetostrictive strain along  $\langle UVW \rangle$
- $\lambda_{si}$  Ideal magnetostrictive strain
- $\mu_0$  Permeability of free space
- $\mu'$  Differential permeability
- $\mu'_{max}$  Maximum differential permeability
- $\sigma$  Electrical conductivity
- $\chi'_{in}$  Initial differential susceptibility
- $\chi'_{max}$  Maximum differential susceptibility
- $\Phi\,$  Magnetic flux
- $\omega$ \* Surface pole density
- ${\mathcal J}\,$  Term characterising nearest-neighbour interactions

#### Modelling: existing models

- A, B Amplitude of fluctuations in ABBM
- k pinning parameter
- $M_{an}$  Anhysteretic magnetisation
- $M_{JS}$  BN jump sum

- $M_{rev}$  Reversible magnetisation
- $< M_{disc} >$  Average BN event size
- v domain wall velocity
- W noise term in ABBM
- $<\epsilon_{\pi}>$  Pinning energy for 180° wall
- $<\epsilon_{pin}>$  Pinning enrgy for wall at arbitrary angle
- $\xi$  Correlation length

#### Modelling: new model

- $A_i$  Total number pinning points of *i*th type per unit volume
- $\mathbf{A}_w$  Wall surface area
- C Constant
- E Fitting error
- $E_0$  Electric field amplitude
- $\mathbf{l}_w$  Wall jump distance
- $l\{H\}$  Distance between pinning sites at field H
- $\langle l \rangle \{H\}$  Domain wall mean free path
- $N\{H\}$  Number of pinning sites of strength  $\geq H$
- $n\{S\}$  Number pinning sites of strength S
- S Pinning site field strength
- $S_b$  Field at which unpinning first occurs
- $\langle S \rangle_i$  Mean value of S for *i*th type of pinning site

- $V\{H\}$  BN voltage at field H
- $V_r\{H\}$  Real  $V\{H\}$
- $V_p\{H\}$  Predicted  $V\{H\}$
- $\langle x \rangle$  Mean value of  $ln\{S\}$  for log-normal distribution
- $\beta$  Parameter depending on angle between adjacent domains
- $\Delta S_i$  Standard deviation of S for *i*th type of pinning site
- $\Delta x$  Standard deviation of  $ln\{S\}$  for log-normal distribution

#### **Orientation Imaging Microscopy**

- $\mathbf{c}_c$  Crystal coordinate system
- $\mathbf{c}_s$  Sample coordinate system
- d Planar spacing
- ${\bf G}\,$  Rotation matrix
- **M** Misorientation matrix
- $\langle UVW \rangle$  Misorientation axis
- $\nu_0$  Brandon ratio proportionality constant
- $\nu_m$  Maximum allowable deviation from ideal coincidence
- $\lambda$  Radiation wavelength
- $\theta$  Misorientation angle
- $\theta_B$  Bragg angle

## ABSTRACT

Power plant components are expected to withstand service at high temperature and pressure for thirty years or more. One of the main failure mechanisms under these conditions is creep. The steel compositions and heat treatments for this application are chosen to confer microstructural stability and creep resistance. Nevertheless, gradual microstructural changes, which eventually degrade the creep properties, occur during the long service life. Conservative design lives are used in power plant, and it is often found that components can be used safely beyond the original design life. However, to benefit from this requires reliable monitoring methods. One such technique involves relating the microstructural state to measurable magnetic properties.

Magnetic domain walls interact energetically with microstructural features such as grain boundaries, carbides and dislocations, and are 'pinned' in place at these sites until a sufficiently large field is applied to free them. When this occurs, the sudden change in magnetisation as the walls move can be detected as a voltage signal (Barkhausen noise). Previous work has suggested that grain boundaries and carbide particles in power plant steels act as pinning sites with characteristic strengths and strength distributions.

In this study, the concept of pinning site strength distributions was used to develop a model for the variation of the Barkhausen noise signal with applied field. This gave a good fit to published data. The modelling parameters characterising pinning site strengths showed good correlations with grain and carbide particle sizes.

New Barkhausen noise data were obtained from tempered power plant steel samples for further model testing. The Orientation Imaging Microscopy (OIM) technique was used to investigate the grain orientations and grain boundary properties in the steel and their possible role in Barkhausen noise behaviour. The model again fitted the data well, and a clear relationship could be seen between the pinning strength parameter and the severity of tempering (as expressed by the Larson-Miller tempering parameter) to which the steel was subjected. The experimental results suggest that the Barkhausen noise characteristics of the steels investigated depend strongly on the strain at grain boundaries. As tempering progresses and the grain boundary dislocation density falls, the pinning strength of the grain boundaries also decreases. A clear difference in Barkhausen noise response could be seen between a  $2\frac{1}{4}$ Cr1Mo traditional power-plant steel and an 11Cr1Mo steel designed for superior heat resistance.

A study of an oxide dispersion strengthened ferrous alloy, in which the microstructure undergoes dramatic coarsening on recrystallisation, was used to investigate further the effects of grain boundaries and particles on Barkhausen noise. The findings from these experiments supported the conclusion that grain boundary strain reduction gave large changes in the observed Barkhausen noise.