Chapter 11

Summary, Conclusions and Suggestions for Further Work

11.1 Summary and conclusions

The existing literature on the dependence of magnetic properties on microstructure has been reviewed. The consensus from this is that particles, dislocations and grain boundaries all affect the domain wall behaviour. The effect of particles depends on their size and is most pronounced when they are evenly spaced. Dislocations interact with domain walls and appear to suppress domain nucleation and annihilation, making a deformed sample more difficult to saturate than an annealed sample of the same material. Grain boundaries appear to exert a weaker pinning effect than carbides or dislocations, but they can nonetheless limit domain wall jump sizes.

In as-quenched or normalised and air-cooled ferritic steel, the RMS Barkhausen noise signal is a small peak at a high applied field. On tempering, this increases in height and moves to a lower field. Prolonged tempering causes splitting into two peaks. This was attributed to separate distributions of pinning strengths from grain boundaries and carbides. On tempering, the pinning strength of grain boundaries decreases, and that of carbides increases.

Using this interpretation, a model has been developed based on two statistical distributions of pinning site strengths. The first attempt at modelling treated these both as normal distributions, and the second (Model 2) used a log-normal distribution for the weaker sites. Both models fitted published data well, but linear relationships were observed between Model 2 fitting parameters and the dimensions of microstructural features.

Samples of austenitised, quenched and tempered $2\frac{1}{4}$Cr1Mo power plant steel were prepared. Changes in microstructure and hardness were slow at lower tempering temperatures, but accelerated at higher temperature. 11Cr1Mo steel samples, which had been heated for several thousands of hours, showed a very gradual change in hardness and no discernible microstructural coarsening. Orientation imaging microscopy observations on $2\frac{1}{4}$Cr1Mo samples tempered at 600°C revealed that the main change occurring was a strain reduction, which appeared as an increase in image quality. Even after tempering for over 250 hours, the relationships from the martensitic structure were preserved. Blocks, consisting of slightly misoriented groups of laths, often had $\Sigma3$ relationships with their neighbours within a packet. It is expected that, as the dislocation density reduces during tempering, it will become progressively easier for a domain wall to move through the structure.

The BN peaks obtained from these samples were different in shape from those observed previously on tempered $2\frac{1}{4}$Cr1Mo steel. Peak-splitting had been expected after prolonged tempering in the $2\frac{1}{4}$Cr1Mo samples, but it was not observed; neither could it be seen in tempered plain-carbon steel samples which were known to give a double peak using other apparatus. It was suspected that the filtering in the apparatus used in this study excluded important low-frequency information. Analysing only low-frequency noise gave evidence of a second peak at high current values.

The BN peak moves toward lower currents with larger Larson-Miller parameter P in the $2\frac{1}{4}$Cr1Mo samples. In the 11Cr1Mo steel, the peak remains at a position close to that of the as-quenched $2\frac{1}{4}$Cr1Mo samples, even after prolonged tempering. This agrees well with the lack of microstructural change and the very small hardness change in this sample.

Model 2 was fitted to the new experimental data, and in all cases, a good fit was obtained. The model parameter characterising the pinning strength of the weaker pinning points decreased with P, following the same curve shape as the peak positions, in the $2\frac{1}{4}$Cr1Mo steel. This weaker distribution is believed to arise from pinning by grain boundaries. Taking into account the optical and OIM measurements, it can be concluded that the pinning strength of this distribution is related to the degree of strain in the microstructure, and in particular the dislocation density at lath and grain boundaries. As this is decreased by tempering, domain wall motion becomes easier. The pinning strength of the distribution attributed to carbides had a minimum at an intermediate value of P. This may be associated with the dissolution of Fe_3C and the precipitation of fine particles of more stable carbides, since it occurs at an appropriate value of P as determined from the carbide stability diagram.

The modelling parameter values also suggested that the number of grain- or lath-boundary pinning sites increased with greater severity of tempering. In reality, the opposite is likely, since lath boundaries become less well defined on tempering. The initial assumptions made when developing the model were very simple, so it may be necessary to modify some of these to obtain a physically meaningful model of the number of sites.

The fitting parameters for the 11Cr1Mo steel lay in a different regime from those of the $2\frac{1}{4}$Cr1Mo steel, and their variations with P could not be determined because all the points had very similar P values. More data, from a wide range of tempering conditions, are needed to study this.

In the ODS alloy, it was possible to isolate the effects of grain boundaries and particles, and to demonstrate that both influence magnetic properties. However, possibly because of the small size of the particles in this material, grain boundaries dominated the Barkhausen noise behaviour. The Barkhausen peak moves from a higher to a lower applied current on recrystallisation from a fine-grained, heavily strained microstructure to coarse columnar grains. This is comparable to the change in peak position occurring on tempering of $2\frac{1}{4}$Cr1Mo steel, and lends support to the interpretation that the $2\frac{1}{4}$Cr1Mo steel BN behaviour is mainly due to effects at grain boundaries.

In summary, the large Barkhausen noise peak observed in these steels is believed, on the basis of experiment and modelling, to originate from domain wall interactions with grain and lath boundaries. During tempering, the dislocation density at these boundaries decreases, and this reduces their pinning strength. The peak therefore shifts to a lower field. The peak position, and the modelling parameter based on this, decrease smoothly with increasing severity of tempering, as characterised by the Larson-Miller parameter.

11.2 Future work

11.2.1 Experimental work

Since there is a clear relationship between BN peak position and P in the $2\frac{1}{4}$Cr1Mo steel, it would be interesting to investigate whether similar relationships exist in other steel compositions, including an extension of the existing work on 11Cr1Mo. Comparisons with samples which are in an unsatisfactory microstructural condition, both overcoarsened and creep-deformed, should be obtained so that criteria for a safe condition can be determined.

Two new sets of BN measuring apparatus, of types (a) and (b) in Figure 4.2, are being built at the University of Newcastle. The filtering ranges of these will be set to allow analysis of low-frequency noise. Repeating the experiments in this study using the new apparatus should enable high-field, low-frequency peaks to be detected where these exist. Careful characterisation of the effects of changing experimental conditions should be carried out to enable repeatability between results. Once this has been achieved, a wide variety of samples can be tested to verify existing interpretations of the role of microstructure in Barkhausen noise, and to gain new insights.

Time limitations prevented a detailed investigation of the carbides in the $2\frac{1}{4}$Cr1Mo steel in this study. Carbon replica measurements can be used to measure particle shapes and sizes, and the identity of the species determined using electron diffraction in the TEM. These can be compared with BN signals obtained from a full frequency range. As Moorthy *et al.* (1998) suggested, it may be possible to relate changes in the BN behaviour to the dissolution of the fine needles of M_2X which are beneficial to creep properties, and the precipitation of spheroidal carbides in their place. This would be extremely useful for estimation of the creep resistance of the microstructure.

Finally, magnetic domain imaging, using the techniques described in § 3.4, could be used to give a more complete understanding of the interaction processes between domain walls and microstructural features in these steels.

11.2.2 Modelling

The Model 2 average pinning site strength values have clear relationships with microstructural or tempering parameters, but the other parameters, characterising the number of sites and the distribution widths, are less obviously related to microstructure. This may well be due to the assumptions used in the model, so it would be useful to test modifications to these, to attempt to produce a more physically reasonable model. For example, it may be necessary to introduce a demagnetising field, a variable number of domain walls, or a more complex arrangement of pinning sites.

Modification of the model fitting program is also necessary to eliminate unphysical solutions. This could take the form of constraints on allowable fitting parameter values. A further suggested alteration to the fitting procedure is discussed in the Appendix.