Influence of microstructural constituents on the hysteresis curves in 0.2%C and 0.45%C steels

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Abstract. Steels with 0.2% and 0.45%C were submitted to different types of heat treatment, leading to different microstructures, with different amounts of the microstructural constituents: ferrite, pearlite and martensite. Measurements of magnetic hysteresis and Barkhausen noise were performed at different frequencies. A relationship was found between coercive force and the volume fraction of the microstructural constituents. The influence of the microstructural constituents on the shape of hysteresis curve is also discussed. The presence of martensite leads to more magnetization reversal by domain rotation, reducing the relevance of domain wall movement as a reversal mechanism. When domain wall movement predominates, permeability increases and coercivity decreases. As a consequence, when the martensite volume fraction increases, coercivity also increases. The results help clarify the relation between microstructure of steels and Barkhausen noise measurements in non-destructive testing.

1. Introduction

Non-destructive evaluation (NDE) is becoming increasingly important in modern industry, contributing both to quality control on the production line and in monitoring equipment during operation. In ferromagnetic materials – carbon steels in general – the microstructural characteristics play a decisive role in determining structure sensitive magnetic properties, for example, magnetic hysteresis curves or Barkhausen noise. For this reason, a better understanding of the relation between microstructure and magnetic properties is always useful. Some time ago, Néel [1] had argued that it is possible to interpret the effects of iron carbide precipitates on the magnetic properties in terms of the pinning of domain walls by the inclusions. Dijkstra and Wert [2] assumed that magnetostatic energy is stored around second phase particles (carbides and inclusions) and that magnetostatic energy can be decreased when domain walls intersect the particles, causing the walls to be pinned by the inclusions. More recently, Jiles [3] presented a comprehensive discussion of the influence of carbon content on the properties of the hysteresis loop for several carbon steels with up to 1wt% C. That work concluded that domain wall motion was mainly influenced by the presence of iron carbide particles, either in lamellae or spheroids, while grain boundaries were of lesser importance. The models of [1,2] were recently
applied to the phenomenon of magnetic aging [4], a decline of the magnetic properties due to the growth of carbide particles.

Since the measurement of Barkhausen noise is an important NDE technique, the present work was undertaken as an initial step to investigate the effect of microstructure on Barkhausen noise and magnetic properties for a couple of important steels: steel containing 0.45wt% C can be hardened by heat treatments or other means, while steel with 0.2 wt%C is a common structural material. The effect of microstructure on Barkhausen noise peak height, position, and frequency spectrum has been considered previously [5-8].

2. Experiment
Two steels were studied in this work, nominal AISI 1020 (actually C: 0.26; Si: 0.19; Mn: 0.59; P: 0.02; S: 0.02 in weight %) and AISI 1045 (actually C: 0.48; Si: 0.24; Mn: 0.80; P: 0.02; S: 0.03 in weight %). Samples were prepared in the form of toroids to facilitate magnetic measurements. All toroids were austenitized at 910˚C (±10˚C). After austenitization they were placed in another furnace for 20 minutes at temperatures of 700˚C, 650˚C, 600˚C, 550˚C, 500˚C and then cooled in air. Normalization consists of complete austenitization followed by air cooling. Heat treatments are listed in the table below.

| Number | Heat Treatment          |
|--------|-------------------------|
| 1      | Normalization           |
| 2      | Water quench            |
| 3      | Oil quench              |
| 4      | Cool from 700˚C         |
| 5      | Cool from 650˚C         |
| 6      | Cool from 600˚C         |
| 7      | Cool from 550˚C         |
| 8      | Cool from 500˚C         |

Metalographic preparation was with Trident and Buehler Microcloth with diamond suspensions of 15, 9, 6, 3, 1, ¼ µm, followed by attack with Nital and examination with a Zeiss AX10 optical microscope and/or a Zeiss EVO 40 scanning electron microscope. After heat treatment, toroidal samples were wound with a primary and secondary for magnetic measurements. A hysteresis loop tracer from Globalmag Ltda. generated a sinusoidal current (f = 0.05 – 400 Hz) which drove the primary. The secondary voltage was integrated to furnish the magnetic induction. The coercive field is always taken to correspond to B = 0. For Barkhausen noise measurements an additional, localized secondary was wound on each toroid. Noise measurements were made on the toroids with a home made system, using a sinusoidal magnetic field varying at 1 Hz. The signal was filtered (5–100 kHz) and amplified, after which noise data were collected (16 bits, 250 000 samples per second) and stored for later analysis. The values presented for the rms Barkhausen noise are the result of averaging over 10 pulses.

3. Magnetic measurements
Hysteresis curves for samples quenched in oil are shown in Fig. 1 for a measuring frequency of 0.5 Hz. Quenching results in the introduction of martensite into the microstructure which has the effect of increasing the coercive field substantially in regard to those samples which were slowly cooled. In Fig.
2. \( H_c \) is shown for two frequencies (0.5 Hz and 1 Hz) for all of the heat treatments. Eddy current effects were very strong and led to large increases in the coercive field. The eddy current effects were larger than the small difference between the coercive fields of 1020 and 1045 steels which were slowly cooled from 500-700°C. Previously, for lamellar pearlite samples, Jiles [3] had encountered \( H_c = 10.5 \) Oe for the 1045 steel, but only \( H_c = 6.5 \) Oe for the 1020 steel. Of course, our steels and those of Jiles are not exactly the same, since both the residual elements and the grain size are different. The amount of Mn and Si should influence the number and size of pinning sites which might lead to increased domain wall pinning and increased \( H_c \), for example.

Fig. 1 Hysteresis curves for 0.5 Hz.                     Fig. 2 \( H_c \) vs. heat treatment.

Eddy current effects lead to a strong dependence of the coercive field upon frequency and eventually result in the distortion of the hysteresis curves. This frequency dependence of \( H_c \) below 20 Hz can be seen in Fig. 3 below.

Initial permeability \( \mu_0 \) and maximum permeability \( \mu_{\text{max}} \) are shown above in Fig. 4 for the 1020 and 1045 steels. The quenched samples (2 and 3) have lower permeability values indicating that wall movement is becoming more difficult. Since martensite consists of nanocrystalline, lens-shaped grains of \( \alpha \)-Fe with carbon inducing stress in the lattice and with a high dislocation density [9], domain wall movement in such nanocrystalline grains is difficult, and domain rotation becomes relevant as an inversion mechanism. This leads to low permeability and high coercivity.

In Fig. 5 below, the rms Barkhausen voltage associated with the two steels is shown. Since the rms Barkhausen noise is related to domain wall movement, this explains the clear relationship between Figs. 4 and 5. The martensite is much harder magnetically than the ferrite-pearlite and its contribution to the Barkhausen noise is smaller than that of the softer material. The 1045 steel has more martensite (Compare Figs. 9 and 10 below.) and therefore shows a reduced Barkhausen noise voltage for samples 2 and 3. A large (magnetic field) separation of the Barkhausen contributions in two-phase steels has been observed previously [7]. Analyzing this two-peaked structure, Kleber \textit{et al.}
[7] found a good correlation between the martensite volume fraction and the Barkhausen noise amplitude. Gür and Çam [8], studying SAE 4140 steel, found a reduced rms Barkhausen contribution from martensite, but much larger contributions from the ferrite-pearlite. Furthermore, the peak fields associated with the several constituents were not widely spaced.

In Fig. 6 we see that the peak fields for the 1020 steel after treatments 2 and 3 are larger than those for the other treatments. But this difference is more striking in the case of the 1045 steel which contains more martensite in its microstructure. See Figs. 9 and 10 below. Recently, a detailed discussion of the factors which influence the peak fields has been reported by Kleber et al. [10] in their study of commercial dual-phase steels. Their results show [10] a linear decrease of $H_p$ with increasing volume fraction of ferrite. This result might be consistent with the larger values of $H_p$ that we observe for the quenched samples (heat treatments 2 and 3) since, for these samples, we find much less ferrite and more martensite. The authors of Ref. 10 attributed the shift in peak position to the distribution of magnetic flux in the two phases and presented a model for the phenomenon which seemed to be consistent with their measurements. The microstructures in the present samples, however, are more complex than those found in [10]. We also see in Fig. 6 that the peak fields are lower than the corresponding coercive fields of Fig. 2 above. This seems to indicate that there is significant domain wall movement before the magnetic field reaches $H_c$.

**Microstructure**

Optical micrographs of the normalized samples are shown in Figs. 7 and 8 below.

Normalized samples do not contain martensite, only pearlite and ferrite. However, normalized hypoeutectoid steels generally show less pro-eutectoid ferrite and a finer pearlite than their
corresponding numbers which have been austenitized and treated isothermally (samples 4 – 8). Thus, from the microstructure, one might have expected \( H_c \) to be slightly greater for the normalized samples (number 1) than for those steels which were treated isothermally. However, the \( H_c \) values found seem to be very similar to those of materials treated in the range 500-700ºC.

Microstructures of the oil-quenched samples are shown in Figs. 9 and 10, as obtained by optical microscopy. The samples with a higher C content always present more martensite when quenched and more pearlite for other heat treatments. Nital leaves the ferrite white in the micrographs, the darker phase being the martensite. The martensite can also be identified by the needle-shaped grains, a consequence of their lenticular shape in three dimensions.

In the samples which were slowly cooled from 500-700ºC, we expect to find ferrite and pearlite. Typical microstructures from materials cooled from 700ºC are found in Figs. 11 and 12 below. The white regions correspond to ferrite and the darker regions with a lamellar structure are pearlite.

Typical microstructures from material cooled from 500ºC are found in Figs. 13 and 14 below. The spacing of the lamellae is slightly smaller than the spacing in the material cooled from 700ºC. The interlamellar distance can be calculated from the Zener equation [11,12]. Thus it might be expected that a smaller value of \( H_c \) would be encountered in the material cooled from 700ºC. A reduction in \( H_c \) with increasing interlamellar distance has been reported by Byeon and Kuwn [13]. However, the effect must be small and we did not observe a significant difference in \( H_c \) between heat treatments 4 and 8.
4. Conclusion
A correlation between microstructure and magnetic properties has been observed which confirms an ordering of magnetic hardness: martensite > pearlite > ferrite. Samples having greater amounts of C have more martensite upon quenching and more pearlite in other heat treatments. Thus the 1045 steel had a larger $H_c$ than the 1020 steel for all of the heat treatments considered.

Martensite is much harder magnetically than the ferrite-pearlite and its contribution to the Barkhausen noise is reduced and displaced in magnetic field. In two-phase steels, the contributions to the Barkhausen noise from martensite and ferrite are well separated in magnetic field and have been used to determine the relative proportions of the two phases and the carbon content of the martensite [7]. In the three constituent steels considered here, the problem is more complex, in part because the contributions of the individual constituents are not so easily separated.

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