Magnetic Barkhausen Noise in quenched carburized steels

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Abstract. Steels with different carbon content, 0.11%C and 0.48%C were submitted to a heat treatment for carburization in the surface. The samples were analyzed after several types of heat treatment, including quenching for producing martensite. The Magnetic Barkhausen Noise (MBN) is directly related to the microstructure. Samples with lower carbon content, have ferrite, a constituent where domain walls can move freely and present higher amplitude in the envelope of MBN. It is also found that the MBN peaks are quite distinct for the samples with martensite, which have lower permeability, and the results suggest that domain rotation contributes as mechanism for reversal of magnetization in martensite. The results also indicate that MBN is very suitable for monitoring the carburizing heat treatment.

1. Introduction
Magnetic Barkhausen Noise is a very important tool for non-destructive characterization [1-4]. Most of the MBN signal is attributed to 180° domain wall movement [1-4]. In steels, microstructural defects like grain boundaries, inclusions and dislocations promote both mechanical and magnetic hardening, increasing the area of hysteresis curve and reducing permeability (especially the initial permeability). This happens because the same defects which “pin” dislocations also may “pin” domain walls. There is strong relationship between microstructure and magnetic properties and this can be used for non-destructive evaluation. However, an important remark should be made: Dislocations are linear defects (i.e., 1 dimension=1D) with ~5 Angstroms of thickness. Domain walls, sometimes considered as planar 2D, are in fact 3D (volume), and have ~1000 Angstroms of thickness (in steels). Thus, atoms in solid solution can pin dislocations, but they cannot pin domain walls. On the other hand, atoms in solid solution can generate stress in the lattice, and this has effect on the hysteresis curve and on the MBN signal [5].

Quenching of steels promotes martensite and other fine microstructural constituents, like bainite. In such fine microstructures domain wall movement is more difficult, and domain rotation can be important as magnetization reversal mechanism. The following Mechanical Hardness relationship holds: Martensite > Pearlite > Ferrite. Martensite microstructure is very hard, due to: (i) Large number of interfaces (ii) High density of dislocations (iii) Carbon in solid solution generating stress in the lattice [6].
The carburizing process is an important industrial process, where a high carbon content layer is produced in the surface, promoting high resistance against abrasion in the surface, after a subsequent quenching. The MBN can be used to non-destructive evaluation of the carburizing process, and formation of that above mentioned high-resistance high-carbon layer, as it will be discussed thoroughly in this study.

**Experimental**

The nominal chemical composition of the studied steels is presented in Table I. The heat treatments were made in 2 steps, with the 1\textsuperscript{st} step having the objective of austenitizing the steel (austenitizing is the name of the heat treatment in the gama-iron (face centered cubic) field of the Fe-C phase diagram).

1\textsuperscript{st} step: (850°C for 2 hours)
2\textsuperscript{nd} step: carburization at 1050°C during 8 hours (in a CO-rich atmosphere)

Group 1: Carburized + quenching in oil. Step 1 + Step 2+ quenching in oil
Group 2: Only Carburized. Step 1 + Step 2, plus cooling in air.
Group 3: Normalized*. Only step 1, followed by a normalization heat treatment
Group 4: As received.

*) When an annealed sample is removed from the furnace and allowed to cool in air, this is called a normalization heat treatment

| Alloy    | C     | Mn    | Si    | P      | S      | Cr    | Ni    | Mo    | V      | Cu    | Al    | Ti    | Nb    | Sn    |
|----------|-------|-------|-------|--------|--------|-------|-------|-------|--------|-------|-------|-------|-------|-------|
| 1048NH   | 0.47  | 0.83  | 0.33  | 0.010  | 0.026  | 0.16  | 0.11  | 0.029 | 0.007  | 0.05  | 0.013 | 0.009 | 0.014 | 0.008 |
| 1011E    | 0.10  | 0.45  | 0.27  | 0.012  | 0.004  | 0.22  | 0.09  | 0.029 | 0.006  | 0.07  | ---0  | 0.010 | 0.014 | 0.009 |

MBN measurements were carried out with a portable equipment, named “Barktech”, developed at our laboratory in USP. Figure 1 shows a simplified sketch of this equipment. The MBN sensor signal was amplified, band pass filtered. Each measurement condition was repeated 10 times.

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Figure 1. Experimental setup showing the U-shaped yoke and sample. The magnetic circuit is also indicated in the figure. It should be noted there are three different coils in the system.
In the Yoke of Figure 1, an alternate current $I$ circulates in a coil, producing a field $H$, and this current has controlled amplitude, shape and frequency. This current produces a magnetization cycle in the studied material. There is also a very sensitive reading coil between the Yoke poles, to detect the magnetic pulses caused by the abrupt movement of the domain walls (The Magnetic Barkhausen Noise). A third coil is placed in the Yoke (see Fig. 1), monitoring the magnetic induction and measuring the induced Voltage $V$. The envelope of MBN is calculated using the MBN signal. A filter is used to eliminate very high frequencies of the envelope. The upper cut-off frequency was 200 Hz. The signals were acquired in the following range of frequencies: 1 Hz - 200 Hz. Microstructures were obtained in SEM Zeiss EVO MA 10, 25 kV. X-Ray Diffraction spectra were measured in equipment Shimadzu 6000, with Co K-alpha radiation.

**Results and Discussion**

The Microstructures of the samples are shown in Fig 2 and Fig. 3. For both samples, 1011 and 1048, the microstructures of groups 3 and 4 are very similar, indicating that the as-received samples probably received the normalizing heat-treatment, which is typical in the steel industry. The microstructure of group 1 is martensitic for both samples 1011 and 1048. The microstructures of samples group 2 is pearlitic, for samples 1001 and 1048. The microstructures of samples 1011, groups 3 and 4 are ferrite (i.e., alpha-iron) with some islands of pearlite, and it should be noted that in ferrite the free movement of domain walls is likely. Nevertheless, the microstructure of samples 1048 is basically pearlite with some islands of ferrite, and the moving of domain walls is more difficult. X-ray diffraction (XRD) has revealed large peak broadening for the samples of group 1(carburized and quenched), which have martensite. This is due to the high density of dislocations and high internal stress in the martensite.

Fig. 2 Microstructure for the samples 1011. Above left group1. Above right group 2. Below left group 3. Below left group 4. Images obtained near the carburized surface for groups 1 and 2.
The MBN envelope (see Fig. 4) is quite distinct for the quenched samples (group 1), which have martensite, and this is in agreement with several other studies [5,7,8]. Martensite consists in lens shaped nanocrystalline grains of alpha-iron supersaturated in carbon [6], and in this fine constituent the domain wall movement is difficult and domain rotation processes may contribute more significantly. Besides, carbon in solid solution introduces stress in the body-centered cubic (bcc) lattice of iron and affects magnetic properties. When martensite is present, it reduces the permeability (see Figure 5). Thus, Figures 4 and 5 confirm each other. The hysteresis (see Fig 5) obtained from the signals of the coils in the yoke is complementary for the MBN data, and indicate the same trend observed with the MBN: samples with lower permeability also have lower RMS of MBN. It has been reported that changing carbon content and pearlite volume fraction, the envelope of MBN is affected [9,10]. The data of Figure 4 also shows that the applied current may have effect on the envelope. For example the envelope of martensitic samples (group 1) present double peak for high current, and only one peak for the lower current, see Fig. 4. The following values (910.7 A/m and 535.7 A/m) of field intensity were chosen because they allowed signal strong enough for the calculation of the envelopes, with also good repeatability. The excitation frequency for the hysteresis (10 Hz, see Fig. 5) was chosen because it was also used for the MBN measurements. The frequency of 10 Hz allows rapid measurements, with good penetration of the applied field. However, when increasing the excitation frequency, the area of the hysteresis increases, due to eddy currents. Thus, eddy currents also affect the shape of the hysteresis, but in this case this effect is small, because the frequency is not very high.
Field A = 910.7 A/m

Field B = 535.7 A/m

Figure 4. MBN envelopes for 1001 and 1048 samples, measured with 2 different currents

Figure 5. Hysteresis curves obtained from the current of the 1st coil and with the Voltage induced in the 3rd coil (see Fig. 1). These hysteresis curves are qualitative, but provide an interesting comparison between the samples, since the samples have the same geometry. This analysis indicates that the martensitic samples (group 1) have lower permeability.

In the Figure 5, hysteresis curves obtained from the magnetic circuit comprising the U-shaped yoke and sample surface are presented (see Fig. 1). A hypothetical $H$ field could be obtained
from the data of figure 5 using the expression: \( H = n \frac{I}{L} \), where \( n \) is the number of turns in the excitation coil, \( I \) is the applied current and \( L \) is the length of the magnetic circuit. A hypothetical magnetic induction \( B \) could also be estimated using data from Fig. 5 with the expression:

\[
B = \frac{1}{nA} \int (\frac{d\phi}{dt}) \, dt : [T]
\]

where: \( n \) is the number of turns in the third coil (see Fig. 1); \( A \) is the transverse area of the yoke \([m^2]\); and \( \frac{d\phi}{dt} \) is the variation of magnetic flux, or the Voltage measured in the third coil (see Fig. 1).

It should be also noted that the analysis presented in Figure 5 only makes sense if the magnetic permeability of the samples is lower than the permeability of the material of the yoke.

**Conclusions**

It is suggested the use of the BxH curve obtained from coils of the yoke as complementary information, besides the MBN. The employed analysis (MBN and Hysteresis with excitation coil) requests samples with identical geometry, because of the magnetic circuit. The qualitative hysteresis obtained from the yoke indicate that the martensitic samples have lower permeability. It is also found that the MBN envelopes are quite distinct for the martensitic samples when compared to the ferritic/pearlitic samples. The results allow establishing a relationship between the MBN and the microstructure, as function of the carbon content, and indicate that MBN is very suitable for monitoring the carburizing heat treatment.

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