Ferrite–Martensite Steels Characterization Using Magnetic Barkhausen Noise Measurements

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Magnetic Barkhausen noise measurements have been carried out to characterize Ferrite-Martensite steels. Using thermal treatments, the volume fraction and the carbon content of martensite were varied in reasonable proportions. The results indicate that the Barkhausen noise measurements can easily allow us to distinguish the two phases, ferrite and martensite. Using a simple mixture rule, we found a good correlation between the martensite volume fraction and the Barkhausen noise amplitude. We observed that the value of the magnetic field for which the Barkhausen noise peak is obtained, correlates with the carbon content of martensite.

We demonstrated that it was possible to use Barkhausen noise measurement to determine both the relative proportion of the two phases (ferrite and martensite) and the carbon content of martensite. The use of this technique could be extended to characterize industrial Dual-Phase steels.

KEY WORDS: dual phase steels; Barkhausen noise; martensite characterization.

1. Introduction

Dual-Phase steels (DP), containing martensite and ferrite, present higher mechanical properties than those of most of the available high strength, low alloy steels. Their superiority is mainly due to the reinforcement of the ductile ferrite matrix by small amounts of martensite islands. Continuous yielding, a low yielding ratio, a high work hardening rate and an important elongation are the main mechanical properties which characterize Dual-Phase steels.1)

One of the most promising uses of these steels is their potential for vehicle parts. DP are generally produced by an inter-critical annealing in the austenite–ferrite region followed by a rapid water quenching to transform all the austenite into martensite phase. During this transformation, the expansion of the austenite to martensite could result in internal stress in ferrite as well as the creation of dislocations that can modify the mechanical properties of Dual-Phase steels.

Due to the multiphase nature of DP steels, most of the usual characterization techniques are unable to make a distinction between the information provided by each of the individual phases, and give a more general information. Recently, using the neutron diffraction technique, Filippone et al have succeeded in determining the martensite volume fraction in DP steels using the ferrite peak broadening,2) whereas Waterschoot et al have studied the low-temperature ageing of martensite in DP steels3) with the same technique. However, even if the results are convincing, such a technique is not easily applicable for non destructive purpose.

There is obviously a lack of experimental tools which could give us information about each phase.

The magnetic Barkhausen noise technique, which is available for ferromagnetic materials, has been successfully employed for the microstructural characterization of steels bars. One of its main advantages is the possibility to separate the information provided by each phase in multiphase materials. A second advantage is that due to the magnetoelastic coupling, this non-destructive technique is sensitive to the stress state inside the materials, and has frequently been used to assess the residual stresses.

The aim of this study is to examine the possibility of using this magnetic technique to determine the different characteristics of Dual-Phase steels. We will present the Barkhausen noise results obtained on model materials (ferrite–martensite steels) with different carbon contents and a high proportion of martensite. Our first step, is to validate Barkhausen noise technique in assessing ferrite-martensite properties in order to apply it to industrial DP steels.

2. Background of the Barkhausen Noise Technique

2.1. Barkhausen Effect

The existence of ferromagnetism, i.e. of a spontaneous magnetization considered as an atomic phenomenon, arises from (1) a combination of the presence of a magnetic moment associated with the spin of an electron; (2) the presence of a field of interaction between electron spins of neighbouring atoms aligning the spin moments parallel to each other.4) From a macroscopic point of view, due to the
minimization of the magnetostatic energy, the magnetic moments of atoms reorganize into specific magnetic structures called Weiss domains. Each domain is characterized by a constant magnetization and is separated from its neighbours by finite frontiers called Bloch walls. When a magnetic field is applied, such magnetic structures are reorganized and if the Bloch walls are not totally free to move under the influence of this field, sudden and irreversible motions occur. Then the local magnetization variations induced give rise to the so-called Barkhausen effect.5)

The recording of the sudden jumps of Bloch walls results in the Barkhausen noise signal which is a signature of the microstructural state of the crystal. Usually, instead of recording these numerous jumps, the envelope of the signal is plotted as a function of the applied magnetic field (Fig. 1). In most cases, the envelope has a single-peak shape and can be characterized by different parameters, such as the maximum noise amplitude (namely BNA) and the corresponding magnetic field (Hpeak).

2.2. Influence of the Metallurgical State of Steels

Many studies have shown that these wall displacements, and thus the Barkhausen effects, are directly linked to the local pinning of walls by obstacles such as inclusions,6) precipitates,7,8) grain boundaries,9,10) as well as dislocations tangles.11,12) The main observation drawn from these studies is that the increase in strength and in number of pinning obstacles leads to an increase of the Barkhausen noise signal. The microstructure becomes magnetically harder and the Barkhausen noise peak shifts to a higher value of the magnetic field.

Furthermore, as the magnetic structure is directly linked to the nature of the metallurgical state, different phases may produce a very different Barkhausen effect. Numerous studies on the Barkhausen noise response of various steels reveal that it is strongly dependent on the existing phases. For example, ferrite and pearlite microstructures have a strong Barkhausen activity located at a low magnetic field,13,14) contrary to a martensite microstructure which has a low Barkhausen emission located at a high field.15)

More recently, Barkhausen noise techniques have also been successfully used for the characterization of new materials such as austempered ductile iron.16)

2.3. Effect of Stress

Stresses, be they internal or external, interfere in the process through magneto-elastic coupling, and so modify the Weiss domain dynamic re-organization. Many studies have observed that, for common steels, a uniaxial tensile stress leads not only to an increase of the Barkhausen noise activity but also to a shift of this activity towards low magnetic fields, whereas a uniaxial compressive stress decreases it, broadens it, and shifts it towards high magnetic fields.17–20) Sometimes, in the latter case, adjacent peaks are observed which are usually attributed to movements of the 90° Bloch walls.

3. Experimental Details

3.1. Barkhausen Noise Measurements Set-up

A diagram of the equipment used to perform the Barkhausen noise measurement is presented in Fig. 2. The magnetic field is applied to the samples using a U-shape core with 1000 turns coil wound around it. The equipment is specially designed to obtain a well-controlled excitation magnetic field varying quasi-linearly with time, with a frequency of 0.5 Hz. A fixed rate of magnetization was imposed on the sample with the greatest of care in order to ensure a perfect reproducibility of the measurements. The magnetic field was measured using a Hall effect probe located at the surface of the sample. The magnetic Barkhausen noise is detected through a 300 turns coil wound around a Polyvinylchloride (PVC) support. The induced voltage is pre-amplified (40 dB), passed through a high-pass filter with a cut-off frequency of 500 Hz. The envelope of the signal is obtained using an analogue Root-Mean-Square (RMS) device with an integrated constant time of 25 ms. Finally, the RMS signal is amplified (40 dB) to obtain a usable signal which can be acquired using a Data Acquisition (DAQ) card plugged into a computer. To suppress the inherent noise due to the electronic environment 10 signals were averaged for each measurement. For each curve, the background noise is removed.
Barkhausen noise is the same for opposite values of the field, only the positive part of the curve is represented.

### 3.2. Description of Steels

The Dual-Phase steels employed are generally made up of less than 20% martensite for 80% ferrite. The carbon content of the martensite phase is generally very high, higher than 0.4% in weight. In order to simulate such a composition, we have chosen three steels with a relatively high carbon content, i.e., 0.34% (steel B), 0.48% (steel C) and 0.63% (steel D). To simulate ferrite materials, we also used a 0.07% carbon content steel (steel A).

The chemical composition of the steels can be found in Table 1. Notice that they contain an important quantity of Manganese, i.e., in between 0.650–0.800 wt%.

Since the Barkhausen noise measurement is sensitive to the size of the sample, each specimen was cut to a perfectly controlled size (0.4 mm × 6 mm × 70 mm).

### 3.3. Thermal Treatments

Ferrite–martensite steels were obtained by intercritical annealing in a salt bath for 5 min at temperatures ranging from 720 to 860°C. In order to slightly change the volume fraction of martensite, a temperature increment of 10°C was applied in the range 750–820°C. A rapid water quench was then performed to transform the austenite into martensite. As the Barkhausen measurement is a surface measurement, the samples were cleaned by immersion in a dilute HCl bath for a few seconds after each thermal treatment.

For some temperatures, several samples were used and no significant difference in the Barkhausen noise signal was observed.

### 4. Results and Discussion

#### 4.1. Barkhausen Noise Response of Ferrite and Martensite Steels

In the assessment of the characteristics of ferrite–martensite steels using Barkhausen noise techniques, we first performed measurements on ferrite and martensite steels. During annealing between 750 and 860°C, the carbon content of the martensite changes drastically whereas that of ferrite is slightly reduced. To simulate changes in the martensite composition which can occur at different annealing temperatures, steels B, C and D were totally austenized, respectively at 860, 850 and 800°C for 5 min and then water quenched.

A ferrite sample was obtained from steel A after an annealing treatment at 700°C followed by a rapid water quench. The carbon content in the ferrite phase is then close to the maximum limit of solubility, i.e., 0.02% in wt.

Barkhausen noise responses are plotted on Figs. 3(a) and 3(b) for ferrite and martensite samples. The Barkhausen noise signal for ferrite steel is fine, located at a low magnetic field (250 A/m) and shows a high amplitude. However, the martensite Barkhausen signature is completely different and clearly depends on the carbon content. Increasing the carbon composition of the martensite results both in a decrease of the signal and in a shift of the peak to a higher magnetic field. The shape is also modified since the peak gets wider.

From these measurements, we have taken two important parameters which are currently used, the maximum Barkhausen noise amplitude (BNA), and the corresponding magnetic field (Hpeak). The first one is related to the jump size of Bloch walls during Weiss domains reorganization, whereas the second one is more directly connected to the magnetic strength necessary to move the Bloch walls from pinning sites.

In Figs. 4 and 5, we have plotted these two quantities as a function of the carbon content in martensite. Error bars have been estimated using the maximum difference obtained between measurements on several samples.

As a first approximation, we can see that these two quantities change linearly with carbon content. Such tendencies can be attributed to a stronger pinning of Bloch walls by the microstructural state of steels. Increasing the carbon content in martensite results both in a higher density of dislocations, and in an increase of the martensite tetragonality. So, fewer walls participate in the magnetic reorganization, and the ones which can move, need a stronger magnetic field to do it: the martensite phase becomes magnetically harder.

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**Table 1. Composition of the steels (in wt%)**

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>S</th>
<th>N</th>
<th>Mn</th>
<th>P</th>
<th>Si</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr</th>
<th>Al</th>
<th>As</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel A</td>
<td>0.071</td>
<td>0.005</td>
<td>0.007</td>
<td>0.437</td>
<td>0.012</td>
<td>0.009</td>
<td>0.006</td>
<td>0.006</td>
<td>0.014</td>
<td>0.058</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Steel B</td>
<td>0.343</td>
<td>0.030</td>
<td>0.008</td>
<td>0.790</td>
<td>0.029</td>
<td>0.166</td>
<td>0.186</td>
<td>0.095</td>
<td>0.055</td>
<td>0.005</td>
<td>0.041</td>
</tr>
<tr>
<td>Steel C</td>
<td>0.476</td>
<td>0.003</td>
<td>0.004</td>
<td>0.654</td>
<td>0.012</td>
<td>0.020</td>
<td>0.022</td>
<td>0.024</td>
<td>0.221</td>
<td>0.007</td>
<td>0.002</td>
</tr>
<tr>
<td>Steel D</td>
<td>0.634</td>
<td>0.032</td>
<td>0.005</td>
<td>0.593</td>
<td>0.010</td>
<td>0.020</td>
<td>0.010</td>
<td>0.053</td>
<td>0.053</td>
<td>&lt;0.001</td>
<td>0.032</td>
</tr>
</tbody>
</table>

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**Fig. 3.** Barkhausen noise response of (a) steel A and (b) steels B, C and D. Steel A is a ferrite steel whereas steel B, C and D are martensite ones.
Using linear regression, we found that the amplitude of the BN signal is linked to the carbon content of martensite by the following relation:

\[
BNA_{\alpha} = 5.3 - 5.7 [C_{\alpha}] \quad (1)
\]

where \([C_{\alpha}]\) is the carbon content of martensite (in wt%).

For these experimental conditions, the signal is expected to vanish for a carbon content of martensite equal to:

\[
[C_{\alpha}]_{\text{crit}} = \frac{5.3}{5.7} = 0.93 \quad (2)
\]

The relative scattering between the 3 steels may be attributed to their difference in chemical composition, particularly the manganese content. For example, it is known that manganese atoms reduce the density of dislocations in the martensite phases,\(^\text{11}\) and so would change the Barkhausen noise signature.

These preliminary results show two important features: (1) the ferrite and martensite phases have distinguishable Barkhausen noise responses, (2) the Barkhausen noise signal of the martensite samples is a function of the martensite carbon content.

### 4.2. Barkhausen Noise Response of Ferrite–Martensite Steels

In order to obtain ferrite–martensite structures, the steels have been annealed at temperatures ranging from 720 to 860°C for steel B, from 720 to 820°C for steel C, and then water quenched.

#### 4.2.1. Microstructures Analysis

Samples were etched with a nital solution and, using image analysis, the martensite volume fraction was determined for steels containing 0.34% (steel B) and 0.48% (steel C) carbon. Some photographs of the resulting microstructures are shown in Fig. 6.

The volume fraction of steel B and C are plotted as a function of the annealing temperature in Fig. 7. Due to the high carbon content of the two steels, the lower volume fraction which can be assessed is close to 50%. The temperature of transition from the ferrite-austenite domain to the austenite domain is estimated to be 790°C for steel C and 800°C for steel B, whereas the eutectoid temperature is located between 730 and 750°C.

![Fig. 6. Optical photographs of steels B and C after intercritical annealing at temperatures ranging from 750 to 800°C. Samples etched with a nital solution. Steel B: (a) \(T=750°C\), (b) \(T=790°C\), (c) \(T=800°C\), and Steel C: (d) \(T=750°C\), (e) \(T=770°C\), (f) \(T=800°C\).](image-url)
Increasing the temperature in the intercritical region, changes the volume fraction of martensite, and its carbon content. These two quantities are linked to each other and by making some reasonable hypothesis a simple expression between them can be found: using a simple lever rule in the Fe–C equilibrium phase diagram, and assuming that (1) the carbon content in ferrite is negligible compared to the carbon content of martensite for all the temperature used in this study, (2) the volume fraction of martensite is close to its weight fraction, one can get the following relation:

\[ [C_{\alpha}] = \frac{[C]}{f_{\alpha'}} \] ................................(3)

where \([C_{\alpha}]\) is the martensite carbon content, \([C]\) the carbon content of the steel, and \(f_{\alpha'}\) the volume fraction of martensite.

4.2.2. Barkhausen Noise Signals

Magnetic Barkhausen noise measurements were performed on the ferrite–martensite steels. The results are presented in Figs. 8 and 9.

At 720°C, no martensite was detected in the two steels. Microstructural characterization reveals the presence of ferrite and pearlite, which means that the eutectoid temperature was not reached. The Barkhausen noise signal has a single-peak shape due to the magnetic response of pearlite which is very close to that of ferrite.

Increasing the temperature to 750°C results in the appearance of a second peak found at a high magnetic field in the Barkhausen noise response. It can be attributed to the martensite phase being formed. We can also notice that the ferrite peak decreases in amplitude and shifts to a higher magnetic field if we compare it to the former peak at 720°C.

As the temperature, and thus the volume fraction of martensite increases, the peak of martensite changes. It becomes narrower, greater in amplitude and shifts to a lower magnetic field. On the contrary, the ferrite peak broadens, shifts to a higher field and decreases in amplitude.

4.2.3. Evolution of the Barkhausen Noise Amplitude with the Martensite Volume Fraction

We plotted the Barkhausen Noise Amplitude (BNA) of the measured signal as a function of the martensite volume fraction determined previously. The results are presented in Figs. 10 and 11. Linear dependencies are found between the amplitude of the peaks of ferrite and the amount of martensite. The same remark can be made about the amplitude of the peak of martensite.

Regarding the ferrite peak, the variations remain the same regardless of the steels used (same slope and ordinate at origin). These results mean that a simple mixture rule applies for the peak amplitude with respect to the volume fraction of ferrite \(f_{\alpha'}\) (and thus martensite), i.e.:

\[ \text{BNA}_{\alpha'} = k f_{\alpha'} = k (1 - f_{\alpha'}) \] ................................(4)

where \(k\) is a proportionality coefficient and \(f_{\alpha'}\) is the volume fraction of martensite.

Using linear regression, we found that \(k=7.3\). From this simple mixture rule we can deduce that it is possible to assess the relative proportion of the two phases by Barkhausen noise measurements.

The situation is different concerning martensite. Even if the slopes of the curves are nearly identical, a shift can be observed between the two steels. Keeping in mind that changing the annealing temperature changes the martensite volume fraction, but also the carbon content of the martensite, one can try to find the origin of such a shift.
First, we previously found for martensite samples (Fig 5), that BNA varies linearly with the carbon content of martensite (see Eq. (1)).

Using Eq. (3), one can rewrite this expression as a function of the martensite volume fraction:

\[ BNA = 5.7 \frac{[C]}{f_{\text{crit}}} \] ..........................(5)

Second, as for the ferrite peak, one can suppose that a simple mixture rule may exist between the amplitude of the martensite peak and the volume fraction, hence:

\[ BNA_{\alpha} = K f_{\text{crit}} \] ..........................(6)

where \( K \) is a proportionality coefficient.

As previously mentioned, changing the temperature changes, on the one hand, the martensite volume fraction, and on the other hand the carbon content of martensite. Taking into account the influence of these 2 quantities on the BNA, and multiplying Eqs. (5) and (6) leads to:

\[ BNA_{\alpha} = K f_{\text{crit}} \left( 5.3 - 5.7 \frac{[C]}{f_{\text{crit}}} \right) \] ..........................(7)

which may be rewritten as:

\[ BNA_{\alpha} = -5.7K[C] + 5.3K f_{\text{crit}} \] ..........................(8)

Equation (8) expresses a linear dependency between the amplitude of the Barkhausen noise signal and the martensite volume fraction, but, more interestingly, the shift observed between the two steels originates from their difference in carbon content \([C]\).

Using relation (8), we calculated the four values of the proportionality coefficient \( K \) deduced, first from the slope, and second from the ordinate at origin obtained from the curve in Fig. 11. The results shown in Table 2 are in good agreement.

Expression (8) shows that when the volume fraction drops below a critical value \( f_{\text{crit}} \), no Barkhausen noise signal of the martensite phase is expected, i.e. \( BNA = 0 \). For our experimental conditions, we found that it is a linear function of the carbon content of the steel:

\[ f_{\text{crit}} = \frac{5.7}{5.3} [C] = \frac{[C]}{[C]_{\text{crit}}} \] ..........................(9)

Table 2 presents the critical volume fraction obtained from Eq. (9) compared to the one obtained using curve fitting (cf. Fig. 11). Once again, the agreement is fairly good.

It is important to note that this critical quantity is directly linked to the experimental parameters, particularly the magnetization rate, and thus it can be easily improved using ad-hoc conditions.

4.2.4. Evolution of the Barkhausen Noise Peak Positions

i) Martensite Peaks

As the two phases are magnetic, we first assume that the field necessary to move the Bloch walls does not depend on the volume fraction of each of the magnetic phases. Consequently, the observed shift of the martensite peak position is directly linked to the carbon quantity left inside the martensite.

We have extracted the positions of the peaks of martensite as a function of the carbon content in martensite, calculated using volume fraction and Eq. (3). The results are plotted in Fig. 12 for steels B and C.

As observed for martensite steels (cf. Fig. 4), linear dependencies are found. The slope values are of the same order and comparable to the ones obtained previously. However, an offset between these two curves can be observed. It can be explained by the chemical compositions of the two steels. As mentioned previously, alloying elements and especially manganese atoms modify the density of dislocations in martensite needles. The lower manganese content of steel C leads to a martensite phase magnetically harder than that of steel B. Consequently, for a fixed amount of carbon in martensite, a higher field is necessary to move Bloch wall, and the Barkhausen noise peak shifts to a higher magnetic field.
The most significant conclusion deduced from these results is that it is relatively easy to determine the amount of carbon in martensite using Barkhausen noise measurement in ferrite–martensite steels. Due to the effect of the alloyed elements, it is evident that such a possibility would require a previous calibration.

ii) Ferrite Peaks

Surprisingly, as the annealing temperature increases, the ferrite peak shifts to stronger magnetic fields. Unlike martensite, it cannot be explained by its carbon content, as it decreases as the temperature increases. Consequently, no magnetic hardening is expected and one has to put forward other causes for such a behavior.

There are two main factors which could influence the ferrite peak position, as discussed below.

First, during the transformation of austenite to martensite, the volume expansion of martensite could induce stresses in the ferrite grains. Consequently, when transformation is finished, the ferrite grains are submitted to residual internal stresses. Depending on the sign of such stresses, the Barkhausen noise signal changes and a shift of the BN peaks may be observed. If the residual stress in the direction of the applied field is compressive, the peaks move to a stronger field.

Second, the adaptation to the plastic strain which is generated by the volume expansion of martensite, creates a huge amount of dislocations inside the grains. This induces pinning obstacles, and, as for the martensite, renders the microstructure magnetically harder. As a result, the peak shifts to a higher field.

Raising martensite volume fraction, on one hand generates more residual internal stresses, and on the other hand, increases the density of dislocations. Consequently, a stronger magnetic field is needed to move the Bloch walls in ferrite, as the volume of martensite increases.

Whatever the mechanisms considered to be responsible for the shift of the ferrite peaks, one has to keep in mind the fact that the amplitude of the BN should also be modified. Regarding the linear relations and the good agreements found previously, we can deduce that the effect of dislocations or residual internal stresses are second order effects on the Barkhausen noise amplitude compared to the effect of a change in the ferrite volume fraction.

If such results are confirmed, magnetic Barkhausen noise measurements may also be good indicators of residual internal stresses and the density of dislocations existing in the ferrite phase. Complementary studies need to be carried out to confirm these possibilities.

5. Conclusions

Barkhausen noise measurements have been successfully used for the characterization of Ferrite–Martensite steels. Using thermal treatments on different steels, we found that the BN signal was very sensitive to the martensite volume fraction and to the carbon content.

Regarding the simple relations found, we concluded that the measurement of the Barkhausen noise signal in such steels can lead to the determination of:
- the martensite (and ferrite) volume fraction deduced from the ferrite peak amplitude
- the carbon content of the steel deduced from the martensite peak amplitude
- the carbon content in martensite from the martensite peak amplitude as well as its field position

By using the ferrite peak position, it may be possible to assess the dislocation density or residual internal stress in ferrite grains, but further studies need to be done to confirm such a potentiality.

Finally, these results obtained on ferrite–martensite steels have to be extended to Dual-Phase steels with a lower martensite volume fraction, in order to show the feasibility of using such techniques for a non-destructive characterization of industrial DP steels.

REFERENCES