Magnetic Barkhausen Noise Measurement Potentialities for Metallurgical Transformations Characterization in Multi-Phase High Strength Steels

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Abstract. Even if numerous techniques are already used in order to characterize multi-phase steels, it seems to be difficult to obtain specific information provided by each phase. In this context, magnetic Barkhausen noise measurements have been used in order to characterize metallurgical transformations in multi-phase High Strength (SH) steels.

We identified specific responses for ferrite or pearlite, bainite and martensite constituents and the main parameters influencing the Barkhausen response. We show that it is possible, first to quantify the proportion of granular ferrite in ferrite-martensite or in industrial DP steels and second to identify the main metallurgical transformations during an isothermal holding of a TRIP steel.

Introduction

Industrial High Strength multiphase steels are composed of different metallurgical constituents. According to their proportions, different families are defined. The two main families are the Dual-Phase (DP) and the TRansformation Induced Plasticity (TRIP) steels. Because of their specific microstructures, these steels present higher mechanical properties than those of the available High Strength low alloy steels. These properties make them suitable for automotive applications, in order to decrease the weight of pieces.
In particular, industrial DP steels are mainly composed of a ductile ferrite matrix in which small martensite (and sometimes bainite) islands are dispersed. TRIP steels present a growing interest for their high strength after forming. This kind of steel is mainly composed of three metallurgical constituents : a majority of ferrite with small amounts of bainite and retained austenite.

Multiphase steels are usually characterized using several techniques such as microhardness, X-ray diffraction, mechanical tests [1,2], etc. These different techniques are unable to make a distinction between information provided by each constituent. Experimental tools which could give us information about each phase are rare [3]. Furthermore, in an industrial point of view, there is no non-destructive application.
The Barkhausen noise measurement is a magnetic non-destructive technique which can be used to characterize ferromagnetic materials. The signal obtained is sensitive not only to the microstructural state of material but also to the stress state inside. Because this

technique can give us information provided by each phase, it is suitable to multiphase High Strength steels characterization.

The aim of this study is to examine the possibility of using this magnetic technique to characterize some microstructural aspects of different metallurgical states obtained on industrial sheets or after thermal treatments. In a first investigation performed on ferrite-martensite and DP steels, we were able to evaluate the proportion and the composition of each phase in such a microstructure [4, 5]. In this work, we used the potentialities of this technique in order to follow the decomposition of austenite in a TRIP steel, during different thermal treatments.

1. Barkhausen Noise Measurement and Identification of Specific Metallurgical Constituents

1.1 Barkhausen noise technique

Ferromagnetic materials present a magnetic microstructure composed of Weiss domains characterized by a constant magnetization and separated by finite frontiers called Bloch walls. When a magnetic field is applied, a reorganization of this magnetic microstructure occurs because of energetic considerations. This phenomenon requires Bloch walls movements which are made by discontinuous jumps. The recording of these sudden jumps results in the Barkhausen noise signal [6]. Usually, the Root Mean Square signal (RMS) which corresponds to its envelope, is plotted as a function of the applied magnetic field in order to quantify the results obtained. In most cases, this envelope has a single-peak shape and can be characterized by different parameters, such as the maximum noise amplitude (BNA) and the corresponding magnetic field (Hpeak) [5].
The discontinuous jumps of Bloch walls are due to their local pinning by different obstacles such as inclusions [7], precipitates [8], grain boundaries [9] or dislocations tangles [10]. When the number of these pinning obstacles increases, the Barkhausen noise amplitude increases too. When the interaction of these obstacles with Bloch walls increases, the Barkhausen noise peak shifts to higher values of magnetic field. Because the magnetic structure is directly linked to the nature of metallurgical state, one Barkhausen noise signal exists for each phase [11,12].
Internal and external stresses also influence the Barkhausen noise signal. For positive magnetostrictive materials as we study in this work, a uniaxial tensile stress leads to an increase of the Barkhausen noise amplitude and a shift of the peak towards low magnetic field whereas a decrease of the amplitude associated to a shift of the peak to high magnetic field and sometimes an appearance of adjacent peaks, is attributed to a uniaxial compressive stress [13].

1.2 Specificity of Barkhausen noise response for several metallurgical constituents

Some authors have studied the evolution of the Barkhausen noise signal as a function of metallurgical states. For example, Saquet et al. [14] have shown that different signals are obtained for several metallurgical states. Ferrite signal is located at very low magnetic field. For martensite sheet, the peak is located at higher magnetic field than for the other constituents and its amplitude is very low. This typical Barkhausen response is due to the huge magnetic hardness of this metallurgical constituent. These results confirm the possibility of identifying specific characteristics for each state.
However, we have to take into account that the carbon content of each sample can influence its Barkhausen noise response. For ferrite, the carbon content is very low (between 0 and

0.02 %), then its role on the Barkhausen noise is very weak. For martensite, we have shown in a previous work that both the field position and the amplitude of the Barkhausen peak are linked to the carbon content [4]. When the carbon content increases, the martensite microstructure becomes magnetically harder and the amplitude of the peak decreases whereas its magnetic field position increases.

2. Experimental Details

2.1 Barkhausen noise device

The Barkhausen noise set-up has been schematized in a previous work [5]. The magnetic field, with a specific frequency (0.1 Hz for Ferrite-martensite and Dual-Phase steels and 0.5 Hz for TRIP steel), is applied to the sample using a U-shape core with a 1000 turns coil wound around it. This applied magnetic field is measured using a Hall effect probe located at the surface of the sample. The magnetic response of the sample is detected through a 300 turns coil wound around it. This signal is pre-amplified (40dB), passed through a high-pass filter with a cut-off frequency of 500 Hz. The envelope of the Barkhausen noise signal is obtained using an analogue Root-Mean-Square (RMS) device with an integrated constant time of 25 ms. Finally, this signal is amplified (40 dB) to obtain a result which can be acquired using a Data Acquisition (DAQ) card plugged into a computer.
For each curve, 10 signals were averaged and the background noise was removed. As the Barkhausen noise is symmetrical in relation to null magnetic field, only the positive part of the curve is plotted.

2.2 Steels and thermal treatments

For ferrite-martensite steels, we have chosen relatively high carbon content steels. An example of these results is given with the steel A (0.48 % C). For industrial Dual-Phase steels, we have chosen different grades (B1, B2, B3, B4 and B5) containing variable carbon contents, but less than steel A. Finally, a TRIP steel C was used for austenite decomposition study. The chemical compositions of each one can be found in table 1.

Table 1. Composition of steels [wt 10^{-3}.%]

Element	C	S	N	Mn	P	Si	Cu	Ni	Cr	Al	Mo	Ti
Steel A	476	3	4	654	12	20	22	24	221	7		
Steel B1	85	7	4	1515	7	333	14	19	216	35	2	
Steel B2	91	3	5	1260	13	129	9	23	519	32	1	
Steel B3	118	1	6	1412	14	368			203	39	59	
Steel B4	141	1	5	1916	16	207	7	23	203	24	6	24
Steel B5	179			1917	20	370				25		87
Steel C	210	3	5	1779	8	1528	14	19	25	38	10	

The as received steel A was annealed in the intercritical range, i.e. at a temperature between 720°C and 860°C, in a salt bath for 5 minutes and then water quenched to transform austenite into martensite. By this way, we obtain different ferrite-martensite microstructures with several martensite proportions. The Barkhausen activity of martensite being quite low, we have chosen a relatively high carbon content steel in order to obtain microstructures containing a high proportion of martensite. Thus, it is possible to detect its Barkhausen noise response.
Industrial DP steels were supplied by ARCELOR Research, after industrial thermal treatment. Different grades of DP are tested and each one corresponds to a specific

proportion of constituents. Ferrite proportion in industrial DP steels is higher than those in ferrite-martensite steels.
Different isochronous thermal treatments were applied on the steel C : austenitization at high temperature, holding in the bainitic transformation temperature range and finally water quenching. The austenitization temperature was approximately 1000 °C during 2 min in a furnace, whereas the bainitic holding is realized at different temperatures (between 275 and 525 °C) in salt baths, during 1 and 8 min.

After these isochronous treatments, each sample was cleaned by immersion in a dilute hydrofluoric bath in order to remove the decarburized layer (approximately 100 µm removed).

3. Results

3.1 Ferrite-martensite and industrial Dual-Phase steels

For ferrite-martensite steel, we obtained variable two-peak shape signals which depend on martensite proportion and carbon content (Fig. 1) [4].

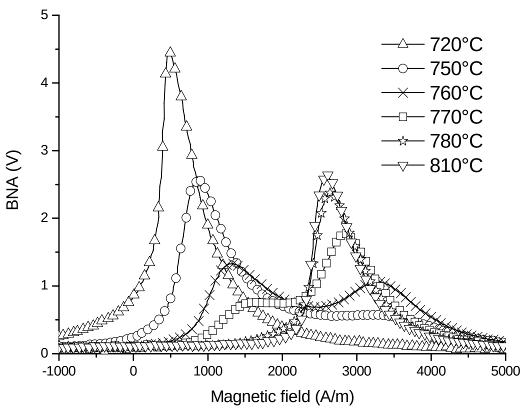

Figure 1. Barkhausen noise signals of ferrite-martensite steels as a function of the intercritical annealing temperature

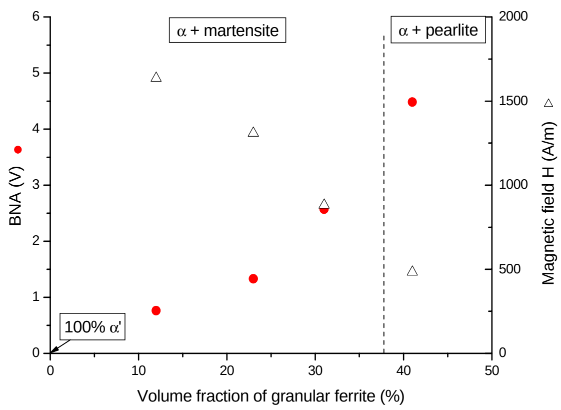

Figure 2. Barkhausen noise amplitude and peak position of ferrite as a function of its proportion for ferrite-martensite steels

With an annealing at 720°C, we observed one peak which corresponds to superposed granular ferrite (that is to say ferrite which is not included in pearlite) and pearlite responses. When the annealing temperature increases, a second peak appears at high magnetic field which can be attributed to martensite phase. Finally, at very high temperature, there is only martensite and the signal has a single-peak shape.
Because martensite proportion in industrial DP steels is very small and its Barkhausen noise activity is very low, we focus on the evolution of granular ferrite peak amplitude for all steels. The evolution of granular ferrite peak amplitude and its magnetic field position are represented in Fig. 2 as a function of its proportion (assessed by micrography analyses after Nital etching). Good correlations are found for the field position and the amplitude of the ferrite peak with the volume fraction of granular ferrite. This phenomenon is noticed in the ferrite-martensite field, but also continuously in the ferrite-pearlite one.

The Barkhausen noise measurements performed on the industrial DP steels are presented in Fig. 3 [5].

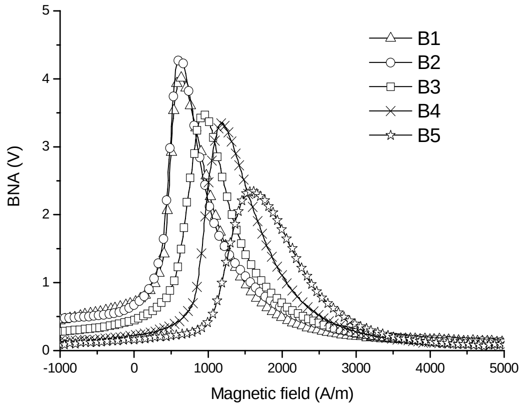

Figure 3. Barkhausen noise signals for different industrial DP steels

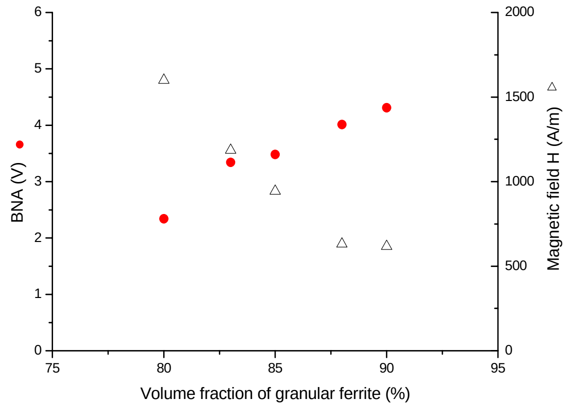

Figure 4. Barkhausen noise amplitude and peak position as a function of the proportion of granular ferrite for industrial DP steels

All Barkhausen signals have a single-peak shape which is representative of the microstructural state of each steel, mainly composed of granular ferrite and, in minority, martensite and bainite. Furthermore, we can notice that the profile of the curve is narrower or wider, depending on the investigated steel.

For the five steels, the evolution of the amplitude and the corresponding magnetic field is represented in Fig. 4 as a function of granular ferrite proportion (assessed by micrography analyses after Lepera etching). Even if the five steels do not present exactly the same chemical composition, we found linear dependencies with the granular ferrite proportion for the two parameters.

3.2 Decomposition of austenite during isothermal holding (TRIP steel)

Optical and Scanning Electron Microscopy were performed on each metallurgical state but no quantitative assessment of phases proportions was possible, due to the difficulty to distinguish bainite and martensite.

However, for 8 min treatments, we find a majority of martensite and lower bainite for low temperatures of bainitic holding whereas at higher temperatures, granular ferrite and pearlite are mainly observed.

In Fig. 5, we plot the Barkhausen noise signal versus the applied magnetic field for different holding temperatures in the case of an isochronous treatment of 8 min. We can notice that the evolutions of the Barkhausen noise signal as a function of the temperature of the bainitic holding are significant. It is representative of the microstructural variations due to the different thermal treatments.

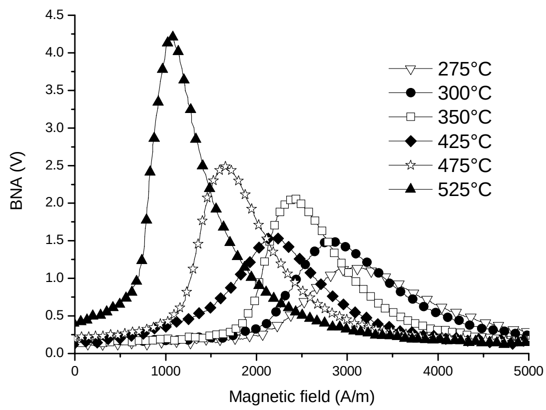

Figure 5. Barkhausen noise signals for isochronous treatments of 8 min

In Fig. 6 and 7, BNA and Hpeak are plotted as a function of the bainitic holding temperature, for the two times.

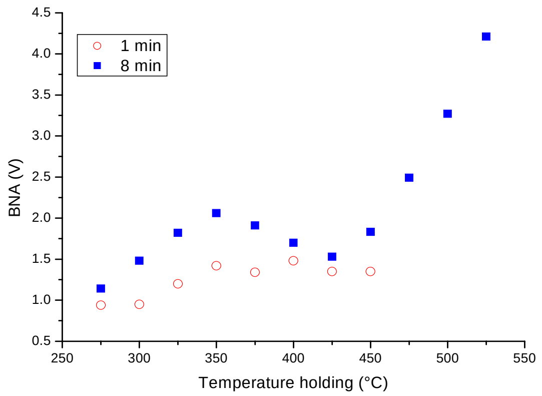

Figure 6. BNA as a function of the holding temperature

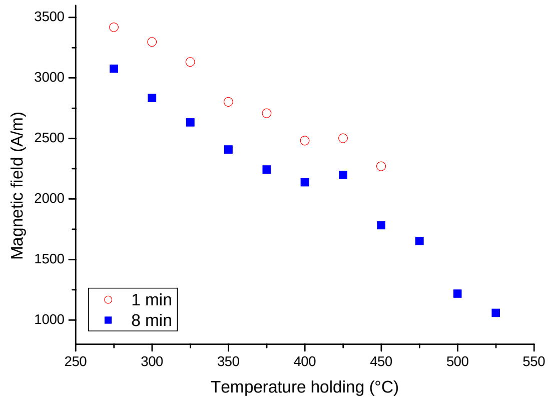

Figure 7. Hpeak as a function of the holding temperature

At low temperatures the BNA increases until approximately 350 °C, then it decreases. For higher temperatures above 450°C, the signal rises again rapidly until 525 °C, the temperature at which the experiments were stopped.
Concerning the corresponding magnetic field (Hpeak), it decreases continuously all along the temperature range and a small slope change is observed at approximately 450 °C.

4. Discussion

Globally, BNA is strongly dependent on the number of pinning obstacles met by Bloch walls. Hpeak is preferentially linked to the nature of obstacles. BNA is thus strongly dependent on the phase proportion whereas Hpeak is usually linked to the microstructural state of this phase.

Concerning ferrite-martensite steels, by performing different intercritical annealing, not only phases proportion changes but also their carbon contents. The martensite proportion increases when the temperature becomes higher, and in the same time, its carbon content decreases. The evolutions of martensite peak position correspond to this latter phenomenon. On the other hand, if granular ferrite proportion decreases when the temperature grows, its carbon content remains very low. Its increasing magnetic field could be attributed to an other phenomenon which should be representative of the martensite presence. This behavior highlights the interest of studying the evolution of ferrite peak to the detriment of martensite one. The origin of the influence of martensite on ferrite magnetic response is not identified at this time and can have different origins such as the introduction of dislocations in ferrite or the modification of the local magnetic field in each phase.
However, for ferrite-martensite steels containing a quite high proportion of martensite, there is a good correlation between ferrite peak parameters and its proportion.
Concerning industrial Dual-Phase steels, as for ferrite-martensite steels, we can see that the granular ferrite phase is also influenced by the presence of the other constituents (martensite and bainite). We found a linear evolution of the BNA with the granular ferrite proportion. Furthermore, linear dependencies are also found between the magnetic field position and the granular ferrite proportion.

In view of this result, after calibration, the determination of the magnetic field position as well as the amplitude of the Barkhausen noise signal of a Ferrite-martensite or an industrial DP steel can provide information about granular ferrite proportion.

Concerning the possibilities to follow austenite decomposition during isothermal holdings, general TTT diagrams shape can provide information about the microstructure evolutions. For very short times, the microstructure is only composed of metastable austenite. After a longer holding time determined by the temperature, the austenite transformation begins. Depending on the temperature, the product of decomposition is composed of lower or upper bainite for lower temperatures and pearlite for higher temperatures. The corresponding proportion increases with the holding time. Finally, during the water quenching, the remained austenite is transformed into martensite and some residual austenite could be present at ambient temperature. These theoretical affirmations are widely confirmed by the micrographs observations. For a specific small range of intermediate temperatures, the beginning of any transformation only occurs at long times.

We can suppose that the evolution of BNA is representative of the volume proportion of the main metallurgical constituent present in each sample. For the lowest temperatures (275 to 450 °C), bainite is the main constituent and the variations of the BNA should follow the variations of bainite proportion. For higher temperatures, pearlite becomes the main constituent and its proportion begins to increase. The transition from a microstructure mainly composed of bainite to an other one mainly composed of pearlite seems to be characterized by a slope change of the curve (approximately 450 °C in this study). This last phenomenon could be also due to the presence of residual austenite which is a non ferromagnetic constituent and constitutes a screen for the magnetic signal.

The corresponding magnetic field is more specifically determined by the nature of the obstacles encountered by Bloch walls. Strong obstacles correspond to a high magnetic field whereas weak ones give rise to a signal located at low magnetic field. According to the metallurgical constituent, these obstacles are not the same. The pearlite response is located at low magnetic field whereas the martensite one at high magnetic field. Bainite response is globally intermediate but however, as for martensite response, it mainly depends on its carbon content. It is reasonable to consider that the lower bainite is magnetically harder than the upper one [15,16]. At low temperatures, there is a majority of lower bainite leading to a peak located at high magnetic field. When the temperature grows, the bainite microstructure softens and progressively becomes upper bainite. This evolution seems to be characterized by the continuous decreasing of Hpeak with the increasing temperature. For higher temperatures, the decreasing of the magnetic field with the temperature can be interpreted by the softening of pearlite when the holding temperature increases. We can notice the slight increasing of the magnetic field around 450 °C.

Finally, the results obtained for the two holding times are very similar. The shift between the results obtained for 1 and 8 min is not surprising. Concerning BNA evolutions, for a same temperature, the proportion of bainite must be higher for 8 min than for 1 min (Fig. 6). This effect is characterized by a higher value of BNA for 8 min than for 1 min, at a same holding temperature. The corresponding magnetic field present also a shift which must be due to the higher magnetic hardness for 1 min samples (Fig. 7).

5. Summary

Barkhausen noise measurements have been successfully used for the characterization of ferrite-martensite and industrial Dual-Phase steels. For the two kinds of steel, we found that the BN signal was very sensitive to the volume fraction of granular ferrite. The measurement of the Barkhausen noise signal in such steels can lead to the determination of the granular ferrite volume fraction deduced from the magnetic field position as well as the amplitude of the peak.

We show that Barkhausen noise measurement can be also used to study austenite decomposition during a thermal treatment. The amplitude of the signal seems to be representative of the evolution of proportion of the main constituents, that is to say the bainite proportion in the bainitic domain and the pearlite one in the pearlitic domain. The corresponding magnetic field (Hpeak) characterizes the evolution of the microstructure hardness. After calibration, which could be realized using an other technique of characterization - for example by dilatometry [17] - the Barkhausen noise measurements should make up a strong tool in order to quantify the isothermal decomposition of austenite.

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