Characterisation of Quenched and Tempered Steels by Magnetic Barkhausen Noise Method

Kemal DAVUT, C. Hakan GÜR, Middle East Technical Univ., Metallurgical and Materials Eng. Dept., Ankara, Turkey
Xavier KLEBER Gr. d'Etudes de Metall. Phys. des Materiaux- Gr. Métaux et Alliages, INSA Lyon, France

Abstract. The aim of this work is to characterise the microstructures of quenched and tempered steels non-destructively by the Magnetic Barkhausen Noise method. Disk shaped specimens were prepared from SAE 4140 steel bars. All specimens were austenitised at 860°C/30 min. and water-quenched identically. The quenched specimens were then tempered at various temperatures between 200°C and 600°C. Formation of the desired microstructures was demonstrated by metallographic examinations and hardness measurements. In all specimens the position, shape and amplitude of the Barkhausen signals were evaluated using a commercial system. The results show that as tempering temperature increases, the magneto-elastic parameter peak increases due to the enhancement of domain wall displacement with softening of the martensite.
Keywords: Steel, quenching, tempering, microstructure, Barkhausen Noise

1. Introduction

Steels are widely utilised in different industries, usually in the form of quenched and tempered components. Tempering in the range of 200°C to 700°C relieves residual stresses and improves toughness and ductility by modifying the microstructure of the quenched steel. For consistency and less dependence on time, quenched steel components generally tempered for 1 to 2 hours. If the principal desired property is hardness or wear resistance, the part is tempered at about 200°C; if the primary requirement is toughness, the part is then tempered above 400°C. Residual stresses are relieved almost completely when the tempering temperature reaches 500°C [1].

In order to provide longer service life with higher performance of steel components, quality control is essential. There is a growing need for non-destructive characterisation of steel components. Magnetic Barkhausen Noise (MBN) measurement provides a good alternative to the traditional methods in terms of fastness and accuracy.

Ferromagnetic materials below their Curie temperature retain a large spontaneous magnetic moment due to the cooperative alignment of unpaired electron spins along a common direction. Oppositely magnetized domains divided by domain (Bloch) walls form to minimize the magnetic energy. The change in magnetisation, caused by the application of external magnetic field, takes place by movement of the boundaries between domains in weak fields or by rotation of the direction of magnetisation in strong fields. On removing the field, the magnetisation again declines to zero if there is no hindrance to Bloch wall motion [2-4].

When a variable external field influences a ferromagnetic material, irreversible jumps of domain walls cause the formation of Barkhausen noise. High resolution examination of hysteresis cycles of ferromagnetic materials reveals discontinuous flux changes due to discontinuous domain wall motion as magnetic field strength is increased. Microstructural features such as dislocations, inclusions and grain boundaries pin the domain walls. When the strength of externally applied magnetic field reaches the critical level, motion of the domain wall continues by Barkhausen jumps. These jumps can be detected as the voltage pulses induced in a pick-up coil positioned close to the surface. These signals are amplified, filtered and then processed using a software to establish the graph of the MBN signal versus magnetic field strength (MBN fingerprint). Microstructures can be characterised by analyzing the parameters such as peak value and peak position of MBN signal, the root mean square value of the Barkhausen signal (r.m.s.).

Changes in microstructure and/or residual stresses effects the Barkhausen signals. Various studies have been published on characterisation of steel microstructures by MBN method. MBN in low carbon was found to be strongly dependent on the grain size. The maximum amplitude of the MBN voltage decreases with grain size due to reduced domain density and preferred nucleation sites [5]. Grain boundary misorientation effects MBN as well [6]. Important changes have been observed in the MBN response during martensite decomposition due to tempering of steels [7-9]. The effect of tempering was also studied for case carburized steel, and a correlation between hardness depth profile and MBN was found [10].

The aim of this study is to investigate the effect of tempering on MBN. The samples consisting of as-quenched martensite and tempered martensite were obtained by various heat treatments. By applying the same austenitisation and quenching procedure prior to the tempering treatments, other microstructural effects such as austenitic grain size and deformation texture were eliminated.

2. Experimental

The specimens of 7 mm-thick and 22 mm diameter were prepared from the hot rolled SAE 4140 bar. Table 1 gives the chemical composition of the steel used. All the cutting and grinding operations were done prior to the heat treatments in order to avoid surface machining residual stresses. Austenitisation was done under controlled atmosphere to avoid oxidation and decarburisation. All specimens were quenched in water after austenitisation at 860^{o}C for 30 minutes. Then, specimens were separately tempered at 200^{o}C, 300^{o}C, 400^{o}C, 500^{o}C and 600^{o}C for 90 minutes. One specimen was left as-quenched.

Table 1. Chemical Composition of the SAE 4140 Steel (wt%)

C	Cr	Mo	Mn	Si	P	S	Fe
0.475	0.942	0.224	0.840	0.202	0.023	0.015	Bal.

Before metallographic investigation, the samples were finely ground, polished with diamond paste and etched with 2% Nital. The through-thickness sections of the specimens were examined using optical microscope. For each specimen an average hardness value was determined by measuring Vickers hardness at different locations.

MBN measurements were performed using a commercial system (Rollscan, μscan 500-2). The sensor S1-138-13-01 was used for the MBN measurements. A sinusoidal cyclic magnetic field with an excitation frequency of 125 Hz was induced in a small volume of the specimen via a ferrite core C-coil. The Barkhausen signals were filtered with a wide band-pass filter (0.1-1000 kHz), amplified, and then, analyzed using the Rollscan-software. The peak magnetizing voltage was 10V.

3. Results and Discussions

3.1 Microstructure and Hardness

Representative micrographs (Figure 1) and hardness values (Table 2) of the samples show that typical martensitic structure (Fig.1 a), and tempered structures (Fig.1 b-f) were successfully obtained.

Martensite has a tetragonal lattice with interstitial carbon in solid solution and high dislocation density formed by shear, that makes it the hardest of all specimens. A transition carbide (ε-carbide) and low-C martensite occur during tempering up to 250^{o}C. At higher tempering temperatures up to 400^{o}C cementite precipitates start to nucleate, and the dislocation density reduces. In addition, martensite loses its tetragonality, and low-C martensite becomes b.c.c. ferrite. At the tempering temperatures above 400^{o}C, carbide precipitates coarsen and spheroidise, and ferrite starts to recrystallise [11]. In addition, residual stresses decrease during tempering.

When cooling rate shows significant variations as going from surface to interior, the phase content and the residual stress state along the thickness of the specimen may differ. In such cases, the hardness and the microstructure of the surface may not represent the whole structure.

Microstructural investigations showed that the thickness of the samples used in this study allowed the formation of desired microstructure uniformly along a penetration depth of the MBN activity, which usually varies between 0.01 and 1.5 mm depending on the analyzing frequency. The higher the analyzing frequency, the lower would be the penetration depth.

Any texture due to variations in grain shape and rolling bands does not exist. To eliminate the effect of variations in the prior austenite grain size, all specimens have been austenitised and quenched identically.

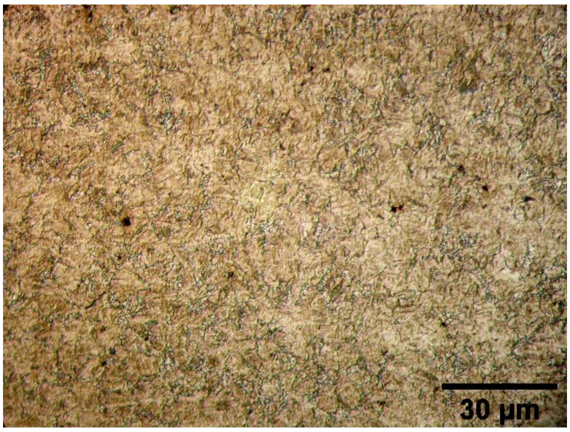
3.2 Barkhausen Noise

The results show that magnetic Barkhausen noise is influenced by the tempering which, as a function of the temperature, causes changes in dislocation density, lattice straining (i.e., micro residual stresses) and the morphology and size of cementite, and corresponding variations in hardness. The results are in agreement with those of the previous studies [7, 9, 10].

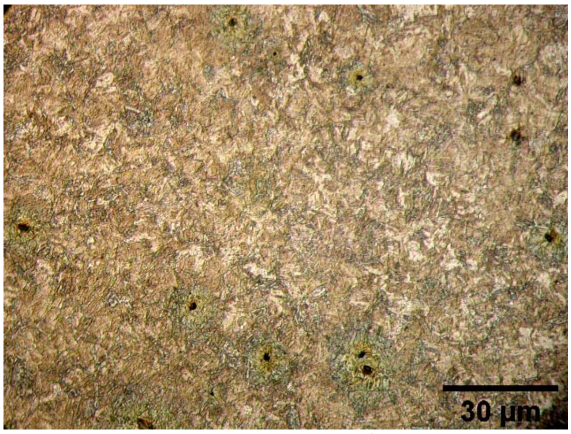
Under the effect of an alternating magnetic field, a representative magnetic hysteresis loop was induced in the small volume measured due to the energy loss with the irreversible process of magnetisation. This irreversible process is strongly related to the dynamic behaviour of domains, i.e., nucleation, annihilation and growth of domains. Grain/lathe boundaries, dislocations and precipitates affect this dynamic behaviour. Consequently, the number of domain walls moving at a given instant and the mean free path of the domain wall displacement decide the MBN peak height.

Table 2 – Hardness Values and MBN Parameters of the Specimens

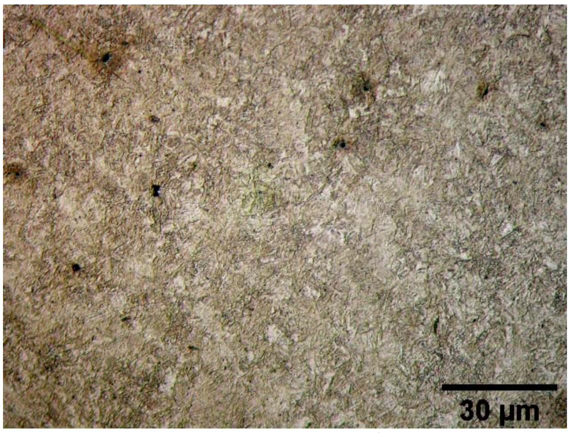
Specimen	Hardness (HV)	r.m.s. (mV)	Peak height	Peak position (% max. magnetic field)
As-quenched	556	2.29	4.29	43.23
200^{o}C-tempered	507	5.72	12.80	33.65
300^{o}C-tempered	492	6.87	15.00	29.35
400^{o}C-tempered	464	7.21	16.42	30.35
500^{o}C-tempered	298	13.50	29.80	22.8
600^{o}C-tempered	205	15.31	33.52	10.27



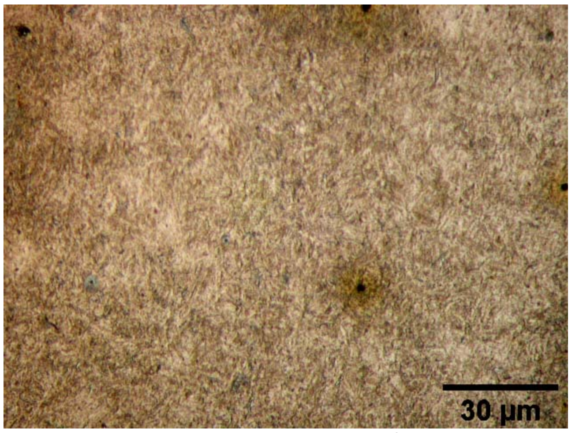
a – As-quenched



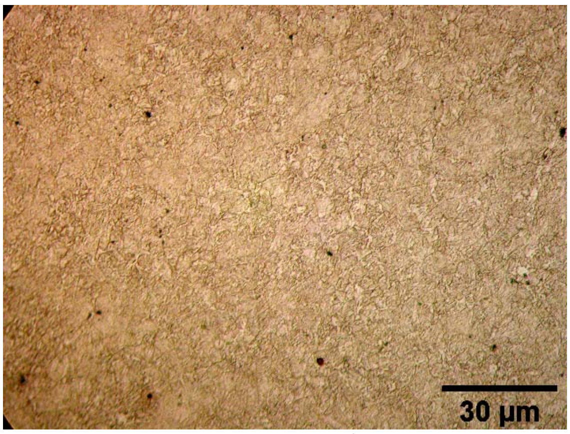
b – Tempered at 200^{o}C



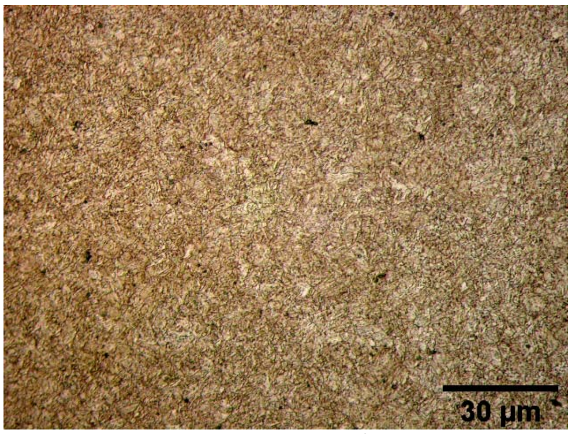
c – Tempered at 300^{o}C



d – Tempered at 400^{o}C



e – Tempered at 500^{o}C



f – Tempered at 600^{o}C

Figure 1. Representative Micrographs of the Samples

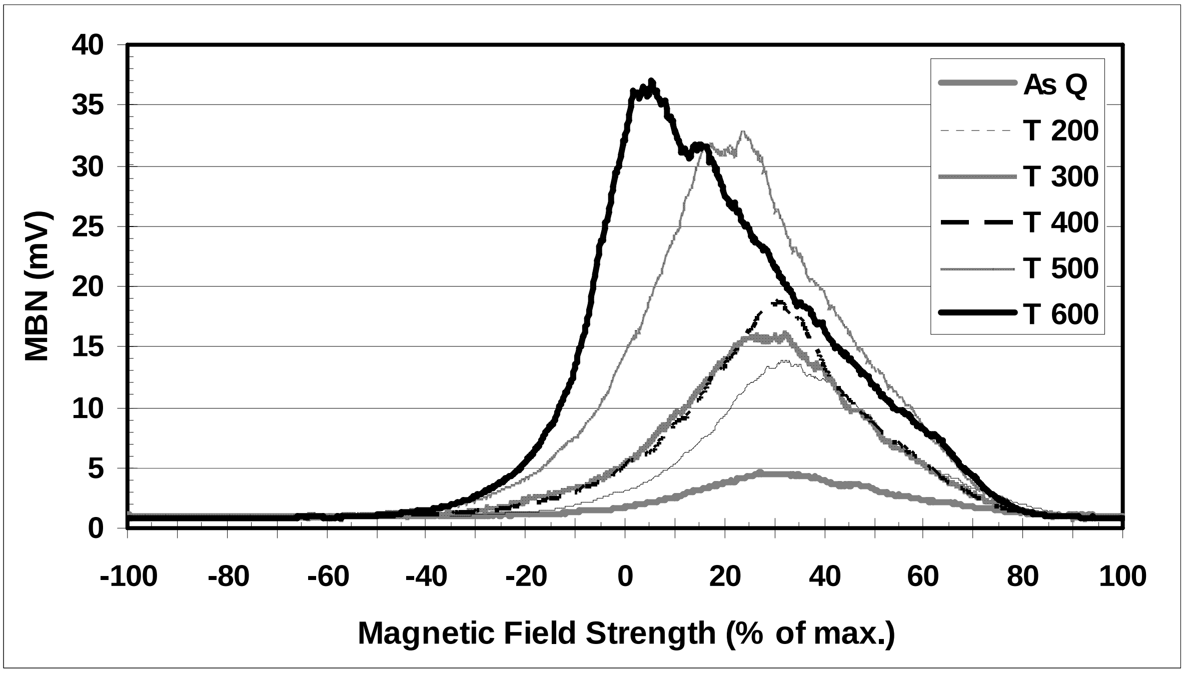


Figure 2. MBN Fingerprints of the Samples

Figure 2 shows the graph for MBN signal versus applied field strength (MBN fingerprints). The as-quenched sample has the weakest MBN peak positioned at the highest field linked with the high coercivity of martensite. Tempering up to 400^oC slightly increases the peak height whereas tempering at 500^oC and 600^oC increases peak height drastically. Moreover, the peak position of the signal shifts to the lower values of magnetic field due to tempering. As the tempering temperature increases, the low amplitude broad peak of as-quenched martensite transforms into a high amplitude peak situated at low magnetic field. The average peak position of the as-quenched martensite is about 43%, and it shifts to 10% for the one tempered at 600^oC.

In the as-quenched specimen, the body-centered tetragonal structure of martensite determines the domain structure. Since the magnetic structure consists of very small domains due to small needles, relative volume occupied by a domain wall is larger. Besides, high dislocation density in the martensite laths acts as a barrier to the movement of domain walls. A strong field is required for the reversal of magnetisation because of low domain wall displacements and difficulty in nucleating domain walls. Presence of micro residual stresses in the martensite needles has an additional effect on reduction of the MBN response.

Tempering at 200^oC changes the microstructure very slightly. Although ε-carbides form, the microstructure is still needle shaped. Therefore, the height and position of the MBN peak do not change significantly. During tempering at 300^oC and 400^oC, cementite replaces ε-carbides, the crystal structure of martensite loses its tetragonality, and dislocation density reduces further. Corresponding magnetisation orientation is no longer favoured and reverse domain nucleation and subsequent domain wall motions take place at lower magnetic fields. All these factors make the domain wall motion easier, and therefore, the amplitude of the MBN peak increases.

In tempering at 500^oC and 600^oC, carbides start spheroidising and recrystallisation of ferrite begins. In parallel to the progressive coarsening of the microstructure, the average size of the domain walls increases. These morphological changes and almost complete relaxation of residual stresses result in a drastic increase in the MBN peak and a clear shift to lower external magnetic field in the peak position by reducing the resistance to the nucleation and movement of domains.

In the tempered steels, the major barriers to domain wall motion are magnetic free poles at the interfaces between ferrite matrix and carbide precipitate and at the grain boundaries. Therefore, the magnetisation involves irreversible movement of domain walls in two stages that occur over a range of critical field strengths: (i) overcoming the resistance of grain boundary free poles and small obstacles such as dislocations; and (ii) overcoming the stronger obstacles such as carbide precipitates at higher field. MBN measurements give a single peak when the ranges of critical field strength for these stages overlap, and their mean values are close to each other [11]. In low temperature tempering, due to incomplete dissolution of martensite and fine ε-carbides, needle like cementite may cause such overlapping. However, the samples tempered at 500^{o}C and 600^{o}C show two-peak behaviour indicating separation of mean values of critical field strengths. Recrystallisation of ferrite at higher temperatures reduces the grain boundary energy, and increases the mean free path of domain wall displacement; hence the field required for unpinning of domain wall from the grain boundary reduces. Cementite precipitates increase free pole density at the matrix-carbide interface, and require higher field for domain wall movement. Therefore, the first peak at lower field strength is due to the irreversible movement of the domain walls existing at the ferrite grain boundaries; and irreversible motion of the domain walls overcoming carbide particles results in the second peak at higher field.

3.3 Hardness Correlation

The raw data consists of a series of voltage pulses and associated magnetic field values. The response of r.m.s. of the noise signals over several field cycles to the changes in microstructure is similar to that of MBN peak height. It is seen in Fig.1 that the as-quenched specimen (the hardest one) has the lowest r.m.s. value. As tempering temperature increases, in contrast to the decrease in hardness, r.m.s. value increases. Pinned domain walls due to high dislocation density and small martensite needles cause lower r.m.s. values. As tempering temperature increases dislocation density decreases, micro residual stresses diminish and the magnetic structure comes close to those of a ferrite. Thus, r.m.s. value increases due to the enhancement of domain wall displacement with softening of martensite. Figure 3 shows the correlation graph between the r.m.s. values of the MBN signal with the hardness of specimens. The regression analysis shows an excellent correlation between the r.m.s. values with hardness.

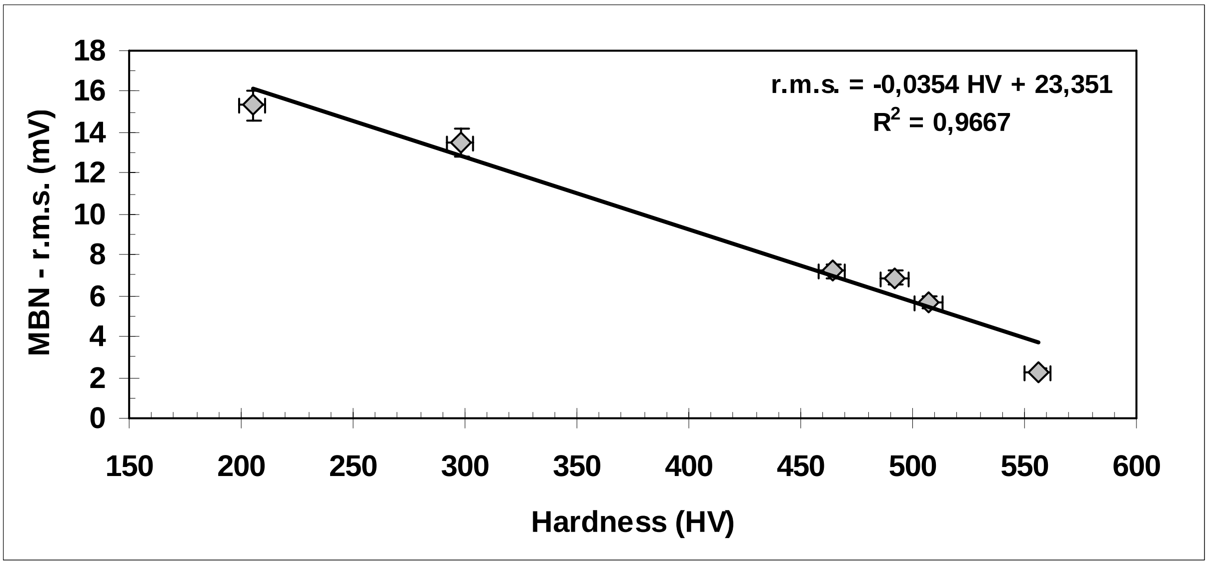


Figure 3. Correlation of MBN-r.m.s. Values with Hardness

4. Conclusions

Microstructures of the quenched and tempered SAE 4140 specimens were characterised by Magnetic Barkhausen Noise measurements. Application of identical austenitisation and quenching procedures eliminated the influence of other effects on the magnetic properties.

MBN method is a powerful tool for evaluating different stages of tempering. In the as-quenched sample, pinned domain walls due to high dislocation density and small martensite needles cause low MBN activity; and MBN peak is at higher magnetic fields due to small domain wall displacements and difficulty in domain nucleation. As tempering temperature increases dislocation density decreases, micro residual stresses diminish and the magnetic structure comes close to those of a ferrite. Thus, MBN activity gets higher due to the enhancement of domain wall displacement with softening of martensite. An excellent correlation exists between the r.m.s. values and hardness. Via establishing the quantitative relationships between MBN parameters the microstructural parameters, this method can be utilised efficiently and effectively for evaluating the hardness and the microstructure of the steel components.

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