$\label{eq:microstructural characterization of hypoeutectoid steels quenched from the A_{e1}$ - A_{e3} intercritical temperature range by magnetic barkhausen noise technique

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ABSTRACT

$\label{eq:microstructural characterization of hypoeutectoid steels quenched from the A_{e1} - A_{e3} \mbox{ intercritical temperature range by magnetic barkhausen noise technique}$

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This thesis aims to examine the possibility of using Magnetic Barkhausen Noise technique in characterizing the ferritic-martensitic microstructure of hypoeutectoid steels quenched from the intercritical temperature range. For this purpose, rectangular specimens were prepared from SAE 1020, 1040 and 1060 steels. The specimens were heated at different temperatures within the intercritical temperature range and then quenched into water. Microstructures of the specimens were characterized by metallographic examinations and hardness measurements. The measurements of the Magnetic Barkhausen Noise (MBN) were performed by using both Rollscan and µSCAN sensor connectors. It was seen that, for specimens having identical carbon content, Barkhausen emission decreased as the heating temperature increased. Moreover, in specimens heated at the same temperature, Barkhausen emission decreased as the carbon content of the specimen increased. In both cases, the decrease in Barkhausen emission is associated with the increase in martensite content. The results indicate that MBN is inversely proportional to hardness and that MBN is very sensitive to the microstructural condition of the material. It has been shown that using MBN is a powerful tool for evaluating the microstructure of hypoeutectoid steels quenched from the intercritical temperature range and that the use of this technique could be extended to characterize industrial dual phase steels.

Keywords: Hypoeutectoid Steel, Ferrite, Martensite, Microstructure, Magnetic Barkhausen Noise

A_{e1} – A_{e3} KRİTİK SICAKLIK ARALIĞINDAN SU VERİLEN ÖTEKTOİD-ALTI ÇELİKLERDE MANYETİK BARKHAUSEN GÜRÜLTÜSÜ TEKNİĞİ İLE MİKROYAPI KARAKTERİZASYONU

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Bu tezin amacı Manyetik Barkhausen Gürültüsü (MBG) tekniğinin Ae1 - Ae3 kritik sıcaklık aralığından su verilen ötektoid-altı çeliklerin ferritik-martensitik iç yapılarının karakterizasyonu için kullanılabilme olasılığını incelemektir. Bu amaçla, SAE 1020, 1040 ve 1060 çeliklerinden dikdörtgen numuneler hazırlandı. Bu numuneler, Ae1 - Ae3 kritik sıcaklık aralığında farklı sıcaklıklarda ısıtıldıktan sonra su verildi. Numunelerin iç yapıları metalografik incelemeler ve sertlik ölçümleriyle karakterize edildi. MBG ölçümleri hem Rollscan hem de µSCAN üniteleri kullanılarak yapıldı. Aynı karbon oranına sahip numunelerde, ısıtılma sıcaklığı arttıkça Barkhausen emisyonunun düştüğü gözlendi. Ayrıca, aynı sıcaklıkta ısıtılan numunelerde, numunenin karbon oranı arttıkça, Barkhausen emisyonu azaldı. Elde edilen sonuçlar MBG'nün sertlikle ters orantılı olduğunu ve malzemenin mikroyapısına oldukça duyarlı olduğunu gösterdi. Bu çalışma, MBG tekniğinin Ae1 -Ae3 kritik sıcaklık aralığından su verilen ötektoid-altı çeliklerin mikroyapılarını değerlendirmek için uygun bir yöntem olduğunu göstermiştir. Bu teknik, özellikle otomotiv endüstrisinde kullanılan çift fazlı çeliklerin karakterizasyonu için de kullanılabilir.

Anahtar Kelimeler: Ötektoid-altı Çelik, Ferrit, Martensit, Mikroyapı, Manyetik Barkhausen Gürültüsü

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CHAPTER 1

INTRODUCTION

The aim of this study, which is the first study conducted using the Magnetic Barkhausen Noise equipment in Türkiye, is to validate the efficient use of Magnetic Barkhausen Noise technique in characterizing the ferritic-martensitic microstructure of hypoeutectoid steels quenched from the intercritical temperature range.

Magnetic Barkhausen Noise analysis method is based on a concept of inductive measurement of a noise-like signal, generated when magnetic field is applied to a ferromagnetic sample. After a German scientist Professor Heinrich Barkhausen who explained the nature of this phenomenon in 1919, this signal is called Barkhausen noise.

Ferromagnetic materials consist of small magnetic regions resembling individual bar magnets called domains. Each domain is magnetized along a certain crystallographic easy direction of magnetization and is characterized by a constant magnetization. Domains are separated from their neighbours by finite frontiers called domain walls, which are also known as Bloch walls. Figure 1.1. shows magnetic domains in the grains of a polycrystalline material and a Bloch wall separating two domains. When a magnetic field is applied, domain walls move due to the growth in volume of favorably oriented, preexisting domains at the expense of those less favorably oriented. The result is a change in the overall magnetization of the sample.

If a coil of conducting wire is placed near the sample while the domain wall moves, the resulting change in magnetization will induce an electrical pulse in the coil. When the electrical pulses produced by all domain movements are added together, a noise-like signal called Barkhausen noise is generated [1].



Figure 1.1. (a) A qualitative sketch of magnetic domains in a polycrystalline material.(**b**) The magnetic moments in adjoining atoms change direction continuously across the boundary between domains [2].



Figure 1.2. A typical magnetization curve, with B, the flux density, appearing to be a continuous function of H, the magnetic field [3].

Prof. Barkhausen proved that the magnetization process, which is characterized by the hysteresis curve, in fact is not continuous, but is made up of small, abrupt steps caused when the magnetic domains move under an applied magnetic field. Figure1.2. shows the small, discontinuous changes of B as H varies. These discontinuous changes are a result of the Barkhausen effect, i.e. small magnetization jumps due to domain walls becoming pinned and released from microstuctural obstacles such as grain boundaries, second phase particles, and non-metallic inclusions. Each abrupt jump produces a brief burst of magnetic noise which can be detected and analyzed [3].

Barkhausen noise has a power spectrum starting from the magnetizing frequency and extending beyond 2 MHz in most materials. It is exponentially damped as a function of distance it has traveled inside the material. This is primarily due to the eddy current damping experienced by the propagating electromagnetic fields that domain wall movements create. The extent of damping determines the depth from which information can be obtained, i.e. measurement depth. The main factors affecting this depth are

- i) frequency range of the Barkhausen noise signal analyzed,
- ii) conductivity and permeability of the test material.

Measurement depths for practical applications vary between 0.01 and 1.5 mm.

Two important material characteristics affect the intensity of the Barkhausen noise signal. One is the presence and distribution of elastic stresses which will influence the way domains choose and lock into their easy direction of magnetization. This phenomenon of elastic properties interacting with domain structure and magnetic properties of material is called a magnetoelastic interaction. As a result of magnetoelastic interaction, in materials with positive magnetic anisotropy (iron, most steels and cobalt), compressive stresses will decrease the intensity of Barkhausen noise while tensile stresses increase it. This fact can be exploited so that by measuring the intensity of Barkhausen noise the amount of residual stress can be determined. The measurement also defines the direction of principal stresses.

The other important material characteristic affecting Barkhausen noise is the microstructure of the sample. This effect can be broadly described in terms of hardness: the noise intensity continuously decreases in microstructures characterized by increasing hardness. In this way, Barkhausen noise measurements provide information on the microstructural condition of the material [1].

The Barkhausen noise signal is a signature of the microstructural state of the crystal. Usually, the envelope of the signal is plotted as a function of the applied magnetic field. The envelope generally 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).



Figure 1.3. Typical Barkhausen noise signal with the RMS envelope. BNA represents the maximum amplitude of the envelope and Hpeak the corresponding magnetic field [4].

Hypoeutectoid steels show similarities with those steels used in obtaining dual phase (ferritic-martensitic) steels which are important for the automotive industry since they have better mechanical properties than those of many commercial high strength low alloy steels. Dual phase steels, which are generally produced by an intercritical annealing in the austenite-ferrite region followed by a rapid water quench, exhibit exceptional strength and formability. The high formability of dual phase steels is due to low yield to tensile strength ratio with high work hardening rates, promoting uniform elongation.

In addition to high ductility, dual phase steels also exhibit a strong bake hardening response and have excellent fatigue resistance. Besides, they demonstrate a positive response to strain rates, critical to the crash performance of the material in a vehicle during impact. Having all of these properties that help to improve fuel economy and vehicle performance, dual phase steels are currently materials of commercial interest for the automotive industry and they have already found numerous applications including safety critical products such as side impact bars and wheel rims [5-7].

Dual phase steels are characterised by a matrix of fine ferrite containing small islands of martensite. The hard martensite particles provide substantial strengthening while the ductile ferrite matrix gives good formability. To produce a dual phase microstructure, the equilibrium pearlite phase needs to be eliminated, with austenite being encouraged to form martensite by rapid cooling.

The simplest method for producing dual phase microstructures is to anneal a ferrite/pearlite steel in the intercritical temperature range. The annealing temperature is controlled within the ferrite plus austenite two phase region, such that much of the room temperature ferrite phase remains. The pearlite reverts to carbon rich austenite. When the steel is then quenched from the annealing temperature the austenite proportion is sufficiently hardenable to transform into martensite [8].

In this study the hypoeutectoid steel specimens were also heated in the intercritical temperature range and then quenched into water in order to imitate dual phase steel production. The aim is to examine the possibility of using Magnetic Barkhausen Noise technique to characterize the microstructure of hypoeutectoid steels quenched from the intercritical temperature range and to form a basis for the future use of this technique in non-destructive characterization of industrial dual phase steels.

CHAPTER 2

LITERATURE SURVEY

Magnetic Barkhausen Noise (MBN) technique has proved its viability for characterization of microstructures and it is considered as a valuable nondestructive evaluation (NDE) technique for microstructural characterization of ferromagnetic materials [9-17]. The dual sensitivity of the phenomenon to stresses and to microstructure on which this study is focused, gives a wide range of potential applications to the technique. For instance, it has already been used to assess the quality of heat treatment [18], to evaluate stresses [19,20], to detect defects [21,22] or to inspect surface conditions [23,24] in steel components and in other magnetic materials.

The effect of crystalline microstructure features on MBN has been widely investigated in steels, and the influence of grain size [22,25,26], carbide precipitates [13,27], ferrite, pearlite and martensite phases [13,28,29], and dislocations [26,30] can be found in the literature. Since the magnetic structure is directly linked to the nature of the metallurgical state, different phases may produce a very different Barkhausen effect. Several studies on the Barkhausen noise response of various steels have proved that it is strongly dependent on the existing phases. Different Barkhausen emission (BE) profiles, i.e. plots of BE signal against applied magnetizing field, obtained from the same type of material imply the existence of alternative forms of microstructures. For instance, ferrite and pearlite microstructures have a strong Barkhausen activity located at a low magnetic field [31,32], whereas a martensite microstructure has a low Barkhausen emission located at a high field [33].

In a study conducted by Mèszáros, Káldor and Hidasi, the application possibilities of Barkhausen energy measuring method and system for determining the amount of ferromagnetic phase in two different alloys were investigated. As a result, the applied Barkhausen noise measuring method was proved to be an accurate and reliable nondestructive method for determining the amount of the ferromagnetic phase in the studied alloys. It was found to be an effective tool for studying the transformation mechanism of the austenite to $\dot{\alpha}$ -phase during cold work and the reverse transformation of $\dot{\alpha}$ -phase back to austenite due to heat treatment [34].

In another study conducted by Sullivan, Cotterell, Tanner and Mèszáros, magnetic Barkhausen noise technique was employed to characterise ferritic stainless steel samples. In this study, MBN was shown to decrease with increasing permanent material deformation. It was found that the inverse of MBN (RMS) is linearly proportional to hardness. It was also found that MBN was strongly dependent on the microstructural condition of the material and it was shown that MBN can be a useful nondestructive evaluation technique for a means to characterise the microstructural state of ferritic stainless steels [35].

Moorthy, Vaidyanathan, Laha, Jayakumar, Rao and Raj conducted a study in which they used MBN measurements to characterise different microstructural regions such as coarse grain region, fine grain region, intercritical structure within the heat affected zone (HAZ) of two different steel weldments. This study showed the possibility of measuring the extent of different microstructural regions within the HAZ using miniature MBN probes with prior calibration [36]. It was also observed that MBN level decreases with increase in hardness corresponding to different microstructures. A similar relationship between hardness, was also found by Yi, Lee and Kim [37].

Yet another study conducted by Okazaki, Ueno, Furuya, Spearing and Hagood investigated the possibility to detect the phase transformation of stress-induced martensite in a ferromagnetic shape memory alloy by using Barkhausen noise method. As a result of this study, Barkhausen noise method turned out to be a powerful technique for non-destructive evaluation of the phase transformation of ferromagnetic shape memory alloy [38].

Saquet, Chicois and Vincent undertook a study to establish the characteristics of MBN in plain steels for various crystalline microstructures. First the MBN fingerprints of single constituent steels (ferrite, pearlite and martensite) were studied and then examples of MBN characteristics for more complex microstructures were presented. It was shown that microstructure of plain steels had a strong influence on the Barkhausen noise features such as MBN peak height, position, frequency range and relative amplitude of MBN. Furthermore, it was found that the MBN activity in martensite is characterized by a very low amplitude and a high frequency range and it was shown that using MBN is a powerful tool for evaluating microstructure of plain steels [39].

Mitra, Govindaraju and Jiles also observed that the Barkhausen emissions are strongly affected by microstructure and they showed that coarse pearlitic microstructures exhibit large RMS voltage, whereas, bainite and spheroidized structures have lower amplitude of Barkhausen emissions [40]. Kameda showed that the MB signal was sensitive to the microstructure change induced during tempering treatments [41]. Ng et al. observed an increase of the MBN signal with the carbon content (from 0.13 to 0.97 wt %) [42].

Magnetic Barkhausen noise has been used to characterize the microstructures in quenched and tempered 0.2% carbon steel by Moorthy, Vaidyanathan, Jayakumar and Raj. In this study, the effect of microstructural changes, the influence of dissolution of martensite and the precipitation of cementite on MBN were explained. It was shown that there was a remarkable correlation of tempered microstructure of 0.2% carbon steel with MBN signal. It was also observed that MBN generation was strongly influenced by the dissolution of martensite and precipitation of cementite particles. The variation in grain size and the cementite particle size could be clearly observed from the variation in the MBN peak height and peak position values. This study showed that MBN measurements could be used to evaluate different stages of tempering in ferritic steels [43].

In a study conducted by Ng, Cho, Wong, Chan, Ma and Lo, it has been found that the BE signal, which is proportional to the number of unpinning events of domain walls, is more intense in samples with small grains which have been annealed at lower temperatures, than in those with large grains which have been annealed at higher temperatures. This study also showed that the BE technique can be used to detect recrystallization temperature of steel samples by exploiting the significant drop of the overall BE signal level due to recrystallization [44].

The effect of tempering on the MBN signal profile was studied in casecarburised EN36 steel using a range of magnetic excitation frequencies and a number of frequency ranges for analysis of the MBN signal by Moorthy, Shaw and Evans. In this study, it has been shown that the MBN level increases with tempering due to coarsening of the microstructure. Moreover, it was found that the systematic variations in the MBN peak height values in different analyzing frequency ranges were consistent with the microstructural gradient. An empirical relationship has also been established between the hardness-depth profile and the MBN measurements [45].

A model of the microstructural defect influence on the magnetic Barkhausen noise in plain steels was presented in a study conducted by Pèrez-Benitez, Capó-Sánchez, Anglada-Rivera and Padovese. This model is also able to describe the influence of the 90° domain wall and the second phase particles on MBN. In this study, it has also been shown that MBN technique is very useful for the carbon content characterization in commercial steels [46]. Kleber, Hug, Merlin and Soler conducted a study in which magnetic Barkhausen noise measurements were carried out to characterize Ferrite-Martensite steels. As a result of this study, it was concluded that the two phases, ferrite and martensite, can be easily distinguished by Barkhausen noise measurements. A good correlation between the martensite volume fraction and the Barkhausen noise amplitude was found. It was observed that the value of the magnetic field for which the Barkhausen noise peak is obtained, correlates with the carbon content of martensite. This study 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. It was concluded that the use of this technique could be extended to characterize industrial dual phase steels [4].

In addition to the numerous studies conducted on microstructural characterization using MBN technique and on the influence of microstructural phases, various studies have still been continuing on understanding the influence of the microstructural features of some special steels, such as dual-phase and triple-phase steels on MBN.

CHAPTER 3

EXPERIMENTAL METHODS

3.1 Material

The samples were prepared from plain carbon steels: SAE 1020, 1040 and 1060. The steels and their respective compositions are shown in Table 3.1. The samples were machined in identical rectangular form of size $24 \times 10 \times 8$ mm. In order to avoid surface machining residual stresses to which MBN is very sensitive, all the machining operations were performed prior to heat treatments.

Table 3.1. Chemical compositions of SAE 1020, 1040, 1060 steels (wt %)

Steel	С	Mn	Si	Ni	Cr	Mo	Cu	Р	S	V	Fe
SAE											
1020	0,189	0,594	0,147	0,154	0,086	0,027	0,312	0,018	0,029	0,013	balance
SAE											
1040	0,392	0,782	0,423	0,131	0,226	0,023	0,197	0,022	0,004	0,014	balance
SAE											
1060	0,614	0,66	0,258	0,087	0,121	0,046	0,145	0,013	0,021	0,013	balance

3.2 Metallographic Investigation

For the metallographic examination, the samples were ground, polished and etched in a 2% Nital solution to reveal the microstructure. Optical micrographs and scanning electron microscope (SEM) photographs of the samples were taken to correlate the magnetic properties with the microstructure.

3.3 Heat Treatment

In order to investigate the effect of different microstructures on MBN measurements, various heat treatment procedures were applied to the samples. Details of the heat treatments are summarized in Table 3.2.

Tab	le 3.2.	Heat	treatment	schedu	ile.
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Steel	Heating Temperature for 1h	Quenching
	730°C	
G 4 F 1000	750°C	
SAE 1020	780°C	
	820°C	23°C water
	730°C	
SAE 1040	750°C	
	780°C	
SAE 1060	750°C	
	740°C	
SAE 1040	760°C	
	780°C	5°C water
SAE 1060	740°C	
SAE 1000	750°C	

3.3 Hardness Test

Hardness of the specimens was determined by measuring Rockwell hardness C, HRC. The measurements of the Rockwell C scale hardness on the rectangular type specimens were made using a 150 kg load. The hardness values given are the average of at least three measurements on each sample, with the total scatter being no more than ± 2 HRC.

3.5 Magnetic Barkhausen Noise Measurement

Before MBN measurements, absence of residual magnetism on the specimens was verified by Gaussmeter measurements. Since the specimens were heated at temperatures which are above or very close to their Curie temperature, they lost their permanent magnetism and became paramagnetic. This demagnetized state also prevailed after quenching and the MBN measurements were conducted when the specimens were in such a state.

Barkhausen noise signal was measured with μ SCAN 500-2 equipment (manufactured by Stresstech Inc.). A picture of the equipment can be seen in Figure 3.1. and a schematic diagram is also presented in Figure 3.2. Measurements were carried out by using the sensor, S1-138-13-01. μ SCAN 500 central unit has two sensor connectors: Rollscan and μ SCAN. In this study, measurements were carried out using both sensor connectors. In Rollscan measurements, the magnetizing frequency was 125 Hz and the filter passed frequencies from 70 to 200 kHz.

In μ SCAN measurements, an excitation magnetic field with a frequency of 125 Hz was obtained. Magnetizing voltage, which adjusts the magnitude of magnetizing field applied to the specimen, was set to 10 V. Sampling frequency, the parameter determining how many samples per second are stored for signal analysis was set as 2 MHz. Number of bursts, the parameter determining how many magnetizing half cycles will be stored for signal analysis, was set as 186, the highest possible number of half cycles when the magnetizing frequency is 125 Hz and the sampling frequency is 2 MHz. The measured Barkhausen emission signals are filtered (pass-band: 0.5-500 kHz) and amplified (voltage gain of 20 dB), and are plotted as a function of the magnetizing field over a hysteresis cycle in order to produce a BE profile.

In order to be able to see the effect of frequency on Barkhausen noise measurements, Barkhausen noise response of a group of specimens were measured by setting the magnetizing frequency first to 125 Hz, then to 50 Hz and finally to 5 Hz. Number of bursts was set as 186 when the magnetizing frequency was 125 Hz, as 74 when the magnetizing frequency was 50 Hz and as 6 when the magnetizing frequency was 5 Hz. The other parameters remained the same.



Figure 3.1. µSCAN equipment [1].



Figure 3.2. Block diagram of µSCAN 500 system.

CHAPTER 4

RESULTS & DISCUSSION

4.1 Results of Hardness Measurements

When a specimen is heated at a temperature within the intercritical temperature region, much of the room temperature ferrite phase remains. The pearlite reverts to carbon rich austenite. When the steel is then quenched from the heating temperature, the austenite proportion is expected to transform into martensite. When the lever rule is applied, the amounts of austenite (γ) and ferrite (α) phases that are expected to be present in a given specimen which is heated at a given temperature within the intercritical temperature region can be found. Table 4.1. shows the expected austenite and ferrite contents of the specimens.

Table 4.1. The austenite and ferrite contents of the specimens heated at temperatures within the intercritical temperature region.

Specimen-Heating	%γ	% a
Temperature (°C)	(calculated)	(calculated)
SAE 1020 – 730	24	76
SAE 1020 – 750	28	72
SAE 1020 – 780	40	60
SAE 1020 – 820	61	39
SAE 1040 – 730	52	48
SAE 1040 – 750	61	39
SAE 1040 – 780	84	16
SAE 1060 – 750	93	7

It is seen that as the heating temperature within the intercritical temperature region is increased, the amount of austenite formed increases for the same type of specimens. Similarly, as the carbon content of the specimen is increased when all specimens are heated at the same temperature, the amount of austenite present in the specimen again increases. Therefore, after quenching more martensite is expected to form in those specimens heated at higher temperatures and also in those having a higher carbon content. Accordingly, hardness values of the specimens that contain more martensite are expected to be higher.

Results of the hardness measurements and the theoretical hardness values are given in Table 4.2. The measured hardness values are in agreement with the theoretical hardness values found by first determining the percentage of phases (namely austenite and ferrite) at the given temperatures by using lever rule and then multiplying the percentage of phases with the corresponding hardness values [47] assuming that all of the austenite transformed into martensite. The agreement between the theoretical and actual hardness values indicates that the martensite content of the specimens increased with increasing heating temperature and that martensite content also increased with increasing carbon content for the specimens heated at the same temperature.

Only in the 1040 specimen which was heated at 730°C and then quenched into 23°C water, the actual hardness value is quite lower than that of the theoretical hardness value. Metallographic examination also showed that this specimen had a ferritic-pearlitic microstructure. The occurrence of a ferritic-pearlitic microstructure that led to a low hardness value in this specimen was most probably due to decarburization since the heat treatments could not be performed in an atmosphere controlled furnace.

Specimen-HeatingTheoreticalTemperature (°C)Hardness(HRC)		Actual Hardness (HRC)		
	(1110)			
SAE 1020 - 730	15,8	$14,2 \pm 0,8$		
SAE 1020 - 780	23,0	$23,1 \pm 1,2$		
SAE 1020 - 820	32,5	$32,2 \pm 1,8$		
SAE 1040 - 730	34,6	$11,6 \pm 0,3$		
SAE 1040 - 750	39,3	$34,3 \pm 1,9$		
SAE 1040 - 780	51,5	57,8 ± 1,1		
SAE 1020 - 750	17,6	$16,3 \pm 0,5$		
SAE 1040 - 750	39,3	$53,7 \pm 2,0$		
SAE 1060 - 750	61,0	$64,2 \pm 1,6$		

 Table 4.2. Theoretical and actual hardness values.

Figures 4.1. (a) and (b) show that hardness increased with increasing heating temperature and also that hardness increased with increasing carbon content when the heating temperature is identical for all specimens. This increase in hardness is a result of the increase in martensite content in both cases. SAE 1020 specimens were heated at 730°C, 780°C, 820°C and the theoretical martensite content of these specimens after quenching are 24%, 40%, and 61% respectively. Similarly, SAE 1040 specimens were heated at 730°C, 750°C, 780°C and theoretically the martensite content in these specimens after quenching are expected to be 52%, 61%, and 84% respectively. As a third group of specimens, SAE 1020, 1040 and 1060 specimens were all heated at 750°C and the corresponding martensite content in these specimens are expected to be 28%, 61% and 93% theoretically. In all groups of specimens, martensite content was expected to increase gradually and hardness values were also expected to increase as a result of the increase in martensite content.



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Figure 4.1. Variation of hardness (a) in SAE 1020 and SAE 1040 specimens heated at different temperatures, (b) in SAE 1020, 1040 and 1060 specimens all heated at 750°C and then quenched into 23°C water.

4.2. Results of Metallographic Investigation

The samples were ground, polished and etched in a 2% Nital solution for the metallographic examination. All of the samples were examined under optical microscope and SEM photographs of the samples were also taken to correlate the Barkhausen noise and hardness measurements with the microstructure. The increase in the martensite content of the samples with both increasing heating temperature and increasing carbon content is also revealed by the SEM photographs. Figures 4.2. (a), (b), (c), 4.3. (a), (b), (c) and 4.4. (a), (b), (c) show the SEM photographs of the samples.





Figure 4.2. SEM photographs of SAE 1020 specimens heated at (a) 780°C, (b) 820°C followed by 23°C water quench.



Figure 4.3. SEM photographs of SAE 1040 specimens heated at (a) 730°C, (b) 750°C, (c) 780°C followed by 23°C water quench.



Figure 4.4. SEM photographs of (a) SAE 1020, (b) SAE 1040, (c) SAE 1060 specimens heated at 750°C followed by 23°C water quench.
4.3. Results of Rollscan Measurements

Rollscan is a passive sensor which has a band pass filter with a range of 70-200 kHz. This filter range implies that the frequency components which are smaller than 70 kHz and those which are higher than 200 kHz are filtered off mathematically. Rollscan reads the magnetic parameter (MP) value of the specimens. Soft magnetic materials have a high magnetic response, whereas hard magnetic materials have a low magnetic response. Hence, a high MP value indicates that the specimen is a soft magnetic material, whereas a low MP value indicates that the specimen is a hard magnetic material.

Magnetic parameters derived from Barkhausen emission signals are strongly affected by the changes in the microstructure since the dimensions of the magnetic domains and the domain walls are comparable with the dimensions of such microstructural features as grain boundary, grains, precipitates, etc. Both, the domain wall movement and domain nucleation, are affected by the microstructural features.

Rollscan data for the three specimen groups are presented in Figures 4.5. (a), (b) and (c).

Results of the Rollscan measurements are presented in graphical form in Figures 4.6. (a) and (b). It is seen that the MP value decreases as the heating temperature increases in both SAE 1020 and 1040 specimens. MP values also decrease with increasing carbon content when all the specimens are heated at 750°C.



Figure 4.5. Rollscan data of (a) SAE 1020 specimens heated at 730°C, 780°C, 820°C, (b) SAE 1040 specimens heated at 730°C , 750°C, 780°C, (c) SAE 1020, 1040, 1060 specimens all heated at 750°C.



(a)



Figure 4.6. Variation of MP (a) in SAE 1020 and SAE 1040 specimens heated at different temperatures, (b) in SAE 1020, 1040 and 1060 specimens all heated at 750°C and then quenched into 23°C water.

This study aimed to investigate the microstructures of hypoeutectoid steels quenched from the *intercritical temperature range*. In order to be able to observe meaningful changes in the MBN measurements, for the same type of specimen, heating temperature was increased at least 20°C at a time and similarly when the specimens were heated at the same temperature, carbon content of the specimens was increased 0.2%. Since the intercritical temperature range is narrow, only three different heating temperatures and three specimens having different carbon content could be used. Therefore, the graphs are drawn using three data points.

When an alternating magnetic field is applied to a ferromagnetic material, a magnetic hysteresis loop is induced since the irreversible process of magnetization causes an energy loss. This irreversible process of magnetization is strongly related to the dynamic behaviour of domains in a magnetic field. Magnetic Barkhausen signal reflects the magnetic flux change due to the magnetic moment change during the domain wall motion.

The decrease in MP values is a result of the increase in martensite content, which is also revealed by the increase in hardness values. For martensite, the leading characteristic of Barkhausen noise is that MBN is very weak and it occurs at high magnetization field. The domain structure in martensite is mainly determined by the tetragonality of martensite structure. Figure 4.7. shows the magnetic domains in a martensitic microstructure.



Figure 4.7. A sketch of magnetic domains in a martensitic microstructure.

Due to the small size of martensite needles, the domain wall energy plays an important role, so the relative volume occupied by a wall is larger than in the other phases of steel. Domain walls are pinned due to high dislocation density, therefore the resistance to the growing domains is very high. Domain wall displacements are low and walls are difficult to create since the reversal of magnetization requires a strong field. Hence, the resulting MBN is very weak. Moreover, the second order internal stresses in the needles, which are inherent to the martensitic transformation, may also have an effect via the magneto-elastic coupling [39].

The correlation between MP values and hardness is shown in Figures 4.8. (a) and (b). There is a linear relationship and in Figure 4.8. (a), it is seen that the slopes of the curves are almost the same, however, the relative positions of the curves are different. It means that the effect of heating temperature, which causes a change in the martensite content, is similar on both SAE 1020 and 1040 specimens. The increase in martensite content causes an increase in the hardness of the specimen, while decreasing the MP value.



(a)



Figure 4.8. Correlation between magnetic parameter (MP) and hardness (HRC) (a) in SAE 1020 and 1040 specimens heated at different temperatures, (b) in SAE 1020, 1040 and 1060 specimens all heated at 750°C and then quenched into 23°C water.

4.4. Results of µSCAN Measurements

When a ferromagnetic material is excited by an external magnetic field, the magnetization state of the sample exhibits a sequence of Barkhausen jumps. Magnetic Barkhausen emissions are detected in the form of voltage pulses induced in a sense pick-up coil positioned close to the surface of the material. The amplitude distribution of such pulses depends on the microstructure and residual stress. An analysis of the Barkhausen signal permits evaluation of changes in subsurface material condition. The root mean square (RMS) value of the voltage signal detected from the specimens is given as an output in μ SCAN measurements. RMS voltage, which is obtained by averaging the BE signal over the time for magnetization reversal, gives the average of the Barkhausen activity.

Results of the μ SCAN measurements are shown in Figures 4.9. (a) and (b). It is seen that RMS value decreased as the heating temperature increased in both SAE 1020 and 1040 specimens and it also decreased with increasing carbon content when all the specimens were heated at 750°C. This decrease in RMS values is again a result of the increase in martensite content, just as the decrease seen in the MP values in the Rollscan measurements.

The correlation between RMS values and hardness is shown in Figures 4.10. (a) and (b). It is observed that MBN (RMS) is inversely proportional to hardness. In Figure 4.10. (a), it is again seen that the slopes of the curves are almost the same, however, the relative positions of the curves are different indicating that the effect of heating temperature, i.e. the effect of the change in martensite content, is similar on both SAE 1020 and 1040 specimens.



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Figure 4.9. Variation of RMS Voltage (a) in SAE 1020 and SAE 1040 specimens heated at different temperatures, (b) in SAE 1020, 1040 and 1060 specimens all heated at 750°C and then quenched into 23°C water.





Figure 4.10. Correlation between RMS (V) and Hardness (HRC) (a) in SAE 1020 and 1040 specimens heated at different temperatures, (b) in SAE 1020, 1040 and 1060 specimens all heated at 750°C and then quenched into 23°C water.

Another parameter in μ SCAN measurements is the peak position. Peak position indicates the magnetic field at which the peak value of the Barkhausen noise signal is located. The fact that ferrite and pearlite microstructures have a strong Barkhausen activity located at a low magnetic field, whereas a martensite microstructure has a low Barkhausen emission located at a high field has been widely accepted in the literature [31-33]. The findings in this study are also in agreement with this statement. Figures 4.9. (a) and (b) show the decrease in Barkhausen emission and Figures 4.11. (a) and (b) show the increase in the magnitude of the magnetic field at which the peak is located, as the martensite content increases. Hpeak values in the graphs represent the percentage of the applied magnetic field.

In μ SCAN measurements, Barkhausen emission (BE) signals are plotted as a function of the magnetizing field over a hysteresis cycle in order to produce a BE profile. Figures 4.12. (a), (b) and (c) show the Barkhausen Emission (BE) profiles of SAE 1020, 1040 and 1060 specimens all heated at 750°C and then quenched into 23°C water. The vertical axis represents the Barkhausen noise signal in volts and the horizontal axis represents the corresponding magnetic field. The numbers on the horizontal axis represent the percentage of the applied magnetic field.



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Figure 4.11. Variation of Hpeak (a) in SAE 1020 and SAE 1040 specimens heated at different temperatures, (b) in SAE 1020, 1040 and 1060 specimens all heated at 750°C and then quenched into 23°C water.



Figure 4.12. BE profiles of (a) SAE 1020 specimen, (b) SAE 1040 specimen, (c) SAE 1060 specimen heated at 750°C and then 23°C water quenched.

If the three profiles are examined, it is seen that the peak value of the Barkhausen noise decreased from 15.26 V to 10.80 V and to 9.382 V as the carbon content of the specimen increased. Besides, the magnetic field at which the peak value of the Barkhausen noise signal is located shifted to higher fields as the carbon content of the specimen increased. The lower Barkhausen emission occurring at a higher magnetic field as the carbon content of the specimen increase in the martensite content. Moreover, the distance between the two peaks increased going from the SAE 1020 to SAE 1060 specimen indicating that the SAE 1020 specimen can be more easily magnetized when compared to the SAE 1040 specimen and that the SAE 1040 specimen.

BE profiles are plotted over a hysteresis cycle. The first peak appears at the magnetic field where the saturation magnetization is reached and the second peak is observed at the magnetic field where saturation is achieved in the opposite sense upon continuation of the applied field in the reverse direction. Hence, the distance between the two peaks in the profile corresponds to the distance between the magnetic field at which the saturation magnetization is reached and the magnetic field at which saturation is achieved in the opposite sense in a hysteresis curve.

Soft magnetic materials reach their saturation magnetization with a relatively low applied field, i.e. they are easily magnetized and demagnetized. Whereas, hard magnetic materials have a high resistance to demagnetization and a large magnetic field must be applied in a direction opposite to that of the original field to reduce the magnetization of the specimen to zero after the specimen has reached its saturation magnetization. Thus, the fact that the two peaks are close to one another in a BE profile indicates that the specimen can be easily magnetized. On the other hand, the two peaks' being far away from each other indicates that the specimen is hard to magnetize. Therefore, the increase in the distance between the peaks with increasing carbon content can also be considered as an evidence for the increase in martensite content. In μ SCAN measurements, a sample hysteresis curve for the specimen under examination is also produced. Since the μ SCAN 500-2 equipment used in this study is a commercial device, the hysteresis curve produced is not the actual hysteresis curve of the material but rather it is a sample curve produced to give an idea about the magnetic properties of the material. Figure 4.13. (a), (b) and (c) show the hysteresis curves produced for SAE 1020, 1040 and 1060 specimens which were all heated at 750°C and then quenched into 23°C water.

It is seen that the hysteresis curve of the SAE 1020 specimen is thin and narrow which is a characteristic of soft magnetic materials. Whereas, the hysteresis curve of the SAE 1060 specimen resembles those of hard magnetic materials. Moreover, the area within the loop increased from 17.51 to 20.06 and to 29.18 as the carbon content of the specimen increased. Soft magnetic materials have low hysteresis energy loses and they reach their saturation magnetization with a relatively low applied field. Hence, the area within the hysteresis loop of soft magnetic materials is small. On the other hand, hard magnetic materials have high hysteresis energy loses and the area within their hysteresis loop is large, as represented in Figure 4.14. It is also known that martensitic structure is magnetically much harder than ferritic and pearlitic structures. Therefore, the increase in the area within the loop as the carbon content of the specimen increased, indicates that the specimen having a higher carbon content is magnetically harder, which in turn reveals the fact that the martensite content of that specimen is also higher.



Figure 4.13. Sample hysteresis curves of (a) SAE 1020 specimen, (b) SAE 1040 specimen, (c) SAE 1060 specimen heated at 750°C and then 23°C water quenched.



Figure 4.14. Schematic magnetization curves for soft and hard magnetic materials [48].

Two other parameters that need to be investigated in order to be able to understand whether the specimen being examined is a soft or a hard magnetic material are permeability and coercivity. Permeability is the degree of magnetization of a material in response to a magnetic field and coercivity of a ferromagnetic material is the intensity of the magnetic field required to reduce the magnetization of that material to zero after the magnetization of the sample has reached saturation. Characteristically, soft magnetic materials have a high initial permeability and a low coercivity, whereas hard magnetic materials have a low initial permeability and a high coercivity.

 μ SCAN measurements give the permeability and coercivity values of the specimen being examined. However, these values are relative values since the equipment is not capable of measuring absolute permeability and coercivity values. Table 4.3. shows the relative permeability and coercivity values of the SAE 1020, 1040 and 1060 specimens all heated at 750°C and then quenched into 23°C water.

Specimen	Relative Permeability	Relative Coercivity
SAE 1020	117.91	0.102
SAE 1040	89.73	0.178
SAE 1060	81.25	0.371

Table 4.3. Relative permeability and coercivity values of SAE 1020, 1040,1060 specimens all heated at 750°C followed by 23°C water quench.

It is known that the addition of carbon causes an increase in coercivity and a decrease in permeability [25]. The findings in this study are in agreement with this statement. Table 4.3. shows that permeability decreased and coercivity increased as the carbon content of the specimen increased. Steels having martensitic microstructures exhibit the properties of hard magnetic materials. So, martensite microstructures are expected to have a low magnetic response and thus a low permeability. Besides, the reversal of magnetization requires a strong field in martensite microstructures and hence they tend to have high coercivity values. Therefore, the decrease in permeability and the increase in coercivity values as the carbon content of the specimen increased, indicate an increase in the martensite content.

Barkhausen Emission (BE) profiles of SAE 1020 specimens heated at 730°C, 780°C, 820°C and then quenched into 23°C water are shown in Figures 4.15. (a), (b) and (c).



Figure 4.15. BE profiles of SAE 1020 specimens heated at (a) 730°C, (b) 780°C, (c) 820°C and then 23°C water quenched.

If the three profiles are examined, it is seen that the peak value of the Barkhausen noise decreased from 16.06 V to 15.99 V and to 11.89 V as the heating temperature in the intercritical temperature region increased. Besides, the magnitude of the magnetic field at which the peak value of the Barkhausen noise signal is located and the distance between the two peaks increased as the heating temperature increased. These results obtained from the BE profiles indicate an increase in martensite content with the increase in heating temperature. Table 4.4. shows the relative permeability and coercivity values of the SAE 1020 specimens heated at 730°C, 780°C, 820°C and then quenched into 23°C water.

Table 4.4. Relative permeability and coercivity values of SAE 1020 specimens heated at 730°C, 780°C and 820°C followed by 23°C water quench.

Specimen-Heating	Relative Permeability	Relative Coercivity	
Temperature (°C)			
SAE 1020-730	136.19	0.114	
SAE 1020-780	131.79	0.138	
SAE 1020-820	101.83	0.214	

As the heating temperature increased, permeability decreased and coercivity increased. This decrease in permeability and increase in coercivity reveal the increase in martensite content that occurred as a result of the increase in the heating temperature.

4.5. Effect of Frequency on MBN Measurements

In order to see the effect of magnetizing frequency on Barkhausen noise measurements, Barkhausen noise response of SAE 1040 specimens, which were heated at 730°C, 750°C, 780°C and then quenched into 23°C water, were measured by using different magnetizing frequencies. The frequency of magnetizing field applied to the specimens was first set to 125 Hz, then to 50 Hz and finally to 5 Hz. Number of bursts was also adjusted each time the frequency was changed since the maximum number of bursts that can be set changes as the magnetizing frequency changes. The other measurement parameters remained the same for all measurements.

When magnetizing frequency is decreased while the magnetizing voltage is kept constant, magnetization level increases. Besides, when the magnetizing frequency is lower, the measurement depth, i.e. the depth from which information can be obtained, increases. Hardness and microstructure at the surface may not represent the whole structure when cooling rate shows significant variations as going from the surface to the interior. In such cases, differences in the phase content and residual stress state may occur through the thickness. In order to eliminate the effect of this risk, thin specimens were prepared and the results of the measurements conducted using different frequencies, i.e. different measurement depths, showed that the microstructure was uniform through the thickness.



Figure 4.16. Occurrence of Barkhausen event, f_A: analyzing frequency, H: magnetic field strength [49].

The electromagnetic skin depth δ is given by the equation

$$\delta = \frac{1}{\sqrt{\pi f \sigma \mu_0 \mu_r}} \tag{1}$$

where f is the frequency, σ is the conductivity of the material, μ_0 is the permeability of vacuum and μ_r is the relative permeability of the material [50]. Since MBN signal reflects the magnetic flux change, the same relationship can be considered for evaluating the measurement depth in MBN measurements. For plain carbon steels used in this study, the conductivity is $5 \times 10^6 \ \Omega^{-1} \ m^{-1}$ [51]. μ_0 is $4\pi \times 10^{-7}$ H/m [52]. Considering the fact that the low field relative permeability of iron is approximately 500 [50] and analyzing the permeability values obtained as a result of the μ SCAN measurements, μ_r is taken to be 100 when the frequency is 125 Hz and 50 Hz, however, it is taken to be 500 when the frequency is 5 Hz.

When the above equation and values are used, measurement depths when the magnetizing frequency is 125 Hz, 50 Hz and 5 Hz, are found to be approximately 0.8mm, 1mm and 2mm respectively.

Table 4.5. RMS voltage, peak value of Barkhausen noise signal and peak position values of SAE 1040 specimens when the magnetizing frequency used was 125 Hz, 50 Hz and 5 Hz.

C	125 Hz			50 Hz			5 Hz		
Specimen- Heating Temperature(°C)	RMS (V)	Peak (V)	Peak Position	RMS (V)	Peak (V)	Peak Position	RMS (V)	Peak (V)	Peak Position
SAE 1040 - 730	10.40	19.66	0.475	6.958	14.10	3.690	2.091	4.284	9.75
SAE 1040 - 750	5.249	10.53	3.837	3.637	7.735	7.645	1.332	2.628	10.94
SAE 1040 - 780	2.507	4.418	6.063	2.122	3.861	21.82	0.837	1.495	14.25

Table 4.6. Relative permeability and coercivity values of SAE 1040 specimens when the magnetizing frequency used was 125 Hz, 50 Hz and 5 Hz.

Specimen-		50 H	łz	5 Hz		
Heating Temperature (°C)	Relative Permeability	Relative Coercivity	Relative Permeability	Relative Coercivity	Relative Permeability	Relative Coercivity
SAE 1040–730	158.18	0.03	284.38	0,050	936.26	0.090
SAE 1040-750	86.95	0.054	166.40	0.082	587.88	0.090
SAE 1040–780	33.96	0.110	74.71	0.190	310.12	0.114

Tables 4.5. and 4.6. present the results of μ SCAN measurements of SAE 1040 specimens, carried out by using different magnetizing frequencies. Peak position values in the Table 4.5. give the percentage of the applied magnetic field.

It is seen that in all sets of measurements the RMS voltage which gives the average Barkhausen activity and the peak value of the Barkhausen noise signal decreased and the peak position shifted to higher magnetic fields as the heating temperature of the specimen increased. It was seen from the previous metallographic examinations and hardness test results that the martensite content of the specimen after quenching increased as the heating temperature in the intercritical temperature region increased. This increase in martensite content of the SAE 1040 specimens is revealed in the BE profiles by a decrease in the RMS voltage, a decrease in the peak value of the Barkhausen noise signal, an increase in the magnetic field at which the peak is located and an increase in the distance between the two peaks in all sets of measurements, i.e. when the magnetizing frequency was 125 Hz, 50 Hz and 5 Hz. As the heating temperature increased, permeability decreased and coercivity increased in all sets of measurements. That can also be considered as an evidence for the increase in the martensite content that occurred as a result of the increase in the heating temperature.

The only difference observed when the magnetizing frequency was changed was that the RMS voltage and peak Barkhausen noise signal values decreased as the magnetizing frequency decreased. Since the measurement depth increased as the magnetizing frequency decreased, number of voltage pulses decreased and weaker voltage pulses were induced in the sense pick-up coil that was positioned close to the surface of the specimen. There is also an increase in both permeability and coercivity values with the decrease in the magnetizing frequency, which results in an increase in the measurement depth.

4.6. Effect of Faulty Heat Treatment on MBN Measurements

In order to enhance martensite formation, a group of SAE 1040 specimens and a group of SAE 1060 specimens were quenched into 5°C water after being heated at different temperatures within the intercritical temperature region. SAE 1040 specimens were heated at 740°C, 760°C and 780°C. SAE 1060 specimens were heated at 740°C and 750°C. Due to certain problems that occurred during heat treatments, martensite was observed only in SAE 1040 specimen which was heated at 780°C and then quenched into 5°C water and in SAE 1060 specimen which was heated at 750°C and then quenched into 5°C water and in SAE 1060 specimen which was heated at 750°C and then quenched into 5°C water. Although identical heating and quenching procedures were applied to all samples, no martensite formation was observed in the other specimens. SAE 1040 specimens heated at 740°C, 760°C and the SAE 1060 specimen heated at 740°C, which were all quenched into 5°C water after being heated at the given temperatures for one hour, had either a ferritic or a ferritic-pearlitic microstructure.

Theoretically, SAE 1040 specimens heated at 740°C, 760°C and the SAE 1060 specimen heated at 740°C should have contained 57%, 70% and 88% austenite respectively, as can be seen from Table 4.7. Hence, the austenite, which was expected to be formed in these specimens, was expected to transform into martensite after quenching. However, such a transformation did not occur and no martensite was observed in these specimens after quenching. This failure in martensite formation was most probably due to the fact that the heat treatments were not conducted in a protected atmosphere. Since the furnaces were not atmosphere controlled, decarburization might have occurred or there might have been problems with the thermocouples of the furnaces.

Specimen-Heating	%γ	% а	
Temperature (°C)	(calculated)	(calculated)	
SAE 1040 - 740	57	43	
SAE 1040 - 760	70	30	
SAE 1040 - 780	84	16	
SAE 1060 - 740	88	12	
SAE 1060 - 750	93	7	

Table 4.7. The austenite and ferrite contents of SAE 1040 and SAE 1060

 specimens heated at temperatures within the intercritical temperature region.

When the specimens were examined under optical microscope, a ferritic microstructure was observed in SAE 1040 specimens heated at 740°C and 760°C, which were both quenched into 5°C water. Whereas, martensite was clearly observed in the SAE 1040 specimen heated at 780°C and then quenched into 5°C water. Similarly, a ferritic-pearlitic microstructure was observed in the SAE 1060 specimen heated at 740°C and then quenched into 5°C water, however, a martensitic microstructure was seen in the SAE 1060 specimen heated at 750°C and then quenched into 5°C water.

Figures 4.17. (a), (b) and (c) show the SEM photographs of the SAE 1040 specimens and Figures 4.18. (a) and (b) show the optical micrographs of the SAE 1060 specimens.

Still, the MBN measurements of the above mentioned specimens were useful in the sense that they showed the distinct difference between the Barkhausen emission of a ferritic or a ferritic-pearlitic microstructure and that of a martensitic microstructure. Figures 4.19. (a) and (b) show the Rollscan data of the aforementioned SAE 1040 and SAE 1060 specimens.



Figure 4.17. SEM photographs of SAE 1040 specimens heated at (a) 740°C, (b) 760°C, (c) 780°C followed by 5°C water quench.



Figure 4.18. Optical micrographs (x500 magnification) of SAE 1060 specimens heated at (a) 740°C, (b) 750°C followed by 5°C water quench.





Figure 4.19. Rollscan data of (a) SAE 1040 specimens heated at 740° C , 760° C and 780° C, (b) SAE 1060 specimens heated at 740° C and 750° C followed by 5° C water quench.

When the Rollscan data of SAE 1040 specimens is examined, it is seen that the specimen which was heated at 780°C and then quenched into 5°C water, has an MP value 15.7. Such a low MP value is a characteristic of martensitic microstructures. So, the MP value of this specimen indicates that it has a martensitic microstructure, which was also revealed by the metallographic examination. On the other hand, MP values of SAE 1040 specimens heated at 740°C and 760°C, which were both quenched into 5°C water, are both higher than 100. This high magnetic response is a characteristic property of ferritic microstructures. Hence, it is seen that the ferritic microstructures of these specimens, which were observed under optical microscope, could also be determined by Rollscan measurements.

Similarly, when the Rollscan data of SAE 1060 specimens is examined, it is seen that the specimen which was heated at 740°C and then quenched into 5°C water, has quite a high MP value, which is indicative of a microstructure that does not contain martensite. This conclusion drawn from the Rollscan data was evidenced by the metallographic examination which showed that the specimen had a ferritic-pearlitic microstructure. It is also seen that the MP value of the specimen which was heated at 750°C and then quenched into 5°C water, decreased significantly when compared to the SAE 1060 specimen heated at 740°C and then quenched into 5°C water. This decrease in the MP value leads to the conclusion that the latter specimen contains martensite and martensite was actually observed in this specimen under optical microscope.

Barkhausen Emission (BE) profiles of the specimens discussed in this section are presented in Figures 4.20. (a), (b), (c) and 4.21. (a), (b).



(a)







Figure 4.20. BE profiles of SAE 1040 specimens heated at (a) 740°C, (b) 760°C, (c) 780°C and then 5°C water quenched.

When the BE profiles of the SAE 1040 specimens heated at 740°C and 760°C which were both quenched into 5°C water, are examined, it is seen that the peak value of the Barkhausen noise signal was 20.36 V for the first specimen and 27.61 V for the second specimen. The peak value of the Barkhausen noise signal was obtained at a magnetic field which was nearly zero in both specimens. This high peak value of the BN signal occurring at a low applied is a characteristic of the ferritic microstructures as discussed before. Moreover, the two peaks are on top of each other in both profiles indicating that both specimens can be quite easily magnetized as expected from a ferritic microstructure.

When the BE profile of the SAE 1040 specimen heated at 780°C and then quenched into 5°C water, is examined, it is seen that the peak value of the Barkhausen noise signal was 2.842 V, which is quite lower than that of the previous specimens. Besides, this peak value occurred at a quite higher magnetic field when compared to the previous specimens. This weaker noise occurring at higher magnetization field is an indication of the martensitic microstructure of the latter specimen. It is also observed that the distance between the two peaks increased significantly in the profile of the last specimen when compared to that of the previous two specimens. The large distance between the peaks shows that the last specimen is quite hard to magnetize which can also be considered as an evidence for the martensitic microstructure of this specimen.







Figure 4.21. BE profiles of SAE 1060 specimens heated at (a) 740°C,(b) 750°C and then 5°C water quenched.

When the BE profiles of the SAE 1060 specimens heated at 740°C and 750°C which were both quenched into 5°C water, are examined, it is seen that the peak value of the Barkhausen noise signal was 14.69 V for the first specimen and 12.22 V for the second specimen. There is a decrease in the peak value of the BN signal and a significant increase in the magnetic field at which the peak is located in the latter specimen. Furthermore, the distance between the two peaks also increased significantly. These changes in the profile of the latter specimen are indicative of a microstructure which contains martensite.

The sample hysteresis curves of the specimens under examination are in agreement with the conclusions stated above. Figures 4.22. (a), (b), (c) and 4.23. (a), (b) show the hysteresis curves produced for those specimens.

When the sample hysteresis curves of the SAE 1040 specimens are examined, it is seen that the hysteresis curves of the SAE 1040 specimens heated at 740°C and 760°C which were both quenched into 5°C water, resemble that of a soft magnetic material. Whereas, the hysteresis curve of the SAE 1040 specimen heated at 780°C and then quenched into 5°C water, resemble that of a hard magnetic material. Moreover, coercivity of the latter specimen is quite higher and permeability of the latter specimen is significantly lower than that of the previous two specimens. This high coercivity and low permeability values of the latter specimen indicate that it is a hard magnetic material. Coercivity and permeability values of the first two specimens show that they are soft magnetic materials. These comparisons of the coercivity and permeability values of the specimens once again prove the fact that the first two specimens have a ferritic microstructure, whereas the last specimen has a martensitic microstructure.



Figure 4.22. Sample hysteresis curves of SAE 1040 specimens heated at (a) 740°C, (b) 760°C, (c) 780°C and then quenched into 5°C water.







Figure 4.23. Sample hysteresis curves of SAE 1060 specimens heated at (a) 740°C, (b) 750°C and then quenched into 5°C water.

When the sample hysteresis curves of the SAE 1060 specimens are examined, it is seen that the first hysteresis curve which is produced for the SAE 1060 specimen heated at 740°C and then quenched into 5°C water, resembles that of a soft magnetic material. On the other hand, the second hysteresis curve which is produced for the SAE 1060 specimen heated at 750°C and then quenched into 5°C water, resembles that of a hard magnetic material. Besides, coercivity of the latter specimen is quite higher than that of the former specimen and the permeability of the latter specimen is relatively lower than that of the former specimen. This increase in the coercivity and decrease in the permeability values together with the increase in the area within the hysteresis curve of the latter specimen indicate that this specimen is magnetically harder, which in turn reveals the fact that this specimen contains martensite since martensitic microstructure is magnetically much harder than ferritic and pearlitic microstructures.
CHAPTER 5

CONCLUSION

For the purpose of investigating the microstructures of SAE 1020, 1040 and 1060 specimens quenched from the intercritical temperature range, Barkhausen noise and hardness measurements were carried out. Identical heating and quenching procedures were applied in order to eliminate the influence of the prior-austenite grain size on the magnetic properties.

Both Rollscan and μ SCAN measurements showed that BN signal was very sensitive to the changes in microstructure that occurred as a result of the change in heating temperature or in carbon content. In Rollscan measurements, it was seen that the MP value decreased when heating temperature increased and also when carbon content increased at identical heating temperature. In μ SCAN measurements, it was observed that the RMS value decreased and the peak position shifted to higher magnetic fields when heating temperature increased at identical heating temperature increased and also when carbon content increased at identical heating temperature increased and the peak position shifted to higher magnetic fields when heating temperature increased at identical heating temperature.

It has been shown that the increase in the martensite content causes a decrease in the peak voltage and an increase in the magnetic field at which the peak is located. The weaker noise occurring at higher magnetization field as the martensite content increased is due to the tetragonal structure and the small size of needles in martensite, which impede the movement of domain walls.

It has been concluded that changing the magnetizing frequency does not cause a major change in the results of the MBN measurements when the microstructure is homogeneous through the thickness. Three different magnetizing frequencies were used to measure the MBN response of the same group of specimens and it was seen that in all sets of measurements, the RMS voltage and the peak value of the Barkhausen noise signal decreased, while the peak position shifted to higher magnetic fields as the heating temperature in the intercritical region increased. The only difference observed when the magnetizing frequency changed was that the RMS voltage and peak Barkhausen noise signal values decreased as the magnetizing frequency decreased, due to the increase in the measurement depth.

It has also been shown that the MBN activity in ferritic and ferritic-pearlitic microstructures is characterized by a high amplitude occuring at low applied field, whereas the MBN activity in martensitic microstructures is characterized by a low amplitude occuring at high applied field.

It is also concluded that MP and MBN (RMS) are inversely proportional to hardness. The increase in martensite content causes an increase in the hardness of the specimen, while decreasing the MP and MBN (RMS) values. This inverse relationship can be further investigated in future studies in order to be able to use MBN measurements to predict hardness.

Another important conclusion drawn from this study is that heat treatment, especially in the intercritical temperature region, should be conducted quite carefully and if possible in a protected atmosphere in order to avoid decarburization. Due to certain problems that occurred during the heat treatments, martensite formation could not be observed in several specimens. Thus, it would be better to use atmosphere protected furnaces, thermocouples of which are ensured to be accurate, when such an heat treatment is to be conducted.

Finally, this study showed that using MBN is an appropriate tool for evaluating the microstructures of hypoeutectoid steels quenched from the intercritical temperature range. These results obtained on hypoeutectoid steels have to be extended to dual phase steels in order to show the feasibility of using MBN technique in non-destructive characterization of industrial dual phase steels.

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