The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM)
ISSN: 2602-3199

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2023

Volume 26, Pages 306-316

IConTES 2023: International Conference on Technology, Engineering and Science

Comparative Non-Destructive Investigations of the Effects of Structural Changes After Heat Treatment of Carbon Steel Samples by Magnetic Noise and Vibration Methods

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Abstract: The microstructure of some components that work for a long time at high temperatures changes, which leads to a reduction in their life cycle. Traditionally, the study of the resulting structures is carried out by mechanical tests and metallographic analysis. The application of non-destructive methods increases the possibilities to monitor the quality of the elements and to prevent destruction. The aim of the study is to detect changes in the microstructure after different heat treatment and ageing heating of materials via magnetic noise and vibration methods. To realize the goal, samples of high-quality medium carbon steel were prepared through targeted heat treatments, quenching and tempering at different temperatures, as well as ageing heating at temperatures of 700 ^{o} C for up to 16 hours by vibration and magnetic noise method. The natural frequencies and damping properties of forced longitudinal vibrations of various heat treated steel specimens were obtained and analyzed. Using magnetic noise method, the following parameters were obtained and analyzed: root mean square voltage (RMS), energy and envelope parameters of magnetic noise signals. The comparative application of several non-destructive methods increases the knowledge of methods and materials and makes it possible to establish their sensitivity in studying microstructural characteristics.

Keywords: Comparative non-destructive investigations, Magnetic noise method, Vibration method, Carbon steel, Microstructure change

Introduction

Changes in steel microstructures upon heating and cooling can affect their macroscopic properties. The processes taking place during cooling of austenite affect the type, shape, size of the obtained phases in the heat treatment process. In addition, construction materials in the industry, working under risky conditions and prolonged overheating, deteriorate their properties due to degradation of their microstructure. As a result, the mechanical properties, strength, yield strength and modulus of elasticity and hardness change (Hertzberg, 1996; Okrajni, 2010; Dobrzański et al., 2007). Many studies have been devoted to the influence of microstructure on the mechanical properties of a metal (Zieliński et al., 2017; Ghorbanhossein et al., 2020; Bhardwaj et al.,2020).

The present work presents an investigation of the influence of quenching, tempering and ageing heat treatments on the microstructure of carbon steel samples, as well as changes in their vibrational (resonance frequencies and damping coefficient) and magnetic properties (the activity and magnitude of Barkhausen magnetic noises). Therefore, in the introduction, a brief review of the scientific results in the literature regarding the effect of the materials microstructure on its vibrational and magnetic properties is made.

In physical metallurgy, studies on damping properties provide a better knowledge of the microstructure, micro or macro defects, as well as phase changes that are induced by heat treatment. Luca et al. (2011) presented an investigation of the internal friction and resonance frequencies after quenching of medium carbon ferrite-pearlite steel. After quenching, a decrease in resonance frequencies and modulus of elasticity in longitudinal oscillations was recorded. Hamisi et al. (2018) investigated the effects of ageing on the mechanical and vibrational properties of SA516 carbide steels. The increasing the duration of heating caused degradation of the mechanical properties of the steel specimens. A decrease in their natural frequencies and an increase in damping properties were found. The effects of ageing heat treatment on the vibrational properties of a Mg-6Al-1Zn alloy were examined (Zhang et al., 1993; El-Morsy et al., 2015).

The results of an experimental and numerical study of the microstructural changes on the vibrational properties of CK35 steel was presented (Amirian et al., 2022), (Yazani et al., 2021). Experimental results indicated that the dispersion of spheroidized carbides in the ferritic matrix could increase natural frequencies and damping loss factors (Amirian, et al., 2022; Ahmadpar et al., 2021). Ageing heat treatment in the ferrite structure induces weak changes in the values of natural frequencies Yazani et al., 2021).

The magnetic noise method is applied in the study of the microstructure of ferromagnetic materials. It is based on the phenomenon that occurs when a ferromagnetic material is magnetized (Manh et al., 2020). The structure of a ferromagnetic material consists of magnetic domains separated by domain walls (Manh et al., 2020; Capo-Sanchez et al., 2004). In the absence of a magnetic field, the domains are randomly oriented. When a magnetic field is applied, the magnetic domains align in the direction of applied field by moving their domain walls (Saquet et al., 1999; Ivanova, 2022).

The time-varying magnetic field creates discontinuous magnetization changes in ferromagnetic samples, leading to the generation of magnetic Barkhausen noise (MBN) . These flux changes in the material induce a voltage in a receiver coil mounted near the Barkhausen events (Samimi et al., 2016). MBN technique had been applied for microstructure characterization such as grain boundaries, other phases, inclusions (Manh et al., 2020). The magnetic noise method is sensitive to carbon content (Capo-Sanchez et al., 2004; Samimi et al., 2016) , crystal size (Moorthy et al., 1998; Saquet et al., 1999) and different microstructures such as: ferrite, pearlite and martensite (Moorthy et al., 1998; Ivanova, 2022; Davut et al., 2009; Gur et al., 2007; Zerovnik et al., 2014).

From the overview presented above, it can be seen that the changes in metal structures may influence their mechanical, magnetic and vibration properties. Combining two or more physical research methods complements material knowledge, which is extremely important for equipment operating in risky conditions. This study is a continuation of experiments on medium carbon steel specimens after quenching and tempering (Ivanova, 2022), where using only one non-destructive method results are presented. The current work extends the study of the same type of steel subjected to ageing heat treatment at 700 degrees for 8 and 16 hours. The influence of microstructures changes after quenching, tempering and ageing on the vibration properties and magnetic properties is investigated.

This paper is organized as follows. After the introduction, which makes a brief overview of the scientific achievements on the sensitivity of the vibrational and magnetic noise methods for detecting the microstructural changes during heating, follows the section "Experimental procedure. Materials and methods". The subsection "Materials" describes the steel samples, as well as the preparation for conducting the heat treatments. The subsection "Methods" consists of two parts. The first part describes the basic concepts of vibration method through forced longitudinal vibrations. In the second part, the equipment for magnetic noise research is presented. The results of the research conducted by the two non-destructive methods are followed by a discussion and a conclusion that notes future directions in our research.

Experimental Procedure, Materials, Methods

Materials

The material used in this study is a ferrite-pearlite steel. The chemical composition of the steel was: C 0.34%, Si- 0.37%, Mn - 0.7%, P < 0.035%, Ni < 0.25%, Cr < 0.25%, S < 0.04%, Cu < 0.25%. Eighteen cylindrical steel samples with a diameter of 5.5 mm and a length of 100 mm were prepared and divided into six groups. Three groups were subjected to heat treatment, which consisted of quenching and subsequent tempering at the following temperatures: 150°C /group 2/, 250°C /group 3/, 400°C /group 4/.

The samples from groups 6 and 7 were subjected to ageing heating (spheroidization) at a temperature of 700 degrees Celsius for 8 and 16 hours (Alza, 2020; Harisha, 2018). The heat treatment modes are presented in column 2 of Table 1.

Table 1. Heat treatments of the investigated steel samples

Steel specimens	Heat treatment	Microstructure	Vickers hardness HV
Group 1	Non-heat-treated	Ferrite and pearlite	320
Group 2	Quenching T = 850°C Tempering T = 150°C	Tempered martensite	750
Group 3	Quenching T = 850°C Tempering T = 250°C	Tempered martensite	620
Group 4	Quenching T = 850°C Tempering T = 250°C	Tempered troostite	400
Group 5	Prolonged heat treatment T = 700°C Duration 8 hours	Ferrite and cementite	200
Group 6	Prolonged treatment T = 700°C Duration 16 hours	Ferrite and cementite	160

Methods

Vibration Method through Forced Longitudinal Vibrations

In the present work, experimental modal analysis by forced longitudinal vibrations is used to investigate the internal friction and dynamic characteristics of the samples. The standing waves are formed in the specimens as the result of longitudinal vibration of the sample. Generally, sound velocity in a specimen could be determined from Eq.1 (Chevalier, 2010).

$$\lambda = C/f \tag{1}$$

where C is sound velocity in a specimen, and f is resonance frequency of the first mode of vibration. The wave length λ can be calculated from Eq. 2 :

$$\lambda = \frac{2L}{n} \tag{2}$$

Where L is the length of specimen and n is the number of resonance mode. According to the positions of the node and the two antinodes corresponding to the first mode of vibration, the wave length was equal to the twice of the specimen length. Young's modulus can be obtained from the longitudinal mode vibration (Lord, 2007) . Knowing the values of Young's modulus (E) and geometric dimensions of the rod sample, we can determine the first fundamental frequency (f_1).

$$f_l = \sqrt{\frac{\pi n^2 r^2 E}{4LmK}} \tag{3}$$

Where m and r are the mass and the radius of rod, n is the mode of vibration (n = 1 for the fundamental). K is a correction factor depending on Poisson's ratio (v) and diameter-length ratio of the rod (Lord, 2007).

$$\frac{1}{K} = 1 - \frac{\pi^2 v^2 r^2}{2L^2} \tag{4}$$

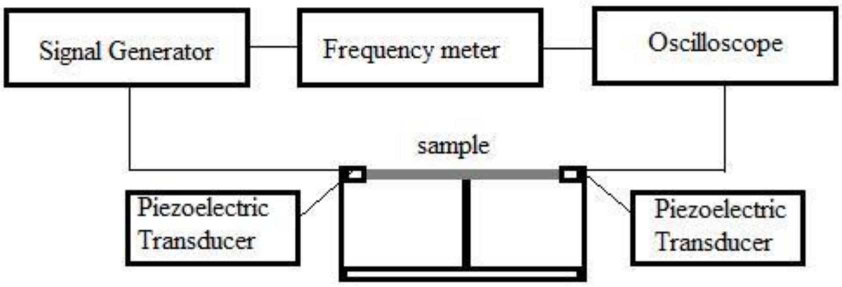


Figure 1. Block scheme of the setup for determining internal friction and damping ratio

when $2r/\lambda = nr/L >> 1$ (Lord, 2007). Figure 1 presents the setup that is applied to investigate the vibration properties of the prepared heat-treated steel samples by longitudinal forced vibration. The specimen was supported in the middle of its length between the excitation and receiving piezoelectric transducers. One transducer excited a sinusoidal vibration at one end of the specimen and the other receiving transducer recorded the response. The resonant frequency of the samples was detected with an oscilloscope and evaluated with a digital frequency meter.

The half-power bandwidth method was used to measure damping loss factor, damping ratio, internal friction and the logarithmic decrement (Hamisi et al., 2018; Yazani, 2021; Maringer, 1966).
The damping loss factor η can be defined as (Yazani, 2021):

$$\eta = \frac{W_2 - W_1}{W_r} \tag{5}$$

Where w_r is the frequency at which the peak of the curve is obtained and w_1 and w_2 are the two frequencies, for which the frequency response is $1/\sqrt{2}$ times the one at resonance. The damping ratio ξ is given by Yazani (2021) and Maringer (1966).

$$\xi = \frac{\eta}{2} \tag{6}$$

The internal friction (Q^{-1}) is obtained using bandwidth ($\Delta w = w_2 - w_1$) and is in the form of Eq. (7) (Hamisi et al., 2018; Yazani, 2021; Maringer, 1966).

$$Q^{-1} = \frac{W_2 - W_1}{W_r} \tag{7}$$

For small damping ($\xi \ll 1$), the logarithmic decrement δ is defined as (Maringer, 1966):

$$\delta = \pi Q^{-1} \tag{8}$$

Magnetic Noise Method Measurements

Acquisition of the raw MBN signal was performed by a MULTITEST-MC 04 device, given in detail in (Velev et al., 2009; Ivanova, 2022). The apparatus consists of a magnetizing module, a converter for recording magnetic noise signals and measuring the magnetic noise voltage, and a signal amplifier with several bandpass filters. A sinusoidal magnetization with a frequency of 76 Hz was applied. A cyclic sinusoidal field was induced in the sample at different magnetizing currents. Barkhausen signals were visualized and recorded using a VT DSO 2810 digital oscilloscope at a sampling rate of 100 MSPS and Multi-Instruments Pro 3.9 data acquisition software.

The software can extracted the RMS value of the Barkhausen noise (V_{RMS}) as well as the peak value (Vp). The MBN envelopes were reconstructed on based on the filtered MBN signals. The position of peak of MBN envelopes of was also analyzed. The time of appearance of MBN was calculated from envelope peak relative to the zero value of the magnetizing field. All MBN parameters were obtained by averaging sixteen consecutive pulses (eight hysteresis cycles).

Experimental Procedure

The prepared steel specimens were subjected to the heat treatments described in Table 1. After the heat treatment, the samples were cleaned of the oxide layer by surface treatment and specified in length and diameter.

Since the natural frequencies and damping properties are to be compared and analyzed, the samples should have the same dimensions and weights. The length (L=100 mm) and diameter (d=5 mm) of the samples were measured with an accuracy of 0.02 mm. After the preparation of the samples, the first resonance frequency of longitudinal oscillations was calculated. Two specimens from each group given in Table 1 are subjected to non-destructive evaluation by vibration analysis and Barkhausen magnetic noise testing.

Results and Discussion

Results of Performed Heat Treatments

The microstructure of investigated samples is shown in figure 2. The initial ferrite-pearlite structure before heat treatment is given in figure 2a. The martensitic structure obtained after quenching and low-temperature tempering T=150 ^{o}C can be observed in figure 2 b. Changes in the structure after these heat treatments can be explained by the decomposition of martensite and the beginning of the formation of iron carbides (Totten, 2006).

Martensitic structure after quenching and tempering at T=250 ^{o} C is given in figure 2c. The processes taking place during heating in the temperature range of 200-250 ^{o} C are mainly explained by the breakdown of residual austenite, as a result of which the internal stresses in the structure and hardness are reduced (Totten, 2006; (Ivanova, 2022).

The microstructure after tempering at T=350^{o} C is a finely dispersed ferrite-cementite mixture (tempered troostite) and is shown in figure 2 d. During tempering at and above 500 ^{o}C, the formed carbides coagulate and as a result a dispersed ferrite-cementite mixture is fixed, which is called tempered sorbite (figure 2e), (Ivanova, 2022). After applying a prolonged heat treatment at 700^{o}C, the pearlite regions are decomposed and the structure consists of ferrite and cementite, which are distributed in the form of irregular islands (Harisha, 2018). Hardness dropped significantly to 200-160 HV.

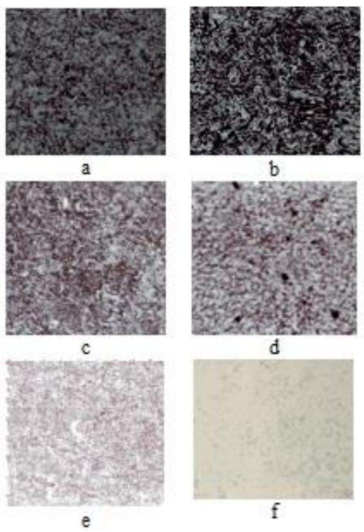


Figure 2. Microstructure of the tested samples: initial ferrite and pearlite microstructure before heat treatment (a), martensitic structure after quenching and low-temperature tempering –Group 2 (b), martensitic structure (quenching and tempering at T = 250°C)-Group 3 (c), tempered troostite – Group 4 (d), tempered sorbite - structure after quenching and tempering at T = 600°C (e); heat treated sample at 700 °C for 8 hours.

Results of the Vibration Tests

As mentioned before, two samples of each heat treatment were prepared for the vibration test. It should be noted that the samples are clamped midway between the emitting and receiving piezoelectric transducers. The emitter excites sinusoidal oscillations at one end of the sample. By changing the frequency of the sine wave through a sweep generator, the occurrence of system resonance was detected and recorded by an oscilloscope. The tests on each specimen were repeated three times and the results for the resonance frequencies were averaged.

Figure 3 shows the resulting resonance and the first resonant frequency obtained from a non-heat treated steel specimen. The vibration test results are summarized in Table 2, where are listed the values of the resonant frequencies and the widths of the resonant curves. Column 5 in Table 2 gives the relative change in the first resonance frequency compared to the initial state. Table 3 shows the experimental damping coefficients, calculated using Eqs. (6-8).

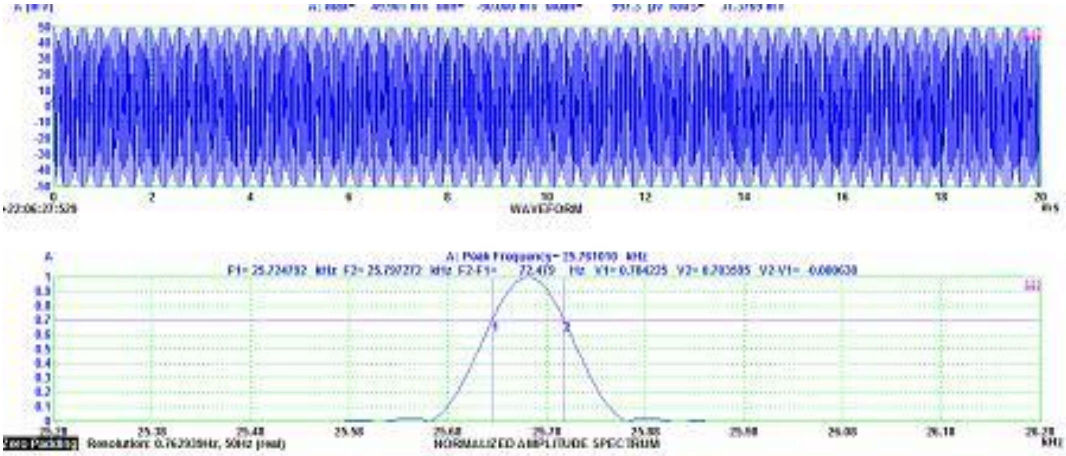


Figure 3. Results from vibration test for non-heated steel sample.

Table 2. First natural frequency of investigated steel samples

Group samples	f_r,kHz	f_1, kHz	f_2,kHz	Difference $\frac{f_{r1}-f_{ri}}{f_{r1}}$,%
1	25.761	25.724	25.797	0
2	25.437	25.400	25.474	-1.26
3	25.610	25.574	25.646	-0.59
4	25.791	25.754	25.827	0.12
5	25.810	25.764	25.837	0.19
6	25.853	25.817	25.890	0.36

According to the first natural frequency in Table 2, it is observed that after the quenching and tempering process at temperatures of 150 ^{o}C and 250 ^{o}C (groups 2 and 3), the natural frequencies are reduced compared to the untreated samples. The relative difference is the largest in group 2 and reaches 1.26%. This is probably caused by the state of internal stresses created during quenching and confirms the results in (Luca et al., 2011). Martensite is characterized by a high density of dislocations and high internal stresses. This also affects the internal friction. The logarithmic decrement increases on average by about 2.6% after quenching compared to the initial state.

Table 3. Damping coefficients of samples

Group samples	Damping ratio ξ (x10^{-3})	Internal friction Q^{-1}(x10^{-3})	Log. decrement δ(x10^{-3})
1	1.414	2.829	8.882
2	1.451	2.902	9.112
3	1.408	2.815	8.840
4	1.412	2.820	8.850
5	1.415	2.829	8.884
6	1.417	2.833	8.896

In the samples subjected to prolonged heating at 700^{o} C for 8 and 16 hours, a slight increase in natural frequencies was observed respectively 0.2% and 0.36%, compared to the initial state. According to (Yazani, 2021), this is due to the deposition of carbides during the heating of 8 h and 16 h duration. As a result of the heat treatment, the pearlite is destroyed and the structure of iron carbide layer is heterogeneously deposited on the surfaces and grows (Ahmadpar et al., 2021). The microstructure becomes softer and as a result the internal friction, the damping coefficient increases slightly (Yazani, 2021; Ahmadpar et al., 2021).

Results of MBN measurements

Figure 4 shows a part of recordings during magnetization and appearing magnetic noises at a magnetizing current of 200 mA. Depending on the various microstructures, different values of peaks (Vp) and shapes of MBN signals can be observed. The maximum amplitude and RMS values of the magnetic noises depend on the magnitude of the jumps of the magnetic domains obtained at different magnetizing currents (Ivanova, 2022).

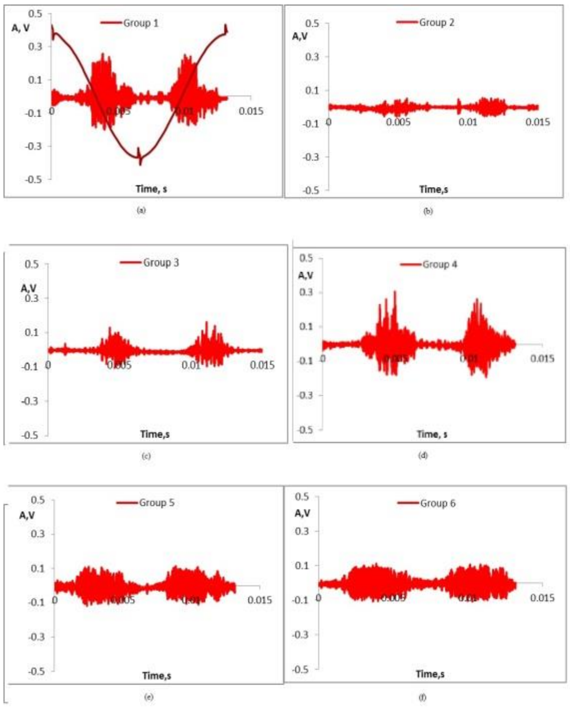
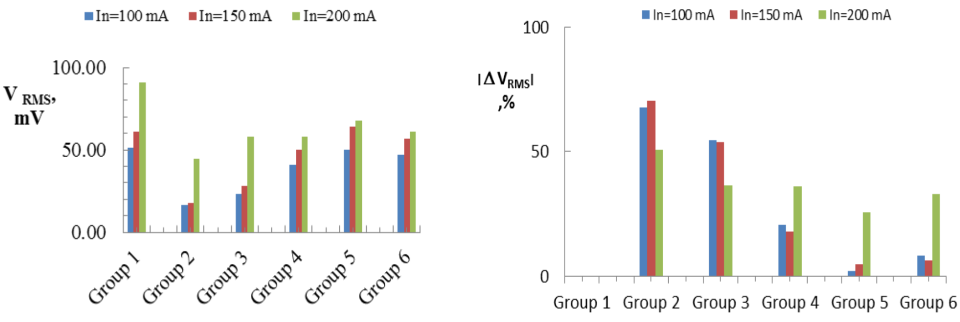


Figure 4. Magnetic noise signals at a magnetizing current of 200 mA: specimens group 1 (a), group 2 (b), group 3(c), group 4 (d), group 5 (e), group 6 (f)

Figure 5a presents the variation of RMS values of the magnetic noise on the magnetizing current *In* for the studied steel samples. The values are well distinguishable. As the magnetizing current increases, the RMS value increases. The samples subjected to quenching and low-temperature tempering, which have a structure of tempered martensite, show the lowest RMS values. In the remaining groups of samples (3, 4), an increase in the root mean square values is noticeable. This can be explained by the type of microstructures obtained: tempered martensite and troostite.

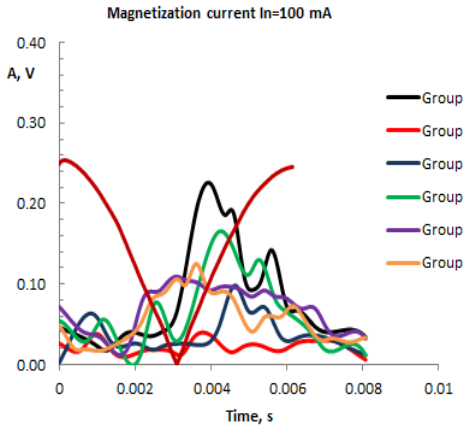
According to the research, the RMS values of the samples after prolonged heating are close to those of group 4. Figure 5 b shows the relative change of the studied parameter ($|\Delta V_{RMS}| = (V_{RMS,i} - V_{RMS,1})/V_{RMS,1}$, compared to the original untreated group 1. A very good sensitivity of the results was found at a magnetizing current of 200 mA. The relative variation in $|\Delta V_{RMS}|$ after quenching and low-temperature tempering is 50%, and after ageing treatments is from 25 to 33%.



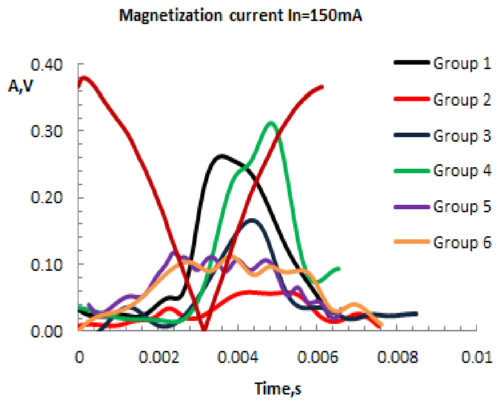
a) b)

Figure 5. Dependence of RMS parameter on the magnetizing current

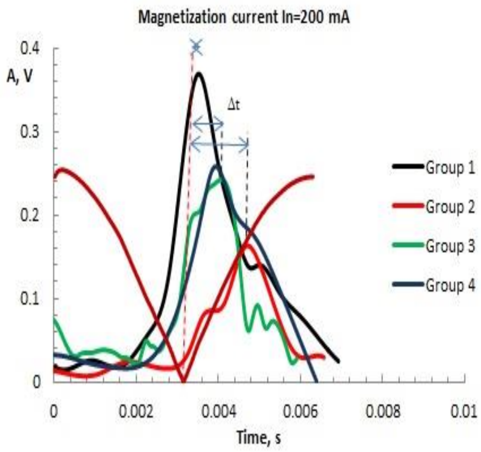
In the case of half a cycle of magnetization, the sample is demagnetized and then magnetized in the opposite direction. This activates the movement of the domain walls, which overcome the obstacles posed by the different microstructural phases. The nature of the MBN signals is a consequence of the nonuniform jumps of the domain walls during the remagnetization of the ferromagnetic material. This process depends on the microstructural features, different phases, boundaries between crystallites, the presence, shape and size of carbide inclusions, etc. (Davut et al., 2009; Gur et al., 2007; Zerovnik et al., 2014). Figure 6 plots MBN envelopes from studied samples at three values of the magnetizing current, respectively 100 mA (a), 150 mA (b) and 200 mA (c).



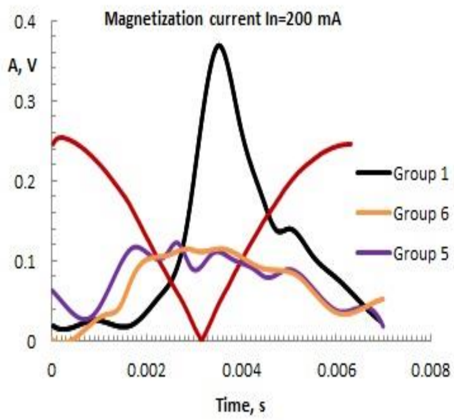
a)



b)



c)



d)

Figure 6. MBN envelopes for investigated steel samples: a) Magnetization current *In* = 100 mA; b) Magnetization current *In* = 150 mA; c) Magnetization current *In* = 200 mA; d) Results for groups 5 and 6 at *In* = 200 mA

At all three values of the magnetizing current for the samples of groups 2, 3 and 4, the following can be observed: i) different peak values; ii) shift of the MBN peak at zero value of the magnetizing field; iii) difference in MBN envelope shapes. In the tempered martensitic structures (group 2), the peak value is small and MBN envelope is wider. In the transition from tempered martensite with high hardness to a finer ferrite-pearlite structure such as troostite, an increase in the peak values, a sharpening of the MBN envelop is observed. The MBN envelopes for the samples subjected to ageing heating for 8 and 16 h are broadest and without pronounced peak values (Figure 6d).

Table 4. Parameters of MBN signals for different heat treated samples

Group samples	MaxMBN (Vp),V	Shift of MBN peak, Δt,s	Normalized MBN energy
Group 1	0.37	0.0003	1
Group 2	0.16	0.0015	0.20
Group 3	0.26	0.0007	0.58
Group 4	0.24	0.0008	0.48
Group 5	0.12	-	0.29
Group 6	0.12	-	0.27

To estimate the MBN in samples with different types of microstructure, the energy parameter can also be used. It is defined as the time integral of the MBN signal squared over the voltage. Table 4 gives parameters that characterize magnetic noise signals , which are: the peak height, the shift in the peak position of MBN envelope

($\Delta\ t$) and the MBN energy. The results for MBN energy are normalized to the energy of the unheated samples. The changes of MBN parameters when changing the heat treatment mode and microstructure are given in figures 7a, b and c.

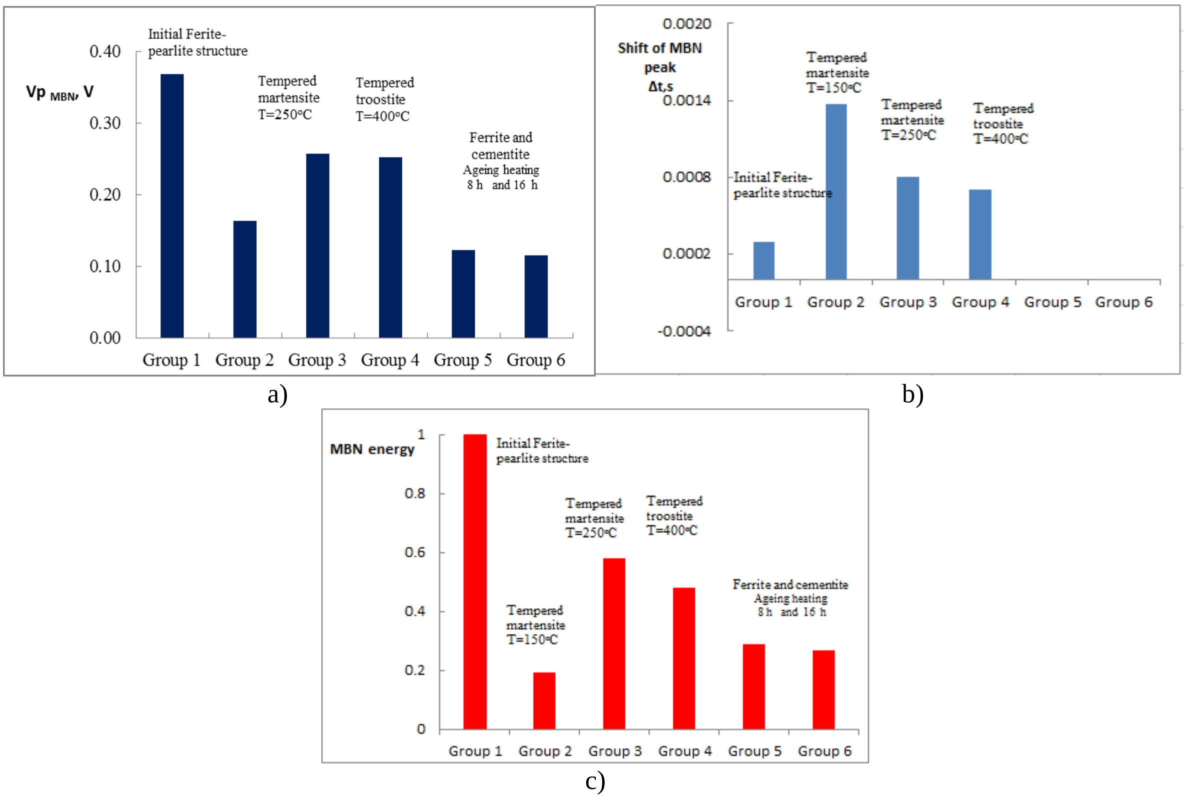


Figure 7. Parameters of MBN signals for different heat treated samples at magnetization current 200 mA:
a) MBN envelope peak height; b) Shift of MBN peak; c) MBN energy

According to the literatures (Ivanova, 2022), (Davut et al., 2009), (Zerovnik et al., 2014) when magnetizing martensite, very small domains are created due to the characteristics of martensite, which has high internal stresses. Therefore, for movement of the domains, a larger value of the magnetizing field is required. From figure 7 a it can be seen that the resulting peak value of the magnetic noise is smaller and is at relatively larger values of the magnetizing current. The energy of MBN signal is the smallest, as can be noted in figure 7c. As the martensitic microstructure changes to a finer ferrite-pearlite structure such as troostite, remagnetization becomes easier. The amplitude of the MBN signal increases and the energy decreases, as can be noted in figures 7a and 7c. In the figure 7b, one can see the differences in the time delays of the samples in different states. The time delay is measured from the highest point of the envelope to the sinusoid of the magnetizing current. For specimens with higher hardness, a longer time delay is obtained. After quenching and tempering, the time of occurrence of magnetic noise increases.

The peak values of the MBN envelopes of groups 5 and 6 heated at 700 degrees for 8 and 16 hours are the smallest and are approximately 0.1 V. The MBN envelopes are the widest and very different from the others for groups 1 to 4. The energy is almost 3 times less compared to the energy of the untreated group. These results are probably due to the degradation of the microstructure, as well as the decarburization of the surface layer of the samples as a result of the prolonged heat treatment.

Conclusion

As a result of the conducted research, the sensitivity of vibration and magnetic noise methods to the microstructural changes of medium carbon steel after various types of heat treatments was established. Some vibration and magnetic properties of steel samples after quenching and tempering were obtained. The microstructural changes after ageing treatment at 700 ^{o}C for 8 and 16 hours were also investigated. The vibration method is less sensitive to microstructure changes than the magnetic noise method. After quenching and tempering, the first resonance frequency was found to increase by about 1%, and the internal friction and logarithmic decrement by almost 2.6%. The prolonged heat treatment causes a slight increase in resonance frequency and damping properties.

In the magnetic noise method study, various parameters such as RMS and parameters of BMN envelopes were used to characterize the microstructure after quenching and tempering and ageing. The method is very sensitive to changes in the microstructure. The relative change in RMS value after quenching and low tempering is 50%. The method is also sensitive to structural changes after prolonged heat treatments. The change in RMS value is about 30%. A multiparameter approach of MBN signals was used for analysis. It was found that with the combination of parameters: MBN envelope peak height, peak shift and signal energy, the different microstructures can be quality characterized and recognized. The use of the two different physically based non-destructive methods complements the knowledge of the materials and allows an application of the methods according to their sensitivity.

Recommendations

The presented research can provide new knowledge to students and engineers to put into practice non-destructive evaluation of construction materials.

Scientific Ethics Declaration

The author declares that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the author.

Acknowledgements or Notes

* This article was presented as a poster presentation at the International Conference on Technology, Engineering and Science (www.icontes.net) held in Antalya/Turkey on November 16-19, 2023.

* This work has been accomplished with the financial support by the Grant No BG05M2OP001-1.002-0011-C02 financed by the Science and Education for Smart Growth Operational Program (2014-2020) and co-financed by the European Union through the European structural and Investment funds.

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To cite this article:

Ivanova, Y.P. (2023). Comparative non-destructive investigations of the effects of structural changes after heat treatment of Carbon Steel Samples by Magnetic Noise and Vibration Methods. *The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 26,* 306-316.