APPLICATION OF MECHANICAL BARKHAUSEN NOISE IN ASSESMENT OF X20 STEEL PROPERTIES AFTER HEAT TREATMENT

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X20CrMoV12.1 steel samples after various heat treatments (austenitizing 1050°C, for 1 h and tempering 720-780°C for 15-240 min) were examined. Magnetic hysteresis loops, mechanical hardness, magnetic coercivity, magnetic and mechanical Barkhausen noise measurements were made. It was found that mechanical Barkhausen noise intensity envelopes are more sensitive to the heat treatment conditions than other classical properties. It was revealed that tempering at a temperature of 780 °C for duration of more than 30 minutes is different in character than other examined heat treatment conditions. Fresh martensite may be formed in theses conditions. It is possible that the differences in magnetic and mechanical Barkhausen noise properties in the measured samples are due to higher sensitivity of non-180 degree magnetic domain walls to tempering.

Keywords: mechanical Barkhausen noise, tempering, martensitic steels, heat treatment, non-destructive testing, magnetic properties

1 INTRODUCTION

Mechanical Barkhausen noise (MeBN) is a mechanical analogue of magnetic Barkhausen noise (MBN) and is due to jumps of non 180 deg domain walls (DW) when sample is mechanically loaded. DW are pinned by dislocation tangles, precipitates and grain boundaries. MeBN intensity evaluated by means of RMS value of voltage induced in pick-up coil is proportional to the volume swept by DW during Barkhausen jump and to the rate of these jumps occurring at the certain stress level (which is the stress necessary for a jump to occur). A plot of MeBN intensity versus applied stress is thus a source of information about the population of pinning sites of certain pinning stress levels.

The MeBN has been applied to determine elastic limit of steel [1] and to monitor fatigue tests [2, 3]. It provides also information about internal stress distribution function, as was shown in [4]. This possible using plot of MeBN intensity obtained during the first load.

In this study we examine the effect of heat treatment of X20CrMoV12.1 steel (X20) on several magnetic properties such as magnetic flux density hysteresis loop, magnetic coercivity, MBN and MeBN intensity envelopes. Mechanical hardness was also measured. Standard heat treatment of this steel consists of austenitizing followed by high-temperature tempering. However the suggested heat treatment conditions allow for a range of tempering temperatures and durations.

2 EXPERIMENTAL

2.1 Samples

16 samples were cut from an X20-grade (X20CrMoV12.1 German standard, 20H12M1F Polish standard) steel tube of dimensions: 14 mm of wall thickness and 126 mm of inner diameter. Samples were machined into long bars having dimensions of (140±2)×(11±1)×(5±0.5) mm.

Various heat treatments were applied in order to determine the effects of tempering on magnetic properties of steel in question. Heat treatment procedure allows different tempering times and temperatures as an industrial standard ((730°C-780°C)[5] [6] 700°C [7], hence analogous conditions are examined.

All of the samples were austenitized at a temperature of 1050°C for duration of 60 minutes and then air quenched. One sample was left in the as quenched state (named as 0-0). The remaining 15 samples underwent tempering, each at a different temperature (720°C, 750°C and 780°C) and for a different duration (15, 30, 60, 120 and 240 min). The tempering was followed by air quenching. Heat treatment conditions for every sample are summarized in Table 1. The mill scale resulting from heat treatment was mechanically removed from the surface of every sample.

Tab. 1. Sample state names associated to the tempering time and temperature

	15 min	30 min	60 min	120 min	240 min
720°C	1-0	1-1	1-2	1-3	1-4
750°C	2-0	2-1	2-2	2-3	2-4
780°C	3-0	3-1	3-2	3-3	3-4

2.2 Experimental setups

2.2.1 COERCIVITY

The magnetic coercivity (HC) measurements were carried out using a magnetizing coil with a slow (less than 1 Hz) rate of change of current, pick-up coil wound over the sample and a Fe-Si yoke closing the magnetic circuit. The magnetizing current intensity and the voltage induced in the pick-up coil were registered using a fast DAQ board.

2.2.2 MAGNETIC BARKHAUSEN NOISE

The magnetic Barkhausen noise (MBN) was measured using the same apparatus for magnetisation as was used in magnetic hysteresis loop measurement with the addition of MBN signal analyzer, described in [8]. This analyzer

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provides average MBN signal from pick-up coil wounded directly on the sample around the sample.

2.2.3 MECHANICAL BARKHAUSEN NOISE
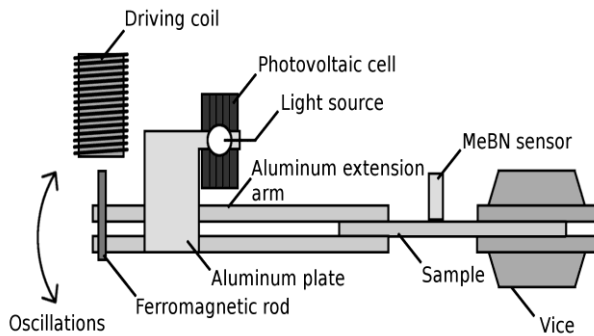
The MeBN experimental setup is shown in Fig. 1.

Fig. 1. Block diagram of the MeBN mechanical part set

Normal mode of oscillations (NMO) of the sample-extension arm system is induced by applying a magnetic pull on a ferromagnetic rod mounted at the end of the extension arm. The pulling impulse duration is less than 15 ms, which is shorter than the quarter of the period of NMO (the period was usually around 100 ms). This means that the pull exerted on the system is only accelerating the system. Driving coil is powered by current from a discharge from a 11 mF capacitor bank. Such driving system allows for a very high repeatability of amplitude of oscillations.

Strain level ε on the sample surface close to pick-up coil position is calculated from a voltage signal acquired from two photovoltaic cells working in a differential mode. This strain is proportional to the difference of voltage signals from the photovoltaic cells. This optical system of strain measurements was calibrated using a strain gauge glued onto the sample surface in position opposite to the MeBN sensor. Surface stress σ is calculated from the surface strain ε assuming Young's modulus of 220 GPa. This signal is further digitally filtered by a low-pass FFT filter. The MeBN sensor contains pick-up coil (with ferrite core) attached to the sample surface. The as induced MeBN voltage, after analog processing (high-pass filtering and amplification), is acquired by a fast (1 MHz) DAQ board.

2.3 Signal analysis in MeBN measurements

Results of sequence of digital signal analysis procedure of MeBN signal is shown in Fig. 2. The as acquired from DAQ signal is named U_0 (plot 1). It consists of series of voltage pulses. Firstly, this signal is filtered (bandpass 3kHz-130kHz) and a root mean square (RMS) is calculated (U_{RMS}, plot 2, not to scale). Environmental noise is subtracted RMS-wise using formula (1). This results in the final U_{MeBN} signal (plot 3).

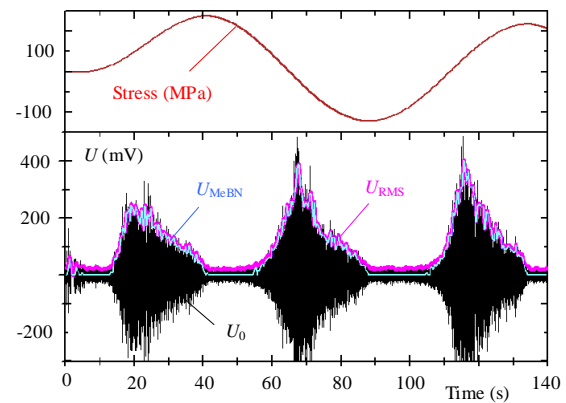$$U_{MeBN} = \sqrt{U_{RMS}^2 - U_{noise}^2} \qquad (1)$$

Fig. 2. Example of digital analysis of MeBN signal, U_0 - as measured MeBN signal, U_{RMS} - RMS of the filtered U_0 signal, U_{MeBN} - U_{RMS} signal after environmental noise was subtracted

As rate at which the stress is applied varies with time and the RMS time constant does not, the U_{MeBN} signal alone may be misleading due to the fact that the original signal consists of discrete pulses rather than being continuous. Thus a correction procedure should be used.

It can be assumed that the number N_T of MeBN events per cycle is constant and that the MeBN pulse heights within a given small number T_{RMS} of recorded data values are nearly constant and have a value of h. If it is assumed that pulses are evenly spaced it may be thus found that the RMS value of a discrete function $F(n)$ - representing recorded dataset, which consists of pulses amplitudes h and zeros, over T_{RMS} points is:

$$RMS(t) = \sqrt{\frac{\sum_{i=0}^{T_{RMS}} F(t+i)^2}{T_{RMS}}} = \sqrt{\frac{N_P h^2}{T_{RMS}}} = \sqrt{f}\sqrt{N_t h^2} \qquad (2)$$

where f is the frequency of oscillations, $N_P = f N_T T_{RMS}$ is the number of pulses in the T_{RMS} set of points. In our experiment RMS time constant is 0.1 ms and the oscillation period is of order of 100 ms. The frequency of oscillations used in (2) is the measure of the rate at which pulses appear. The stress rate (time-derivative of stress) is proportional to the rate at which MeBN pulses appear. This leads to the correction procedure implemented in RMS calculations (3).

$$U_{RMS}(t) = \frac{1}{\sqrt{\dfrac{\partial \sigma(t)}{\partial t}}} \sqrt{\frac{\sum_{i=0}^{T_{RMS}} U_0(t+i)^2}{T_{RMS}}} \qquad (3)$$

3 RESULTS

Magnetic coercivity as a function of tempering time and temperature is shown in Figure 3. The results shown are scaled to the coercivity of the non-tempered sample. Coercivity decreases monotonously as tempering time increases. For every tempering time HC is lower for samples tempered at lower temperatures. The exception from that are the samples tempered at 780°C: for these samples

when they were tempered for duration of more than 60 minutes coercivity increased.
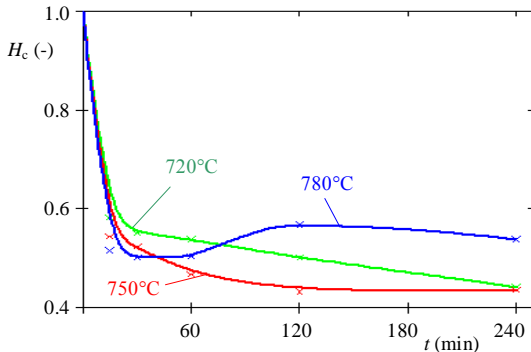

Fig. 3. Magnetic coercivity (HC) as a function of tempering time, results scaled to the coercivity of the non-tempered (0-0) sample
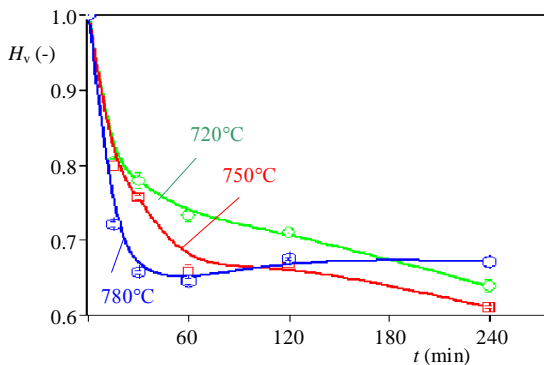

Fig. 4. Mechanical hardness (Vickers scale, HV) as a function of tempering time, results scaled to the hardness of the non-tempered (0-0) sample

Fig. 4 presents mechanical hardness (HV) as a function of tempering time and temperature. The results are scaled to the hardness of the non-tempered sample. One can find that HV exhibits a very similar behavior as HC. Again, samples tempered for more than 60 minutes at a temperature of 780°C are the exception, as their hardness increases with tempering time.
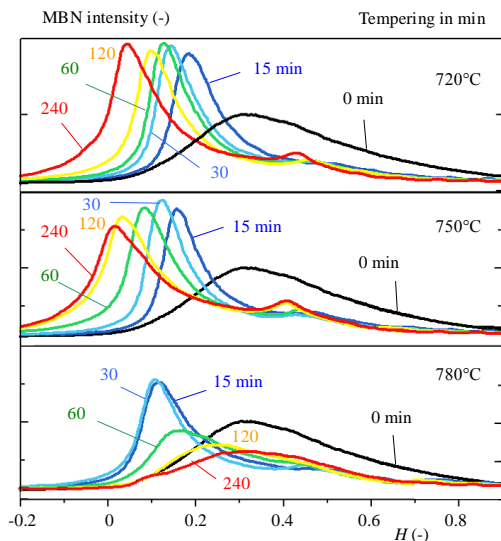

Fig. 5. Envelopes of MBN intensity (increasing field strength) for samples with different heat treatment applied, MBN intensity and magnetic field strength in arbitrary units

Figure 5 reveals the differences of MBN properties between samples tempered at 780°C and those tempered at 720°C and at 750°C. For lower temperatures, MBN intensity peaks have approximately the same height and their position shifts to lower magnetic field strengths for longer tempering time. It is due to lower coercivity of samples tempered for longer time. The second small MBN peak appears at high magnetic field strengths. It is visible in the envelopes of MBN intensity for samples: 1-3, 1-4, 2-2, 2-3, 2-4. It appears also in a sample 3-1. Samples tempered at a temperature of 780°C for a duration of 60 minutes (and more) have their MBN intensity peaks lower in amplitude and more shifted towards higher magnetic field strengths. Sample 3-4 has a similar envelope of MBN intensity to that of sample 0-0 (non-tempered).

Differences between MeBN properties between samples tempered at 780°C and those at lower temperatures are evident in Fig. 6. Envelopes of MeBN intensity are shown for both the first positive (tensile) and negative (compressive) stress loading. One can find that the peak position for samples not tempered enough (*ie* 1-0, 2-0) is out of the range of the as applied. With increasing tempering time peak position shifts to lower absolute stress values and peak height increases. Samples tempered at a temperature of 780°C have peaks in the examined stress range until a tempering time of 60 minutes is reached. Samples 3-0 and 3-1 show similarities in the shape of envelope and peak size and position. Samples: 3-2, 3-3 and 3-4, are also similar to each other. Sample 3-4 has the lowest amplitude of MeBN intensity envelope of all tempered samples examined. No MeBN signal was observed from the non-tempered sample.
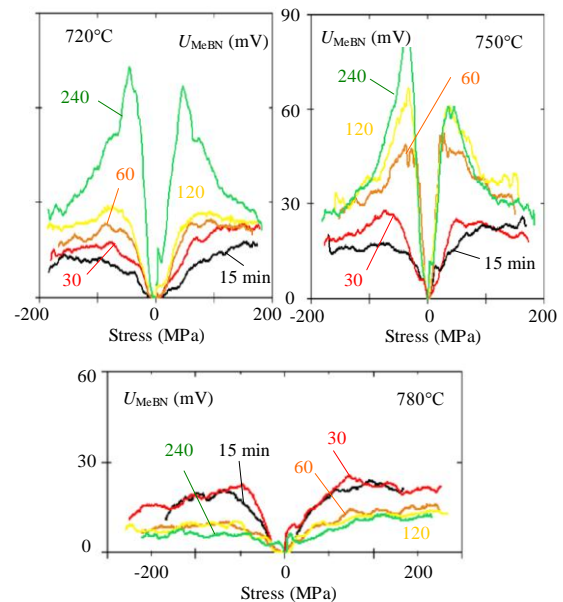

Fig. 6. Envelopes of MeBN intensity, with stress-rate correction applied, as obtained for the first stress loading after demagnetization, for all the heat treatments applied

Also an asymmetry in MeBN intensity envelopes obtained from negative and positive stress loading is observed. It is important to stress that the sample 2-4, with

the biggest asymmetry observed, has had MeBN measured in different probe-sample position configurations and that this effect is consistent within all of the examined configurations. It is thus not an effect of damaged (*ie* plastically deformed) sample surface.

One can find that mechanical Barkhausen noise intensity envelopes are the most sensitive parameter to the heat treatment applied from all the examined properties, such as: magnetic flux density hysteresis loops, magnetic coercivity, mechanical hardness or magnetic Barkhausen noise intensity envelopes. MeBN exhibit peak size and position changes while those of MBN do not vary significantly in size and only their position changes. As MeBN is due to non-180 degree DW activity and MBN to due to all DW it is possible to state that non-180 degree DW are more affected by the heat treatment than 180 degree walls.

A second peak appears in MBN intensity envelopes for more tempered samples (1-3, 1-4, 2-2, 2-3, 2-4) and is more distinct with tempering time. It is possible that this is attributed to the growth of a new phase in material, magnetically different (higher coercivity) than the rest of material.

The shape of hysteresis loops of magnetic flux density and MBN intensity envelopes for samples tempered at a temperature of 780°C becomes similar to the shape of non-tempered sample. At first coercivity and mechanical hardness decrease and MBN peak position shifts towards lower magnetic field strengths. With tempering times of more than 30 minutes the process reverses. Also, MBN intensity amplitude decreases. As for MeBN, for samples 3-0, 3-1, the shape is very alike and it is also found that the shape of MeBN intensity envelope of samples 3-2, 3-3, 3-4 is similar. The latter are lower in amplitude and have their peaks at higher stress levels. Non-tempered sample does not emit MeBN (no amplitude). As it was stated in [5], in higher tempering temperatures, a fresh martensite may be produced which may be the cause of such behavior.

Another interesting feature is the similarity of hysteresis loops for samples 3-0 and 3-2, with only coercivity slightly higher for 3-0. HV, MBN and MeBN are very different for these two samples. It may be explained knowing that the response from all of the material volume affects magnetic hysteresis loops and the other properties are surface related. The process that induces these differences might affect only surface of the material at first, with most of the volume unaffected.

It is known that the density of dislocations decreases about 10 times after 1 h of tempering at a temperature of 750°C [9]. As dislocation tangles are pinning sites for DW and they contribute to coercivity and mechanical hardness, their decrease affects all these parameters. MeBN intensity envelopes peak shifts towards lower stress levels (lower pinning forces) and its amplitude increases (more pinning sites of lower stress level). MBN intensity envelopes peak shifts towards lower magnetic field strengths (again lower pinning forces).

4 CONCLUSIONS

1. Our studies have found that tempering at a temperature of 780°C for a duration of more than 30 minutes differs significantly in character (whether magnetic properties or mechanical hardness is in question).

2. MeBN properties are more sensitive to the heat treatment applied than MBN properties are. As MBN signal originates from the activity of 180 and non-180 degree DW it is a blend of these two. The MeBN changes with heat treatment more in character than the MBN signal does. It makes evident that the non-180 degree DW are 'more' sensitive to the microstructure changes due tempering. These changes mean also modification of local stress barriers because non-180 degree DW position and mobility is stress dependent. It is not a case of 180 deg DW.

3. MeBN peak shifts in function of tempering parameters. This should be attributed to the change of the most common DW pinning sites stress level. Higher stress level shifts MeBN peak toward higher level of external stress and vice versa.

4. The as described results reveal that MeBN measurement can be treated as complementary to traditional like magnetic measurements, allowing direct insight to residual stress level distribution.

REFERENCES

[1] KHARITONOV, Y.N.: Determination of the elastic limit in ferromagnetic materials using the mechanical Barkhausen effect, Russian Physics Journal 10(12) (1967), 71-73

[2] RUUSKANEN, P. — KETTUNEN, P.: Effect of cyclic straining of the harmonic amplitude spectrum of magnetoelastically generated voltage u_B, Mater Sci Eng A 1991,142(1), 125–33

[3] SOULTAN, M. — KLEBER, X. — CHICOIS, J. — VINCENT, A.: Mechanical Barkhausen noise during fatigue of iron, NDT and E Int. 39 (2006), 493-498

[4] AUGUSTYNIAK, B. — DEGAUQUE, J.: Microstructure inspection by means of mechanical Barkhausen effect analysis, Journal de Physique 6 (8) (1996), 527-530

[5] BOJINOV, V. — HALD, J — LANGER, E.W.: Microstructural Investigations in Steel X20 CrMoV 12 1 After Nonstandard Heat Treatment. II. Varied Tempering Temperature, Scand. J. Metall. 18(5) (1989), 221-225

[6] SKOBIR, D.A. — GODEC, M. — JENKO, M. — MARKOLI, B.: Characterization of the carbides in the steel X20CrMoV12.1 used in thermal power plants, Surf. Interface Anal. 40(3-4) (2008), 513-517

[7] THOMSON, R.C. — BHADESHIA, H.K.D.H.: Carbide precipitation in 12Cr1MoV power plant steel, Metall. Mater. Trans. A 23(4) (1992), 1171-1179

[8] SABLIK, M.J. — AUGUSTYNIAK, B.: The effect of mechanical stress on a Barkhausen noise signal integrated across a cycle ramped magnetic field, J. Appl. Phys. 79 (2) (1996), 963-972

[9] PESICKA, J. — KUZEL, R. — DRONHOFER, A. — EGGELER, G.: The evolution of dislocation density during heat treatment and creep of tempered martensite ferritic steels, Acta. Mater. 51 (16) (2003), 4847-4862

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