52, 1, pp. 181-188, Warsaw 2014

DETERMINATION OF MECHANICAL PROPERTIES OF P91 STEEL BY MEANS OF MAGNETIC BARKHAUSEN EMISSION

Katarzyna Makowska

Motor Transport Institute, Warsaw, Poland, e-mail: katarzyna.makowska@its.waw.pl

ZBIGNIEW L. KOWALEWSKI

Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland and Motor Transport Institute, Warsaw, Poland, email: zkowalew@ippt.pan.pl; zbigniew.kowalewski@its.waw.pl

Bolesław Augustyniak, Leszek Piotrowski

Gdansk University of Technology, Gdańsk, Poland e-mail: bolek@mif.pg.gda.pl; lesio@mif.pg.gda.pl

In this work, an attempt at determination of mechanical properties by means of a method based on magnetic Barkhausen emission measurements was proposed. The specimens made of P91 steel were subjected to creep or plastic flow which were interrupted after a range of selected time periods in order to achieve specimens with an increasing level of strain. Subsequently, measurements of magnetic Barkhausen emission were carried out, and then static tensile tests were performed in order to check variations of basic mechanical parameters. It is shown that evident relationships between the yield point/ultimate tensile strength and some parameters of the Barkhausen emission exist.

Key words: mechanical properties, magnetic Barkhausen effect, plastic strain

1. Introduction

The method based on measurements of the magnetic Barkhausen emission/noise (MBE) might be applied to diagnostics of parts made of ferromagnetic materials. The MBE is a result of the irreversible movement of magnetic domain walls during a magnetisation cycle (Blaow et al., 2007). The domain walls are pinned by microstructural barriers (like grain boundaries, precipitation, dislocation tangles) and released abruptly in the changing magnetic field (Jiles, 2000). The Barkhausen jumps of the domain walls are detected as voltage pulses induced in a magnetic reading head or in a searching coil.

Due to sensitivity of the MBE to the microstructure of the material, this technique is commonly applied to provide information about different material properties, i.e. material structure (Saquet et al., 1999; Kleber et al., 2008), grain size (Yamuara et al., 2001), texture (Augustyniak, 2003), as well as mechanical properties, i.e. hardness (Gorkunov et al., 2000), residual stress (Vahista and Paul, 2009) or ultimate tensile stress (Kleber et al., 2008). For example, Kleber et al. (2008) achieved a linear relationship between the volume fraction of ferrite and amplitude of MBE peak in a lower magnetic field intensity for a steel with carbon content of $343 \times 10^{-3}\%$ C, whereas Yamuara et al. (2001) found a linear dependency between rms of the magnetic Barkhausen emission and grain size for pure iron.

Moreover, microstructural degradation during creep (Mohopatra et~al., 2008) or plastic deformation (O'Sullivan et~al., 2004) of the material may be detected by means of the MBE. Intensity of the MBE is very sensitive to material damage development, particularly at the early stage of degradation (Mohapatra et~al., 2010). Mohopatra et~al. (2008) examined 5Cr-0.5Mo steel subjected to creep at $600^{\circ}\text{C}/60\,\text{MPa}$ by means of the MBE method. The same test was also

performed on specimens cut out from a tube made also from 5Cr-0.5Mo steel. The rms voltage of the magnetic Barkhausen signal for the virgin specimens decreased at the initial stage of the creep life due to the newly formed carbides. For the specimen cut from the tube exploited by 15 years the growth of carbides had already taken place, and the rms voltage of MBE increased even during the initial stage of laboratory creep testing. As fast as the void started to form in the specimens (both virgin and service exposed), the rate of the increase of the rms voltage of the Barkhausen signal started to decrease.

The MBE might be also used to estimate mechanical properties. Kleber *et al.* (2008) achieved a linear relationship between the ultimate tensile stress of five commercial DP steels (DP450, DP500, DP600, DP780, DP1000) and MBE amplitude as well as MBE peak position for example. Trillon *et al.* (2012) found linear relationships between the hardness and MBE peak position of medium-carbon specimens earlier submitted to a standardized Jominy test.

The aim of this work is to evaluate relationships between selected mechanical parameters (yield point, ultimate tensile strength) of P91 steel and parameters coming from the non-destructive magnetic method (amplitude and integral of the MBE). The thesis of this work is that the basic mechanical properties as the yield point or ultimate tensile stress could be estimated on the basis magnetic parameters only, without conducting of a static tensile test. It would be possible in the case when functional relationships between parameters coming from non-destructive and destructive methods would be found. The relationships between parameters coming from considered magnetic and mechanical tests could be varied for different types of steel in dependence on, among others, carbon content, grain size and microstructure of the steel. Non-destructive measurements may replace conventional mechanical tests in special cases, for example when the cutting out specimens from an installation or other devices is impossible. Determination of mechanical properties using magnetic parameters can be very helpful in estimation of the stage of material degradation. This issue is especially important for the power industry applications since it may lead to reduction of maintenance inspections.

2. Experimental procedure

P91 low-carbon steel, typically used in power industry for structural components such as plates and tubes, was tested. Its chemical composition is shown in Table 1.

Table 1.	Chemical	composition	of P91	steel
----------	----------	-------------	--------	-------

С	Si	Mn	Р	S	Cr	Мо
0.085	0.27	0.30	0.015	< 0.01	8.2	0.86
Ni	Al	Cu	Ti	Nb	V	Fe
0.16	0.010	0.14	< 0.01	0.098	0.19	rest

The microstructure of the material in the as-received state is presented in Fig. 1.

P91 steel consists of a tempered martensite phase (Fig. 1). According to Panait *et al.* (2009) the microstructure of P91 steel is stabilized by $M_{23}C_6$ carbides (where M = Cr, Fe, Mo) and MX carbonitrides (where M = V, Nb and X = C, N).

A creep test $(T=500^{\circ}\text{C}, \sigma=290\,\text{MPa})$ and a tensile test $(T=25^{\circ}\text{C}, V=1\,\text{mm/min})$ were performed on plain specimens having a rectangular cross section of $5\,\text{mm} \times 7\,\text{mm}$ and gauge length of $40\,\text{mm}$. The creep process was interrupted to obtain different deformation levels i.e.: 0.85%, 3.15%, 4.60%, 5.90%, 7.90% and 9.30%. In the case of the tensile test, the deformation levels, were as follows: 2.00%, 3.00%, 4.50%, 5.50%, 7.50%, 9.00% and 10.50%.

The magnetic Barkhausen emission signal was measured using the measuring set whoose diagram is presented in Fig. 2.

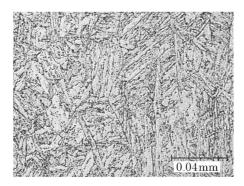


Fig. 1. Microstructure of P91 steel, etched state (2%HNO $_3$ + etlhyl alcohol), light microscope, magnification $500\times$

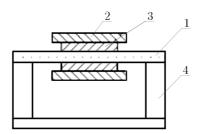


Fig. 2. The measuring set for magnetic properties and Barkhausen emission investigations (1-specimen, 2-solenoid, 3-pick-up coil, 4-core)

Specimen (1) was magnetized by solenoid (2). Core (4) was used to close magnetic flux. Pick-up coil (3) provided the induced voltage signal U_0 . This voltage contains two components: the low frequency component (related to the main change of the magnetic flux) and a high frequency component due to the Barkhausen effect. It should be stressed that the second one is some orders smaller than the first one. The voltage signal U_0 was integrated in order to obtain the hysteresis loop B(H). In order to evaluate intensity of the MBE, the second component of U_0 was separated by means of a high-pass filter. The MBE intensity envelopes of this voltage were calculated as the rms value U_b according to the equation (Augustyniak, 2003)

$$U_b = \sqrt{\frac{1}{\tau} \int_0^{\tau} U_{tb1}^2(t) dt}$$
 (2.1)

where U_b is the root mean square of the coil output voltage, U_{tb1} – fast-variable component defining the voltage separated by means of the high-pass filter from induced voltage in the pick-up coil, τ – integration time.

Next, the parameter Ub_{pp} – evaluated as the difference between the maximum Ub_{max} and the lowest value Ub_{min} of U_b was calculated using the relation

$$Ub_{pp} = Ub_{max} - Ub_{min} (2.2)$$

The second descriptor of MBE – the integral over a half-period of the voltage U_{sb} (signal U_b corrected on the noise level) was also calculated using formula

$$Int(Ub) = \int_{-U_{g\,max}}^{+U_{g\,max}} U_{sb} dU_g$$

$$(2.3)$$

where

$$U_{sb} = \sqrt{U_b^2 - U_{tb}^2} (2.4)$$

The parameter U_{tb} is a root mean square of the background voltage. U_g is a voltage proportional to the magnetizing current intensity. The magnetic parameters were normalized in respect to their values for the non-deformed specimen $(Ub_{pp\,norm}, Int(Ub)_{norm})$.

After magnetic measurements, static tensile tests at room temperature were performed in order to evaluate the yield point and ultimate tensile strength. Determination of relationships between the parameters obtained from mechanical (destructive) and magnetic (non-destructive) tests was the last point of the experimental programme.

3. Results

Figure 3 presents the MBE envelopes obtained for P91 steel at three extreme stages: (a) non-deformed, (b) after plastic deformation by a tensile test and (c) after a creep test (with the highest deformation levels for each type of damage). These plots reveal variation of MBE intensity for one period of magnetization with an evident hysteresis. The arrows depict the 'direction' of magnetic field change.

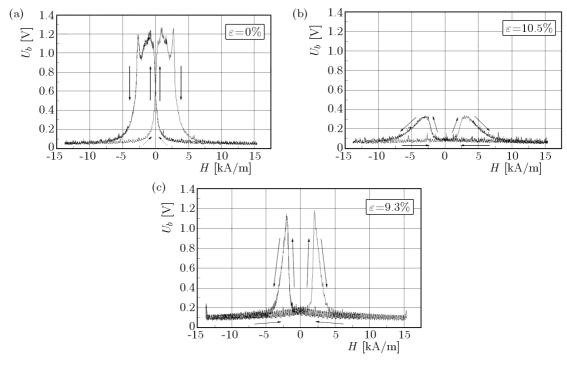
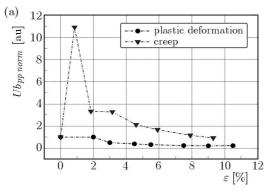


Fig. 3. The rms envelopes of the magnetic Barkhausen emission for P91 steel: (a) non-deformed, (b) after plastic deformation up to 10.5%, (c) after creep up to 9.3%

In the case of the non-deformed specimen, the MBE intensity plot reveals broad maximum with two peaks which can be attributed to 'soft' and 'hard' magnetic phases. In P91 steel, these phases can be attributed to micrograins with low and high density of dislocations (Pesicka et al., 2003). In comparison with the non-deformed specimen, the Barkhausen rms envelopes of the strained specimens (Figs. 3b-c) have different shapes. The two-peak broad maximum observed in the non-deformed specimen transforms to a single maximum for specimens strained up to 10.5% in the tensile test and up to 9.3% in the creep test. The shape (height and width) of that maximum varies for all the deformed samples.

Figures 4a,b present how two magnetic parameters calculated from these envelopes $(Ub_{pp\,norm}$ and $Int(Ub)_{norm})$ depend on the deformation level.



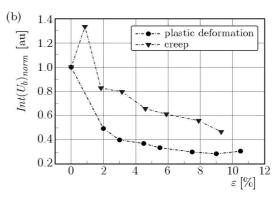


Fig. 4. Amplitude of the magnetic Barkhausen emission versus pre-strain for P91 steel (a); integral of a half-period voltage signal of the magnetic Barkhausen emission versus pre-strain for P91 steel (b)

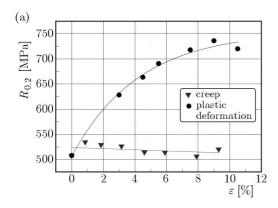
The amplitude (Ub_{pp}) of the Barkhausen emission of specimens after the creep process seems to be more sensitive to the strain level than of those after plastic deformation achieved (Fig. 4a). In the case of the creep test, the abrupt peak of the parameter Ub_{pp} is observed for the deformation level of about 1%, and – subsequently, a decrease down to nearly the initial level appears. For specimens deformed by means of plastic deformation, only a slight and monotonic decrease of this parameter is observed. However, the lack of data around the deformation level of 1% does not allow one to conclude explicitly whether a maximum of Ub_{pp} in this case also occurs or not.

A decrease of the magnetic Barkhausen emission integrals Int(Ub) for plastic deformation can be observed in Fig. 4b. However, its values for specimens after creep are systematically higher in comparison with those after plastic deformation. Additionally, the plot for the creep test demonstrates also a single maximum at low values of deformation.

For both types of damage tests, the variations of the MBE amplitude (Ub_{pp}) and integral, (Int(Ub)) depend on changes occurring in the microstructure. We argue that performed plastic deformation of P91 steel leads to an increase of dislocation density. A relatively high level of plastic deformation (above 2%) is probably associated with formation of extensive dislocation tangles which reduces the mean free path of domain wall displacement as well as increases the domain wall pining force (Baldev et al., 2001). Movement of the domain walls becomes less effective, and the MBE signal intensity decreases monotonically (O'Sullivan et al., 2004).

In the case of creep experiment, the observed initial increase of MBE intensity is probably related to the abrupt decrease of dislocation density at martensite subgrains (tempering process). Material recovery by dislocations cross slips also occurs. The applied stress of a high level (290 MPa) leads also, at the same time, to poligonization of the material by climb of dislocations. The second process is the hardening of the steel by introducing high density dislocation tangles to the material. The significant amount of dislocation tangles impedes much more movement of the magnetic domain walls, and the amount of Barkhausen jumps decreases as it was in the case of plastic flow at room temperature. It should be thus mentioned that the performed creep experiment is a mixed process consisting of 'creep' and 'plastic deformation' due to a high stress level.

These differences in P91 microstructure after plastic flow and creep experiments should be revealed by mechanical tests. The results of static tensile tests of pre-strained specimens are presented in Fig. 5a (yield point) and in Fig. 5b (ultimate tensile strength). The ultimate tensile strength of P91 steel subjected to creep decreases very much whereas the yield point of this material is insensitive to the deformation level. In the case of plastic flow, a prior deformation leads to the hardening effect: both mechanical parameters increase.



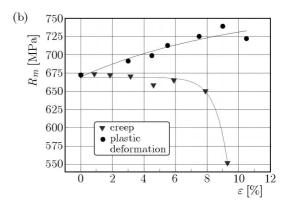
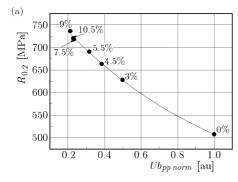


Fig. 5. Variation of yield point (a) and ultimate tensile strength (b) of P91 steel

It should be noticed that, generally, higher values of magnetic parameters are related to lower $R_{0.2}$ and R_m (creep process) and conversely – lower values of the magnetic parameters are related to higher values of $R_{0.2}$ and R_m (plastic deformation).

In the next step of the experimental programme possible, relations between the mechanical and magnetic parameters are evaluated. Since the yield point of P91 steel subjected to creep was insensitive to the deformation level induced by this process, it is not considered in the further analysis.

Figures 6 and 7 show relationships between two magnetic parameters of MBE (Ub_{pp}) and $Int(U_b)$ and two mechanical parameters (yield point and ultimate stress). The magnetic parameters are normalized to their values for the non-deformed specimen. The numbers in figures denote the level of prior deformation. Figures 6a,b allow one to conclude that both parameters (amplitude Ub_{pp} and integral $Int(U_b)$) of the Barkhausen noise may be used to estimate the level of the yield point of plastically deformed specimens.



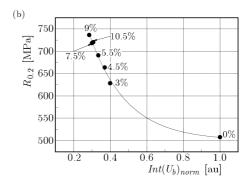
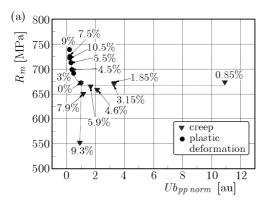


Fig. 6. Variation of the yield point of P91 steel versus amplitude of the magnetic Barkhausen emission $Ub_{pp\,norm}$ (a) and versus integral over a half-period voltage signal of the magnetic Barkhausen emission $Int(Ub)_{norm}$ (b)

Also, the ultimate tensile strength of P91 steel subjected to prior plastic flow may be determined using the relationship between R_m and $Ub_{pp\,norm}$ or $Int(Ub)_{norm}$ (Figs. 7a,b) but only for the flow experiment. It has to be emphasised that a non unique relationship between R_m and $Ub_{pp\,norm}$ and $Int(Ub)_{norm}$ was found for the steel pre-strained by creep.

The relations in Figs. 6 and 7 may be explained as follows. Steel after plastic deformation with higher $R_{0.2}$ and R_m is characterised by lower values of the magnetic parameters because it contains more dislocation tangles that impede movement of the domain walls. On the other hand, higher values of the magnetic parameters are related to lower R_m of steel after creep.

Our results make evident that MBE intensity varies very much due to microstructure modification but in different ways: its intensity decreases monotonically after plastic flow (for de-



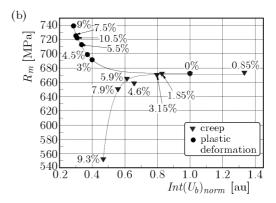


Fig. 7. Variation of the ultimate tensile strength of P91 steel versus amplitude of the magnetic Barkhausen emission $Ub_{pp\;norm}$ (a) and versus integral over a half-period voltage signal of the magnetic Barkhausen emission (b)

formation higher than 2%) and increases (several times) and then decreases during creep. Such a non-monotonic function makes the direct estimation of the mechanical state impossible when only one magnetic parameter is used for such estimation. Addressing the issue of practical application of MBE measurement for the assessment of mechanical properties of damaged steel, we argue that it is possible but analysing at least two magnetic parameters with respect to their initial values. One can find from Figs. 7a,b that a relative decrease of Ub_{pp} and $Int(U_b)$ denotes plastic flow, while important increase of $Int(U_b)$ is specific for the early stage of creep damage. The most difficult case – when advanced creep is in question – can be detected knowing that the level of Ub_{pp} is higher or close to its initial value, while the level of $Int(U_b)$ decreases. It should be stressed that such analysis can be enhanced by watching the variation of MBE peak shape.

4. Conclusions

The magnetic Barkhausen effect can be helpful for the assessment of selected basic mechanical parameters like the yield point or ultimate tensile strength for example.

It is shown that the yield point of P91 steel subjected to plastic flow might be determined by means of the amplitude/integral calculated from the rms magnetic Barkhausen emission envelopes. Since the yield point of P91 steel was insensitive to the creep pre-strain deformations, such relationships cannot be formulated for such a kind of damage. The yield point as well as the ultimate tensile strength of the tested steel can be determined on the basis of the Barkhausen noise properties using simultaneously two magnetic parameters Ub_{pp} and $Int(U_b)$.

Acknowledgments

The research was performed within the financial support of the Motor Transport Institute (No. 6019/CBM) and the Ministry of Science and Higher Education of Poland (grant No. R15004904).

References

- 1. Augustyniak B., 2003, Magnetoelactic Effects and their Application in the Non-Destructive Testing of Materials (in Polish), Wydawnictwo Politechniki Gdańskiej, Gdańsk, Poland
- 2. Baldev R., Jayakumar T., Moorthy V., Vaidyanatham S., 2001, Characterisation of microstructures, deformation and fatigue damage in different steels using magnetic Barkhausen emission technique, *Russian Journal of Nondestructive Testing*, 37, 11, 789-798

- 3. Blaow M., Evans J. T., Shaw B. A., 2007, The effect of microstructure and applied stress on magnetic Barkhausen emission in induction hardened steel, *Journal of Materials Science*, **42**, 4364-4371
- 4. Gorkunov E.S., Dragoshanskii Yu.N., Mikhovski M., 2000, Barkhausen noise and its utilization in structural analysis of ferromagnetic materials (Review article V), Russian Journal of Nondestructive Testing, 36, 6, 389-417
- 5. JILES D.C., 2000, Dynamics of domain magnetization and the Barkhausen effect, *Czechoslovak Journal of Physics*, **50**, 893-988
- 6. Kleber X., Hug-Amalric A., Merlin J., 2008, Evaluation of proportion of phases and mechanical strength of two-phase steels using Barkhausen noise measurements: application to commercial dual-phase steel, *Metallurgical and Materials Transactions A*, 1308-volume 39A
- 7. Mohapatra J.N., Bandyopadhyay N.R., Gunjan M.K., Mitra A., 2010, Study of high-temperature ageing and creep on bainitic 5Cr-0.5Mo steel by magnetic NDE techniques, *Journal of Magnetism and Magnetic Materials*, **322**, 589-595
- 8. Mohopatra J.N., Ray A.K., Swaminathan J., Mitra A., 2008, Creep behaviour study of virgin and service exposed 5Cr-0.5Mo steel using magnetic Barkhausen emissions technique, *Journal of Magnetism and Magnetic Materials*, **320**, 2284-2290
- 9. O'Sullivan D., Cotterell M., Cassidy S., Tanner D.A., Mészáros O., 2004, Magneto-acoustic emission for the characterisation of ferritic stainless steel microstructural state, *Journal of Magnetism and Magnetic Materials*, **271**, 381-389
- 10. Panait C., Bendick W., Fuchsmann A., Gourgues-Lorenzon A.-F., Besson J., 2009, Study of the microstructure of the Grade 91 steel after more than 100'000h of creep exposure at 600°, 2nd ECCC Creep Conference, Zurich, Switzerland
- 11. Pesicka J., Kuzel R., Dronhofer A., Eggeler G., 2003, The evolution of dislocation density during heat treatment and creep of tempered martensite ferritic steels, *Acta Materialia*, **51**, 4847-4862
- 12. SAQUET O., CHICOIS J., VINCENT A., 1999, Barkhausen noise from plain carbon steel: analysis of the influence of microstructure, *Materials Science and Engineering*, **A269**, 3-82
- 13. Trillon A., Deneuville F., Petit S., Bisiaux B., 2012, Magnetic Barkhausen noise for hardness checking on steel, 18th World Conference on Nondestructive Testing, 16-20 April 2012, Durban, South Africa
- 14. Vahista M., Paul S., 2009, Correlation between surface integrity of ground with Barkhausen noise parameters and hysteresis loop characteristics, *Materials and Design*, **30**, 1595-1603
- 15. Yamuara S., Furuya Y., Watanabe T., 2001, The effect of grain boundary microstructure on Barkhausen noise in ferromagnetic materials, *Acta Materialia*, **49**, 3019-3027

Manuscript received April 25, 2013; accepted for print August 22, 2013