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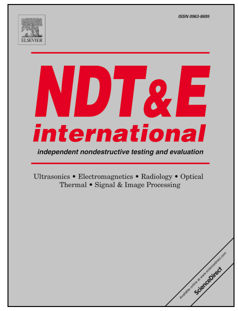

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Comparison of magnetic nondestructive methods applied for inspection of steel degradation

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Abstract

A series of low carbon steel specimens is investigated in the frame of a chain of magnetic non-destructive measurements on round robin samples, organized by the Universal Network for Magnetic Non-Destructive Evaluation. The samples have been plastically deformed by cold rolling to five consecutive stages of deformation. They were examined by several different nondestructive magnetic methods and the results were compared with each other and with the destructive mechanical measurements of Vickers

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hardness and ductile-brittle transition temperature. Very well correlated and qualitatively very similar relations between the magnetic and mechanical parameters were found in all cases, and for all the methods. Difference between the results of different measurements was found only in the sensitivity of the measurement with respect to the independent parameter, which is a promising message for the potential practical applications and possible future standardization of magnetic nondestructive methods.

Keywords: Magnetic NDT, Magnetic hysteresis, Steel degradation

Introduction

Magnetic measurements are frequently used for characterization of changes in the structure of ferromagnetic materials, because magnetization processes are closely related to their microstructure. This fact also makes magnetic measurements an obvious candidate for non-destructive testing, for detection and characterization of any defects in materials and in manufactured products made of such materials, see e.g. [1,2,3]. Structural non-magnetic properties of ferromagnetic materials have been non-destructively tested using different magnetic methods for a long time with fair success.

Nowadays, steel degradation of reactor pressure vessels presents a very important and urgent problem of nuclear industry. No effective *nondestructive* method for reliable detection of the steel ductile-brittle transition was commonly established and code accepted yet. Degradation of the pressure vessel steel is caused by joint influence of the long time exposure of the material to elevated temperature, to neutron irradiation and to mechanical pressure. The action of each of those factors contributes to modification of microstructure of the steel, and magnetic methods are expected to bring an acceptable solution of the problem. The 3MA technique (Micromagnetic Multiparameter Microstructure and Stress Analysis), which is a combination of Barkhausen noise, incremental permeability, upper harmonic analysis of the magnetic tangential field and eddy current testing [4] in a project of the German nuclear safety research program has documented the ability to nondestructively characterize the ductile-to-brittle transition temperature at Charpy specimens. The samples were pressure vessel steel, base material and weldments of German and WWER nuclear power plants exposed to different neutron fluences [5,6].

Structure-sensitive properties of minor hysteresis loops have been recently compared with Vickers hardness and ductile-brittle transition temperature (DBTT) obtained by Charpy impact test for cold rolled low carbon steel and a good relationship between mechanical and magnetic properties was found [7]. The measurements were done on magnetically closed picture-frame samples. Similar measurements on bar (Charpy) samples, with similarly good results were performed by applying Magnetic Adaptive Testing [8].

For helping general use of magnetic methods in practical application and to promote the joint efforts of magnetic community to choose the best methods, an informal network of many research workers in many countries all over the world, interested in this area, has been organized on Prof. Seiki Takahashi's (Iwate University, Japan) initiative [9]. Main goal of the Universal Network for Magnetic Nondestructive Evaluation (UNMNDE) is to concentrate research power in this important area, to improve efficiency of the information exchange, to prove applicability of magnetic ND methods, to develop new methods, to investigate theoretically the observed phenomena, to find new application possibilities, to fasten cooperation with industrial companies, to organize workshops for experts in this area and to organize new projects for introduction of magnetic methods into industrial application. NDE of steel degradation before any crack initiation would be one of the targets in Universal Network concerning nuclear plants, thermal and electric plants, chemical plants, mass transportation, bridges and gas pipelines.

One of the most important targets of UNMNDE is the systematic comparison of different magnetic nondestructive measurements techniques. Each of the applied methods has its own advantages and limitations. Recently there have been performed some efforts for making comparison between different magnetic methods. In [10] three popular magnetic NDT technologies were reviewed: magnetic flux leakage (MFL), magnetic Barkhausen noise (MBN) and recently developed metal magnetic memory (MMM). In [11] major hysteresis loop measurement, Barkhausen noise measurement and minor hysteresis loop analysis (Magnetic Adaptive Testing, MAT) – were applied in plastically deformed series of transformation induced plasticity (TRIP) steel samples. Good correlation was found between magnetic characteristics, measured by different ways. In [12] Magnetic Barkhausen Noise measurements, magnetic minor loops Power Scaling Laws (PSL) and Magnetic Adaptive Testing were compared with each other on the same series of neutron irradiated nuclear reactor pressure vessel steel material. MAT was found the most sensitive of the applied methods and also it showed the most straightforward linear correlation with the independently measured traditional destructive DBTT and Vickers hardness. The other two magnetic methods (MBN and PSL) were less sensitive than MAT, and besides they correlated neither with DBTT, nor with HV, nor with each other.

For even wider scale of systematic comparison a series of round robin samples was prepared, which was circulated among the participating laboratories. The same series of samples was measured by each lab within the network. Each member performed his own measurements by use of the same samples and the measured results are being compared within this paper with each other and with destructively measured mechanical properties (Vickers hardness, tensile deformation, Charpy impact test). To our knowledge, such a large scale comparison of different magnetic methods, performed on the *same* samples, till now has not been performed.

We believe that our efforts will help the practical application of magnetic nondestructive measurement methods. The main purpose of this paper is to do the comparison of different magnetic methods and to draw conclusions about the correlation of magnetic parameters.

Samples

Five samples were prepared for the magnetic measurements. The material of them is low-carbon steel with 0.16 wt.% of C, 0.20 wt.% of Si, 0.44 wt.% of Mn, with the rest of Fe. Convenient pieces of the material were cold-rolled down to plastic deformation, ε, of 0, 5, 10, 20 and 40%, and then the samples of desired dimensions and shapes of rectangular blocks were carefully machined from the deformed steel. The shape and dimensions of the block samples can be seen in Fig. 1. A V-shaped notch was machined at one face of each sample, so that they can also be used for Charpy mechanical tests. The largest dimension (axis) of Charpy specimens is along the direction of rolling.

Transmission electron microscopy (TEM) revealed modified microstructure of the deformed steel, accumulation of dislocations (from 10^9 to 10^{10} cm^{-2}) in particular, which corresponds to the expected and observed increase in mechanical and magnetic hardness of the deformed material. The TEM micrographs of the five different samples are shown in Fig. 2. TEM micrographs reveal different microstructures on the surface samples. These pictures are shown only to illustrate that significant changes occur in the microstructure, which will be then reflected in mechanical hardening and in magnetic parameters. To analyse the TEM pictures and to find correlation between dislocation density and NDT parameters is out of the purpose of the present work.

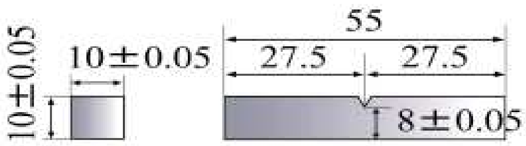

***Fig. 1**. Dimensions (in mm) and shape of the measured samples. (Each sample had a V-notch at one face.)*

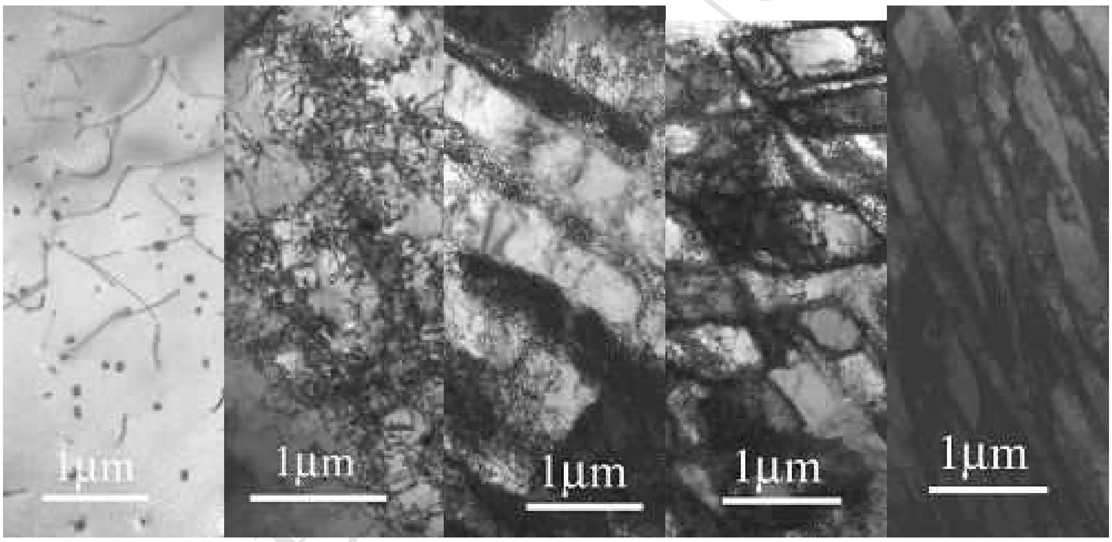

***Fig. 2**: TEM micrographs of the steel material cold rolled to the thickness reduction of 0, 5, 10, 20 and 40% (from left to right) [7].*

Vickers hardness of the deformed material was measured with the standard Vickers indentation technique. The applied load was 300g. Five samples were tested for each rolling reduction and 10 indents were taken for each sample.

Charpy impact test was performed with a pendulum of 27.6 kg and the lift angle of 138.5^{O} in the 201÷363 K temperature range. Five V-notched Charpy samples were tested at each temperature and both the largest and the smallest values of the absorption energy were eliminated when averaging the data. The ductile-brittle transition temperature, DBTT, was defined as the midpoint between the low toughness brittle- and the high toughness ductile-fracture regimes.

Magnetic methods

Different nondestructive magnetic methods were used for the characterization of the same set of the above described, cold rolled low carbon steel material and nine different magnetic parameters were evaluated based on these methods. The measurements were performed at three different laboratories. These methods are the following:

- Measurement of the coercive field, H_{c0}, based on the analysis of set of quasistatic minor hysteresis loops, performed at Department of Materials Science and Engineering, Faculty of Engineering, Iwate University, Morioka, Japan (S. Kobayashi).
- Coefficients of power law relations between minor loop parameters, *WRO*, performed at Department of Materials Science and Engineering, Faculty of Engineering, Iwate University, Morioka, Japan (S. Kobayashi).
- Coefficients of power law relations between minor loop parameters, *WFO*, performed at Department of Materials Science and Engineering, Faculty of Engineering, Iwate University, Morioka, Japan (S. Kobayashi).
- Coercive field of saturation loop, $H_c(MHL)$, performed at Ghent University, Ghent, Belgium (L. Dupre).

- Maximum of relative permeability along virgin curve, μ_{max}, performed at Ghent University, Ghent, Belgium (L. Dupre).
- Hysteresis loss of saturation loop, *W*, performed at Ghent University, Ghent, Belgium (L. Dupre).
- Magnetic Adaptive Testing, *MAT*, performed at Institute of Physics ASCR, Prague, Czech Republic (I. Tomáš)

In the following a brief information is given about these methods.

Analysis of set of quasistatic minor hysteresis loops

Magnetic measurements were performed using an apparatus designed for measuring Charpy impact test pieces [13]. A closed magnetic circuit is composed of Fe-3%Si magnetic yokes, test-piece support rods made of 2%V-permendur, and a test piece itself fixed by upper and lower support rods at the center of the apparatus. A 2227-turn exciting coil to apply a magnetic field parallel to the long axis of a test piece, two B coils (50 X 2 turns) to measure a magnetic flux density, and four H coils (400 X 4 turns) in series connection to detect magnetic field near the sample surface, are placed near the sample position. H coils face to one of four different surfaces of the test piece to get an average value of the field intensity. A triangular wave current of 0.5 Hz obtained from a function generator was applied to a bipolar power supply. The bipolar power supply converts the voltage to current and amplifies the current. The amplified current was applied to the excitation coil to generate a cyclic magnetic field and magnetize the specimen. The induced voltage of H and B coils were amplified with a gain of 20 and 60 dB, respectively, and then purified by a low-pass filter with a cut-off frequency of 40 Hz. These signals were converted from analogue to digital data by an AD converter and were integrated with respect to time to calculate H and B.

A set of minor hysteresis loops with various magnetic field amplitudes H_a up to 6.5kA/m (totally, 70 minor loops) was measured by changing H_a step by step. Before measuring each minor loop, the sample was demagnetized to obtain symmetrical minor loops. For analysis of the minor loops, we introduced several minor-loop parameters; minor-loop maximum flux density B_a*, minor-loop coercive force H_c*, minor-loop remanent flux density B_R*, minor-loop hysteresis loss W_F*, minor-loop remanence work W_R*, where W_R* is the area enclosed by a minor loop in the second quadrant [7].

We examined three relation curves between obtained minor-loop parameters, i.e. W_F*-B_a*, W_R*-B_R*, and H_c*-B_R* curves, which were least-squares fitted to scaling power laws, $W_F^*=W_F^0(B_a^*/B_s)^{nF}$, $W_R^*=W_R^0(B_R^*/B_R)^{nR}$, and $H_c^*=H_c^0(B_R^*/B_R)^{nc}$, respectively. Here, W_F^0, W_R^0, and H_c^0 are minor-loop coefficients, which are sensitive indicator of defect density; B_s and B_R are maximum and remanent flux density of the major loop, and were set to 1.8 and 1.0 T, respectively. Note that the first relation between W_F* and B_a* with nF=1.6 is well known as the Steinmetz law [14]. n_F, n_R, and n_c are power-law exponents, and n_F = 1.74±0.03, n_R = 1.68±0.01, and n_c = 0.48±0.02, irrespective of the level of rolling reduction in the present measurements. These exponent values are consistent with n_F~n_R~1.5, n_c~0.45 obtained for a magnetically closed frame sample of Fe and its alloy [15]; Slightly different values for Charpy test samples may be attributed to magnetic properties of yokes and/or high frequency of a cyclic field. The least-squares fits were typically performed using minor loops with B_a* = 0.2 – 0.9 T, at which magnetic flux density exhibits a steep increase with applied field and irreversible motion of Bloch wall plays a dominant role for magnetization process.

Saturation loop measurement

The core elements of the measurement setup applied at Ghent University and based on the unidirectional field-metric method are typically: (1) a closed magnetic circuit comprising the ferromagnetic material under test; (2) local H-sensors; and (3) two windings, placed around the material under test: an outer excitation winding is used to apply the time-dependent magnetic field H(t), and secondly an inner measurement winding, wound as close as possible around the sample under test is used to determine the resulting flux density B(t). The identification of the magnetic polarization $J(t)=\mu_0 M(t)$ in the sample is by adding an air flux compensator in hardware [16,17].

A linear power amplifier supplies the current to generate a unidirectional magnetic field in the excitation coil, and the magnetic circuit magnetizes the sample under test as uniform as possible. At each time point, the resulting magnetic field H(t) and polarization J(t) $=\mu_0 M(t)$ are measured with appropriate sensors and if necessary analog signal conditioning is applied on the measured signals. With the experimental setup based on the field-metric method the constitutive relation J(H) can be determined for excitation frequencies in the range of $f\approx 0$ (quasi-static) to typically 1 kHz. For the work presented in this paper we focus on quasi-static measurements. However, one should be cautious when interpreting the measured J- and H-values, since this interpretation is inherently connected to the underlying experimental principles of the field-metric method. The inductive method used to measure the polarization $J(t)=\mu_0 M(t)$ inherently results in an average magnetization value, $M_{meas}=M_{avg}$, averaged out over the cross section (perpendicular to the applied magnetic field) of the sample, whereas both direct (H-sensors) and indirect methods (Ampere's law and a predefined magnetic path length) to determine the magnetic field H(t) inherently result in the magnetic field value at the surface of the sample, $H_{meas}=H_{surf}$.

The dimensions and the shape of Charpy samples are standardized: the specimens are 55 mm long bars with a square cross section of 10 mm by 10 mm, and with a V-shaped notch of 2 mm deep introduced in the middle of one of the long sample sides. For samples with such dimensions its magnetic characterization is not straight-forward. Indeed, as the Charpy samples are short, miniature closing yokes have to be used. Here closing yokes are chosen with an inner width of 40 mm between the yoke legs. Moreover, since the cross section of the Charpy samples is large, and of comparable size to the cross section of suitable closing yokes, the reluctance of the closing yokes cannot be neglected and therefore the appropriate method to determine the effective magnetic field at the surface is based on the local measurement of the tangential field followed by extrapolation towards the Charpy samples surface. Indeed, due to the finite dimensions of the H-sensors, the magnetic field in air cannot be measured exactly at the surface. To overcome this, three local H-measurements (Hall probes) in the surrounding air can be performed at different distances close to the surface of the Charpy samples (active Hall elements at 1,25, 2.1 and 2.85 mm from surface) , and the tangential component of the magnetic field strength at the outer surface of the samples is then estimated by linear extrapolation towards the sample surface [18,19]. This approach is justified by the observation that in a small region close to the sample surface, the magnetic field increases weakly and more or less in a linear fashion with distance away from the sample surface. The three Hall sensors are placed in such a way to measure the magnetic field component along the longitudinal direction above the sample surface, when the notched sample side is facing down.

For the considered Charpy samples, field-metric measurements are performed. Each time, a set of 40 first order symmetric quasistatic magnetization loops is measured (for a range of field amplitudes) at an excitation frequency of 0.05 Hz. Notice that at this frequency, $M_{meas}=M_{avg}=M_{surf}$ as well as $H_{meas}=H_{surf}=H_{avg}$ The virgin curve is constructed from the tip values of these symmetric quasistatic loops. The experimentally obtained magnetization loops are then analyzed based on the conventional saturation loop and virgin curve parameters. This results in the coercive field H_c and hysteresis loss W at technical saturation and in the maximum value for the differential permeability of the virgin curve.

Magnetic Adaptive Testing

This method is a special type of magnetic hysteresis measurements, which is based on systematic measurement and evaluation of magnetic minor hysteresis loops [20,21]. The samples are periodically magnetized with step-wise increasing amplitudes. A specially designed Permeameter with a magnetizing yoke was applied for measurement of families of minor loops of the magnetic circuit differential permeability. The magnetizing coil, wound on the yoke, gets a triangular waveform current with step-wise increasing amplitudes and with a fixed slope-magnitude in all the triangles. This produces time-variation of the effective field, $F_i(t)$, in the magnetizing circuit and voltage signal is induced in the pick-up coil wound also on the yoke,. As long as $F_i(t)$ sweeps linearly with time, the voltage signal $U(F_i(t), A_j)$, in the pick-up coil is proportional to the mean differential permeability, $\mu(F_i(t), A_j)$, of the magnetic circuit. Symbol A_j denotes amplitude of the sweeping field, $F_i(t)$, and $B(F_i(t), A_j)$ is the present value of the (mean) magnetic induction in the circuit. Similarly to the case of major hysteresis loop measurement, MAT measurements could also be regarded as pseudo-static with negligibly small effect of eddy-current on the magnetization curves, because of the low slope of the increase of the excitation current.

The Permeameter works under full control of a PC computer. The experimental raw data are processed by an evaluation program, which interpolates the data into a regular square grid of elements, $\mu_{ij}\equiv\mu(F_i,A_j)$, of a μ-matrix with a pre-selected field-step. Each μ_{ij}-element represents one "MAT-descriptor" of the investigated material structure variation. The consecutive series of μ-matrices, each taken for one sample with a value of the strain, ε_k, of the series of the more-and-more deformed steel, describes the magnetic reflection of the material plastic deformation. The series of matrices is processed by another program, which normalizes them by a chosen reference matrix, and arranges all the mutually corresponding elements μ_{ij} of all the evaluated μ-matrices into a table of $\mu_{ij}(\varepsilon)$-*degradation functions*.

In MAT measurements with an attached yoke we deal with non-uniform magnetization. As a consequence, the magnetizing field inside the sample cannot be easily calculated, we cannot use the magnetizing *field* coordinates (F_i, A_j), but we use the magnetizing *current* coordinates (I_{Fi}, I_{Aj}) instead. Besides, with the non-uniform magnetic circuits we cannot speak about the signal of pick-up coil to be proportional to the differential permeability of the *material*, but evidently we deal with an *effective* differential permeability of the whole magnetic *circuit* instead. As MAT is a *relative* method, the current coordinates are also well applicable for identification of the mutually corresponding magnetic states of the samples to be related and compared.

MAT degradation functions of the investigated samples were evaluated and those, optimized for description of the studied dependences, were expressed as functions of the rolling reduction, of the Vickers hardness and of the ductile brittle transition temperature. Optimization means that those degradation functions were chosen from the large data pool, which were the most sensitive with respect to the change of the independent parameter, and at the same time they were highly repeatable, and in such a way the most reliable.

The samples were measured by attaching an inspection head to the surface of each sample. Lateral dimensions of the yoke can be characterized by its cross-section: S=10x8 mm^2, the total outside length 27 mm, and the total outside height of the bow 26 mm. The magnetizing coil was wound on the bow of the yoke, with N=200 turns and the pick-up coil was wound on one of the yoke legs with n=40 turns. The inspection head was attached along the length of the block samples (magnetizing the samples along the rolling direction), to the smooth surface at the face opposite to the V-notch. The main experimental conditions were given by the rate of change of the magnetizing current, I_F: dI_F/dt=93.8mA/s, and the maximum current amplitude I_A=50mA. Reciprocal value of the differential permeability (1/μ) of the magnetic circuit was determined and is shown in the following graphs. The plotted curves correspond to the minor loop with the current amplitude I_A=40mA, at the value of the magnetizing current I_F=20mA.

The correlation between material degradation and magnetic parameters does not depend on the material of the yoke and on the type of magnetization (if the magnetizing yoke is put on the sample surface or if the samples are magnetized in a magnetizing coil), as it was shown in [22]. Also, if the parameters of MAT measurement are properly chosen, then they practically do not depend on temperature in the T = 20 – 180 ^{o}C range, as published in [23].

As for the slope of the magnetizing field, there is no universal advice how to chose it and what region of degradation functions to use for the most successful MAT nondestructive tests in individual cases. The concrete adaptation is completely governed by properties of the investigated material degradation and by the level of noise and the available rate of change of the magnetizing field of the used measuring technique. However, generally speaking it can be stated, that as long as the MAT degradation functions are picked-up from localities of the field coordinates where the local differential permeability is *high* (frequently close to the maximum permeability at the used minor loop), then the (usually "reciprocal") degradation functions possess *very high sensitivity*, but level of their sensitivity is *strongly field-slope-dependent*. On the other hand, if the MAT degradation functions are chosen from localities of the field coordinates where the material is closer to saturation and the local differential permeability is *low*, sensitivity of such degradation functions is *not extremely high*, but it is *little dependent on the applied magnetizing field-slope* [24].

Results

Each technique provides one output parameter. Fig. 3 shows within one graph the results of all magnetic measurements, which were described in section „Magnetic methods", as functions of the rolling reduction. For making the proper comparison, all measured values are given in relative units. The reason of this normalization is, that we can compare the results of measurements, performed at different laboratories within one graph only such a way. For instance, result of H_c measurement is given in Oe, μ_{max} is a dimensionless value by itself, MAT descriptors are given is arbitrary unit, etc. But if all measured values, obtained by different methods are normalized by the corresponding value of the reference (0 % rolled) sample, we can easily compare the results of different measurements. In Fig. 3 the numbers show, how many percentage of the given parameter was increased with respect to the reference (0 % rolled) sample due to cold rolling. The same relative units of the magnetic parameters will be used then in the following graphs, too.

An exponential function can be fitted on the measured curve in form of

$$Y = A * exp(-x/t) + y_0$$

Where Y is the given magnetic parameter, and x is the rolling reduction. For instance, in case of MAT data, given in Fig. 3: A=-268.5, t=10.76 and y_0=270, regression is 0.997.

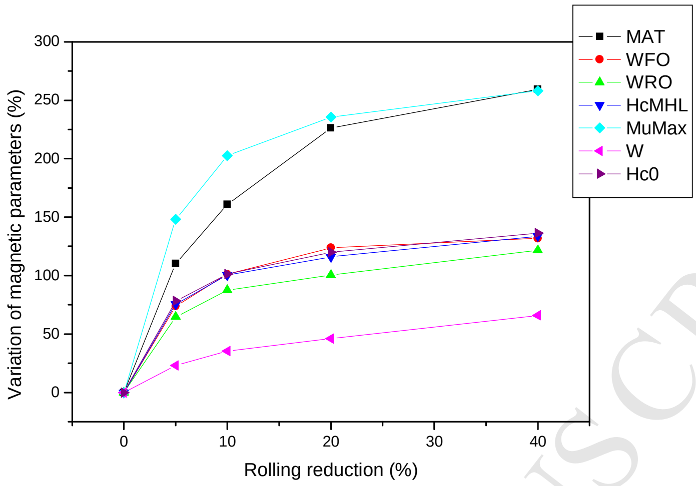

***Fig. 3:** The relative change of the investigated magnetic parameters as functions of rolling reduction. The meaning of the parameters are given in section „Magnetic methods”.*

The same magnetic parameters are shown as functions of Vickers hardness, too, in Fig. 4.

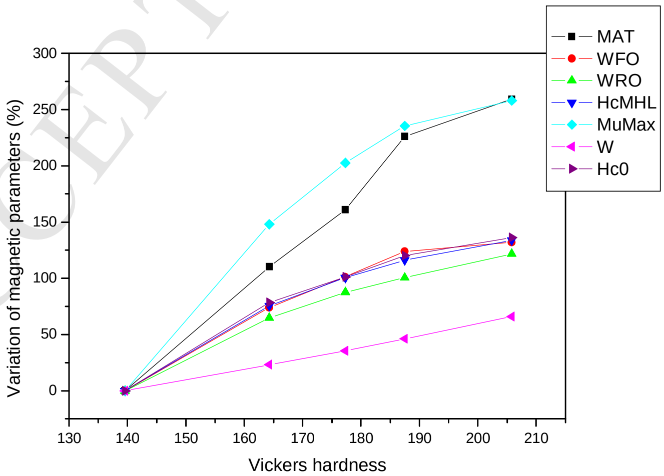

***Fig. 4**: The relative change of the investigated magnetic parameters as functions of Vickers hardness. The meaning of the parameters are given in section „Magnetic methods”*

The correlation between magnetic parameters and Vickers hardness is closely linear. It is demonstrated in Fig. 5, where linear fit was used for the parameters. For the better reading of this figure only several magnetic parameters (five of the total seven) are shown, otherwise the middle region of the figure would become too confusing. Linear fit was found as the best one. The slope of the linear fit (given in Table 1.) gives data about the sensitivity of the different methods, discussed in this paper. The regression of the linear fit to Vickers hardness values is also given in Table 1. for all investigated methods.

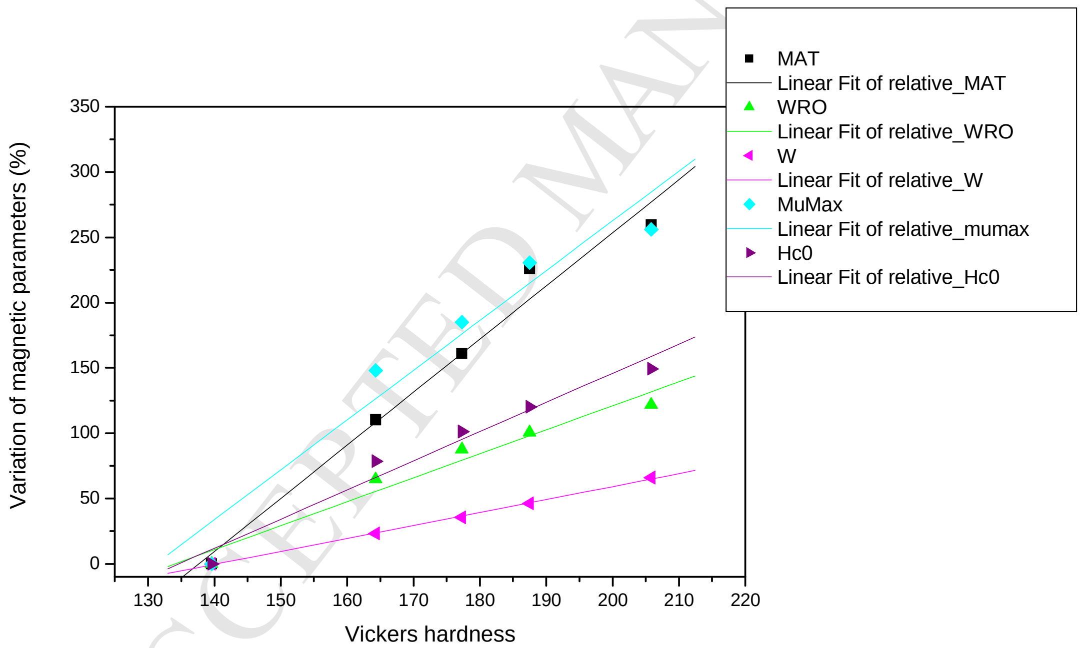

Fig. 5: *The relative change of some investigated magnetic parameters as functions of Vickers hardness, and linear fits of the data. The meaning of the parameters are given in section „Magnetic methods”.*

Table 1.

Regression of linear fit to HV values of different magnetic parameters.

Method	MAT	WFO	WRO	H_cMHL	μ_{Max}	W	H_{c0}
Slope of the linear fit	4.07	2.06	1.84	1.90	3.82	1.01	2.07
R	0.998	0.962	0.979	0.967	0.936	0.999	0.967

The same magnetic parameters are shown as functions of ductile brittle transition temperature in Fig. 6.

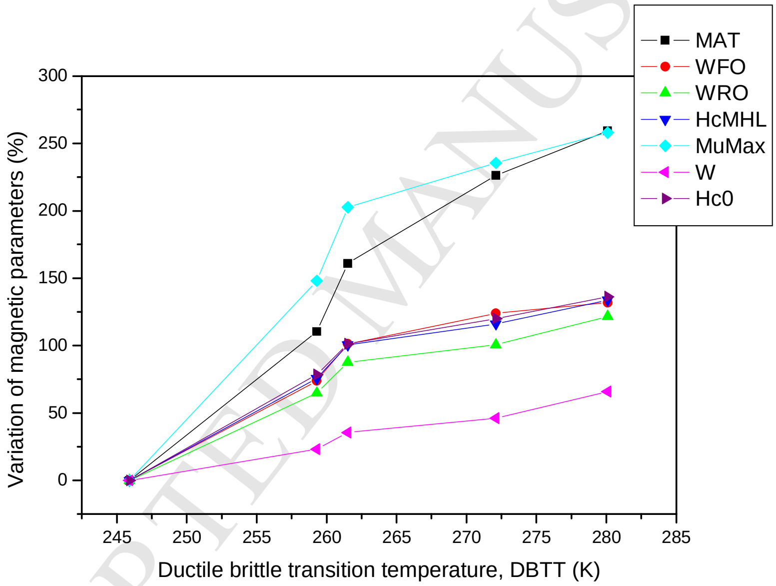

Fig. 6: *The relative change of the investigated magnetic parameters as functions of ductile brittle transition temperature, DBTT. The meaning of the parameters are given in section „Magnetic methods".*

The correlation between magnetic parameters and ductile brittle transition temperature is closely linear, similar to the case of hardness. It is clearly seen in Fig. 7, where linear fit was used for the parameters. Again, for the better reading of this figure only several magnetic parameters (five of the total seven) are shown, otherwise the middle region of the figure would become too confusing

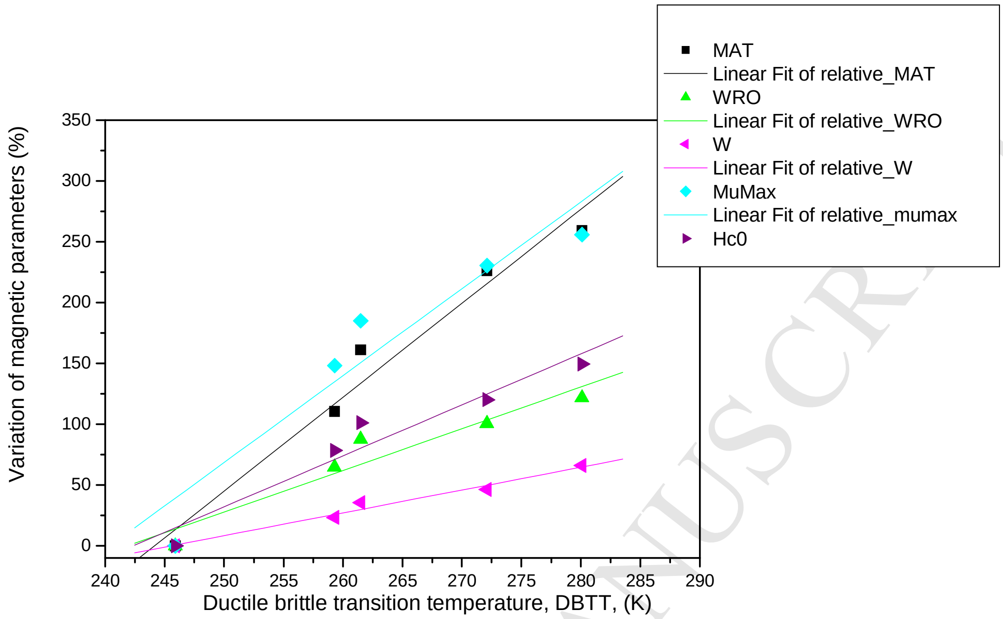

Fig. 7: *The relative change of some investigated magnetic parameters as functions of ductile brittle transition temperature, DBTT, and linear fits of the data. The meaning of the parameters are given in section „Magnetic methods”.*

Anisotropy behaviour

The material, from which the measured samples were cut, was cold rolled. This cold rolling introduces an anisotropy. This work is discussing the results of measurements performed on block shape samples (Charpy samples). These samples can be reasonably magnetized only along its long (55 mm) axis, and the above given measurements were done in such a way. However, plate samples with dimensions of 60 x 40 x 10 mm3 were also prepared and they were measured as well. Some difference, which shows the anisotropy behaviour was observed if the samples were magnetized parallel with or perpendicular to rolling direction. Fig. 8 demonstrates this anisotropy. It is seen that the effect is not significant and the correlation between magnetic parameters and rolling reduction is very similar to each other.

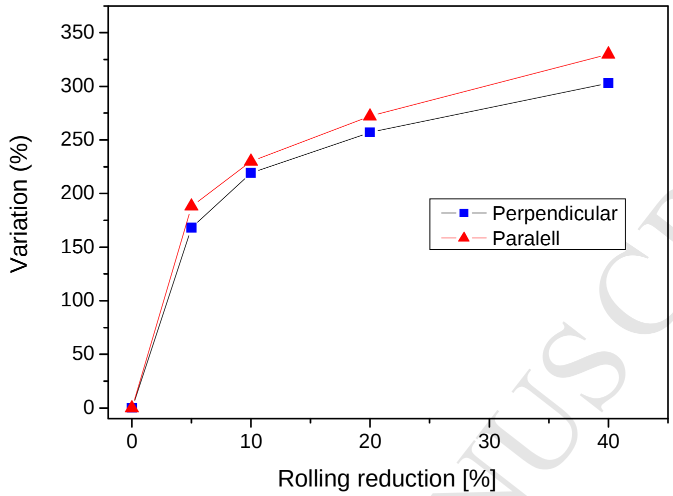

Fig. 8. *The relative change of the optimally chosen MAT parameters as functions of the rolling reduction for the parallel and perpendicular orientation of the magnetizing field with respect to rolling direction.*

Discussion

The material degradation induces the formation of nanoscale defects such as precipitates, dislocation loops, solute clusters, solute-vacancy clusters, etc. These defects act as pinning center for dislocations and disturb their movement, resulting in changes of mechanical properties; an increase of hardness, DBTT, yield strength as well as a decrease of ductility, upper shelf energy. The shape of hysteresis loops is changed depending on the size, density, distribution, and kinds of defects. According to an earlier theory for micromagnetism [25], the arrangement of magnetization is determined so as to minimize magnetic Gibbs free energy consisting of exchange energy, magnetocrystalline anisotropy energy, magnetostatic energy, and magnetoelastic energy. In ferromagnets including lattice defects, the Gibbs free energy is lowered when domain walls are located at lattice defects and the defects act as obstacles to the domain wall motion.

All measurements, but relative permeability measurement along virgin curve provide hysteresis behaviour of the material. Evidently the geometry of the NDT probes are not the same and the measurement conditions are not similar. Consequently, outputs taken from the original results of partners are different. Furthermore, the hysteresis losses by using different methods, are evaluated at different magnetization levels. The comparison has sense – as done in this work – if first we determine the most sensitive parameter of each method, regardless, how it was evaluated and then we compare each of the optimal parameters, as presented e.g. in Fig. 3.

The first and most important message of the present work is, that all the investigated magnetic methods result in very similar correlations between the nondestructively measured magnetic quantities and the destructively determined mechanical parameters. Difference was found only in the sensitivity of methods: certain methods are more sensitive and certain methods are less sensitive, but even in the „worst" case the sensitivity is fairly enough for the reliable detection of degradation of the material. This conclusion is very promising for further application of magnetic NDE: if any method is chosen for solving a given problem, the qualitative result is the same. Evidently, for successful practical application the magnetic parameters should be first calibrated with independently measured mechanical data.

Very significant influence of the rolling reduction on the magnetic parameters was found, regardless on the chosen method. Magnetic characteristics increase monotonously as functions of the rolling reduction. All the investigated magnetic methods describe magnetic reflection of the investigated material modifications.

As it is seen on Figs. 5 and 7, very regular, linear correlations were found, with low scatter of the points between the magnetic characteristics and other mechanical parameters, which are frequently used in industry for the characterization of the material integrity.

The values are referred to the 0% rolling, which seems to be the most appropriate way of evaluation of how the material degrades by the rolling. 0% rolling means the virgin sample. This way of interpretation makes it possible on one side to follow the material degradation due to cold rolling, but on the other side it is also the best way, how to reliably compare results of different measurements performed on the same series of samples.

By application of the magnetic methods, relatively small differences between characteristics of the investigated samples can be determined more easily and more sensitively than by the conventional destructive methods. In case of the Vickers hardness, the change due to cold rolling, which appears between the reference sample and the most plastically deformed sample was 48 %, while the less sensitive magnetic parameters revealed 65 %, the most sensitive 260 % difference. The same correlation for the ductile brittle transition temperature is even more pronounced: the DBBT changes by 15 % due to 40 % rolling reduction, while the same ratio for the less sensitive magnetic parameters is again 65 %, for the most sensitive one is again 260 %.

In principle if different types of magnetic measurements are performed, the heterogeneity properties (depth) of the sample have to be taken into account. However, in our case we do not need to deal much with this problem, because the size and geometry of the measured samples ensures that practically the same volume is magnetized during the measurement in all investigated cases.

This correlation can be used successfully for the calibration of a next measuring device. On the base of these Figures, it is possible to get direct, quantitative information about mechanical degradation of the investigated samples, if the measurement is performed on a known material and compared with the data of a reference sample.

Presently, the drawback of all of the micromagnetic techniques is, that there is no standardization yet, according which all different measurements would be performed and according which all the results would be rigorously compared as for their sensitivity. However, the fact, that we got qualitatively the same correlation between independent destructively measured parameters and different magnetic parameters, in spite of the different excitation systems (sinusoidal, triangle, frequency, maximum magnetic field strength), of the different receiving systems (receiver coil on yoke leg, Hall-element, receiver coil as pick-up coil with high spatial resolution,…) and of different signal analysis (bandpass-, highpass-, lowpass-filtering, rectifying, peak-value, …) is rather promising for making steps towards a future standardization. This is the most important message of the present work.

For the potential future application of magnetic NDE it is very important to prove that even in the case of magnetically open samples (as is the case of the investigated Charpy bar samples) proper information can be obtained about the degradation that happened in the samples, regardless on the chosen way of magnetic measurement.

As mentioned already in the Introduction, none of the methods, considered in this work is new. Each methods can have advantages and drawbacks. A significant advantage of MAT, WRO and WFO is, that the parameters are evaluted from the set of minor hysteresis loops. It means, that if these methods are applied there is no need for magnetic saturation of the sample. The magnetic saturation is extremely difficult – due to the factor of demagnetization - or simply unpossible if large size samples are measured. This is also valid for the μ_{max} measurement.

Conclusions

Seven different, nondestructive magnetic methods were applied to determine the correlation between magnetic parameters and independently measured mechanical characteristics on the same series of cold rolled low carbon steel samples. Practically the same and very good correlation was found in all cases, regardless on the concrete applied method. The *character* of variation of some parameters are linked to the intrinsic properties of the sample, they are not dependent of the used magnetic NDT technique, as it was proved in this work. This is one of the most important messages of the present work.

Difference between the results of different measurements was found only in the sensitivity of the measurement with respect to the independent parameter. This is the other important aim of this paper, i.e. to show the sensitivity of each electromagnetic technique and to compare them with each other. As it was shown, there are signifant differences in the sensitiviies, as can be clearly seen in all figures, or given numerically in Tabl 1. Magnetic adaptive testing and measurement of the maximum of relative permeability along virgin curve are the most sensitive methods and determination of hysteresis loss of saturation loop is the less sensitive one.

The above mentioned facts are believed as a very good and promising message for the potential practical applications

The presented results give also a good chance to determine the level of embrittlement of ferromagnetic steel objects (e.g. of the nuclear pressure vessel surveillance specimens) due to their heavy-duty industrial service period, with the aid of non-destructive magnetic methods.

This analysis of different magnetic methods can be a first step towards standardization of magnetic NDE.

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Obituary

During the writing of this manuscript Prof. Seiki Takahashi, the coauthor of the work, and the founder of UNMNDE, passed away. Because of this very sad event, authors of this work would like to dedicate this paper to the memory of Professor Seiki Takahashi. He was an outstanding scientist, the founder and the first Chair of the academic UNMNDE society, where he tried to contribute to the security of nuclear power plants and of other important areas of industry. He significantly contributed to the understanding of hysteresis phenomenon and magnetic losses and in general to the magnetic nondestructive evaluation of ferromagnetic materials. We will miss his knowledge of magnetic materials and also his kind, friendly and open personality.

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Figure captions

Fig. 1: Dimensions (in mm) and shape of the measured samples. (Each sample had a V-notch at one face.)

Fig. 2: TEM micrographs of the steel material cold rolled to the thickness reduction of 0, 5, 10, 20 and 40% (from left to right) [7].

Fig. 3: The relative change of the investigated magnetic parameters as functions of rolling reduction. The meaning of the parameters are given in section „Magnetic methods".

Fig. 4: The relative change of the investigated magnetic parameters as functions of Vickers hardness. The meaning of the parameters are given in section „Magnetic methods".

Fig. 5: The relative change of some investigated magnetic parameters as functions of Vickers hardness, and linear fits of the data. The meaning of the parameters are given in section „Magnetic methods".

Fig. 6: The relative change of the investigated magnetic parameters as functions of ductile brittle transition temperature, DBTT. The meaning of the parameters are given in section „Magnetic methods".

Fig. 7: The relative change of some investigated magnetic parameters as functions of ductile brittle transition temperature, DBTT, and linear fits of the data. The meaning of the parameters are given in section „Magnetic methods".

Fig. 8. The relative change of the optimally chosen MAT parameters as functions of the rolling reduction for the parallel and perpendicular orientation of the magnetizing field with respect to rolling direction.

Highlights

- Magnetic nondestructive methods were applied for investigation of plastically deformed low carbon steel.
- The same series of samples were measured at different laboratories by seven different nondestructive magnetic methods.
- Results were compared with each other and with the destructive mechanical measurements of Vickers hardness and ductile-brittle transition temperature.
- Very good correlation was found between the destructively measured and magnetically measured quanties.
- The results of different magnetic measurements correlate with each other very well.