

COMPARISON OF SELECTED METHODS FOR NON-DESTRUCTIVE EVALUATION OF STRUCTURAL DEGRADATION OF CONSTRUCTIONAL FERROMAGNETIC MATERIALS

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In this study, two advanced methods out of several utilised for nondestructive inspection of ferromagnetic constructional materials were compared. Already well-established, yet novel method, Magnetic Adaptive Testing (MAT), was put into the confrontation with brand new approach based on examination of the relaxation phenomena associated with domain wall motion. Early experiments have revealed that in case of the samples made of standard steels subjected to long-term annealing MAT sometimes brings ambiguous, chaotic results very difficult to interpret, whereas the relaxation processes appear to correlate strongly and in a simple manner with annealing time acting as an agent representing artificial ageing of material. Hence the detailed analysis of relaxation processes seems to be a new, promising way for non-destructive inspection of structural degradation of this type of materials even when MAT fails. Thus, the offer of magnetic non-destructive evaluation methods for industrial practice extends.

Keywords: non-destructive evaluation, structural degradation, ferromagnetic constructional materials

2 THEORETICAL BASIS

1 INTRODUCTION

Diverse methods based on the analysis of the changes of various (electro)magnetic parameters associated with the structural degradation related to the long-term action of *eg* elevated temperatures, mechanical load, irradiation, *etc* are used for non-destructive evaluation (NDE) of constructional, usually electrically conductive, ferromagnetic materials. Some of them are suitable for the detection of macroscopic defects, such as visible cracks, gaps, *etc* (*eg* Eddy Current Testing - ECT, [1]), whereas some other methods (including well-adopted, yet under extensive development, Barkhausen Noise Analysis - BNA, [2]) allow to investigate even the microstructural variations. In general, all of these methods have some advantages as well as limitations.

Many studies confirmed the relationship between classical integral hysteretic parameters, such as, *eg*, the coercivity and remanent flux density and, *eg*, the density of dislocations present in the material, but the sensitivity of these parameters to structural changes associated with various factors causing at a final stage even fatigue damages was often relatively low and the interpretation of experimental results could sometimes be unclear, [3]. Therefore, several other advanced techniques that seem to have promising perspective for these purposes in industrial practice worked out at our department will be discussed in this study. One of them, invented and developed firstly by I. Tomáš and further improved by him and his co-workers is Magnetic Adaptive Testing - MAT, [3, 4], will be compared with a novel technique based on direct experimental observation of the dynamics of magnetization processes (especially domain wall motions) taking place in the samples magnetized under sophisticatedly defined conditions, [5, 6].

Obviously, the magnetization processes taking place in the ferromagnetic materials with changing exciting field are associated among many other things, with the presence of various structural defects, residual stresses, *etc*. This knowledge supports the effort to find an explicit correlation between such structural damages, especially of a microscopic scale, and experimentally obtained magnetization curves. To keep all the things as simple as possible, one needs to eliminate the influence of too many input variables, therefore the operational conditions during the magnetizing the samples should be chosen in such a way, that only one type of magnetization process (preferably domain wall motion) is dominant. This requirement is preferable from the point of view of hardware as well, since avoiding too large exciting currents and/or signal conditioning circuitry needed (high-power supplies, preamplifiers, filters, *etc*) allows to build up the instrumentation with an emphasis on compactness and portability, allowing the use of such devices routinely in industrial environment.

2.1 Magnetic Adaptive Testing

MAT method is based on the determination of a large set of "local" differential magnetic quantities (*eg*, differential permeability, Preisach-like model parameters, *etc*) parameterized with respect to properly chosen operating point in a two-dimensional discretized space of possible values, [3,4]. Properly chosen differential parameters were confirmed by many studies [3,4,7] to be far more sensitive to the structural changes than integral ones. For this reason, MAT appears to be very progressive, using potential of latest hardware and software yet relatively simple method for non-destructive inspection of constructional ferromagnetic materials.

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To find the set of proper magnetic parameters the series of minor hysteresis loops has to be measured. The relative differential permeability, one of quantities highly sensitive to microstructural changes is defined as

$$\mu_{\text{diff}}(t) = \frac{1}{\mu_0} \frac{dB(t)}{dH(t)} = \frac{1}{\mu_0} \frac{\frac{dB(t)}{dt}}{\frac{dH(t)}{dt}}, \quad (1)$$

where μ_0 is the vacuum permeability and $dB(t)/dt \approx v_{\text{ind}}(t)$. If the same constant field change rate $|dH(t)/dt| = c$ is provided for each minor loop and all the conditions for homogeneous magnetization are satisfied, then the differential permeability is directly proportional to the voltage $v_{\text{ind}}(t)$ induced in pick-up coil with N_2 sensing turns, sample cross-sectional area A ,

$$\mu_{\text{diff}}(t) = \pm \frac{1}{\mu_0 N_2 A c} v_{\text{ind}}(t) \quad (2)$$

“+” sign is applicable for increasing and “-” for decreasing field $H(t)$. Since the applied field is a piecewise linear function of time $H(t) \approx \pm ct$, the time instant as a variable can be substituted by the applied field and thus the differential permeability can be understood as the function of applied field h_A . The permeability belonging to the particular minor hysteresis loop can be characterized by the second variable - exciting field amplitude h_B . The loops are thus to be measured as a function of applied field h_A which is altered within a set of exciting field amplitudes h_B changing with step Δh_B . The obtained magnetic parameters can be arranged into the matrices whose elements are determined by two values - discrete field value h_{Ai} and minor loop amplitude h_{Bj} , [3].

After proper discretization of applied field, the permeability matrix corresponding to the set of minor hysteresis loops can be determined by the elements

$$\mu_{ij} = \mu_{\text{diff}}(h_{Ai}, h_{Bj}) \quad (3)$$

The comparison of the matrices of magnetic parameters obtained *before* and *after* varied types of defined loading is the fundamental idea of MAT method. The comparison reveals a subset of matrix elements most sensitive to structural changes owing to long-term action of various influencing factors, *ie* can be used as “operating” values of h_A and h_B . Thus, the obtained data are evaluated as *relative* changes with respect to some defined initial state of the materials represented by an initial matrix of parameters (either as a ratio or as a subtraction of actual parameters after the material is subjected to some defined load $\mu_{ij,load}$ and initial values $\mu_{ij,0}$ prior to any load applied). As a result, the experimental equipment needs not to be calibrated absolutely with metrological precision, since any systematic errors minimise each other. On the other hand, obtained values have to be put into correlation with the data found by any of absolute, usually destructive methods to find proper material calibration curves.

2.2 Magnetic relaxation processes associated with domain wall motion

As shown in *eg* [8], the domain wall motion can be analyzed by means of damped forced harmonic oscillator

$$m_w \frac{d^2 x(t)}{dt^2} + \beta_w \frac{dx(t)}{dt} + \alpha_w x(t) = \nu H(t) \quad (4)$$

with $x(t)$ being the distance of domain wall from its equilibrium position given by the local minimum of energy, m_w is the effective mass of the domain walls, β_w is the motional damping coefficient, α_w is the restoring coefficient associated with the force acting on the wall when disturbed from equilibrium in the direction x and $\nu H(t)$ is the driving force due to applied field $H(t)$. Since the change of magnetic flux density is proportional to the change of wall position, solving Eq. (4) for $x(t)$ gives the time dependence of the flux density $B(t)$. Total damping coefficient consists of several terms; two of them belong to intrinsic relaxation (eddy current damping and spin-relaxation damping) meanwhile one (which is of interest here) is associated with structural relaxation, [9]. If one knows the waveform of exciting field $H(t)$, finding general solution to (4) is a standard route offering the expression for $x(t)$ consisting of two parts - a transient part (solution to the homogeneous second-order differential equation) and a steady-state part (particular solution to the inhomogeneous equation), which must be used jointly to fit the physical boundary conditions.

To reveal pure relaxation processes, the samples have to be magnetized by piecewise linear exciting field $H(t)$, which is at a particular time instant t_{hold} held at the same value H_{hold} with corresponding flux density $B_{\text{hold}} = B(t = t_{\text{hold}})$ as an initial condition for a sufficiently long time needed for the flux density to reach a final steady-state value B_{steady} . Typically, regardless of H_{hold} value, the first-order exponential-like behaviour of the magnetic flux density $B(t)$ given within the interval of $t \in \langle t_{\text{hold}}; t_{\text{hold}} + T \rangle$ as

$$B(t) = B_{\text{hold}} + \Delta B \left[1 - \exp\left(-\frac{t - t_{\text{hold}}}{\tau}\right) \right] \quad (5)$$

with one dominant time constant τ can be seen, [5]. The induced voltage is then

$$v_{\text{ind}}(t) = \frac{N_2 A}{\tau} \Delta B \exp\left(-\frac{t - t_{\text{hold}}}{\tau}\right) \quad (6)$$

3 EXPERIMENTAL WORK

The samples made of standard steel were subjected to artificial ageing by means of thermal treatment - annealing for 2, 4, 6, 20, 40 and 60 hours at the temperature of 753 K. First 2 hours served for removal of any residual stresses remained after hot- and cold-working of the samples. In order to minimize the influence of parasitic effects, such as, *eg* the demagnetizing fields, internal field inhomogeneity *etc*, whilst achieving sufficient signal levels, magnetically

closed ring-shaped samples with proper geometry (average inner diameter $d = 45.7$ mm, outer diameter $D = 55.7$ mm and sample height $h = 4.79$ mm) were used for the measurement of magnetic characteristics with the aim to verify whether the suggested principles of NDE could possibly work. The samples were thermally treated in a commercially available furnace, the dimensions were verified after annealing as well. Customized computer-controlled experimental equipment based on well-known principles allowing control of exciting field waveform via analogue feedback introduced from the current-sensing resistor was utilized for the magnetization of samples as well as for data acquisition, see *eg* [10]. As of hardware, crucial components of the experimental set-up are mostly LXI-based instruments allowing remote monitoring of the objects subjected to the testing almost everywhere, where LAN/WAN is available. So, the problems associated with the placement of the tested objects in an environment unsafe for the staff or difficult-to-access, *etc* can be avoided.

The control software is permanently optimized from the point of view of further signal processing, handling large datasets, supplementary digital feedback algorithm stability and reliability, *etc*. The waveforms of $H(t)$ and $v_{ind}(t)$ were taken as single-shots using the arbitrary waveform generator in triggered burst mode. Subsequently, the magnetic flux density $B(t)$ was obtained by means of numerical integration of induced voltage. In case of direct evaluation of domain wall motion, the exciting field was in fact DC, ‘frozen’ at constant value H_{hold} during sufficiently long time interval (related to the time constant(s)) during which the transients are in progress. Thus, only pure relaxation phenomena linked with damped motion of domain walls towards steady-state equilibrium (associated with domain wall pinning and bowing) were observed. All the experimental data were obtained at the same value of $H_m = 1500$ A/m. This value was chosen as the compromise - the sample is close to saturation and the exciting peak current is not too high. Moreover, one of the goals of these experiments was the comparison with MAT, where the fields close to saturation are optimal as well. Also other values of H_m were tested, similar behavior was observed.

RESULTS AND DISCUSSION

4.1 MAT experiments

An example of discretized values of the differential permeabilities as well as those *difference-normalized* to the first reference values (the first matrix in the left figure) at particular operating points represented by discrete h_{Ai} and h_{Bj} organized into triangular matrices are shown in Fig. 1. The values of difference-normalized matrix elements in Fig. 1b were calculated simply as the differences between the differential permeabilities after applied defined load and the reference matrix elements, $\Delta\mu_{ij} = \mu_{ij,load} - \mu_{ij,0}$. The obtained data at each discrete operating point were ana-

lyzed with respect to various criteria, such as *eg* the sensitivity, stability (the measure of the fluctuation of the values in the nearest neighborhood), linearity and monotonicity. For these purposes a customized software developed in commercially available development environment was used, [11].

Detailed data examination revealed somewhat chaotic behavior of the dependencies of differential permeabilities upon the annealing time t_a as a measure of artificial sample ageing. The curves were mostly non-monotonous, non-linear and the sensitivity was far less than expected based on our long-term experience in utilizing this method. Similar results were obtained when the matrices were *ratio-normalized*. As such, in this *very particular* case MAT seems to be not a good choice for non-destructive inspection of sample degradation. A possible explanation could be associated with the fact that observed changes of the shape of hysteresis loops (in accordance with only weak changes of both coercivity and remanent flux density) reflecting the heat treatment of given material are insignificant, (see further).

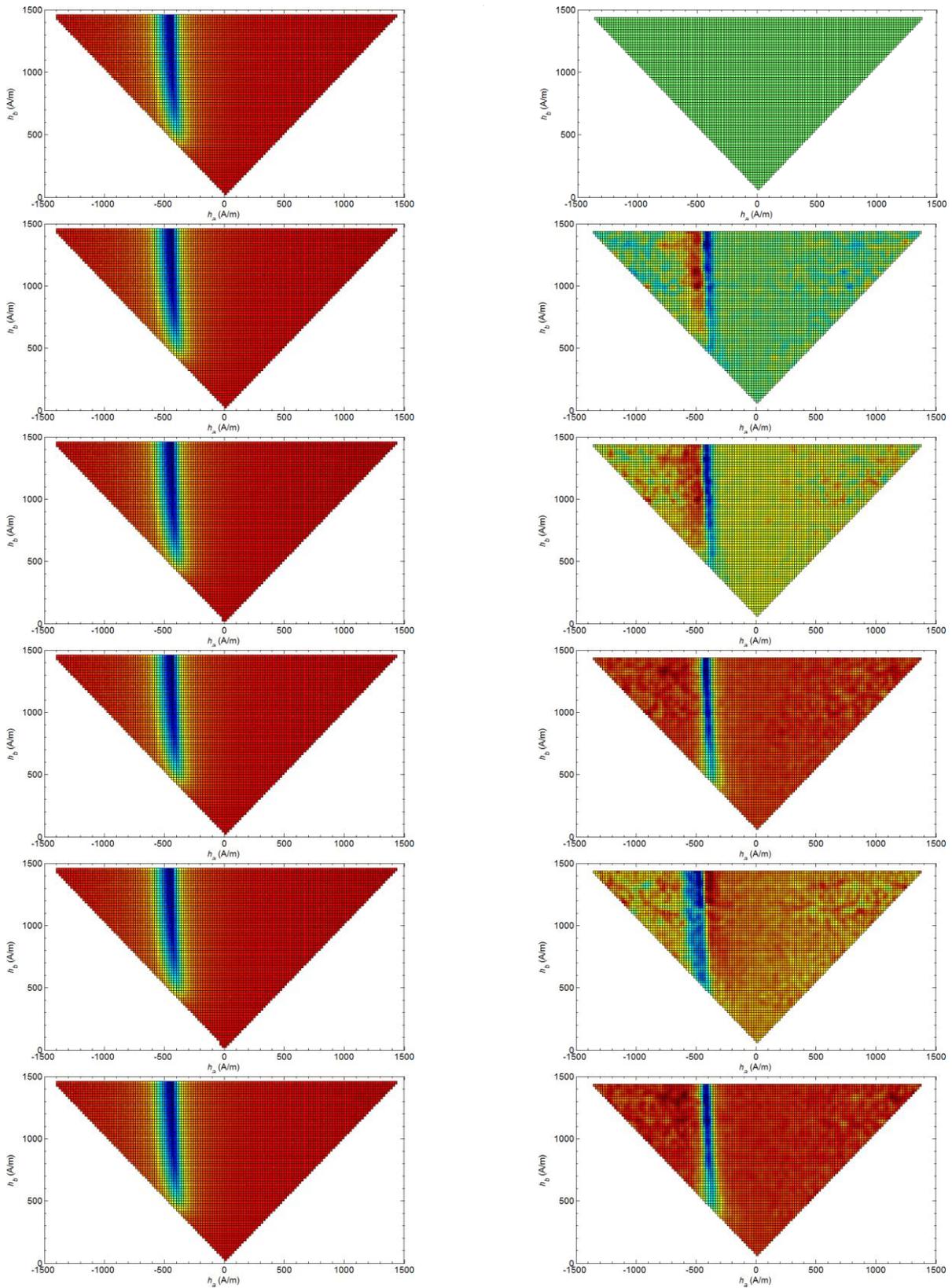
4.2 Relaxation processes

The illustration of typical measured waveforms of exciting field, flux density and induced voltage waveforms related to the magnetization conditions as described in Section 2.2 is displayed in Fig. 2a (taken from [6]) and Fig 2b. Extra triangular cycles of exciting field (not shown in Fig. 2) without constant (steady) part were added before these waveforms to minimize the influence of magnetic accommodation.

Also, $\Delta B = B_{steady} - B_{hold}$ and $\Delta v_{ind} = v_{ind,steady} - v_{ind,hold}$ are indicated by corresponding arrows in Fig. 2b, c, d. Since $v_{ind,steady} = 0$ if holding interval T is enough for the transients to vanish, Δv_{ind} can be expressed as

$$\Delta v_{ind} = -\frac{N_2 A}{\tau} \Delta B$$

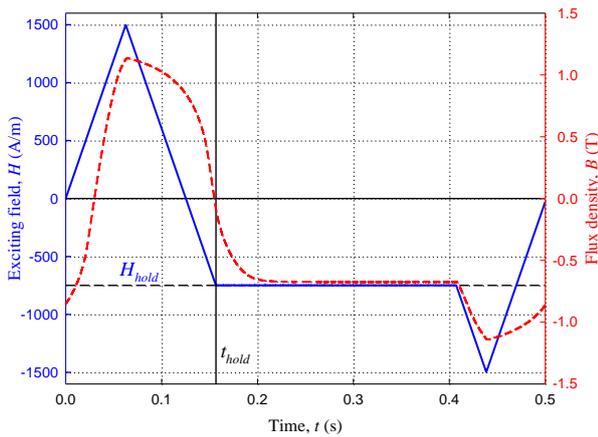
As revealed by the experiments, the peak of induced voltage Δv_{ind} almost does not depend on annealing time, only H_{hold}/H_m ratio is important, Fig. 3. Consequently, the relationship between ΔB and τ should be almost linear, *ie* the shape of $\Delta B(H_{hold}/H_m)$ dependence should be similar to $\tau(H_{hold}/H_m)$ dependence, keeping in mind negative constant of proportionality ($-N_2 A$), Fig. 4. The time constants can be calculated either from experimental data of ΔB and Δv_{ind} using Eq. (7) or by multiparametric fitting of the waveforms within $t \in \langle t_{hold}; t_{hold} + T \rangle$ using Eq. (5). As follows from theory, the time constant $\tau = m_w/\beta_w$ is linked to microstructure via m_w and β_w . Thus, the parameter ΔB , known from the experiment and directly proportional to τ appears to be a good indicator of structural changes associated with thermal ageing, [5, 6, 8]



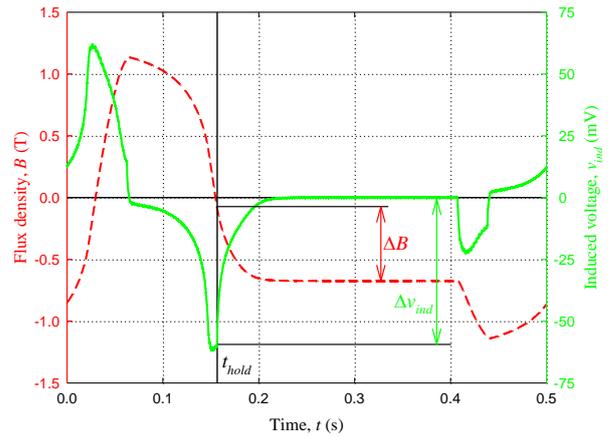
(a) Matrices of differential permeability $\mu_{ij} = \mu_{diff}(h_{Ai}, h_{Bj})$.
 Matrices of differential permeability $\mu_{ij} = \mu_{diff}(h_{Ai}, h_{Bj})$.

(b) Difference-normalized matrices $\Delta\mu_{ij} = \Delta\mu_{diff}(h_{Ai}, h_{Bj})$

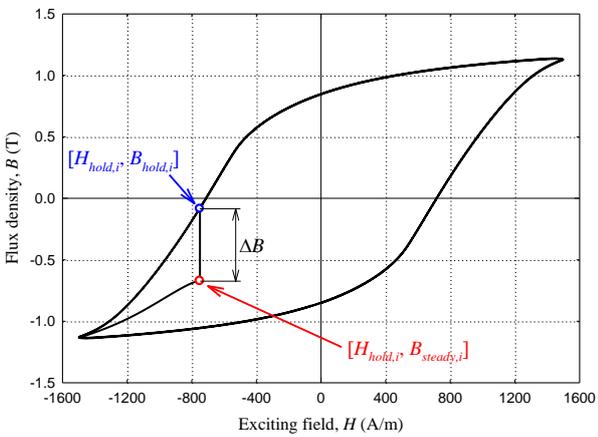
Fig. 1. Discretized matrices of differential permeability $\mu_{ij,load}$ (a) - and difference-normalized matrices related to the first matrix $\Delta\mu_{ij} = \mu_{ij,load} - \mu_{ij,0}$ (b) - of the samples thermally treated for 2, 4, 6, 20, 40 and 60 hours respectively (1st to 6th row) at the temperature of 753 K ; the reference parameters $\mu_{ij,0}$ for 2 hours of annealing are given in the matrix placed in the first row on the left.



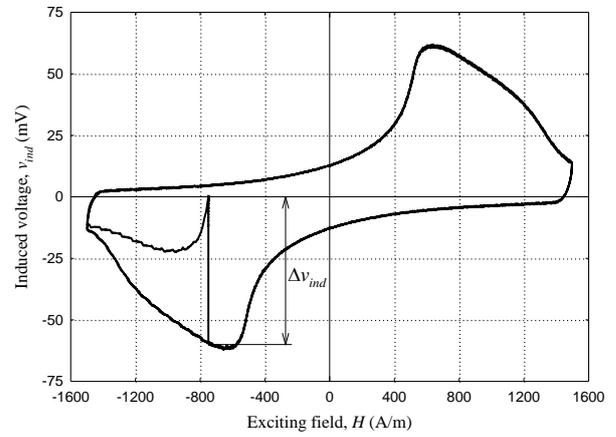
(a) Exciting field (blue, solid line) and corresponding flux density waveform (red, dashed line)



(b) Induced voltage (green, solid line) and flux density waveform (red, dashed line) found by numerical integration



(c) Hysteresis loop with steady period



(d) Induced voltage with steady period

Fig. 2. Important signal waveforms. (a), (b) - at $f = 4$ Hz, $H_m = 1500$ A·m⁻¹ and $H_{hold} = 0.5H_m$, (c) - hysteresis loop related to signals shown in Fig. 2a and, (d) - induced voltage as a function of applied field corresponding to solid waveforms in Fig. 2a and Fig. 2b

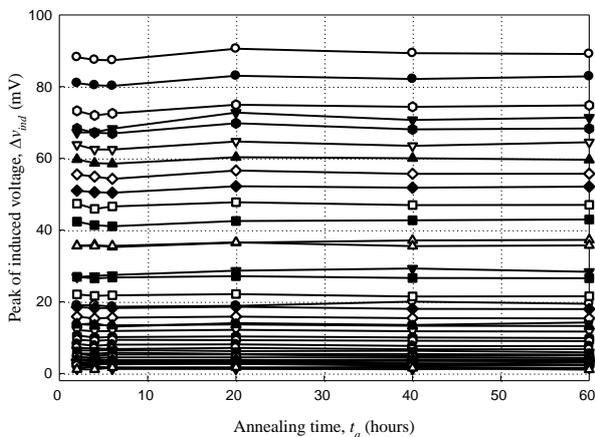


Fig. 3. The difference Δv_{ind} as a function of annealing time t_a . and parameter of the curves is H_{hold}/H_m ratio - the highest peak (uppermost curve) observed at $H_{hold}/H_m = -0.35$

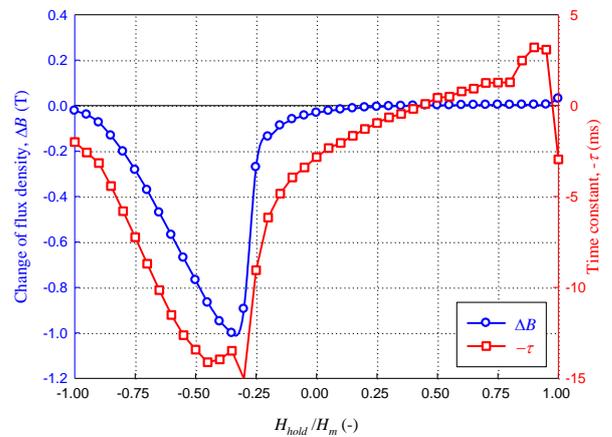


Fig. 4. The difference ΔB and time constant τ as a function of H_{hold}/H_m ratio. The time constant values are calculated using (7) from experimental data of ΔB and Δv_{ind}

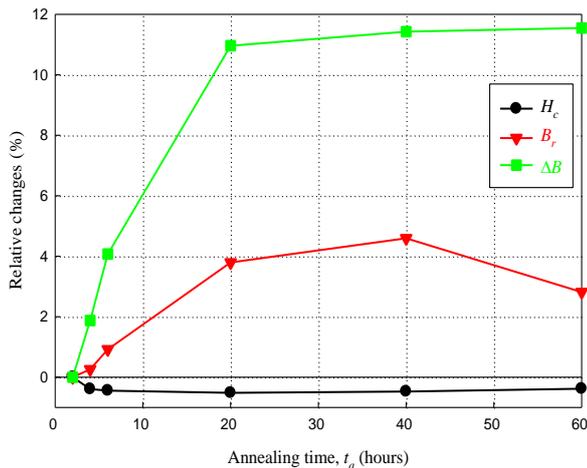


Fig. 5. Relative changes of hysteric parameters H_c , B_r as well as ΔB found at $H_{hold}/H_m = -0.3$ as a function of annealing time t_a

For illustration, the changes of ΔB as well as classical hysteric parameters often used for non-destructive evaluation, such as the coercivity H_c and remanent flux density B_r , determined from quasi-static hysteresis loops measured at $f = 1$ Hz and $H_m = 1500$ A.m⁻¹ related to the reference values after 2 hours of annealing as a function of ageing time are in Fig. 5. Only ΔB at $H_{hold}/H_m = -0.3$ most sensitive to thermal treatment is shown. The most significant changes were observed for the parameter ΔB , with maximum change almost 12%, meanwhile the change of B_r was about 4.6% and H_c was almost insensitive to thermal treatment with the change of -0.5% only.

5 CONCLUSIONS

The initial experiments focused on the analysis of relaxation processes associated with domain wall motion show significant sensitivity of properly chosen set of parameters of the obtained induced voltage/flux density waveform to the annealing time of the samples subjected to artificial aging at elevated temperatures comparing to the sensitivity of MAT, where the differential permeability values at properly chosen operating points out of the whole set structured into special triangular matrix usually offers very good outcome. Hence the detailed analysis of relaxation processes seems to be a promising way for non-destructive inspection of structural degradation of this type of materials even when MAT fails. Thus, the offer of magnetic non-destructive evaluation methods for industrial practice extends. Nevertheless, one should keep in mind that bringing this approach into an industrial practice is associated with the difficulties related to the fact that the experiments on closed samples are difficult or even impossible. Thus, careful verification of knowledge obtained taking into account

well-known side effects associated with the measurement on magnetically open samples (such as demagnetisation field, ill-defined air gaps, etc) needs to be carried out, as discussed eg in [3] and [7].

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