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INVESTIGATION OF THERMALLY AGED SAMPLES BY MAGNETIC ADAPTIVE TESTING

Gábor Vértesy * − Ivan Tomáš ** − Satoru Kobayashi * − Yasuhiro Kamada *****

Two series of thermally aged Fe-1 wt % Cu alloys were investigated by the method of Magnetic Adaptive Testing, typical by its low required magnetization of the samples. The samples were prepared for modelling neutron irradiation and thermal ageing in nuclear reactor pressure vessels. Sensitive correlation was found between magnetic descriptors and ageing time. Based on these results, Magnetic Adaptive Testing is suggested as a highly promising non-destructive tool for monitoring the influence of irradiation of surveillance samples in pressure vessels of nuclear reactors. Results of the non-destructive magnetic tests were also compared with the destructive mechanical measurements of Vickers hardness.

Keywords: nondestructive testing, magnetic adaptive testing, irradiation embrittlement, thermal ageing, minor hysteresis loops

1 INTRODUCTION

Magnetic measurements are frequently used for characterization of changes in structure of ferromagnetic materials, because their magnetization processes are closely related to microstructure of the materials. This fact also makes magnetic measurements an evident candidate for non-destructive testing, for detection and characterization of any modification and/or defects in materials and in products manufactured from such materials [1,2,3]. Structural non-magnetic properties of ferromagnetic materials have been non-destructively tested using traditional hysteresis methods since long time with fair success. A number of techniques have been suggested, developed and currently used in industry, see *eg* [4,5]. They are mostly based on detection of structural variations via the classical parameters of major hysteresis loops.

An alternative, more sensitive and more experimentally friendly approach to this topic, the Magnetic Adaptive Testing (MAT) method was considered recently [6]. In contrast to the traditional hysteresis tests, where every sample is characterized only by its single major hysteresis loop, MAT investigates a complex set of minor hysteresis loops (from a minimum amplitude of the magnetizing field, with the amplitude increasing by regular steps, up to the material-saturating major amplitude) for each sample of the measured series. The method was successfully tried out for a number of ferromagnetic materials and a number of modifications of their properties (*eg* [7-9]).

In the present work this method was applied on two series of thermally aged Fe-1 wt. % Cu alloy samples 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 [10]. The samples were prepared for modelling neutron irradiation and thermal ageing in nuclear reactor pressure vessels.

2 EXPERIMENTAL

It is known that by thermally ageing the Fe-1 wt % Cu samples at 773 K, Cu precipitates are formed in the Fe metal matrix.

Two series of the Fe-Cu alloys were annealed at 1123 K for 5 hours, followed by water quenching. Then one part of samples was cold-rolled down to 10% deformation and isothermally aged at 773 K for t = 0, 50, 500 and 5000 min. The other part of samples was aged similarly, but without previous cold rolling (0% deformation).

Vickers hardness of the deformed material was measured with the standard Vickers indentation technique. The applied load was 300 g, 15 indents were taken for each ageing condition.

Two millimeter thick ring-shaped samples were prepared having 18 mm outer and 12 mm inner diameter, respectively. The samples were equipped with a magnetizing coil (60 turns) and a pick-up coil (90 turns) each.

A specially designed Permeameter [11] was applied for measurement of families of minor loops of the magnetic circuit differential permeability. The magnetizing coil wound on the sample 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, $h_a(t)$, in the magnetizing circuit and a signal is induced in the pick-up coil. As long as $h_a(t)$ sweeps linearly with time between minor loops field amplitudes, $\pm h_b$, the voltage signal $U(h_a,h_b)$, in the pick-up coil is proportional to the differential permeability, $\mu(h_a,h_b)$, of the magnetic circuit

$$\mu(h_a,h_b) \propto U(h_a,h_b) \propto \frac{\partial B(h_a,h_b)}{\partial h_a} \frac{\partial h_a}{\partial t}$$

The Permeameter works under full control of a PC computer, which sends the steering information to the function generator, and collects the measured data. An input-output

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* Hungarian Academy of Sciences, Research Institute for Technical Physics and Materials Science, Konkoly Thege Miklós út 29-33, H-1121 Budapest, Hungary; vertesyg@mfa.kfki.hu ** Institute of Physics, Academy of Sciences of the Czech Republic, 18221 Praha, Na Slovance 2, Czech Republic; tomas@fzu.cz
*** Non Destructive Evaluation and Science Research Center, Faculty of Engineering, Iwate University, Japan; koba@iwate-u.ac.jp

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data acquisition card accomplishes the measurement. The computer registers data-files for each measured family of the minor "permeability" loops, corresponding to each measured sample. They also contain detailed information about all the pre-selected parameters of the voltage signal induced in the pick-up coil. The rate of change of the magnetizing field was 440 $Am^{-1}s^{-1}$.

The described regime of the Permeameter yields a characteristic signal in the pick-up coil. The signal values start at the origin of the plot (the magnetic circuit was demagnetized before the measurement), then it increases into positive values (up to the positive starting field amplitude, $+1*\Delta h_b$), then it drops down into negative values as the applied field changes the direction of its rate, it proceeds in the negative values until the negative starting field amplitude $-1*\Delta h_b$ is reached, changes its rate direction and polarity again, raises up to $+2*\Delta h_b$, etc.

Every measured set of minor hysteresis loops is then recomputed into a matrix of elements, positioned by a couple of field coordinates (h_a and h_b), where h_b is amplitude of the minor loop in question and h_a is the field-position of the matrix element on the loop. Each succession of matrix elements with the same coordinates, of all the samples, ordered according to the increasing material degradation (here with the ageing time, t) is called a succession of MAT-degradation-descriptors or simply a MAT-degradation-function. The optimal (most sensitive and most reliable) descriptors are used for testing of the structural modifications of the material in question.

3 RESULTS

Illustrating the primary measured quantities, the signals of the pick-up coil of the 10% deformed samples are shown in Fig. 1.

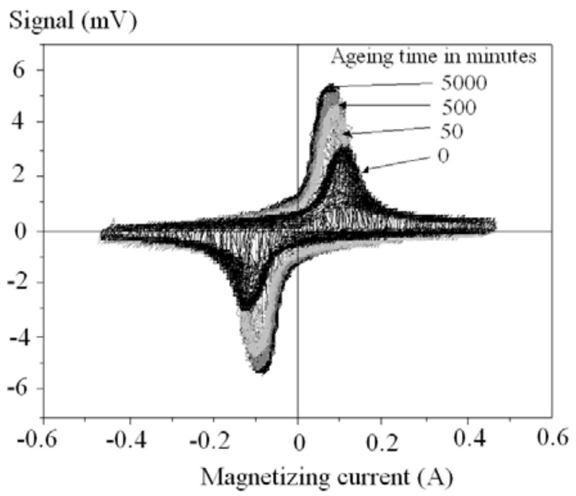

Fig. 1. The signal of the pick-up coil from the measurement of 10% compressed series of samples.

Influence of the cold rolling, as reflected by Magnetic Adaptive Testing is shown in Fig. 2. Here the optimal degradation function, determined by the proper values of the differential permeability, μ_{ij}, is shown as a function of the cold rolling strain. The first samples of each of the two sample-series (those samples, which were *not* thermally aged) are compared with each other. The two-point experimental degradation function of Fig. 2 is normalized by the value corresponding to the not-rolled sample.

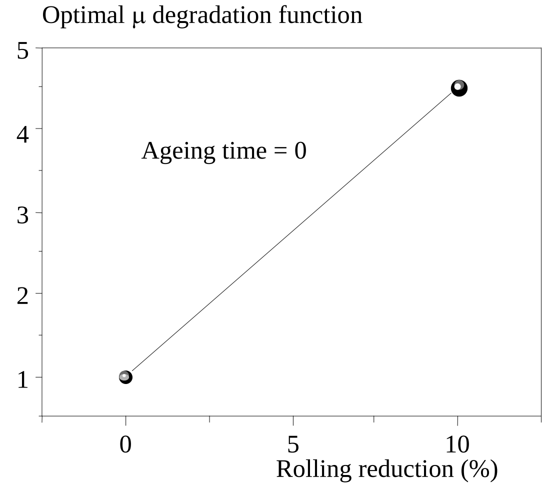

Fig. 2. The most sensitive μ_{ij}-degradation function as a plot of the rolling reduction for the thermally not-aged samples.

The influence of thermal ageing can be seen in Fig. 3. The most sensitive $\mu_{ij}(t)$ -degradation functions (see below) of the two sample series (with 10% and with 0% deformation) were determined and illustrated. Each MAT descriptor was normalized within the same series of samples, *ie* the samples with $t = 0$ were used for normalization of each degradation function.

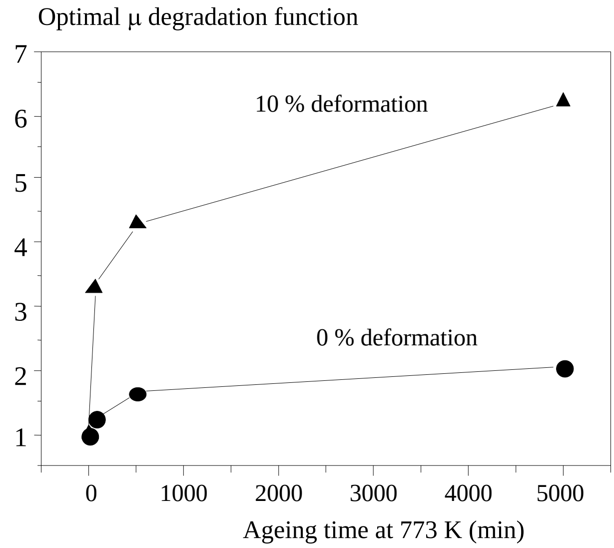

Fig. 3. The most sensitive μ_{ij}-degradation functions as functions of the ageing time (in each series the degradation functions are normalized by the first sample within the series).

The permeability matrices had been calculated from the measured data, and then the matrices evaluation program compared relative sensitivity of all the individual $\mu_{ij}(t) = = \mu(h_{ai}, h_{bj}, t)$ -degradation functions in a 3D plot, referred to as a sensitivity map. The lighter is the color at the coordinates, the higher is sensitivity of the respective degradation function. The most responsive part of the sensitivity map of the 10% deformed samples is shown in Fig. 4 as the whitest area. The crossed lines in Fig. 4 indicate the position of the (h_{ai},h_{bj}) -coordinates ($h_a \approx 60$ A/m, $h_b \approx 90$ A/m), which determine the μ_{ij} -degradation function used in Fig. 3. The neighboring points along the h_b -scale (*ie* along the minor loops amplitude variation) would result in almost the same values, however. This makes the choice of the proper, sensitive descriptor to be very reliable.

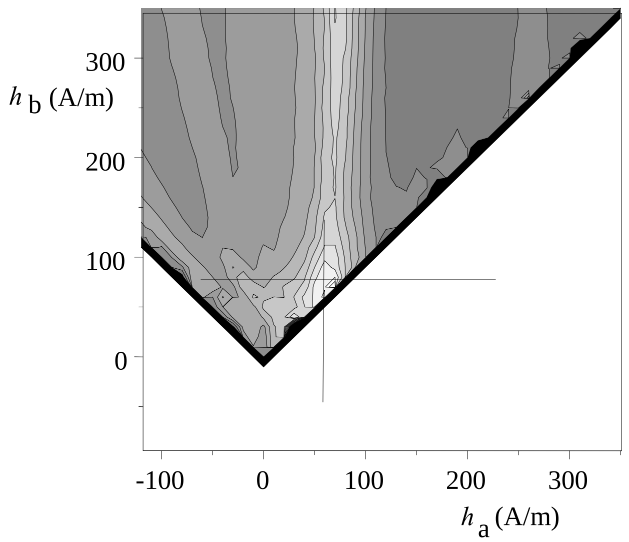

Fig. 4. Map of relative sensitivity of the μ_{ij}-degradation functions, $\mu_{ij}(t) \equiv \mu(h_{ai},h_{bj},t)$. The crossing point of the lines indicate the field coordinates of the $\mu_{ij}(t)$ -degradation function used in Fig. 3.

Vickers hardness values, measured on the samples confirmed that the material was substantially changed. The result of the Vickers hardness measurements is shown in Fig. 5.

4 DISCUSSION

A definite modification both in Vickers hardness and in magnetic descriptors was observed due to cold rolling. It is seen (from Fig. 5) that cold rolling caused a 33% increase in the Vickers hardness. Applying magnetic adaptive testing, a 350% (4.5 times, as illustrated in Fig. 2) increase in the properly chosen magnetic descriptors was found. This fact indicates that the nondestructive magnetic method can be a very sensitive technique for detection of material degradation.

Heat treatment also caused significant changes in the structure of the investigated materials. Very regular and monotonous correlation was found between the ageing time and magnetic descriptors with low scatter of points (see Fig. 3). It is worth of mentioning that modification of magnetically measured material parameters (ageing) is much more pronounced if the samples were deformed before the thermal processing. In the case of not deformed samples 100% increase (with respect to the reference sample), while in the case of 10% deformed sample 520% increase was detected.

As a consequence of the thermal ageing, material of the samples became mechanically harder with the increase of the ageing time, as can be followed by the Vickers hardness values of the samples. The heat treatment caused 64% and 27% increase in Vickers hardness for 0% and for 10% deformed samples, respectively.

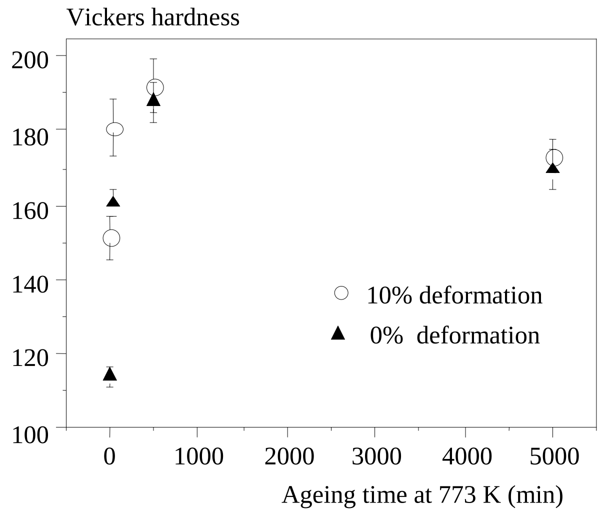

Fig. 5. The averaged Vickers hardness values of the thermally aged Fe-Cu alloys, for samples with and without previous cold rolling (0 and 10 % deformation)

However, no monotonous increase of Vickers hardness with respect to ageing time was found (see Fig. 5), and the difference between the deformed and not deformed case is not so pronounced as in the case of MAT measurements. The decreasing trend of hardness at t = 5000 min ageing can be explained by the structural change of Cu precipitates and coarsening (so-called overageing state) [12]. As it was found earlier, thermal ageing caused several structural changes inside the Fe-Cu alloys. Cu precipitation, reduction of Cu solute atoms in matrix, and modification of dislocation state were observed [12-14]. These facts indicate that hardness, which shows a peaking trend (see Fig. 5 or Fig. 2 of [13]) is sensitive to the precipitation. On the other hand, electrical conductivity shows an increasing trend, due to the solute atoms, and a conventional magnetic parameter, the coercivity, a decreasing trend in deformed specimen, which is attributed mainly to the dislocation state [13, 14].

In this case MAT parameters do not show a peaking trend here. In another work, by measuring plastically deformed low carbon steel no peak in Vickers hardness was observed, and a very good and sensitive correlation was found between the plastic deformation and magnetic descriptors, similarly as indicated in Fig. 3 [15]. In contrast to the present case, in this work the correlation between Vickers hardness and MAT parameters was linear, with very low scatter of values. These arguments indicate that the correlation between MAT parameters and other material characteristics should be the subject of future work. Magnetic parameters are strongly sensitive to the modification of dislocation state [14, 15], but it can also be influenced by the modification of precipitation state [16].

Figure 3 is considered as the main result of the present work. Based on this result it can be stated that by measuring a series of minor loops and performing MAT method on the obtained data-pool, reliable and sensitive parameters can be determined. An advantageous and independent outcome of the tested method is the confirmation that these results can be obtained without magnetic saturation of the samples, as evident from the sensitivity map. For the future possible practical application of the method, the monotonous correlation between ageing and MAT descriptors is rather encouraging. However the correct relationship between MAT parameters and material structural parameters should be still studied closely in future.

The method does not give absolute values of the traditional magnetic quantities, because of the not-reached magnetic saturation, but evidently it is able to serve as a powerful tool for comparative measurements, and for detection of changes, which occur in structure of the inspected samples during their lifetime or during a period of their heavy-duty service.

5 CONCLUSIONS

It was found and demonstrated, that the optimal MAT-degradation functions are good indicators of the material degradation of thermally aged Fe-1 wt. % Cu alloys. These parameters seem to be more sensitive than the traditional, destructively performed hardness measurements. Besides, they are acquired at substantially low magnetizing fields than the traditional magnetic hysteresis measurements, where parameters are determined from the major hysteresis loop.

These results give a good chance to determine the level of structural changes of ferromagnetic steel samples (e.g. of the nuclear pressure vessel surveillance specimens) due to neutron irradiation and thermal ageing, with the aid of the non-destructive method of Magnetic Adaptive Testing.

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REFERENCES

[1] JOHNSON, M.J. — LO, C.C.H. — ZHU, B. — CAO, H. — JILES, D.C.: J. Nondestruct. Eval. 20 (2000) 11.

[2] JILES, D.C.: Magnetic methods in nondestructive testing, .H.J.Buschow et al., Ed., Encyclopedia of Materials Science and Technology, Elsevier Press, Oxford, (2001) p.6021.

[3] MÉSZÁROS, I.: Materials Science Forum, 473-474 (2005) 231.

[4] JILES, D.C.: NDT International, 21 (1988) 311.

[5] BLITZ, J., Electrical and magnetic methods of nondestructive testing, Bristol, Adam Hilger IOP Publishing, Ltd., 1991.

[6] TOMÁŠ, I.: J. Magn. Magn. Mat. 268 (2004) 178.

[7] TOMÁŠ, I — STUPAKOV, O — KADLECOVA, J — PEREVERTOV, O: J. Magn. Magn. Mat. 304 (2006) 168.

[8] VÉRTESY, G — UCHIMOTO, T — TAKAGI, T — TOMÁŠ, I — STUPAKOV, O — MÉSZÁROS, I — PÁVO, J: Physica B372 (2006) 156.

[9] VÉRTESY, G — TOMÁŠ, I — MÉSZÁROS, I: J. Magn .Magn. Mat. 310 (2007) 76.

[10] http://www.ndesrc.eng.iwate-u.ac.jp/UniversalNetwork/html/main.html

[11] TOMÁŠ, I. — PEREVERTOV, O.: JSAEM Studies in Applied Electromagnetics and Mechanics, T. Takagi and M. Uesaka (Eds.), 9 (2001) 533.

[12] DESCHAMPS, A. — MILITZER, M — POOLE, W.J.: ISIJ International (Japan), 41 (2001) 196

[13] KAMADA, Y, — NISHINO, Y, — HOSOI, S. — TAMAOKA, S, — IDE, N, — KIKUCHI, H, — KOBAYASHI, S.: Mater. Sci. Eng. A, *in press.*

[14] KAMADA, Y, et al, to be published.

[15] VÉRTESY, G — TOMÁŠ, I — TAKAHASHI, S — KOBAYASHI, S —KAMADA, Y — KIKUCHI, H, NDT & E International, 41 (2008) 252.

[16] VANDENBOSSCHE, L.P. — KONSTANTINOVOC, M.J. — ALMAZOUZI, A. — DUPRÉ, L.R.:J. Phys. D: Appl. Phys. 40 (2007) 4114

Gábor Vértesy (DSc, PhD), born in Budapest, Hungary, in 1951. Graduated from Eötvös Loránd University, Budapest, in 1974 and received his PhD degree from Hungarian Academy of Sciences in 1995. At present he is scientific advisor of Photonics Department of Research Institute for Technical Physics and Materials Science of Hungarian Academy of Sciences. His main field is study of ferromagnetic materials and development of novel nondestructive electromagnetic testing methods for characterization of structural industrial materials.

Ivan Tomáš (RNDr, PhD), born in Prague, Czech Republic, in 1943. Graduated from the Faculty of Mathematics and Physics at Charles University, Prague, in 1967, and received his PhD degree from the Czechoslovak Academy of Sciences in 1973. Presently he is head of the Department of Magnetism at the Institute of Physics of the Academy of Sciences of the Czech Republic, Prague. His main field is ferromagnetism of solids, with a strong interest in magnetic testing of variation and degradation of ferromagnetic industrial materials.

Satoru Kobayashi (PhD in Science), born in Fukushima, Japan, in 1971. Graduated and received his PhD degree from Tokyo University of Science in 2000. At present, he is assistant professor of the NDE&Science Research Center in Iwate University, Morioka. His main field is study of magnetism in solids and development of magnetic nondestructive evaluation method.

Yasuhiro Kamada (PhD in Engineering), born in Yamaguchi, Japan, in 1969. Graduated and received his PhD degree from Nagoya University in 1997. Presently, he is associate professor of the NDE&Science Research Center in Iwate University, Morioka. His main field is study of magnetic and ultrasonic properties of structural materials for development of NDE methods for evaluation of materials degradation.

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