

## Nondestructive Magnetic Analysis of Cladded Steel Blocks in Reactor Applications

George Vickers<sup>\*1</sup>, Alexander Grant<sup>2</sup>, an Spencer & Frederick Gillmore<sup>4</sup>

<sup>\*1,2,3&4</sup>Hungarian Academy of Sciences, Centre for Energy Research

### ABSTRACT

Degradation of nuclear pressure vessel steel material (15H2NMFA) was investigated by a novel magnetic nondestructive testing method, so called Magnetic Adaptive Testing (MAT), which is based on systematic measurement and evaluation of minor magnetic hysteresis loops. The measured samples were thermally treated by a special step cooling procedure, causing embrittlement of the material. It was found that this type of degradation can be easily followed by magnetic measurements. Results of Charpy impact tests were compared with the magnetic parameters. A good, reliable and nearly linear correlation was found between magnetic descriptors and transition temperature. Some results suggest that MAT testing of cladded block samples is possible even through the relatively thick cladding

**Keywords:** steel degradation, nuclear reactor pressure vessel, Magnetic NDT, Magnetic adaptive testing, cladding.

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### INTRODUCTION

Pressurized water reactors are typical in most of the present operating nuclear power plants. The reactor pressure vessel (RPV) is one of the most important safety related part of these plants, and determines the lifetime of them. Elevated temperature, high pressure, neutron radiation, thermal ageing and low cycle fatigue (pressure and thermal) are the main environmental effects which degrade the RPV material properties during service. Safe operation rules require that the material properties never degrade below the safety code requirements. The nuclear reactor pressure vessel are made from high quality steels. During operation the actual properties must be monitored during the whole service life. Their critical parts are the forged rings and their welds at the core level, since the degradation caused by neutron irradiation is the maximum here. The RPV walls are low alloyed ferromagnetic CrNiMo or CrMoV steels.

The material properties of the reactor pressure vessels are periodically monitored by the so called surveillance programmes. The surveillance programmes use sets of encapsulated RPV wall and weld material specimens for mechanical testing and dosimetry foils. The capsules are located inside of the reactor from the start of operation, and exposed to the same (or even a little larger) neutron fluence and thermal conditions as the RPV walls.

All mechanical tests are destructive. One of the testing is the well-known destructive Charpy impact method. Charpy impact tests were introduced as a standard inspection method of material toughness more than one hundred years ago [1]. A swinging hammer breaks the fixed tested sample (with standardised dimensions and shape), and the energy absorbed by the fracture of the specimen is a characteristic properties of the material. In case of low alloyed steels the absorbed energy highly depends on the testing temperature. The engineering practice uses empirically defined values of the absorbed energy, specimen deformation, or rate of the brittle-ductile fracture surfaces as acceptance criteria or evaluates the critical temperature called ductile/brittle transition temperature. The most used criteria is the 41J absorbed energy, that is the temperature where the absorbed energy is 41 J is called the ductile-brittle transition temperature (TTKV41).

However, any nondestructive test, in contrast to the destructive ones, does not measure directly the mechanical properties of the materials. Each nondestructive test, before it can be applied in practice, must be painstakingly and rigorously correlated to the relevant destructive ones in a long, checked and re-checked series of detailed investigations.

This is probably the main reason why a single nondestructive test was able neither to replace the destructive ones so far, nor to be applied in parallel as an auxiliary one. To solve the problem a very sound foundation for verification of the needed correlation is required. Nevertheless, a number of applicable nondestructive tests were suggested, are currently tested, and the mosaics of the necessary correlations are presently slowly built up. Magnetic hysteretic methods, however, occupy a special position among them. Close correspondence between mechanical hardness and magnetic hardness of the same ferromagnetic steel is known: the neutron irradiation changes of the dislocation structure and creates precipitations within the grains. These cause radiation hardening and blocking the domain wall movement, as well. This is the base of the expected correlation. Magnetic methods are technically simple, mobile, inexpensive.

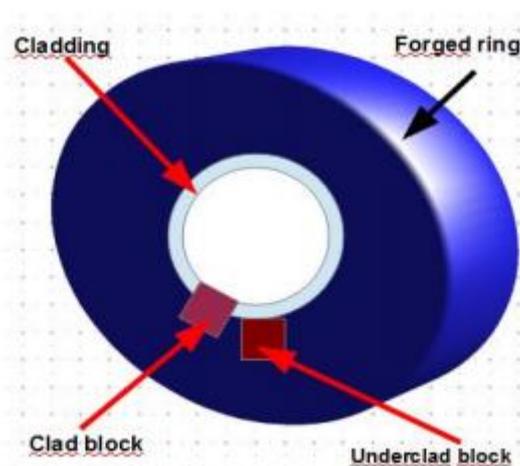
Several successful measurements were published, which proved practical applicability of magnetic methods for quantitative indication of steels embrittlement. Cold rolling [2], and special thermal processing [3] and fast neutron irradiation effect on RPV steels and welds were tested, see e.g. [4-11]. As received materials and irradiated ones with different fluences were studied.

One promising candidate technic used for the measurement and evaluation of steel degradation is the method of Magnetic Adaptive Testing (MAT). This method is based on systematic measurement and evaluation of minor magnetic hysteresis loops. The advantage of MAT method compared with the Barkhausen noise measurement is the deep penetration into the tested material. Further advantage is that the MAT signal is an electric one, allows remote controlled measurements of the radioactive materials. This is a multi-parametric, highly sensitive and robust procedure of magnetic “structuroscopy” introduced recently, see [12,13]. In a previous work of us [14] three series of Charpy samples, made of JRQ, 15K2MFA and 10ChMFT type steels were measured by MAT. The samples were irradiated by  $E > 1$  MeV energy fast neutrons with total neutron fluence of  $1.58 \times 10^{19}$ – $11.9 \times 10^{19}$  n/cm<sup>2</sup>. Regular correlation was found between the optimally chosen MAT degradation functions and the neutron fluence in all three types of the materials. In another work of us [15] Charpy samples were thermally treated by a special step cooling procedure, which caused structural modifications in the material. Charpy impact test were also performed and the results were compared with the magnetic parameters. It was found that this type of degradation can be easily followed by magnetic measurements.

In this work, applicability of MAT is studied for another type of samples. Cladded blocks of reactor presser vessel steels were measured. The influence of the special step cooling procedure was studied and magnetic measurements were performed on both (cladded and not cladded) sides of the samples.

### SAMPLE PREPARATION

Three clad block specimens made of 15H2NMFA material were measured. The chemical composition of the base material is given in Table 1. Blocks were cut from the forged ring as shown in Fig. 1. The size of blocks was 115mm x 50mm x 50mm. Cladding (7-10 mm thick) was on the top of the blocks. One of the measured blocks can be seen in Fig. 2. The cladding on the top is clearly seen. The clad surface of the real pressure vessels is usually made by submerged strip welding (a special way of the submerged arc welding) in three layers. It is basically austenitic structure but also contains 2-8% ferrite (magnetic phase). The surface is either grinded, or roughly machined to provide coupling for the ultrasonic testing.



*Fig. 1: Cutting of clad blocks from forged ring*



Fig. 2: One of the investigated blocks. Cladding is on the top.

Table 1. Chemical composition of 15H2NMFA material

C%	Mn%	Si%	S%	P%	Cr%	Ni%	Mo%	V%	Cu%	Co%	Sb%	Sn	As%
0.16	0.42	0.29	0.08	0.012	1.97	1.29	0.52	0.12	0.12	0.06	0.001	0.003	0.003

**THERMAL TREATMENT**

The samples were aged by thermal treatments. In the literature several step cooling technology can be found. Most of them are used for accelerated evaluation of thermal ageing sensitivity. The main consideration at the selection of the thermal treatment was to produce similar microstructural changes of which occurs at irradiation. Of course to produce the same microstructure as the result of irradiation is impossible by any other ageing technology. Quenching and annealing (selected for this purpose) may result similar hardness as irradiation, but very different microstructure. To avoid the rough change in grain sizes and microstructure the applied treatment temperature should be below the annealing temperature (i.e. 640-655°C) for this material. A step cooling technology elaborated for the 15H2NMFA steels and suits for the above mentioned requirements is known and published [16].

Two types of step cooling were performed. One sample was processed by “Thermal treatment 1”, another pieces was processed by “Thermal treatment 2”, and the third piece was not heat treated, it served as reference sample.

In Thermal treatment 1 slow heating was applied up to 600°C, then 10-20°C step cooling down to 500°C. The time spent on stable temperature at each steps are also given as it follows: 600°C/2h + 590°C/2h + 580°C/4h + 570°C/4h + 560°C/6h + 540°C/12h +530°C/12h + 520°C/12h + 510°C/18h + 500°C/24h. (See Fig. 1/a).

In Thermal treatment 2 slow heating was applied up to 600°C, then 10-20°C step cooling down to 500°C. The time spent on stable temperature at each steps are also given as it follows: 600°C/2h + 590°C/2h + 580°C/4h + 570°C/4h + 560°C/6h + 540°C/12h +530°C/12h + 520°C/12h + 510°C/18h + 500°C/24h + 490°C/144h + 480°C/162h +470°C/96h (See Fig. 1/b).

**CHARPY TRANSITION TEMPERATURE DETERMINATION**

Our final purpose is to compare the magnetic parameters measured in non-destructive way with the Charpy impact transition temperature shifts on irradiated samples. Since irradiated samples are radioactive and they can be tested only in hot laboratories, these thermally aged samples were used to develop the testing technology. Charpy impact testing was used to determine the 41 Joule transition temperature.

At the surface area (just under the cladding) the properties of the forgings are rapidly changing. The clad and unclad blocks cut from the forging surface area are not homogeneous and not isotropic, consequently these blocks can't be characterized by a single transition temperature value, only by a transition temperature distribution in the function of the distance from the forging surface (cladding and base material interface). The standard Charpy transition temperature distribution of the first 5 mm-layer under the surface cannot be measured directly due to the standard specimen size.

The near surface Charpy transition temperature of each block can be evaluated by cutting and Charpy testing only. Standard Charpy transition temperature measurement of the clad-forging interface and heat affected zone (about the first 5 mm) is not possible (study the property distribution within this zone using miniature specimens

or materials with simulated heat treatments may available and correlations can be used to guess the Charpy transition temperature). According to the available literature and data bases the Charpy transition temperatures, given in Table 2, were used for our evaluation. As seen from the Table 2, different TTKV41J values were used for top side and for bottom sides of the blocks.

Table 2. TTKV41J values, given in o C, estimated for top and bottom sides measured blocks

	As received	Thermal treatment 1	Thermal treatment 2
Top (0T)	-75.4 ± 25	-58.8 ± 25	-17.1 ± 25
Bottom (1/4T)	-50.4 ± 9.3	-33.8 ± 18	+8.1 ± 8

It is seen, that these thermal treatments caused a monotonous increase of the transition temperature (means embrittlement).

**MAGNETIC ADAPTIVE TESTING**

MAT investigates a complex set of minor hysteresis loops (from a minimum amplitude of the magnetizing field, with increasing amplitude by regular steps, up to almost saturation) for each sample of the measured series. It follows from the theory of Preisach model of hysteresis [17], that such a set of experimental data contains complex information on hysteresis of the measured material. A specially designed Permeameter with a magnetizing yoke was applied for measurement of families of minor loops of the magnetic circuit differential permeability. The flat samples were magnetized by an attached yoke. Size of the yoke was chosen to fit geometry of the samples: it was a C-shaped laminated Fe-Si transformer core with cross-section S=19mm x 16mm, total outside length 62 mm, and total outside height of the bow 55 mm. The magnetizing coil was wound on the bow of the yoke, with N=150 turns and the pick-up coil was wound on one of the yoke legs with n=50 turns. The magnetizing yoke, placed on the top of the sample (cladding) is shown in Fig. 3.

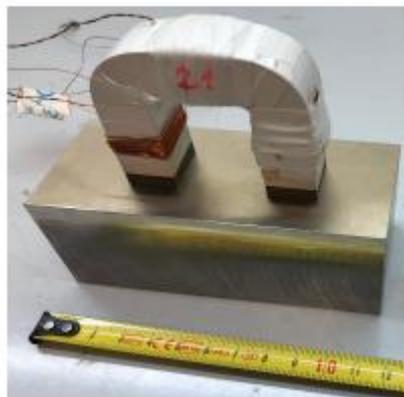


Fig. 3. The magnetizing yoke on the top of the clad sample

The magnetizing coil gets a triangular waveform current with step-wise increasing amplitudes and with a fixed slope magnitude in all the triangles. This produces a triangular time variation of the effective field in the magnetizing circuit and a signal is induced in the pick-up coil. As long as the field sweeps linearly with time, the voltage signal in the pick-up coil is proportional to the differential permeability of the magnetic circuit. The Permeameter works under full control of a PC computer, which registers data-files for each measured family of the minor “permeability loops”.

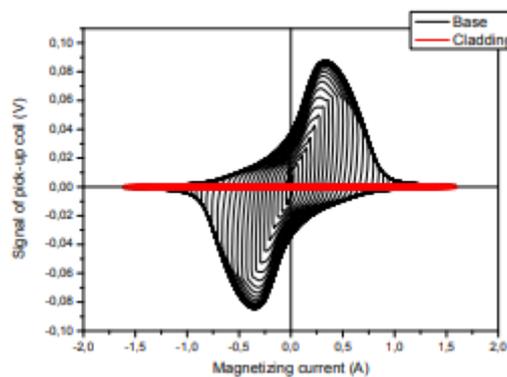
The experimental raw data are processed by an evaluation program, which divides the originally continuous signal of each measured sample into a family of individual permeability half-loops. The program filters experimental noise and interpolates the experimental data into a regular square grid of elements, m ° m(ha,hb), of a m-matrix with a pre-selected field-step. The coordinates of the m -elements represent the actual magnetic field value, ha, on the actual minor loop with amplitude hb. Each m-element represents one “MAT-descriptor” of the investigated material structure variation. The matrices are processed by another evaluation program, which divides values of their elements by corresponding element values of a chosen reference matrix (i.e. matrices standardization), and arranges each set of the mutually corresponding elements m of all the evaluated m-matrices into a m(x)-degradation function. Here x can be any independently measured parameter. In our case

these are the total time of the step cooling on one side, and the transition temperature on other side, determined independently in the samples as described above. For details of the whole MAT procedure see [13].

The samples are magnetized during the measurement by the magnetizing yoke, which is placed on the flat surface of the sample, as shown above. This experimental arrangement means an open magnetic circuit, because some magnetic flux is always scattered at the air gap between the yoke and the sample. The exact value of the magnetic field inside the sample is not known/measured in the used experimental arrangement. Because of this, instead of the magnetic field (given in A/m), the value of the magnetizing current (given in mA) is used as  $h_a$  and  $h_b$  when the  $m^o$   $m(h_a, h_b)$  matrix elements are given.

**RESULTS OF MAGNETIC MEASUREMENTS**

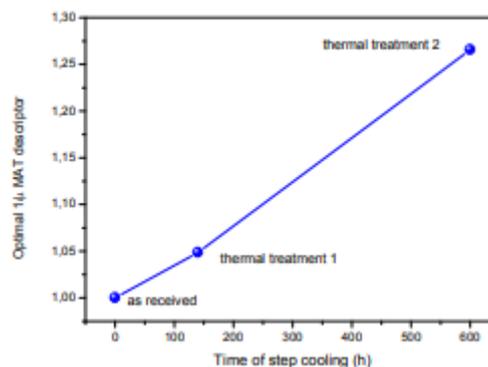
MAT measurements were performed on the three blocks. Both sides of the blocks were measured. The bottom side is the base material, while cladding is on the top side. The measured permeability loops are illustrated in Fig. 4. Regular loops with large amplitude were experienced on base (highly ferromagnetic) material, as shown by black lines in the Figure 4. The situation is different on the top side, which is covered by cladding. Cladding is an austenitic, almost paramagnetic material, which means practically a large air gap between magnetizing yoke and ferromagnetic part of sample. This is the reason of the very weak signal.



*Fig. 4: Permeability loops, measured on two opposite sides of the reference (as received) sample. Base: bottom side (black), cladding: top side (red).*

**Base material**

As the first step of evaluation, MAT descriptors were considered as functions of the time of step cooling. Fig.5 shows the result, obtained on the base material (bottom side). Here the optimally chosen MAT descriptor can be seen as a function of the time of step cooling. The optimally chosen descriptor is characterised by  $1/\mu(h_a=700\text{mA}, h_b=800\text{mA})$  values.



*Fig. 5: Correlation between normalized MAT parameters and time of step cooling, measured on base material.*

As it is seen in the graph, regular, monotonously increasing correlation was found between the optimally chosen MAT degradation function and the time of step cooling. The magnetic parameters, due to 142 hours heat treatment were modified by about 5% compared with the reference sample. The second, 482 hours heat treatment caused additional changes in the structure of the material, the magnetic parameters were modified by about 27% compared with the reference. As the second step of evaluation, MAT descriptors were also considered as functions of the transition temperature. The result is given in Fig. 6. The correlation between

MAT parameters and transition temperature seems to be very close to a linear one. Because of this a linear fit was done on the points and shown in Fig. 6. We are aware that any fit on three points is usually not justified, so this line, which connects the points is considered only as for driving the eye. But the monotonous (at least closely linear) correlation is evident.

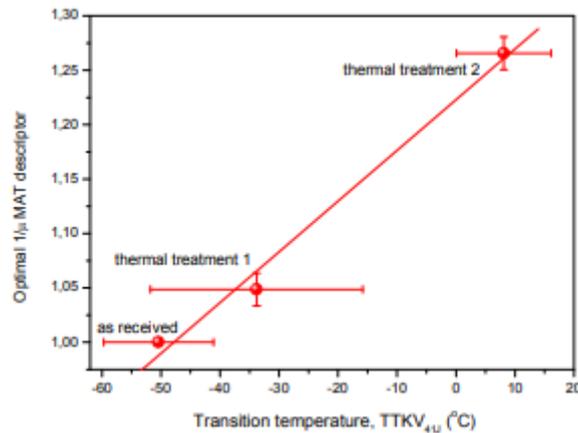


Fig. 6: Correlation between normalized MAT parameters and transition temperature, measured on base material.

**Cladding**

As is it shown in Fig. 4, if the measurement is performed on the cladding (top side) the maximal induced signal is much less (0.0015 V) compared to the case of bottom measurement (0.08 V) by using the same settings of measurement electronics are optimal for the base material, and this way the obtained permeability curves are very noisy. The reason is the very big air gap between ferromagnetic base material and magnetizing yoke. Anyway, some local maximum can be observed even in this case (Fig. 7/a). This maximum is seen better, if one single minor loop is selected (in Fig. 7/a all of the minor loops are seen) as done in Fig. 7/b. (here the noise is also magnified, of course). But, if a smoothing of points is done by adjacent averaging of the points, as shown in Fig. 7/c, the maximum becomes well visible. This fact gives a chance that the MAT evaluation results usable correlation between MAT descriptors and independent parameters even if the measurement is performed through the cladding. The result of evaluation is shown in Fig. 8. Excellent correlation was found between MAT parameters and step cooling time/transition temperature. The result is the same as in case of measurements, performed on the base material.

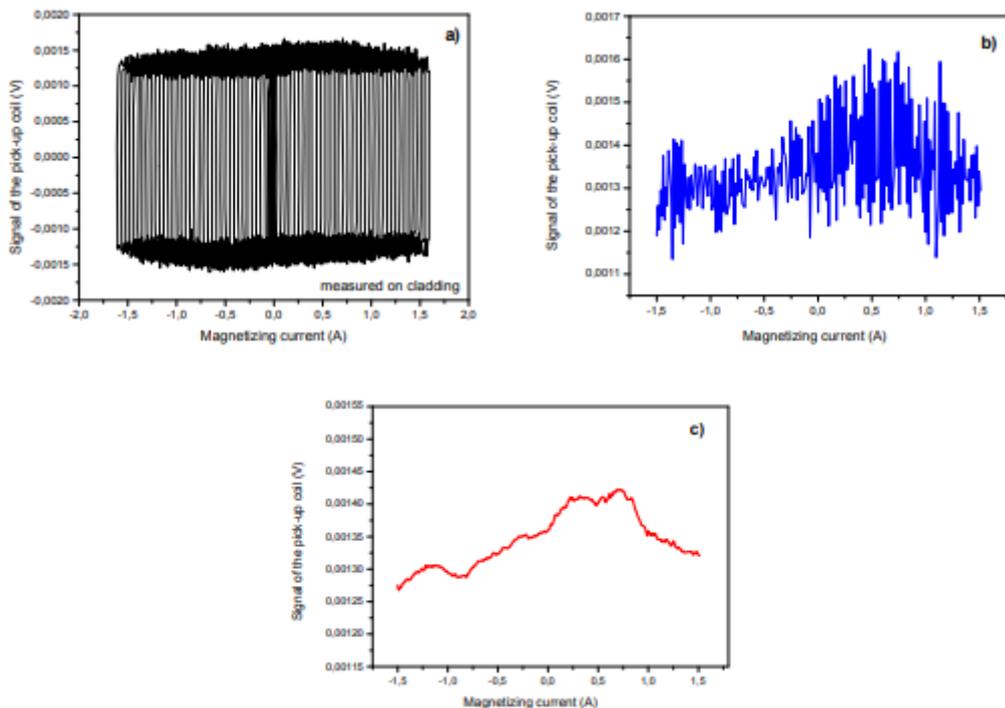


Fig. 7: Permeability curves, measured through the cladding. Original measurement, series of minor loops (a), one single minor loop (b), and smoothed single minor loop (c).

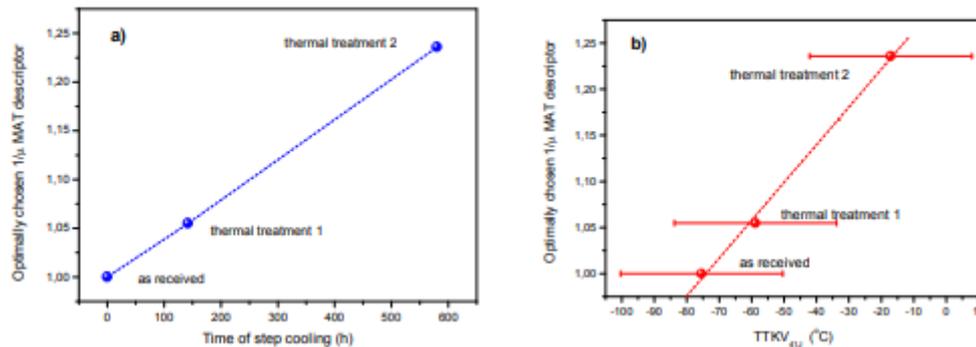


Fig. 8: Correlation between normalized MAT parameters and time of step cooling (a) and between normalized MAT parameters and transition temperature (b), measured on cladding.

## DISCUSSION AND CONCLUSIONS

It was shown that the method of Magnetic Adaptive Testing was found as a sensitive and reliable way to characterize the structural changes in reactor pressure vessel material. A definite and monotonous correlation was found between the time of step cooling process and MAT descriptors. The modification of the transition temperature due to these thermal treatments can also be followed by this nondestructive method, and a closely linear correlation was found between MAT descriptors and Charpy 41 Joule transition temperature. An important and novel result of the present work is, that the ferromagnetic base material can be inspected even through the relatively thick cladding, and the measurement through the cladding result the same correlation between magnetic parameters and independent variable as obtained by direct measurement of base material. This fact proves the unique applicability of MAT in nondestructive inspection of degradation of ferromagnetic objects. Another advantage (compared to traditional magnetic methods) of the MAT method is that the magnetic saturation of the samples is not required. It is possible to apply a small simple yoke for both the magnetization and the measurement of magnetically open samples. This method does not give absolute values of the traditional magnetic quantities, 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.

For the successful application of the MAT method, first it is necessary to make comparative, traditional, destructive measurements on a series of samples, for “teaching” the MAT. This teaching procedure determines the optimum degradation function/functions, and the method of Magnetic adaptive testing is best adapted to the investigated task in this way. Then, this/these chosen optimum degradation functions will serve as sensitive parameters for practical measurements of unknown samples (of the same kind) to be investigated.

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## REFERENCES

- [1] Diego Ferreño, Iñaki Gorrochategui and Federico Gutiérrez-Solana, “Degradation Due to Neutron Embrittlement of Nuclear Vessel Steels: A Critical Review about the Current Experimental and Analytical Techniques to Characterise the Material, with Particular Emphasis on Alternative Methodologies”, chapter in “Nuclear Power - Control, Reliability and Human Factors”, ISBN 9789533075990, Edited by: P.Tsvetkov, published by InTech, September 2011, Available from: <http://www.intechopen.com/articles/show/title/non-destructive-testing-for-ageing-management-of-nuclear-power-components>
- [2] S. Takahashi, S. Kobayashi, H. Kikuchi, Y. Kamada, “Relationship between mechanical and magnetic properties in cold rolled low carbon steel”, J. Appl. Phys. (2006) vol.100, p.113908
- [3] I. Tomáš, J. Kadlecová, R. Konop, M. Dvořáková, “Magnetic nondestructive indication of varied brittleness of 15Ch2MFA steel”, Proceedings of 9th International Conference on Barkhausen Noise and Micromagnetic Testing (ICBM9), Hejnice, Czech Republic, June 2011, ISBN 978-952-67247-4-4 (paperback), ISBN 978-952-67247-5-1 (CD-ROM), pp.55-63

- [4] S. Takahashi, H. Kikuchi, K. Ara, N. Ebine, Y. Kamada, S. Kobayashi, M. Suzuki, "In situ magnetic measurements under neutron radiation in Fe metal and low carbon steel", *J. Appl. Phys.* (2006) Vol. 100, p.023902
- [5] G. Dobmann, I. Altpeter, M. Kopp, M. Rabung, G. Hubschen, "ND-materials characterization of neutron induced embrittlement in German nuclear reactor pressure vessel material by micromagnetic NDT techniques", *Electromagnetic Nondestructive Evaluation (XI)*, 2008, p.54, IOS Press, ISBN 978-1-58603-896-0
- [6] L. Vandebossche, "Magnetic Hysteretic Characterization of Ferromagnetic Materials with Objectives towards Non-Destructive Evaluation of Material Degradation", Gent University, Gent, Belgium, PhD Thesis 2009
- [7] F. Gillemot, S. Pirfo Barroso, "Possibilities and difficulties of the NDE evaluation of irradiation degradation", *Proceedings of 8th International Conference on Barkhausen Noise and Micromagnetic Testing (ICBM8)*, 2010, (ISBN 978-952-67247-2-0)
- [8] S. Pirfo Barroso, M. Horváth, Á. Horváth, "Magnetic measurements for evaluation of radiation damage on nuclear reactor materials", *Nuclear Engineering and Design* 240 (2010) 722-725
- [9] S. Kobayashi, F. Gillemot, Á. Horváth, R. Székely, "Magnetic properties of a highly neutron-irradiated nuclear reactor pressure vessel steel", *Journal of Nuclear Materials* 421 (2012) 112-116
- [10] B. Minov, "Investigation of the hardening in neutron irradiated and thermally aged iron-copper alloys, on the basis of mechanical and magnetic relaxation phenomena", Gent University, Gent, Belgium, PhD Thesis 2012
- [11] G. Dobmann, "Non-Destructive Testing for Ageing Management of Nuclear Power Components", in *Nuclear Power - Control, Reliability and Human Factors*, P.Tsvetkov (Ed.), ISBN: 978-953-307-599-0, InTech (2011), Available from: <http://www.intechopen.com/articles/show/title/non-destructivetesting-for-ageing-management-of-nuclear-power-components>
- [12] I. Tomáš, "Non-Destructive Magnetic Adaptive Testing of Ferromagnetic Materials", *J.Mag.Mag.Mat.* 268/1-2 (2004) 178-185
- [13] I. Tomáš, G. Vértesy, "Magnetic Adaptive Testing", in *Nondestructive Testing Methods and New Applications*, M.Omar (Ed.), ISBN: 978-953-51-0108-6, (2012), InTech: <http://www.intechopen.com/articles/show/title/magnetic-adaptive-testing>
- [14] I. Tomáš, G. Vértesy, F. Gillemot, R. Székely, "Nondestructive Magnetic Adaptive Testing of nuclear reactor pressure vessel steel degradation", *J. Nuclear Materials*, 432 (2013) 371-377
- [15] G. Vértesy, A. Gasparics, I. Szenthe, F. Gillemot, I. Uytendhouwen, "Inspection of reactor steel degradation by magnetic adaptive testing", submitted for publication to *Materials*
- [16] S.U. Potapov, J.R. Hawthorne, "The Effect of Residual Elements on 500°F Irradiation Response of Selected Pressure Vessel Steels and Weldments", *Naval Research Laboratory Rep.* 6803 (1968).
- [17] I.D. Mayergoyz, "Mathematical models of hysteresis", Springer-Verlag, New York, 1991