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# Detection of neutron irradiation induced degradation of reactor steel by magnetic method

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**Abstract.** A novel nondestructive method, based on step by step measurement of minor magnetic hysteresis loops was used to determine the neutron irradiation induced degradation of nuclear reactor pressure vessel steel material. This method is called Magnetic Adaptive Testing (MAT), and two types of reactor steel material were investigated: base material type 22NiMoCr37 and weld material type 18MND5. Charpy geometry samples were irradiated by neutrons at the BR2 in SCK•CEN. The neutron irradiation generated structural changes in the material. As a result of our measurement, it was shown that this degradation could be detected by MAT measurements. Charpy impact tests were also done on the investigated samples and the results of these measurement were compared with the nondestructively determined magnetic quantities. It was shown that a closely linear correlation existed between the ductile-to-brittle transition temperature (DBTT) and MAT parameters.

## 1. Introduction

Pressurized water reactors are applied in majority the nuclear power plants. The reactor pressure vessel (RPV) is the most important part from point of view of safety. The lifetime of the power plant is determined by the lifetime of RPV. It is exposed at high pressure to neutron radiation and is operated at elevated temperature. Reactor pressure vessel material is degraded during service because of these effects. This material is a high quality low alloyed ferromagnetic CrNiMo or CrMoV steel, and its properties must be monitored during the whole lifetime of the power plant. The forged rings and their welds at the core level are the most critical parts. Regular inspection of reactor pressure vessel material is performed by the so called surveillance programmes. During this programme surveillance samples (Charpy samples) placed inside of the reactor are exposed to the same conditions (neutron radiation and thermal conditions) as the pressure vessel walls. Charpy impact, tensile stress and fracture toughness tests are performed on surveillance samples. All these tests are destructive, and sooner or later there are no more samples (especially if the life time of nuclear power plants is extended) for mechanical tests.

The most frequently applied method is the well-known, conventional, mechanical Charpy impact test, which is performed on 55x10x10 mm<sup>3</sup> size samples. Samples are periodically taken out from the reactor pressure vessel and inspected. These tests have been used as a standard testing method of material toughness for a long time [1]. The fixed standard size and shape specimens to be tested are



broken by a swinging hammer, and the absorbed energy by the fracture of the sample is a typical property of the material. This energy depends on the temperature at which the test is being performed. As a definition, ductile-to-brittle transition temperature (DBTT) is the temperature where the absorbed energy is 41 J. One single sample is not enough for the reliable determination of DBTT, usually several (at least a dozen) of surveillance specimens are used for one single DBTT characterization. The surveillance specimens became highly radioactive during irradiation. It means that they can be measured only in hot cell laboratories. This type of inspection is very costly. Because of this application of nondestructive tests instead, or at the beginning parallel with the present surveillance techniques is very important.

There exist numerous nondestructive methods, and those which are based on electromagnetic measurements are encouraging for potential practical applications. Among them magnetic hysteresis methods are rapidly developing and occupy a special position. A close correlation was found between modification of the microstructure of steels due to different influences (e.g. mechanical load, thermal treatment, fatigue, corrosion, etc.) and between magnetic behaviour, which is reflected the best in the magnetic induction of samples under an applied magnetizing field. Domain wall movements  $\delta$  which influence the shape of hysteresis curve  $\delta$  and dislocations in the ferromagnetic material are both affected by the micro-structural defects. This direct correlation between magnetic and mechanical hardness of the same ferromagnetic material is well-known, and also well understood.

Magnetic methods are not expensive, mobile and technically simple. A review of magnetic methods in nondestructive testing is given in [2]. An advantage of these methods is that they can be applied via electrical cables and coils, such a way it is possible to place the necessary electronic equipment outside the protected space where the irradiated, and due to irradiation radioactive specimens are kept. Some measurements were published, which proved the practical applicability of magnetic methods in the inspection of steel degradation. For example cold rolling, special thermal processing and fast neutron irradiation effect on nuclear pressure vessel steels and welds were tested, see e.g. [3-11].

A novel method, Magnetic adaptive testing (MAT) has been developed recently and this technique seems to be a promising candidate for nondestructive inspection of steel degradation. In MAT method series of minor magnetic hysteresis loops are measured [12,13], permeability and/or hysteresis matrices are calculated and the matrix parameters of degraded material are compared with the same parameters of virgin samples. Such a way so called magnetic descriptors are generated, which reflect any modification of material structure with high sensitivity. This procedure is multi-parametric and robust. Details of technique is described detailed in [13].

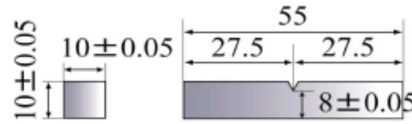
In a previous work of us [14], different samples, made of JRQ, 15CH2MFA and 10ChMFT type steels were investigated by MAT. The samples were irradiated by  $E > 1$  MeV energy fast neutrons with total neutron fluence of  $1.58 \times 10^{19}$   $\delta$   $11.9 \times 10^{19}$  n/cm<sup>2</sup>. Regular correlation was found between the optimally chosen MAT degradation functions and the neutron fluence in all three types of the materials. In the present work the applicability of MAT is shown for the detection of the degradation of the reactor steel, caused by neutron irradiation. Our final purpose is to compare the magnetic parameters measured by this non-destructive way with the Charpy impact testing shifts on irradiated samples.

## 2. Sample preparation

Standard Charpy specimens from 22NiMoCr37 type base and from 18MND5 type weld materials were measured. These steel grades are members of the typical RPV steel groups: Mn-Ni-Mo steels (western RPV design). The geometry of samples is shown in Fig. 1 and the chemical compositions of the samples are given in Table 1.

The samples were prepared, irradiated and measured at the SCKCEN Belgian Nuclear Research Centre. The samples were irradiated in the BR2 reactor at 260 °C temperature by  $E > 1$  MeV energy fast neutrons with total neutron fluence in the range of  $4.02 \times 10^{19}$   $\delta$   $8.95 \times 10^{19}$  n/cm<sup>2</sup>. Since irradiated

samples are radioactive, they can be measured only in hot cell laboratories. Charpy impact testing (ASTM-23-16b [15]) was used to determine the 41 Joule transition temperature ( $T_{41J}$ ). In this paper the ductile-to-brittle transition temperature shift ( $\Delta DBTT$ ) is the difference between the neutron irradiated  $T_{41J}$  and the as-received (base-line)  $T_{41J}$  which is a measure for the embrittlement of the material.



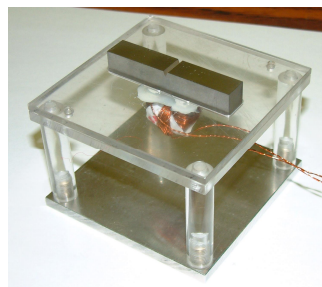
**Figure 1.** Dimensions (in mm) and shape of the measured Charpy samples. (Each sample had a V-notch at one face.)

**Table 1.** Chemical composition as measured by Optical Emission Spectroscopy of the measured materials (values are given in wt%)

Material	C	Si	Mn	P	S	Cr	Mo	Ni	Cu
18MND5 weld	0.09	0.23	1.21	0.018	0.009	0.12	0.49	0.96	0.13
22NiMoCr37 base	0.20	0.25	0.87	0.009	0.007	0.39	0.49	0.85	0.06

### 3. Magnetic adaptive testing

Set of minor hysteresis loops is measured (starting from zero magnetizing field, then magnetizing field is increased step by step up to closely saturated state). This measurement is performed on each sample within the given set of samples. A specially designed equipment is used, which is controlled by a PC. A magnetizing yoke (a C-shaped laminated Fe-Si transformer core) is placed to the sample surface. Size of the yoke fits geometry of the specimen. In our case the cross-section is  $S=10 \times 5 \text{ mm}^2$ , the total outside length is 18 mm, and total outside height of the bow is 22 mm. There are two coils, both of them wound on the yoke. The magnetizing coil has 200 turns and the pick-up coil has 75 turns. The photo of the sample holder, which was built specially for hot cell measurement is shown in Fig. 2. The magnetizing yoke is placed below the sample, the samples can be replaced within the hot cell by manipulator. The whole electronic unit, which controls the measurement and manages the data acquisition is placed outside of the hot cell.



**Figure 2.** The sample holder with a Charpy geometry sample to be measured.

An evaluation program processes the measured data. Experimental noise is filtered by the program and the experimental data are interpolated to a square grid of elements,  $\mu \equiv \mu(h_a, h_b)$ , of a  $\mu$ -matrix (or permeability matrix), where  $h_a$  is the actual magnetic field value and  $h_b$  is the amplitude of the actual minor loop. These  $\mu$ -elements are designated as  $\delta$ MAT-descriptor and they characterize the material structure variation of the investigated specimens.

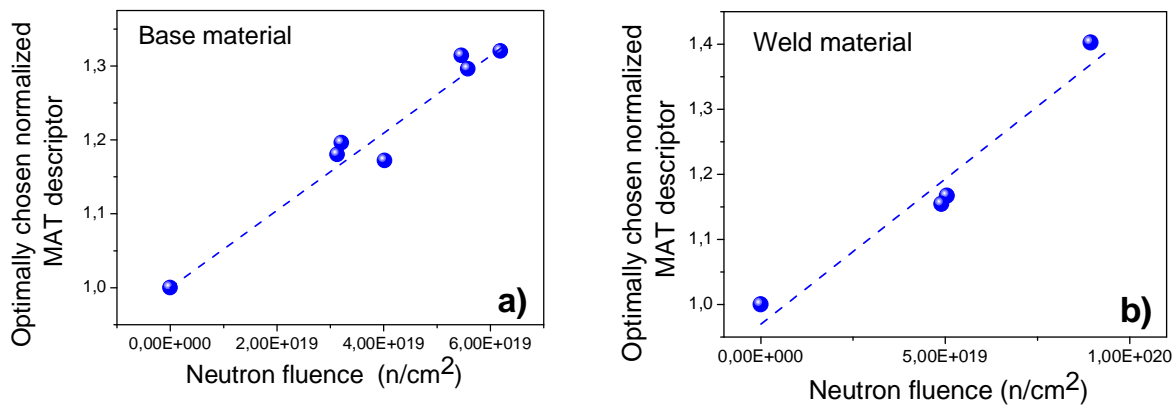
Another evaluation program processes the matrices. All matrix elements are divided by the corresponding element of the reference matrix (matrix elements of the reference sample). As a result,

normalized  $\mu(x)$ -degradation functions are generated, which reflect the magnetic response of the material to any kind of degradation.  $x$  can be any parameter, which characterizes material degradation. In our case these are the neutron fluence on one side, and the shift of the transition temperature ( $\Delta$ DBTT) on the other side, determined independently in the samples. Taking into account the accuracy of the measurement, and analysing the measured MAT parameters, the error of the MAT descriptors is estimated about 5%.

#### 4. Results and discussion

MAT measurements were performed on two series of the above described reactor steel samples before and after neutron irradiation. The matrix elements from the reference (not irradiated) samples were used for normalization. In the graphs (Figs. 3 and 4) shown below the modification of the MAT descriptors due to neutron irradiation are presented (normalized MAT descriptors) as functions of two independent parameters.

In Fig. 3 the optimally chosen MAT descriptor can be seen as a function of the neutron fluence for both materials. The optimal matrix elements were calculated from the measured permeability loops. Optimal means that these descriptor has the largest sensitivity with respect to the independent parameter (neutron fluence and  $\Delta$ DBTT), which characterizes the material degradation, and at the same time they are reliable and well reproducible. The exact description on how these optimal descriptors are determined is given in Ref. [13]. The optimally chosen MAT descriptor is characterised by  $\mu$  ( $h_a=500\text{mA}$ ,  $h_b=1150\text{mA}$ ) values. A linear correlation was found between magnetic parameter and neutron fluence which is monotonously increasing. A 30% increase of the MAT descriptor was detected due to  $6 \times 10^{19} \text{ n/cm}^2$  neutron fluence in case of the base material, compared with the not irradiated sample; and close to 40% increase due to  $8.5 \times 10^{19} \text{ n/cm}^2$  neutron fluence in case of the weld material.



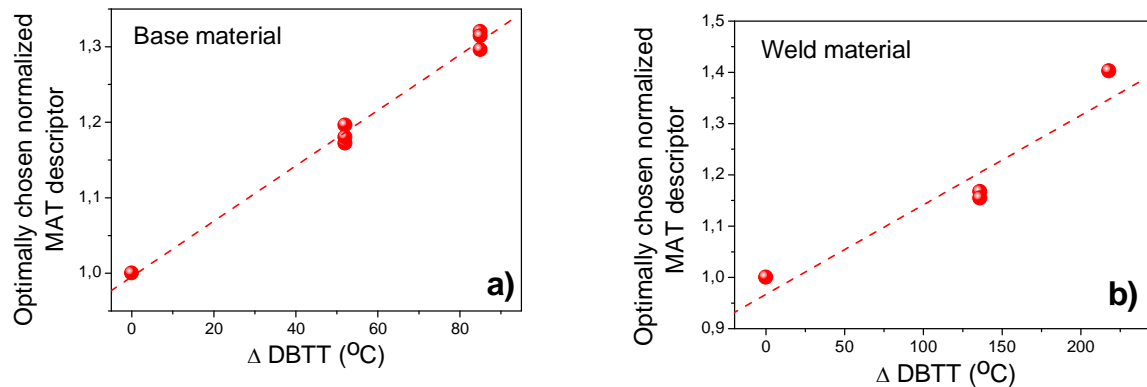
**Figure 3.** Optimally chosen normalized MAT descriptor ( $\mu$  degradation functions) as function of neutron fluence for 22NiMoCr37 type base material (a) and for 18MND5 type weld material (b).

As a second step of evaluation, the optimally chosen MAT descriptors were considered as a function of the transition temperature shift  $\Delta$ DBTT (see Fig. 4) for both materials. The optimally chosen MAT descriptor is characterized by  $\mu$  ( $h_a=500\text{mA}$ ,  $h_b=1150\text{mA}$ ) values in this case, too.

It can be seen that a similar correlation was found between the MAT descriptors on the one hand and the transition temperature shift and the neutron fluence on the other hand. In this case, an averaged value of the transition temperature shift is used for samples that were irradiated with similar (but not exactly same) neutron fluence.

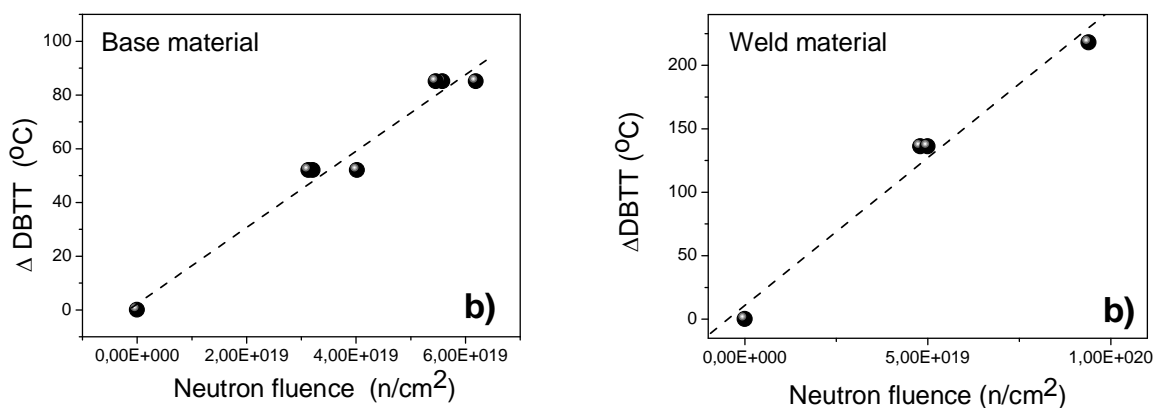
The correlation between the magnetic parameters and the neutron fluence shows that the material properties were changed due to the neutron irradiation. In other cases, see e.g. [14], also a monotonous, but not linearly increasing correlation was found. In general, the neutron fluence values

alone are useless for the characterization of the material degradation, because the radiation damage is measured through the effect of the irradiation on the mechanical properties (amount of hardening and embrittlement). But in this case, for the two investigated series of samples, a similar correlation was found between the optimally chosen MAT descriptors; and the neutron fluence and transition temperature shift. It is not surprising, because if the DBTT is considered as a function of the neutron fluence, the correlation is also linear, as shown for the two materials in Fig. 5. For the correct interpretation of the material embrittlement the modification of the transition temperature is the most important parameter.



**Figure 4.** Optimally chosen normalized MAT descriptor ( $\mu$  degradation functions) as a function of the transition temperature shift for a 22NiMoCr37 type base material (a) and for a 18MND5 type weld material (b).

The linear fit on the measured points is to lead the eye to reflect more visible the character of the correlation. It does not reflect a strict mathematical fitting, because of the limited number of measured points.



**Figure 5.** Correlation between the transition temperature shift and the neutron fluence for a 22NiMoCr37 type base material (a) and for a 18MND5 type weld material (b).

## 5. Conclusions

It was shown that the Magnetic Adaptive Testing method can characterize the structural changes in a reactor pressure vessel material in a sensitive and reliable way. A definite and monotonous correlation was found between the neutron fluence and the MAT descriptors for both types of investigated materials. The modification of the transition temperature due to neutron irradiation can also be

followed by this nondestructive method. A linear correlation was found between the MAT descriptors and the Charpy 41 Joule transition temperature.

As a general conclusion, it was shown that the MAT method is suitable for the nondestructive structural inspection of reactor pressure vessel steel: degradation functions characterize structural changes of this material easily and reliably. Our presently described results are in good coincidence with previous results, performed on other types of reactor steel.

MAT is also an advantageous method, from point of view of measurement, because there is not necessary to saturate the sample magnetically, which is almost impossible in case of an open magnetic circuit. As has been proved above, it is also possible the measurement of highly radioactive specimens. The evaluated magnetic descriptors are not absolute parameters, but MAT is powerful tool to perform comparative measurements, and such a way to detect changes in the material behaviour due to any type of degradation. However, calibration of MAT procedure should be done on a reference series of samples (by comparing magnetic descriptors with independently, destructively measured parameters) and then the degradation of any unknown sample can be determined.

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