# Magnetic measurements to evaluate irradiation effects on Reactor Pressure Vessel high nickel steel

Luigi Debarberis[1], Soraia Pirfo[2, 1] and Ferenc Gillemot[3]

[1] Institute for Energy, Joint Research Centre of the European Commission, P.O.Box 2, 1755ZG Petten, The Netherlands
[2] ASI Consulting, Olvasztar u 22, Erd, H-2030, Hungary
[3] Atomic Energy Research Institute, P.O.Box 49, H-1525, Budapest, Hungary

**Abstract.** With the aim to characterise material changes due to irradiation exposure, the possibility of using magnetic measurements have been continuously studied. In this work, a further study is described dealing with a reactor pressure vessel (RPV) shell of a Russian WWER-1000 nuclear power plant type.
The shell has been cut into blocks and finally several sets of Charpy specimens were manufactured and a large fraction of them exposed to neutron irradiation in the High Flux Reactor (HFR), Petten, of the JRC-IE, in order to assess irradiation effects on the given 15Kh2MNFAA low alloy steel. The irradiation was carried out at 290 ºC and a t a fluence of 1.8 1023 n.m-2 (E>1 MeV).
The scope of this paper is to present the evaluation of the effect of irradiation on magnetic properties and its possible relation to mechanical properties changes, as a consequence of embrittlement on irradiated Charpy specimens. In this case the Charpy specimens were standard full size CV-n type with very well known cutting orientation with reference to the original pressure vessel shell. This is very important since the cutting orientation of specimen often have such strong effect on the magnetic Barkhausen signal to mask the irradiation effects on specimens
The magnetic measurements carried out included Barkhausen signal and the so called $R_X I$ value; which is the relative induction values to changing on intensity of magnetic flux within the specimen. The results highlight the necessity to treat magnetic measurements on Charpy specimens in a statistical way.

## Introduction

Material condition is a key issue in predicting the lifetime evolution and life management programme of structural components. Many components, as the reactor vessel steels (RPV), are subject to long-term temperature and significant neutron irradiation doses. The irradiation effects can lead to the degradation of materials physical and mechanical properties. Such degradations are known to play an important role on structural integrity and on the safety of nuclear power plants, as it is the particular case of embrittlement of material due to neutron irradiation.

A better understanding of the irradiation effects on structural steels will help among others the development of predictive models of evolution of material degradation under irradiation. A thorough understanding of changes on microstructure and dependent factors, e.g. chemical composition, fabrication and thermal treatments involved in neutron embrittlement of steel is of course progressively being pursued. As much more information is gathering from experimental studies a better overall understanding is provided which is also an added value to the theoretician's work.

## Theoretical Basis

Magnetism measurements can be a material fingerprint expected to be sensitive to several of the same material structure features that are modified under irradiation. So it is worthwhile the efforts spent in order to better identify and understand the magnetic measurements and their changes under irradiation, mainly the ferromagnetic ones.

The mechanistic explanation of the observed behaviour on steels exposed to irradiation embrittlement, hardening and others effects are identified as features like irradiation-induced precipitation of impurity, matrix atom displacement damage, intergranular embrittlement due to P segregation at grain boundaries and others. [1]

The domain regions are formed within ferromagnetic material in order to minimise the system energy, energies originated of magnetism itself, as from balance among dipoles, spin orientation, magneto anisotropy and affect by crystalline structure.

Domain walls movements are not smooth but are happening in a jerky way, they were first identified by Professor H. Barkhausen, in 1919, receiving from him the name of Barkhausen noise. The discovered noise is a signal translated in electrical sense voltage or acoustical pressure wave propagation, and finds its origins altogether from the magnetic domain instability towards a new configuration intermingles to structural disorder.

It is generally measured by a system of magnetising yoke and pick-up encircling coil. The measurement system is based on the magnetic induction changes versus time.

Micro-structural irradiation induced features like precipitates and dislocations can affect domain walls formation and movement. That is why several studies had been done on Barkhausen and other micromagnetic measurements of irradiated material; the topic is still far from conclusive but anyhow contributing to elucidate the mechanisms involved in this complex subject. [2, 3, 4]

The MBN (Magnetic Barkhausen noise or emission), is a signal on its own and it has a characteristic profile. The different signal parameters e.g. signal energy, frequency, intensity, amplitude distribution can be evaluated and correlated to the observed material degradation caused by irradiation on the specific material, condition and specimens. The research seems to be promising and much is still to be done as several signal characteristics and experimental methodologies can be tried to assess reactor pressure vessel material state.

At JRC-IE a first effort was dedicated to analyse the Barkhausen signal and induced magnetic response to an applied magnetic field utilising small specimens: KLST mini-Charpy impact test specimens 3 x 4 x 27 mm dimensions. Such small size specimen represents a challenge to the magnetic measurements in irradiated condition; especially in hot-cell environment. [5]

The second part of the research, presented in this paper shows results obtained on as-received and irradiated samples with different cutting orientation in relation to radial, circumferential and transversal direction of a shell of a VVER-1000 reactor pressure vessel.

## Sample specification of RPV Machined Samples

Diverse blocks were cut from the parts of the reactor pressure original shell of the VVER-1000 reactor pressure vessel. The vessel was manufactured from steel 15Kh2NMFAA, forged and heat treated according to Russian specifications and norms. The nominal composition according to the manufacturer data is given in table 1; the steel is high nickel type differently from VVER-440 case.

The pressure vessel has a wall thickness of 280 mm and it is cladded by a corrosion resistant austenitic steel cladding.

**Table 1.** Chemical composition, weight %, of VVER1000 type steel

| C | Mn | Si | P | S | Ni | Cr | Mo | Cu | V |
|---|---|---|---|---|---|---|---|---|---|
| 0.17 | 0.46 | 0.30 | 0.008 | 0.01 | 1.26 | 2.2 | 0.5 | <0.08 | 0.1 |

The extracted blocks were in turn cut on smaller ones from which different samples were machined. Machined samples represent different position on the blocks, thus different locations in the vessel.

The group of samples cut from the blocks are representative layers from different depths along the 280 mm vessel thickness.

Another important factor about the sample cuttings is that they were cut in the different directions related to three main vessel orientations, i.e. radial, circumferential and longitudinal. All this cutting process has been carefully documented and samples identified according to their originating block.

The results presented here are obtained on specimens from different vessel thickness depths of two blocks identified as 1.6 and 4.1 at different longitudinal position. Moreover specimens have been cut from the blocks following different orientations related to main vessel directions; both on as-received as well as on irradiated conditions. The pressure vessel and specimen cut diagram is shown in Figure 1.

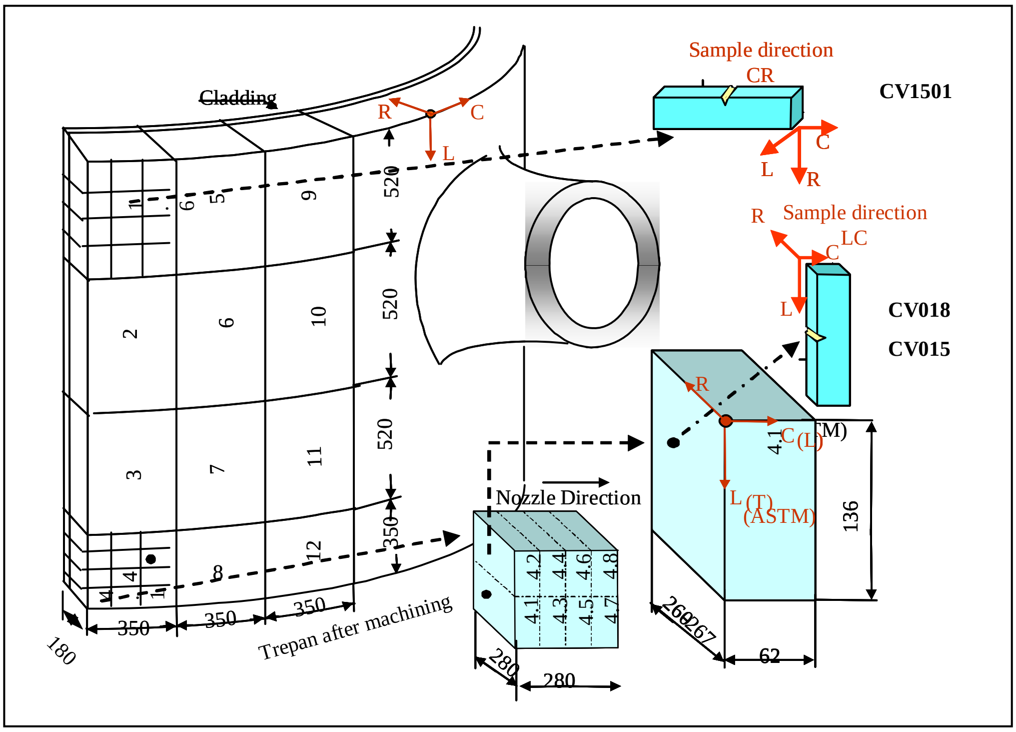


**Figure 1.** Pressure vessel and specimens cut diagram

Thus the distribution of properties in axial and radial directions of the VVER-1000 shell and the effects on irradiation is analysed in this studied.

The samples here examined were consisted of full (*unbroken*) Charpy type. The irradiated samples have been exposed in the High Flux Reactor of Institute for Energy, Joint Research Centre of the European Commission, Petten, The Netherlands. The irradiation was carried out at a fluence of 1.8 $10^{23}$ n.m$^{-2}$ (E>1 MeV) at 290 ºC.

The Charpy specimens have their longer lengths side; 55 mm, oriented in two different directions in according to the block of origin, from which specimen were machined. Also depending on the layer of origin specimens are representative of different depths within the thickness of the vessel wall. Block, layer, its depths, directions and identification are summarised in Table 2.

**Table 2.** Originating block; orientation and samples respective identifications. X is the specimen identification number

| Block Source | Specimen Orientation | Specimen Identification |
|---|---|---|
| 1.6 | Longer side along vessel circumferential direction. | CVX |
| 4.1 | Longer side along vessel longitudinal direction. | CV0X |

## Micromagnetic NDT

*Method*

The magnetic measurements were done by contacting probe which consists of a magnetic yoke and a pick-up coil. Several electromagnetic process influences the captured signal and some can separately be obtained with discriminatory procedures; e.g. filtering.

The Stresstest 20 Barkhausen device (equipment produced by Metalelektro for stress measurements and adapted to the required specifications for material characterisation) offers the following main characteristics:

- provide the unfiltered/response signals
- the excitation frequency are relatively low compaired to others electromagnetic tests
- the excitation frequency options are 10 and 100 Hz
- the signal can be captured at one excitation level or through level scanning

In the *scanning excitation mode* at each excitation level the signal is measured and then the excitation is increased by a certain value, step by step, the signal is again measured and the process proceed by level or excitation intensity being increased till the maximum value. The result is a curve of the measured values versus increasing applied excitation.

Actually the measured values are four: applied excitation signal, output signal before filtering, the signal after filtering consisting of the Barkhausen signal itself and a value named $R_xI$ (Relative value inversely proportional to Induction).

All the signals are monitored by oscilloscope and are recorded to PC. The Barkhausen signal device measures and stores the RMS (*Root Mean Square*) values together with the relative value inversely proportional to the induction, $R_xI$. Such value is a function of the flux intensity, the surface condition, the contact among the joke and the specimen and the material properties, such as the magnetic permeability and conductivity.[3]

Irradiated specimens must be remotely and carefully handled by authorized personal in hot cell conditions. Hot cell measurements requires easily handling, fast and hermetically closed (due to contamination issues) probes.

The same probe was used to test the non-irradiated and irradiated specimens.

The specimens are placed with about their half length contacting the yoke and sensor so that effective measurements are done on half specimen.

To evaluate the irradiation the two opposite faces of the specimen were measured by positioning the specific faces to be measured contacting sensor and magnetic yoke.

The faces were the Charpy specimen faces along the length where no identification number was present. This faces are the one with the notch machined on it, identified as “N” notched face, and the one directly opposite to it where the hammer strike the sample, “B” back face. As only half face is placed on the sensor and the notch is not placed among the yoke poles we can assumes that the notch does not cause a big influence on the flux distribution, even so there is medium boundary proximity, i.e. material surface to air.

## Methodology

A calibration procedure was performed to avoid deviations on measurements. The calibration was done at the start of a testing day or a new specimen series.
Every specimen has been measured three times on each face, N and B faces.

This was done to ensure that a proper contact between the specimens and the magnetising joke would be achieved. Besides monitoring signals for disturbance and the $R_xI$ values this aim was pursued with further statistical trails. In fact a bad contact among the joke and the specimen will be demonstrated to result on flux difference within the specimen and consequently the measured signals. [5]

If at least two of the three results resulted within a very limited scatter than those test results were accepted as valid. Otherwise more measurements were performed to conclude on discarding or accepting them.

As previously mentioned, the applied excitation, the output before filtering and the Barkhausen signal were continuously screened following their profile ready to stop and restart measurement in case of any background interferences or noises. All signals have been recorded and they can be rechecked.

All those procedures are done to guarantee reliability of the measurements.

The measurements were performed with two different excitation frequencies 10 and 100Hz were used; to try to evaluate penetration/response depth. At fix excitation mode, an applied excitation values fixed at 2000 and 450 a.u. for 100 and 10Hz excitation frequency were chosen respectively.

Altogether 12 specimens (9 irradiated, 3 unirradiated) were tested for a total number of 216 individual measurements (2 measured side, 2 frequencies, 3 repeated test, fix and scanning mode) plus the calibration ones.

## Results

### *As-received Condition, Orientation and Depths of Samples*

Firstly the specimens in as-received condition were measured by micromagnetic measurements. The specimens were selected from the blocks with different cutting directions and depths.

Measurements on the different plates cut from different block depths were averaged on each block in order to compare the blocks which represent different specimen orientation to the vessel directions. The final averaged Barkhausen signal and $R_XI$ relative to induction value versus applied excitation on block 4.1 and 1.6 are shown in Figure 2.

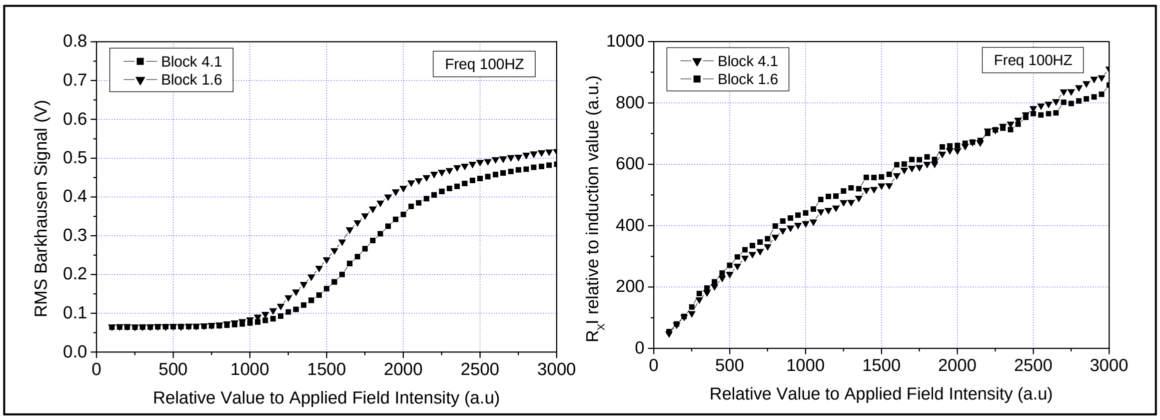


**Figure 2.** Barkhausen signal and RXI relative to induction value versus applied excitation compaired on blocks 4.1 and 1.6

*Irradiated Condition*

The irradiated specimen presented good repeatability of measurements. Actually, the irradiated specimens were cut having longer side along vessel longitudinal direction, CV0X specimens from 4.1 block (block 1.6 is not yet ready as-irradiated).
It is convenient to remember in this case the applied magnetic flux was along longitudinal direction.

In those measurements of irradiated specimen it was evaluated the difference on results according to the measured face, notched and back face. Some specimens presented slightly to none difference among measurements on faces back and notched. Other specimens presented large difference among measurements on faces back and notched.

Figure 3 shows the curve results of Barkhausen and $R_X I$ relative induction measurements with increasing applied magnetic field measured on notched and back faces for one of the irradiated specimens which presented significant difference on measurements (the applied excitation frequency was 100Hz).

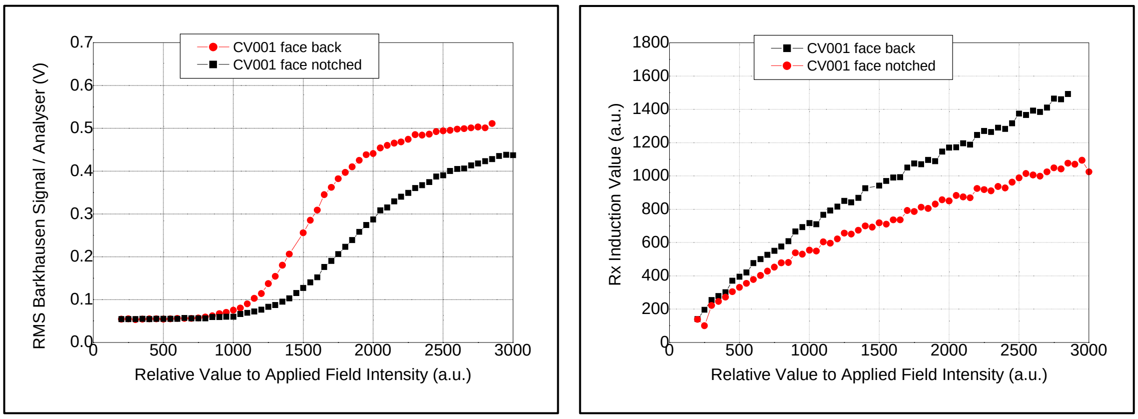


**Figure 3.** Barkhausen signal and RXI relative to induction value versus applied excitation intensity of irradiated specimens measured on two faces (100 Hz)

The results of both Barkhausen and $R_XI$ relative induction measurements at fixed level for irradiated specimen on both faces are respectively shown in Figure 4 (the applied excitation frequency was 100Hz). Figure 5 shows the results on both faces at the applied excitation frequency of 10Hz.

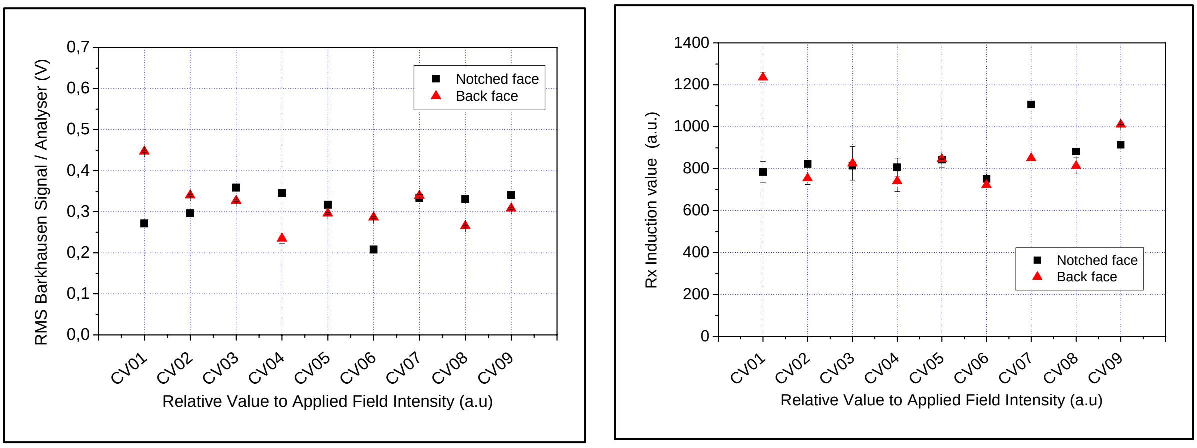


**Figure 4.** Barkhausen signal and RXI relative to induction value versus applied excitation intensity of irradiated specimens measured on two faces (100 Hz)

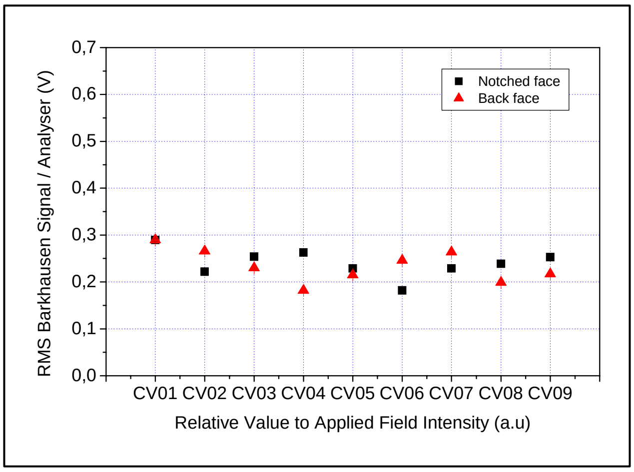


**Figure 5.** Comparison between two faces, back and notched of Barkhausen measurements on irradiated specimens (10Hz)

## Discussion of the Results

It is evident from this study that for good representativity of Barkhausen signal related to Charpy specimens a series of measurements on the same specimen is required, mainly to guarantee that a proper coupling is obtained and allowing discarding measurements on non-proper surfaces (possibility not smooth and planar). This is required until new sensors or methodologies are developed to overcome this problem. We can also say that there is not conclusive answer of the reason that measurements repeatability was better found on irradiated specimens.

On as-received specimens differences due to cut orientation were observed; the Barkhausen noise was larger on specimen CV1501 when compared to the others two samples [6]. In the CV1501 specimens the applied magnetic field was through its side cut along the circumferential direction of the vessel while the others two were along the longitudinal direction. The $R_X$I relative induction value presented so slightly difference among received specimens even from different cut direction.

*Measurements with Variable Level/Intensity*

Results measured by scanning excitation mode were divided in two groups. One group composed of specimens presenting difference among faces and the other group where sample measurements on both face were similar. Figure 6 shows the measurements results of as-received and irradiated samples for which the irradiated samples did not show any difference among faces (back and notched). Applied frequency was 100Hz.

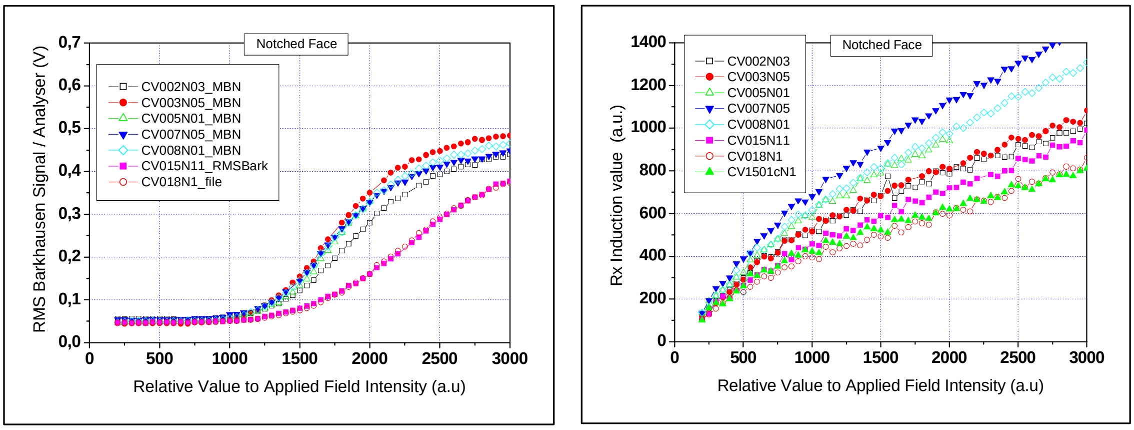


**Figure 6.** Barkhausen signal and RXI relative to induction value versus applied excitation intensity of irradiated and fresh specimens measured on notched face

*Measurements at Fix Level/Intensity*

The results of both Barkhausen and $R_X$I relative induction measurements at fix level for as-received and irradiated were also analysed.

On the evaluation of irradiation effects, as-received and irradiated specimens which had their measurements done on same sample face were compared.

In Figure 7, at left side the Barkhausen and at right side of the figure the $R_X$I measurements are shown. Measurements were done at the back faces of as-received and irradiated specimens, at 100Hz applied frequency with intensity at fix level of 2000.

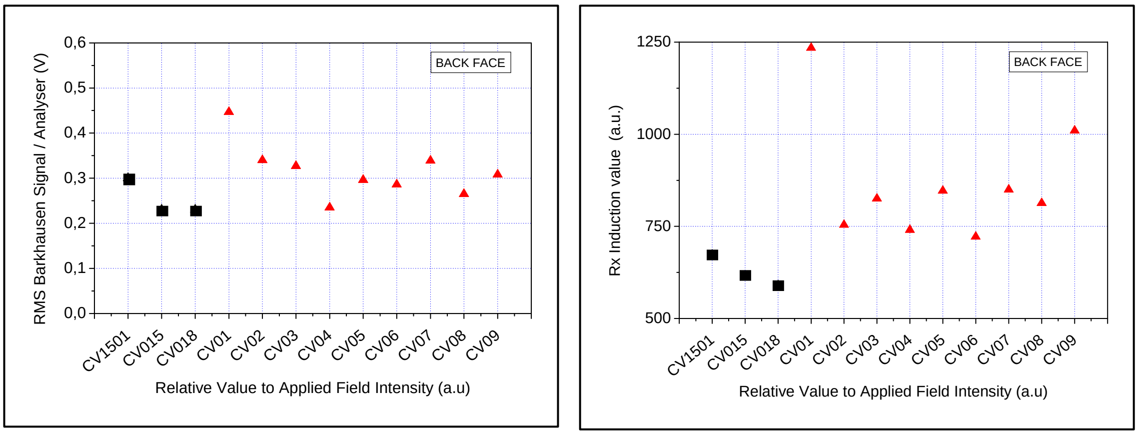


**Figure 7.** Barkhausen (left) and RxI (right) measurements on received (square) and irradiated (triangle) specimens (100Hz, 2000 constant level)

There is a slightly increase on Barkhausen noise measurements clearly seen when considering that as-received specimen CV1501 has a different flux orientation when compared with the other as-received and irradiated samples.

However independently of the considered flux orientation on as-received specimens, $R_XI$ relative induction values were higher on irradiated specimen and different among them.

On irradiated specimens Barkhausen measurements performed by excitation frequency of 10 Hz seems to be less sensitive to the difference among notched and back faces. At 10Hz applied excitation frequency the differences on measurements among as-received and irradiated samples tend to disappear on both measurements of Barkhausen and $R_XI$ values.

## Conclusions

- This study reveals the necessity to perform a statistical treatment when dealing with magnetic measurements of Charpy specimens obtained from reactor pressure vessel in order to achieve realistic representation and observe changes due to the effect of neutron irradiation.
- A slightly increase on Barkhausen noise measurements is clearly seen when considering that as-received specimen CV1501 has a different magnetic flux orientation when compared with the others as-received and irradiated ones.
- However independently of the considered flux orientation on received specimens, RxI relative induction values were higher on irradiated specimen than the received ones.

## References


[1] "Embrittlement, Mechanistic Interpretation of Reactor Pressure Vessel and Internal Materials", Published by European Commission, Joint Research Centre, Institute of Energy, Petten, The Netherlands, 2005. (EUR Report to be published)

[2] Buttle, D.J. Little, E.A., Scruby, C.B., Briggs, G.A.D. and Jakubovics, J.P. *A study of neutron irradiation and Barkhausen emission*, Proc. R. Soc. Lond. A 414, 221-236, UK, 1987.

[3] Sipahi, L.B., Govindaraju, M.R. and Jiles, D.C. *Monitoring neutron embrittlement in nuclear pressure vessel steels using micromagnetic Barkhausen emissions*, J. Appl. Phys. 75(10), 6981-6983, 15 May, 1994.

[4] Pirfo, S., Czoboly, E., Horvát, M. and Uri, G., Debarberis, L. *Micromagnetic and Barkhausen Testing in Irradiated Specimens*, The 6th International Conference of Slovenian Society for Non-destructive Testing "Previous Experience and Current Inovation in NDT", 13-14, September 2001.

[5] S. Pirfo, L. Debarberis, B. Acosta, F. Sevini *Magnetic and Barkhausen signal responses applied on sub-sized Charpy specimens to characterise irradiation effects*, 11th International Symposium on Applied Electromagnetics and Mechanics, Versaille, 12-14 May, 2003.

[6] Luigi Debarberis, Beatriz Acosta, Soraia Pirfo, F. Sevini, A. Kryukov, F. Gillemot A.Chernobaeva, M. Brumovsky, *VVER-1000 Base Metal Reference Steel and its Characterisation*, 17th International Conference on Structural Mechanics in Reactor Technology (SMIRT), Prague, Czech Republic, August, 2003