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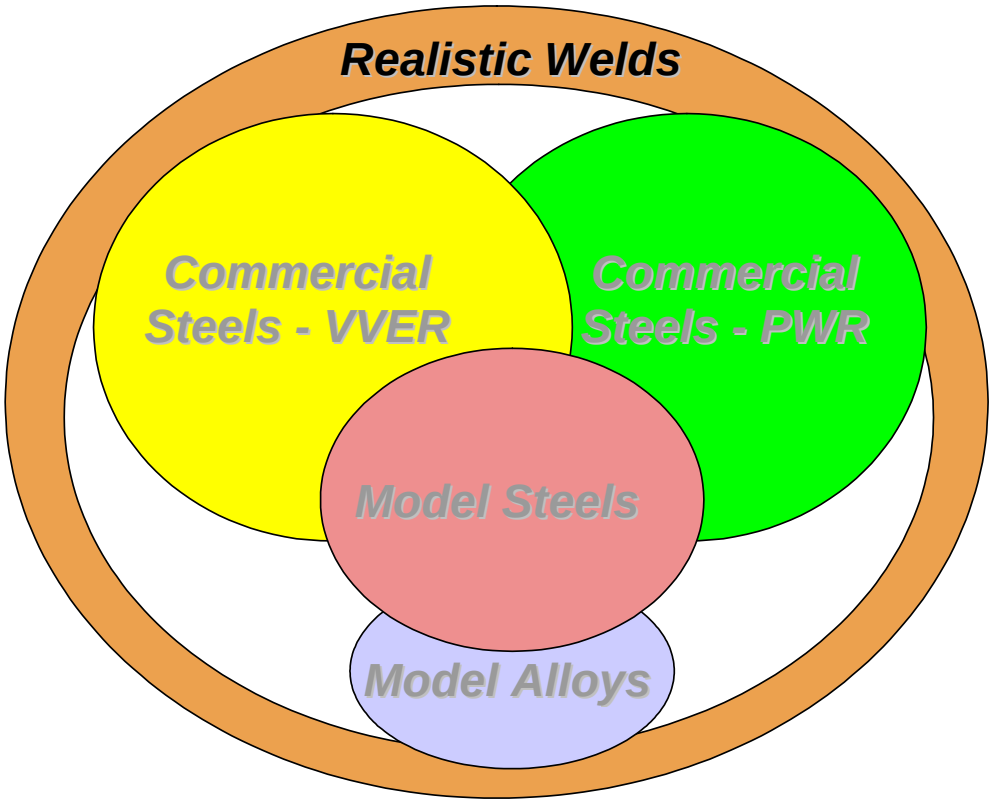
Mechanical and Magnetic Testing of Realistic Welds with Parametric Variation of Ni, Si, Cr and Mn Content

J. Degmova, A. Rito-de-Abreu, T. Heftrich, H. Chantraine, L. Debarberis



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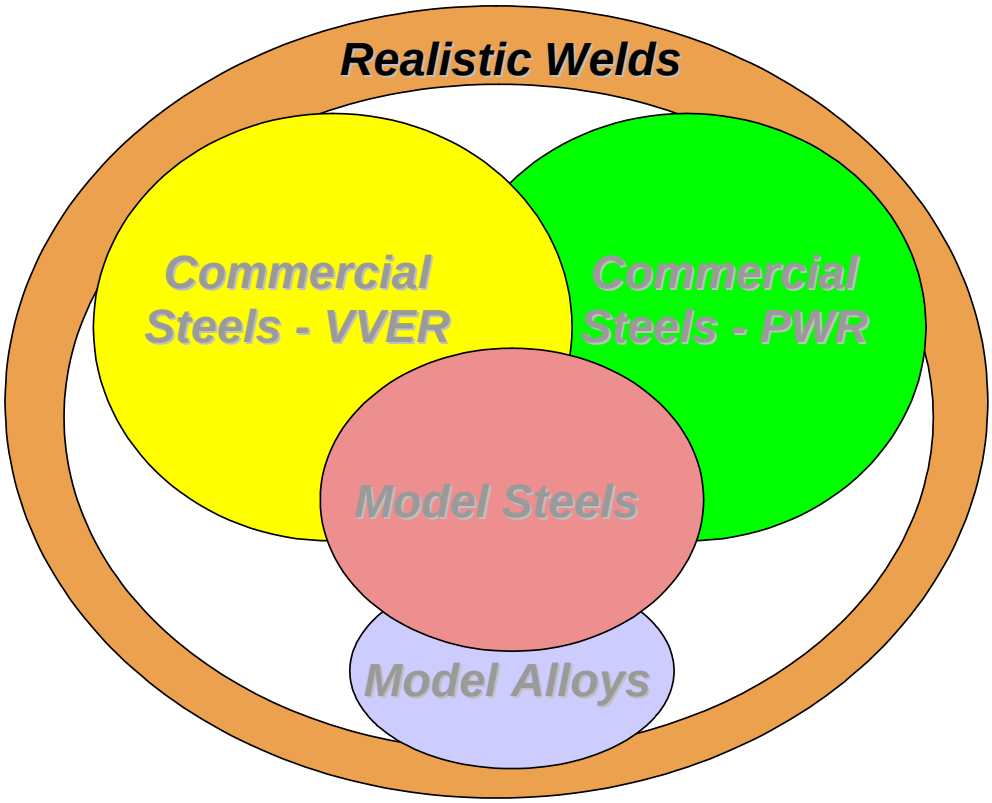

September 2007

Index

1 Abbreviations & Symbols

Charpy V-notch	Charpy Impact Test Specimen
CV samples	Charpy V-notch Samples (10x10x55)
DBTT	Ductile-to-Brittle Transition Temperature
HFR-LYRA	Irradiation Facility
MBN	Magnetic Barkhausen Noise
NPP	Nuclear Power Plant
PWR	Pressurised Water Reactor
RPV	Reactor Pressure Vessel
RMS	Root Mean Square Values

2 Introduction

The Reactor Pressure Vessel (RPV) is the most critical component and its embrittlement poses one of the limiting factors in the lifetime of vessels of today's nuclear power plant (NPP). Due to the efforts to prolong the lifetime of many reactors and increasing safety requests, the investigation of RPV-steels changes going on during long-term operation should have the highest priority. The present work is aimed at the investigation of the role of selected alloying elements in reactor pressure vessel (RPV) radiation damage. It has been found that some deleterious elements may cause synergy effect in producing the complex radiation defects which can lead to the radiation-induced degradation in the mechanical properties of RPV steels. In order to understand the role and influence of Ni, Si, Cr and Mn a spectrum of model realistic welds with parametric variation of these elements was designed out.

The present work reviews the results of mechanical testing (Charpy impact tests) and non-destructive Magnetic Barkhausen Noise measurements of as-cast realistic welds and their correlation.

The next step will be the neutron irradiation of such welds in HFR-LYRA irradiation facility up to a neutron fluence of about 10^{19} n.cm^{-2} and the further comparison of mechanical properties before and after irradiation.

3 Material Characterisation

All primary material preparation was done by Skoda Vyzkum Plzen in Czech republic based on JRC specifications. The nominal base compositions were derived from typical SV12Ch2N2MAA weld with variation of certain elements as Ni, Si, Cr and Mn.

The heat treatment of the tested material was done as follow:

620 ^{o}C/5 h/ furnace up to 400 ^{o}C

620 ^{o}C/5 h/ furnace up to 400 ^{o}C

620 ^{o}C/10 h/ furnace up to 300 ^{o}C/ air

The surface of each weld was machined to the level of the surrounding base and subsequently ultrasonically tested before cutting. After cutting, all individual welds have went to the radiographic examination.

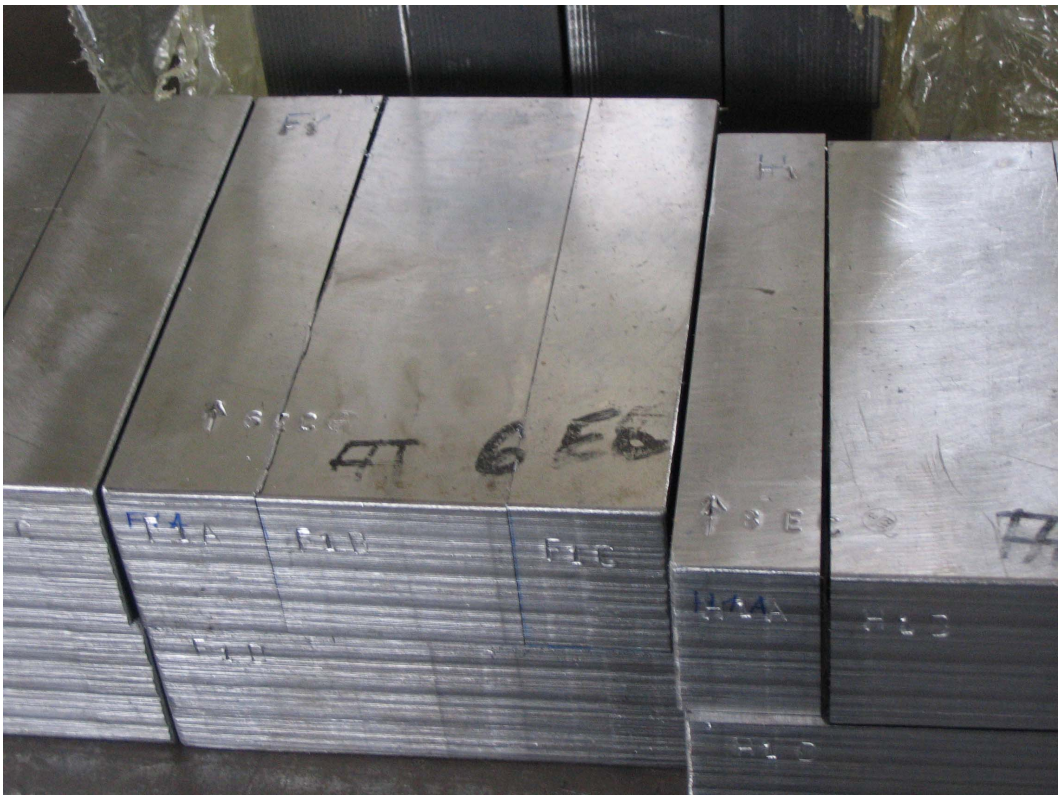

Figure 1: The cut block of realistic weld.

14 kg of material corresponded to one weld. The different welds were numbered as 1 - 8 EC, where each number from 1 to 8 corresponds to the different block of weld (see Fig. 1)

with different composition. Tab. 1 shows the compositional details. The 8 blocks were cut into test specimen according to the cutting scheme shown in Fig. 2: each block was divided into 5 subblocks and cut onto test samples.

All samples were labelled with respect to the following nomenclature:

A . B . X . Y . Z

Where:

A – Material ID (A, B, C,…H)

A = 1EC23

B = 2EC23

C = 3EC23

D = 4EC23

E = 5EC23

F = 6EC23

G = 7EC23

H = 8EC23

B – subblock ID (1, 2, 3, 4, 5)

X – X axis position (1, 2, 3,…)

Y – Y axis position (1, 2, 3,…)

Y – Y axis position (1, 2, 3,…)

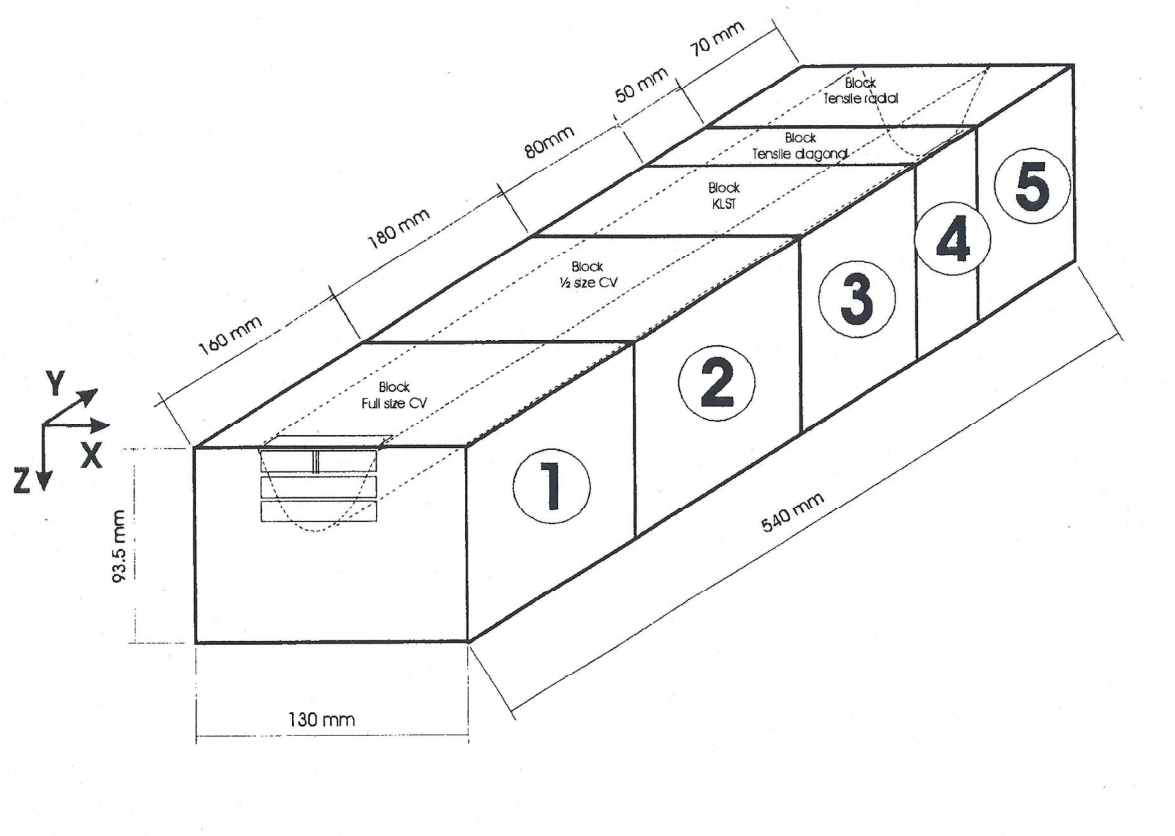

Figure 2: The cutting scheme for block of realistic weld.

Table 1: Results of Analytical Chemistry Testing provided by Skoda Vyzkum Plzen on 8 different heat of SV12Ch2N2MAA based welds.

Designation	C [%]	Mn [%]	Si [%]	P [%]	S [%]	Cr [%]	Ni [%]	Mo [%]	V [%]	Co [%]	As [%]	Cu [%]	Sn [%]	Sb [%]
1 EC 23	0.07	0.57	0.18	0.011	0.007	2.07	1.30	0.59	0.09	0.02	0.004	0.06	0.005	<0.001
2 EC 23	0.06	0.56	0.31	0.007	0.009	2.04	1.59	0.60	0.09	0.02	0.004	0.06	0.005	<0.001
3 EC 23	0.05	0.60	0.32	0.007	0.010	1.95	1.87	0.58	0.08	0.02	0.004	0.06	0.005	<0.001
4 EC 23	0.06	0.72	0.29	0.006	0.009	2.01	1.57	0.59	0.09	0.02	0.004	0.06	0.004	<0.001
5 EC 23	0.05	0.89	0.30	0.006	0.009	2.00	1.94	0.57	0.09	0.02	0.004	0.06	0.004	<0.001
6 EC 23	0.06	1.07	0.29	0.006	0.009	2.04	1.26	0.58	0.09	0.02	0.004	0.06	0.004	<0.001
7 EC 23	0.06	1.07	0.30	0.007	0.0010	2.04	1.57	0.59	0.09	0.02	0.004	0.06	0.005	<0.001
8 EC 23	0.06	1.08	0.32	0.007	0.0010	1.98	1.89	0.58	0.09	0.02	0.004	0.06	0.005	<0.001

The full size Charpy V-notch samples (full size CV) were used for both kind of testing i.e. Magnetic Barkhausen Noise measurements as well as Charpy impact testing (see Figure 3, 4).

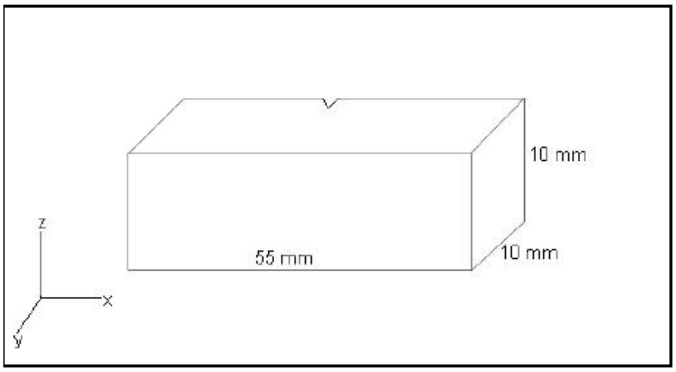

Figure 3: Specimen geometry – ASTM standard V-notch Charpy test specimen [1].

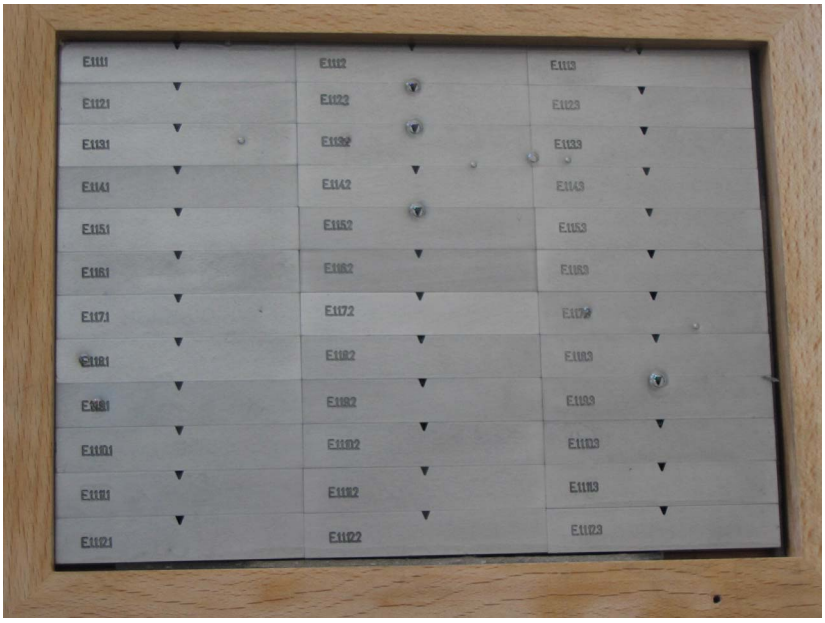

Figure 4: Set of full size CV samples.

4 Experimental Methods Characterisation

4.1 Magnetic Barkhausen Noise (MBN) Technique

Barkhausen technique has his importance because of his capability to characterise materials properties accurately, easily, quickly and without damaging tested materials. Magnetic Barkhausen Noise (MBN) is considered as an important technique for microstructural and mechanical characterization of ferromagnetic materials like feritic steels. The main advantages of this method are that this technique has non-destructive character and it offers the possibility to evaluate samples of various shapes and sizes. MBN is sensitive to various parameters which affect the magnetic domains configuration and domain-wall pinning sites, which are strongly influenced by grain size [2, 3, 4], composition [3, 5, 6, 7,], different phases [5, 8, 9], surface conditions [10, 11], hardness [11, 12], residual stress [12, 13, 14, 15], fatigue and damage [16, 17, 18, 19], magnetic field strength [20, 21, 22] and applied stress [2, 21, 22, 23, 24].

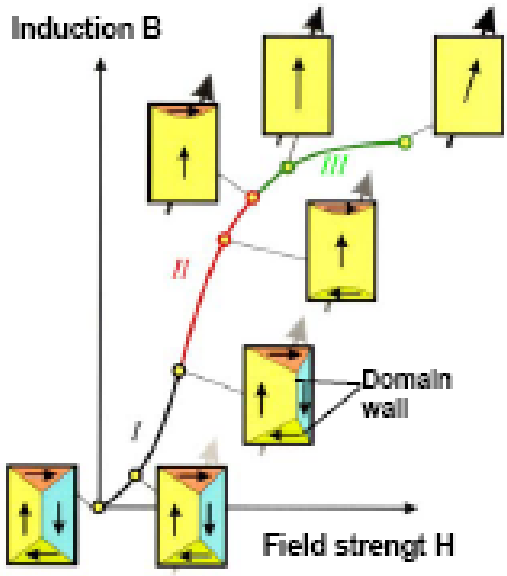

Figure 5: Distribution of magnetisation process along the hysteresis curve [25].

The Magnetic Barkhausen Noise technique is based on concept that the ferromagnetic materials consist of domains which are magnetised along a certain crystallographic easy direction of magnetisation. Domains are separated from one another by boundaries (domain walls). Application of a magnetic field causes the domain walls movement: the domain on one

side of the wall has to increase in size while the domain on the opposite side of the wall shrinks (see Fig. 5).

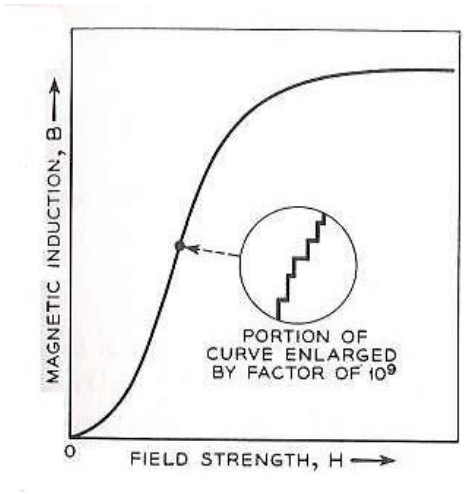

Figure 6: Barkhausen effect [26].

The result is a change in the overall magnetisation of the sample. If a coil of conducting wire is placed near the sample during the domain wall movement, the resulting change in magnetisation will induce an electrical pulse in the coil. In 1919 professor Barkhausen proved that the magnetisation process (hysteresis loop) is not continuous but is build from small steps caused when the magnetic domains move under an applied magnetic field (see Figure 6). When the electrical pulses produced by all domain movements are added together, a noise-like signal called Barkhausen noise is generated (see Figure 7). Barkhausen noise spectrum starts at the magnetising frequency and extends beyond 2 MHz in most ferromagnetic materials. The signal is exponentially damped as a function of travelled distance inside the material.

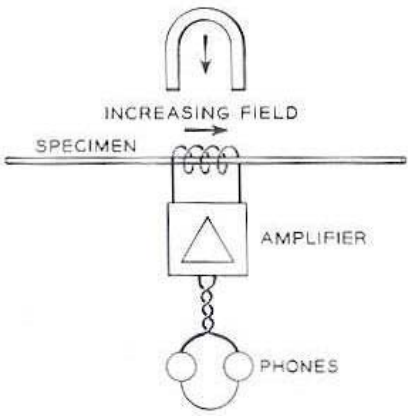

Figure 7: The principle of observing the Barkhausen effect [26].

This is generally caused by the eddy current damping experienced by the propagating electromagnetic fields created by domain wall movements. The extent of damping determines the depth from which information can be obtained (so called measurement depth). There are two main factors affecting this depth: frequency range of the analysed Barkhausen noise signal, and conductivity and permeability of the tested material.

For practical application the measurement depth varies between 0.01 and 1.5 mm [27]. Generally the measurement depth is increasing with the decrease of frequency, but lower frequency gives also lower sensitivity.

A microcomputer based signal analyzer μSCAN 500C (see Figure 8) in combination with a PCI-6111E computer card were used to pick up and to analyse the Barkhausen signal. The μSCAN 500C signal analysis is based on digitizing and storing the analog signal derived from patented sensors (probe type S1-101-13-01).

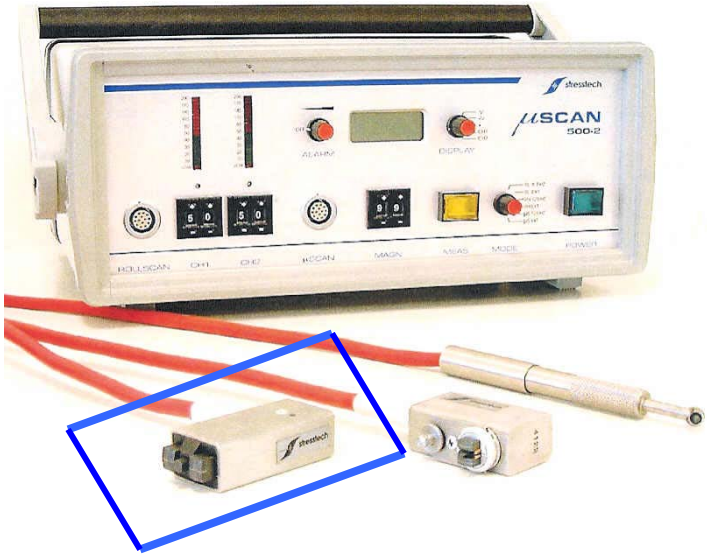

Figure 8: μSCAN 500C analyzer with patented sensor. Sensor used in our experiments is bleu-marked [28].

The magnetic measurements were performed by two different applied field frequencies (10 and 100 Hz) in the way that dept effects could also be taken into account. For Barkhausen excitation a sinusoidal exciting magnetic field with magnetising voltage of 2 and 1 V_{pp} was used. The signal of the pick-up coil was processed by a 5 – 500 kHz band pass filter and amplified with a gain of 20. For complete measuring set up see Table 2. The root mean

square values (RMS) of the noise signal were determined and were used to characterise the realistic welds (and to correlate with DBTT values).

Table 2: Setup for MBN measurements on full size CV samples of realistic welds

Parameters	Values	Values
Magnetizing frequency	10 Hz	100 Hz
Magnetizing voltage	2 Vpp	1 Vpp
Magnetic offset	0	0
Number of bursts	10	10
Sampling frequency	1 MHz	1 MHz
Signal input scale	5 V	5 V
Magnetizing current input	ON	ON
Magnetizing current input scale	1 A	1 A

4.2 Charpy Impact Test Technique

Charpy impact testing is a destructive way to monitor mechanical properties of the materials. It gives information about the material response to the dynamic load. The test consists of breaking a test piece notched in the middle and supported at each end by one hit from a swinging pendulum (see Fig. 9). The energy absorbed during hit is a measure of the impact strength of the tested material [29, 30].

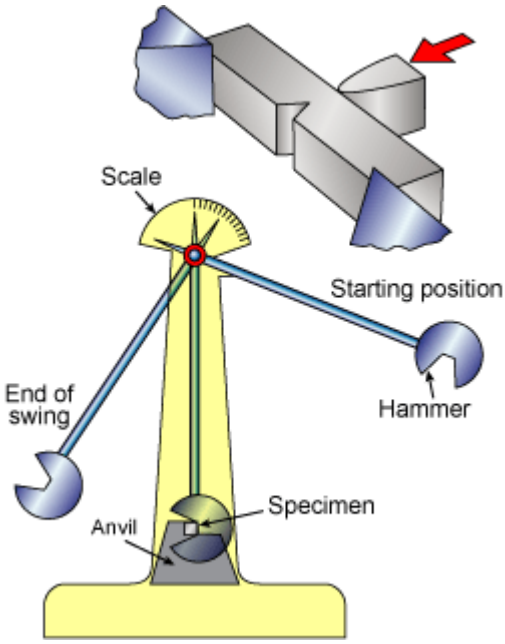

Figure 9: Charpy testing machine. Adopted from [31].

If the testing is done in a wide temperature range, the obtained results can provide us with information as values of transition temperature (DBTT) or upper shelf energy (USE) by drawing so-called "Charpy impact curve", which is the dependence of impact/absorbed energy on testing temperature (see Fig. 10). General shape of Charpy impact curve shows that the fracture of the tested material changes from being ductile on the upper shelf (USE) to brittle on the lower shelf (LSE) with decreasing testing temperature. The temperature at which the fracture behaviour changes from brittle to ductile is so-called "transition temperature" [32].

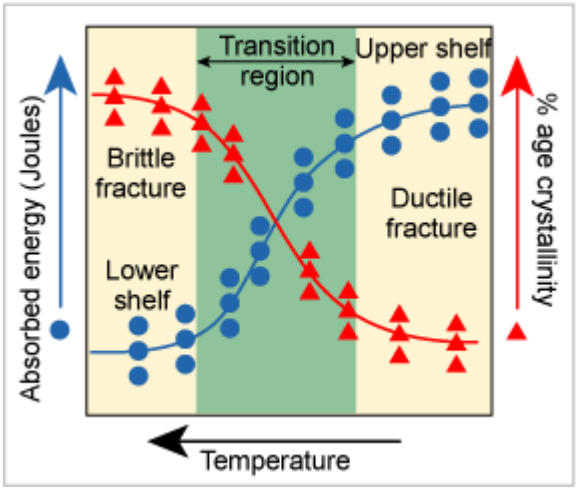

Figure 10: Schematic picture of Charpy-V energy and % age crystallinity curves. Adopted from [31].

The impact testing procedure was performed in the AMES Impact Testing Laboratory of JRC IE Petten. Impact Testing Hammer WOLPERT PW 30 (300 J) equipped by ISO 10 KN tup was used to test Charpy V-notch samples (standard CV). Testing was carried out in the temperature range from – 150 up to 150 °C. Samples were cooled and heated in the INSTRON conditioning chamber for 0.5 h and then transferred to the anvil support of the hammer. Transfer of the sample between the chamber and the anvil was done within 5 s and in order to minimise the changes of the sample's temperature a stainless steel support is used [33]. In addition to the impact energy also the lateral expansion is measured, which is also a measure of the ductility of the sample. When the sample is ductile, the test piece is deformed before breaking. The amount by which the sample deforms is measured and expressed in millimetres of lateral expansion (see Fig. 11). Table 3 shows temperature criteria for CV specimen used for DBTT determination.

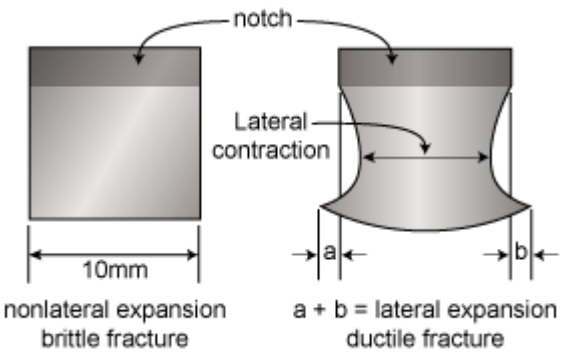

Figure 11: Lateral expansion. Adopted from [31].

Table 3: Transition temperature criteria for CV specimen [34].

Absorbed energy	41 J or 68 J
Lateral expansion	0.9 mm

5 Results & Discussion

5.1 Magnetic Barkhausen Noise Measurements

Table 4 shows the RMS values measured at 10 and 100 Hz for the realistic welds. Decrease of frequency increase a scanning depth and vice versa. So at frequency of 10 Hz we expect to get signal trough the bulk of the sample and at 100 Hz the only surface layer of the sample is scanned. The RMS value for each sample was calculated by averaging the results from two measurements from each side of the notched CV sample. Figures 12, 13, 14 and 15 show the RMS as a function of Si, Mn, Cr and Ni content, respectively.

Table 4: RMS (Root Mean Square) values for 8 realistic welds with compositional details.

Mark	Si (%)	Mn (%)	Cr (%)	Ni (%)	RMS (V) 10 Hz	RMS (V) 100 Hz
A.1.16.1 (A)	0.18	0.57	2.07	1.30	5.05	1.98
B.1.1.8.1 (B)	0.31	0.56	2.04	1.59	7.94	5.30
C.1.1.7.1 (C)	0.32	0.60	1.95	1.87	6.95	4.45
D.1.1.8.1 (D)	0.29	0.72	2.01	1.57	7.87	3.40
E.1.1.8.1 (E)	0.30	0.89	2.00	1.94	11.37	4.98
F.1.1.5.1 (F)	0.29	1.07	2.04	1.26	9.60	4.57
G.1.1.5.1 (G)	0.30	1.07	2.04	1.57	8.06	3.19
H.1.1.4.1 (H)	0.32	1.08	1.98	1.89	7.18	2.48

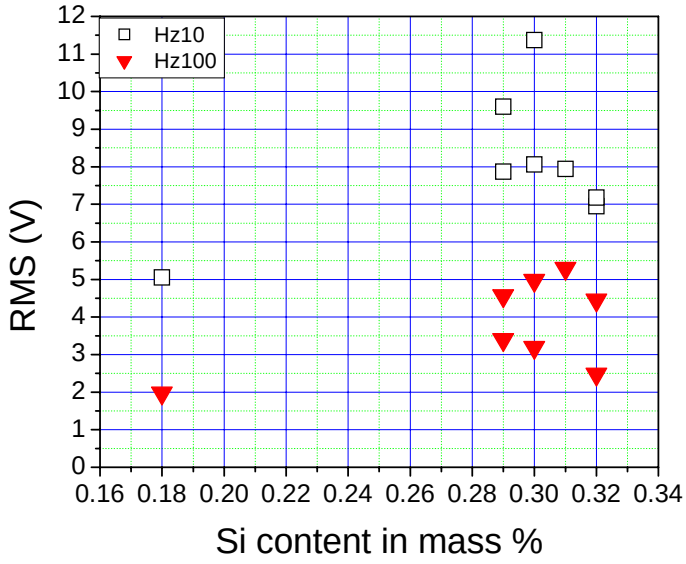

Figure 12: RMS as a function of Si content for 10 Hz and 100 Hz excitation.

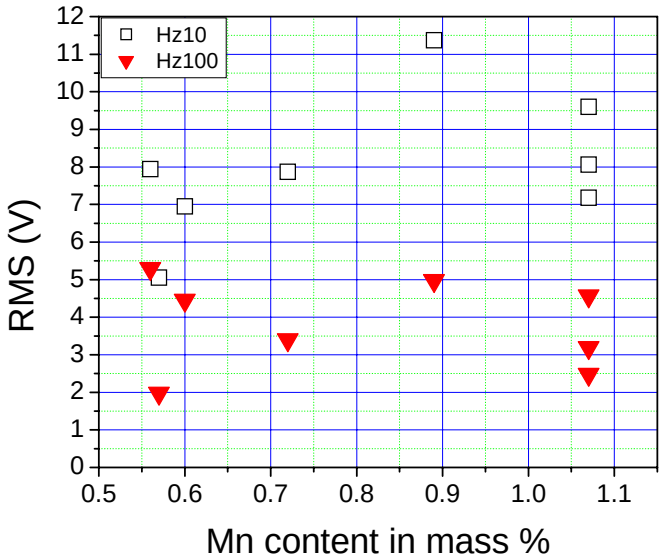

Figure 13: RMS as a function of Mn content for 10 Hz and 100 Hz excitation.

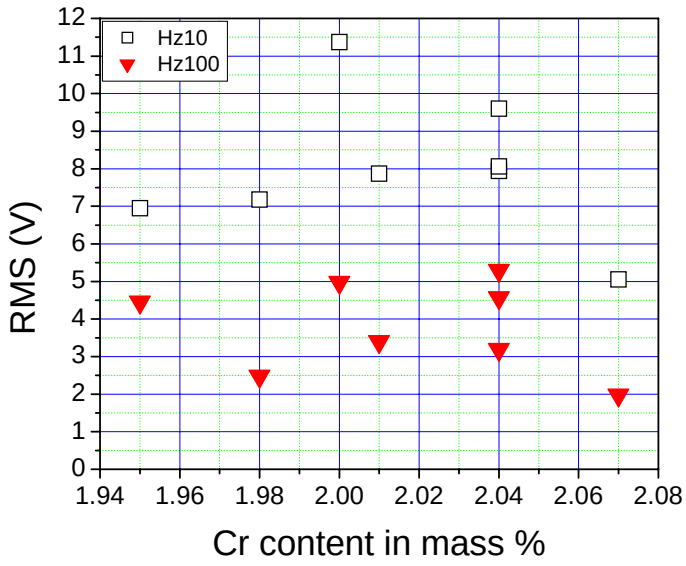

Figure 14: RMS as a function of Cr content for 10 Hz and 100 Hz excitation.

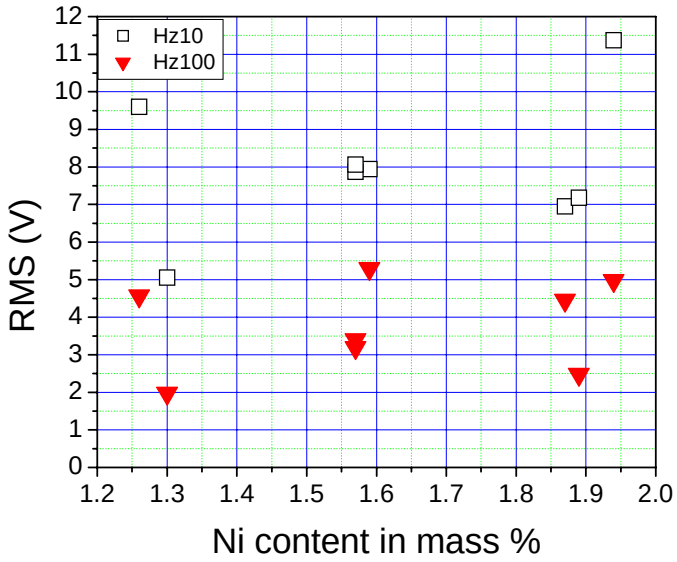

Figure 15: RMS as a function of Ni content for 10 Hz and 100 Hz excitation.

Generally the RMS values observed from 10 Hz measurements are higher in comparison with those obtained from 100 Hz measurements. This could be explained by the depth from which the signal is collected.

In the case of Si content variation, the low Si content (= 1.8 %) significantly decreases the RMS values for both scanning frequency. These values achieve approximately half of the values characteristic for the welds with higher Si (0.29 - 0.32 %).

Variation of Mn composition in the range from 0.57 to 1.08 mass % seems to have no significant influence on RMS values.

The effect of Cr content is visible only in the case of its highest tested percentage, which is 2.07 mass % of Cr in the weld. For both frequencies (10 and 100 Hz) the RMS values decreased almost to their half in comparison with the values characteristic for lower Cr content.

In the case of Ni, the results of MBN measurements are quite scattered and it is not easy to distinguish the Ni effect.

5.2 Charpy Impact Tests

The results of Charpy impact test for realistic welds are listed in Table 7. Figures 16 – 19 show the DBTT values as a function of Si, Mn, Cr and Ni content, respectively.

Table 7: Results of Charpy impact tests for 8 realistic welds with compositional details. DBTT is the ductile to brittle transition temperature, transition temperature criterion is 68 J.

Mark	Si (%)	Mn (%)	Cr (%)	Ni (%)	DBTT (^{o}C)
A.1.16.1 (A)	0.18	0.57	2.07	1.30	-2.56
B.1.1.8.1 (B)	0.31	0.56	2.04	1.59	-16.32
C.1.1.7.1 (C)	0.32	0.60	1.95	1.87	-49.61
D.1.1.8.1 (D)	0.29	0.72	2.01	1.57	-7.86
E.1.1.8.1 (E)	0.30	0.89	2.00	1.94	-15.57
F.1.1.5.1 (F)	0.29	1.07	2.04	1.26	-23.79
G.1.1.5.1 (G)	0.30	1.07	2.04	1.57	-20.68
H.1.1.4.1 (H)	0.32	1.08	1.98	1.89	72.68

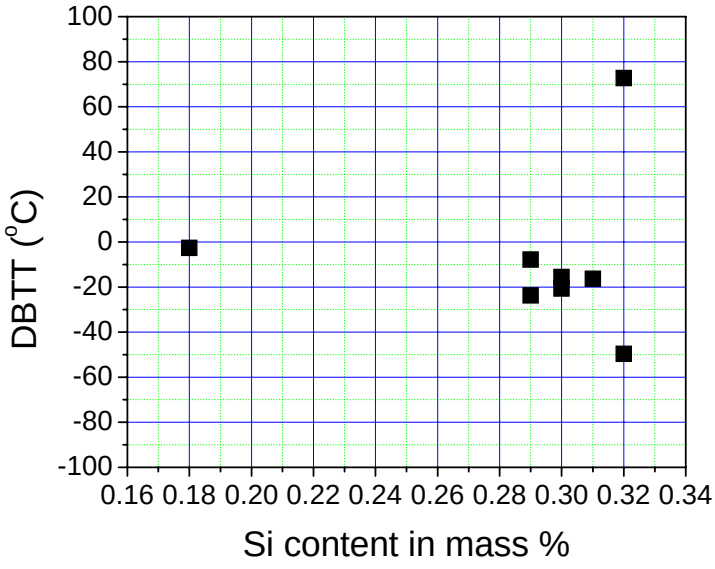

Figure 16: DBTT as a function of Si content.

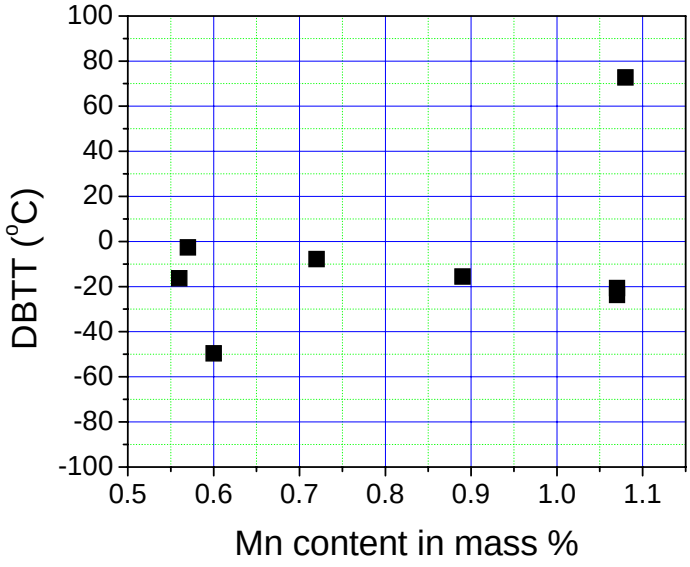

Figure 17: DBTT as a function of Mn content.

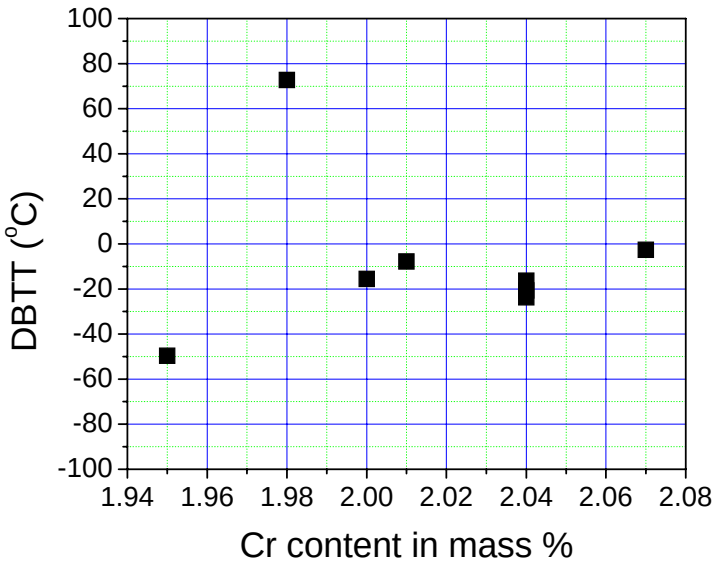

Figure 18: DBTT as a function of Cr content.

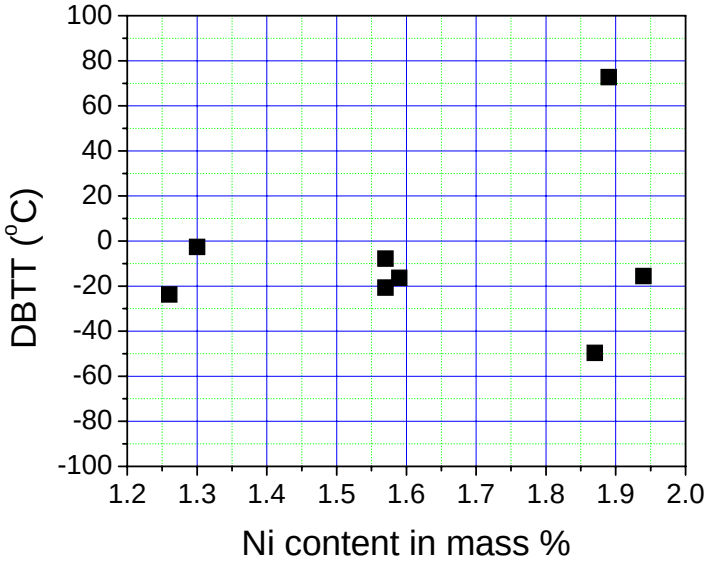

Figure 19: DBTT as a function of Ni content.

The DBTT values are mostly from temperature range -2 to -50 °C, only DBTT measured for sample labelled as H is significantly higher reaching 72.68 °C. This sample combine high mass % of all 4 elements of interest.

An increase in the mass % of Si and Mn leads to the significant decrease of DBTT with only exception for their highest concentrations *i.e.* 0.32 % of Si and 1.08 % of Mn.

DBTT values increase almost linearly with increasing Cr content. Only the concentration of 1.98 % of Cr is an exception: in this case DBTT reaches its highest value: 72.68 °C.

With increasing Ni content, a slight decrease of DBTT values can be observed. Only the value measured for sample labelled as H (where Ni content corresponds to 1.98 mass %) is significantly higher (72.68 °C).

Table 8 and Figure 20 correlate the results of Magnetic Barkhausen Noise (represented by RMS) and Charpy Impact Testing (represented by DBTT) obtained on the realistic welds.

Table 8: RMS and DBTT values for the realistic welds with detailed composition.

Mark	Si (%)	Mn (%)	Cr (%)	Ni (%)	RMS (V) 10 Hz	RMS (V) 100 Hz	DBTT (°C) (68 J)
A.1.16.1 (A)	0.18	0.57	2.07	1.30	5.05	1.98	-2.56
B.1.1.8.1 (B)	0.31	0.56	2.04	1.59	7.94	5.30	-16.32
C.1.1.7.1 (C)	0.32	0.60	1.95	1.87	6.95	4.45	-49.61
D.1.1.8.1 (D)	0.29	0.72	2.01	1.57	7.87	3.40	-7.86
E.1.1.8.1 (E)	0.30	0.89	2.00	1.94	11.37	4.98	-15.57
F.1.1.5.1 (F)	0.29	1.07	2.04	1.26	9.60	4.57	-23.79
G.1.1.5.1 (G)	0.30	1.07	2.04	1.57	8.06	3.19	-20.68
H.1.1.4.1 (H)	0.32	1.08	1.98	1.89	7.18	2.48	72.68

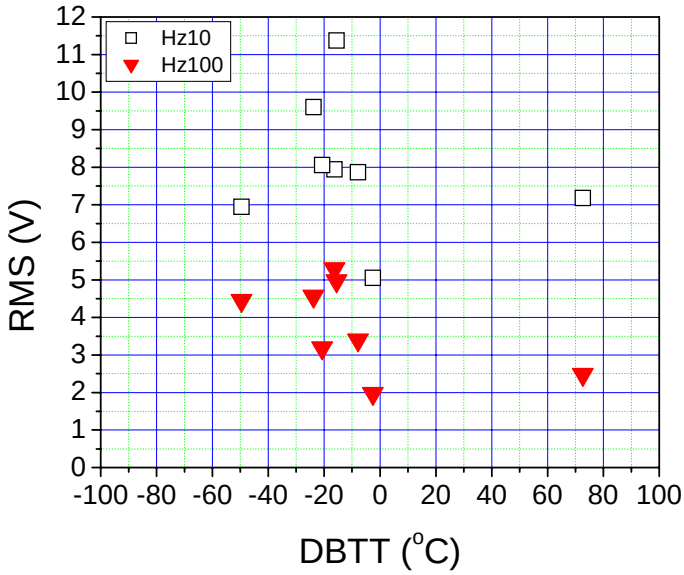

Figure 20: RMS as a function of DBTT for 8 realistic welds.

By comparing the RMS and DBTT values of the 8 realistic welds it is not possible to find a general conclusion. In as-received state of the material, there is no clear relation between these two values in every type of material. The neutron irradiation is expected to have significant influence on this correlation by comparison of DBTT shift with RMS values as a function of welds composition.

6 Conclusions

The results of Barkhausen Noise analysis showed that mostly Si and Cr content variation significantly influences the RMS values. The decrease of Si content in the samples leads to the decrease of RMS. In the case of Cr the situation is different: an increase of Cr content is causing the decrease of RMS values. Morover this effect is visible only for higher Cr percentage.

The DBTT values are mostly within temperature range -2 to -50 °C, only DBTT measured for sample labelled as H is significantly higher reaching 72.68 °C. This sample combine high mass % of all 4 elements of interest.

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Title: Mechanical and Magnetic Testing of Realistic Welds with Parametric Variation of Ni, Si, Cr and Mn Content

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Abstract
In order to understand the role and influence of Cr, Ni, Si and Mn alloying elements on the mechanical and magnetic properties of NPP's materials a large spectrum of welds with parametric variation of alloying elements was prepared in the frame of the SAFELIFE Action of JRC-IE and the AMES Network.
The present report describes their mechanical and micromagnetic properties, results of Magnetic Barkhausen Noise measurements are correlated with the results of Charpy impact tests.

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