Journal of ELECTRICAL ENGINEERING, VOL. 60, NO. 3, 2009, 161–165

MODEL STEELS WITH PARAMETRIC VARIATION OF Ni, Mn, Si AND Cr CONTENT: CORRELATION BETWEEN MAGNETIC BARKHAUSEN NOISE & CHARPY IMPACT TESTS RESULTS

Jarmila Degmová [*, **] — Luigi Debarberis [*]

In order to understand the role and influence of Ni, Mn, Si and Cr alloying elements and certain impurities on the mechanical properties of steels a large spectrum of ferritic steels with parametric variation of alloying elements as well as impurities content were prepared in the frame of the SAFELIFE Action of JRC-IE and the AMES Network. The composition of the 12 prepared model steels were inspired by typical base compositions of WWER-1000 and PWR base metal materials. In the present work the results of Magnetic Barkhausen Noise measurements are discussed and correlated with the results of Charpy impact tests.

K e y w o r d s: model steels, magnetic Barkhausen noise, Charpy impact test

1 INTRODUCTION

Reactor Pressure vessel (RPV) 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. 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 [1, 2]. The problem is rather complex and it is still the subject of large international programmes, IAEA [3], and international projects, REVE-PERFECT [4]. In order to understand the role and influence of Ni, Si, Cr and Mn as alloying elements and certain impurities as Cu and P on the mechanical properties of steels, a large spectrum of ferritic steels with parametric variation of alloying elements, as well as impurities content, were prepared. Thus 12 "model steels" were manufactured started from a basic typical compositions representative of WWER-1000 and PWR reactor pressure vessel base materials.

The present work reviews the results obtained on as-cast model steels to understand the level of correlation between Magnetic Barkhausen Noise and Charpy impact test results and the role of the different alloying elements. The next step will be the completion of the neutron irradiation of such model steels in HFR-LYRA irradiation facility up to a neutron fluence of about $10^{19}\,\mathrm{n\,cm^{-2}}$ and the further comparison of properties before and after irradiation.

2 EXPERIMENTAL DETAILS

All primary material preparation was done by a scientific Russian team while the testing was done by the JRC [5, 6]. The nominal base compositions of the 12 model steels are derived from typical Russian and Western RPV base metal materials (WWER-1000 and PWR). This material matrix choice has been optimised to reveal the possible distinction of different compositions in their sensitivity to the deleterious element components during irradiation. The studied materials include mainly various Ni and Mn combinations and quite narrow range of Si content (see Table 1). This material matrix choice has been optimised to reveal the possible distinction of different compositions in their sensitivity to the deleterious element components during irradiation. The studied materials include mainly various Cr, Ni and Mn combinations and quite narrow range of Si content. All preparations started with melting adjusted target compositions in an open induction furnace (120 kg of bulk metal per cast). After melting, each of the 12 cast ingots was forged into a bar with 50×50 mm cross-section. The sinkhead parts of ingots were subsequently chopped off. Forging was carried at the temperatures ranging from 1200 °C down to 900 °C. The ingots were heated before forging for 4 hours starting from 800 °C. Subsequently the forged bars were hot-rolled into plates about 8 mm thick and 200–210 mm wide. The starting temperature for rolling was 1180 °C with preheating of about 2 hours. Every finished plate was cut into pieces with lengths of 600–700 mm.

The produced 8 mm thick hot-rolled plates were heat treated in order to obtain the similar bainite-martensite microstructure for all materials, independently on their chemical composition. The plates underwent to two different sets of quenching conditions. Firstly melts 1 and 2 (see Table 1) were tempered at 655 °C for 10 hours

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ISSN 1335-3632

Table 1. Chemical composition of the measured model steels (in mass %)

Mark	Melt	C	Si	Mn	Cr	Ni	Mo	V	Cu	S	P
631 (A)	2	0.11	0.28	0.43	2.22	< 0.02	0.71	0.10	0.09	0.008	0.010
632 (B)		0.11	0.26	0.38	2.19	0.99	0.70	0.10	0.10	0.008	0.010
633 (C)		0.12	0.24	0.38	2.13	2.00	0.69	0.10	0.10	0.008	0.010
634 (D)		0.11	0.23	0.83	2.13	2.00	0.68	0.10	0.09	0.008	0.009
641 (E)	2	0.12	0.33	0.77	2.16	1.02	0.70	0.10	0.10	0.008	0.009
642 (F)		0.12	0.33	1.37	2.15	1.02	0.70	0.10	0.10	0.008	0.010
643 (G)		0.11	0.32	1.36	2.06	1.99	0.69	0.10	0.10	0.008	0.009
644 (H)		0.12	0.51	1.31	2.07	2.00	0.69	0.10	0.10	0.008	0.010
301 (K)	3	0.17	0.35	0.78	0.10	0.58	0.64	–	0.07	0.005	0.009
302 (L)		0.18	0.35	0.77	0.08	0.96	0.63	–	0.05	0.005	0.010
303 (M)		0.16	0.37	0.74	0.09	1.90	0.61	–	0.05	0.005	0.010
304 (N)		0.16	0.33	1.27	0.07	1.97	0.63	–	0.06	0.005	0.010

Table 2. RMS and DBTT for 12 model steels, transition temperature criteria are in the brackets.

Mark	631 (A)	632 (B)	633 (C)	634 (D)	641 (E)	642 (F)	643 (G)	644 (H)	301 (K)	302 (L)	303 (M)	304 (N)
RMS (V)	8.51	8.36	7.80	7.40	7.64	7.38	7.58	7.57	5.99	6.86	7.08	6.65
DBTT (°C)	−150 (3.1J)	−160 (1.9J)	−129 (1.9J)	−142 (3.1J)	−150 (3.1J)	−137 (1.9J)	−141 (3.1J)	−112 (1.9J)	−131 (3.1J)	−150 (3.1J)	−150 (3.1J)	−104 (1.9J)

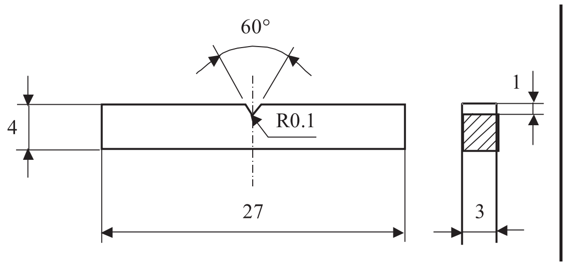

Fig. 1. Specimen geometry [5].

and melt 3 at 640 °C for 10 hours. In order to reach certain strength level for each of the materials the additional heating, hardening and tempering was applied:

melts 1 and 2: heating from 600 up to 940 °C, holding for 20 min, water cooling

melt 3: heating from 600 up to 900 °C, holding for 20 min, water cooling. for more detail see [7].

A second series of additional treatments for the plates 631 (A) and 303 (M) were carried out in order to optimise the material properties (after first thermal treatment the DBTT's values were −6 and −42 °C, respectively, indicating probably non sufficient effect of first heat treatment):

Plate 631: heating up to 940 °C, holding for 20 min, water cooling, tempering at 655 °C for 10 h, at 660 for 2 h, air cooling, tempering at 670 °C for 5 h, air cooling.

Plate 303: heating up to 900 °C, holding for 20 min, water cooling, tempering at 640 °C for 10 h, air cooling, tempering at 640 °C for 5 h, air cooling.

Miniaturised Charpy V-notch samples (KLST) were used for the testing in order to allow minimised amount of wasted materials (see Figure 1).

A microcomputer based signal analyser μSCAN 500C in combination with a PCI-6111E computer card were used to pick up and to analyse the Barkhausen signal. For Barkhausen excitation a sinusoidal exciting magnetic field with magnetising voltage of 10 Vpp 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 [8, 9]. The applied magnetizing frequency was 10 Hz, which avoids sample vibration and ensures a good contact and fixation of the sample to the external magnetizing device. This is required in order to create suitable conditions for further measurements of the irradiated samples in the hot cells using remote manipulators [10, 11].

The root mean square values (RMS) of the noise signal were determined and were used to characterise the model steels (and to correlate with DBTT).

Impact Testing Hammer WOLPERT PW 5 (50 J) equipped by ISO 10 KN tup was used to test miniaturised Charpy V-notch samples (KLST) in the temperature range from −150 up to 150 °C. 1.9 J and 3.1 J were criteria used for DBTT determination [12].

3 RESULTS AND DISCUSSION

Table 2 and Figures 2, 3, 4 show the RMS values measured for the model steels. The results show that the amount of Cr plays an important role *ie* an increase of Cr content in model steels leads to an increase of RMS

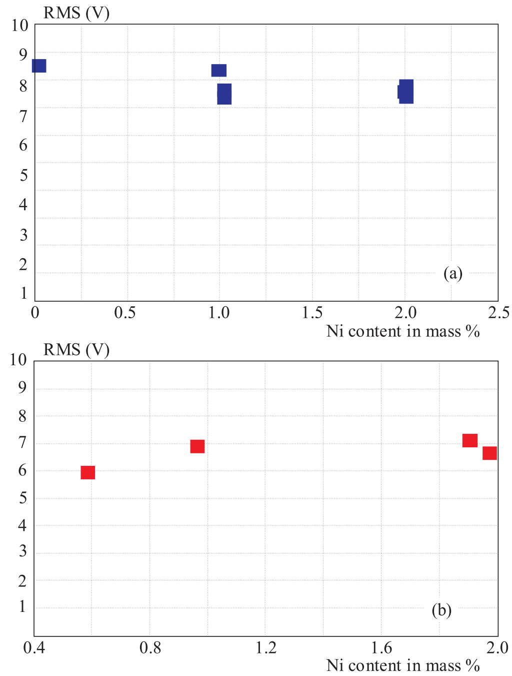

Fig. 2. RMS as a function of Ni content for: (a) – high Cr ($> 2\%$) and (b) – low Cr ($< 0.10\%$) containing steels

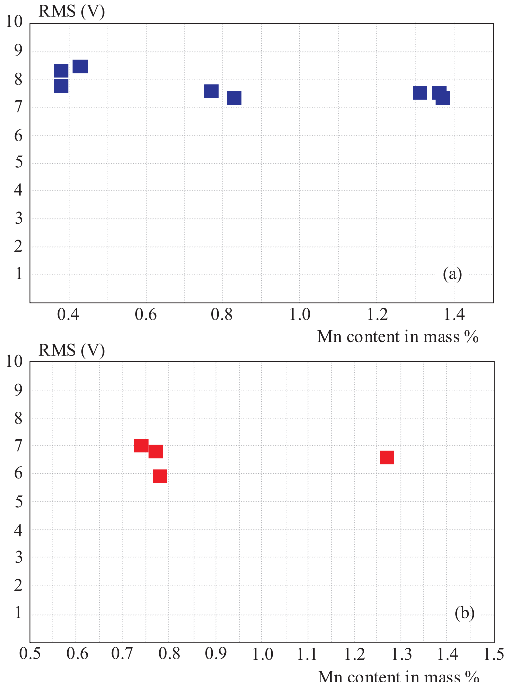

Fig. 3. RMS as a function of Mn content for: (a) – high Cr ($> 2\%$) and (b) – low Cr ($< 0.10\%$) containing steels

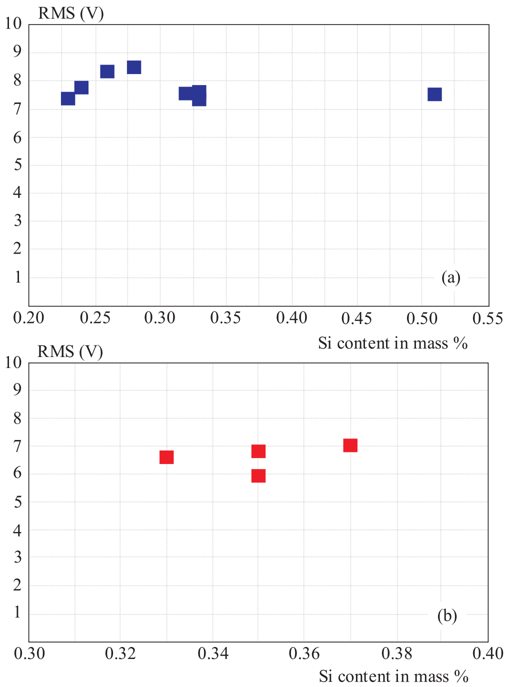

Fig. 4. RMS as a function of Si content for: (a) – high Cr ($> 2\%$), and (b) – low Cr $< 0.10\%$) containing steels

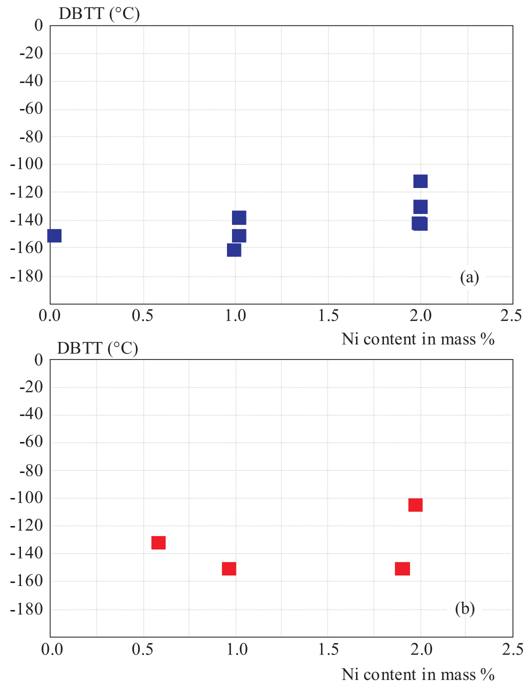

Fig. 5. DBTT as a function of Ni content for: (a) – high Cr ($> 2\%$), and (b) – low Cr ($< 0.10\%$) containing steels

values independently on Mn and Si content. From Table 2 and Figure 2 it can be seen that also the percentage of Ni has influence on RMS values. The RMS value of material labelled as material 631 or A with less than 0.02 mass % of Ni in combination with quite high percentage of Cr (about 2.22 mass %) is the highest one (8.51 V). The second highest value of RMS is obtained for material 632 (B) with high Cr content (about 2.19 mass %) and with just average percentage of Ni (about 0.99 mass %). The lowest value was measured from sample labelled as 304 combining low Cr content (0.07 mass %) with quite high Ni (1.97 mass %) and Mn (1.27 mass %) content.

After 3rd heat treatment the ranges of DBTT differ between high and low Cr containing steels only. When for

Table 3. Chemical composition of the reference materials

ID	C	Si	Mn	Cr	Ni	Mo	V	Cu	S	P	Al
JRQ	0.18	1.42	0.24	0.12	0.84	0.51	0.002	0.14	0.004	0.017	0.012
WWER-1000	0.17	0.30	0.46	2.2	1.26	0.50	0.10	< 0.08	0.01	0.008	–

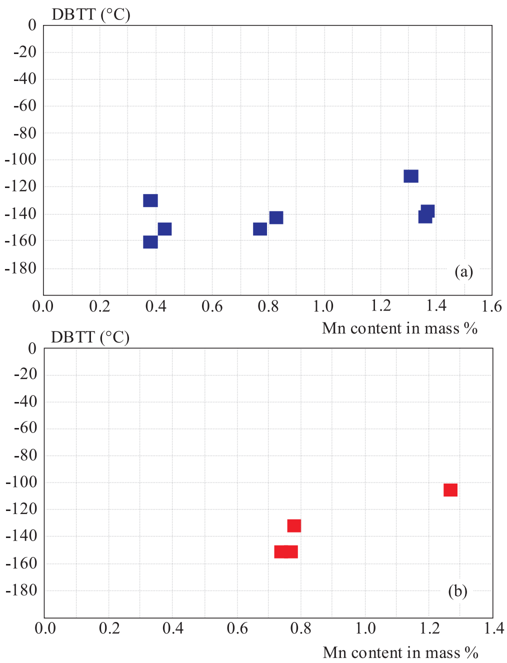

Fig. 6. DBTT as a function of Mn content for: (a) – high Cr ($> 2\%$), and (b) – low Cr ($< 0.10\%$) containing steels

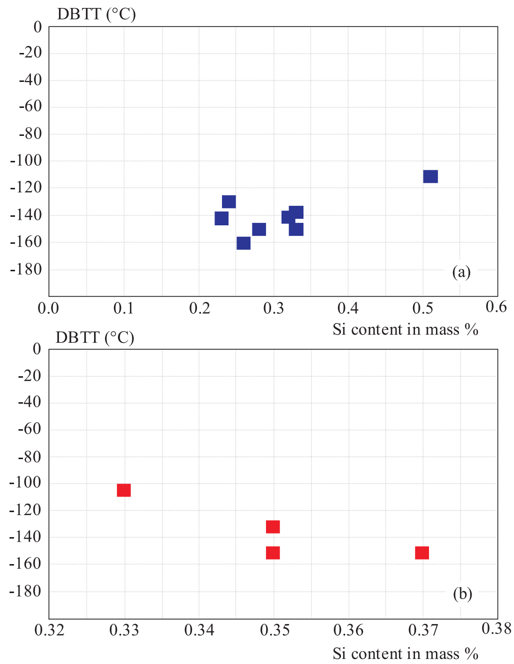

Fig. 7. DBTT as a function of Si content for: (a) – high Cr ($> 2\%$), and (b) low Cr ($< 0.10\%$) containing steels

Table 4. RMS and DBTT for JRQ and WWER-1000 reference materials, transition temperature criteria are in the brackets

Mark	JRQ	WWER-1000
RMS(V)	3.7	6
DBTT (°C)	$-50\,(47J)$	$-25\,(47J)$

high Cr containing steels the DBTT range from $-160\,$°C to $-112\,$°C, in the case of low Cr containing steels the DBTT's range from $-150\,$°C to $-104\,$°C. The lowest value of DBTT is reached for few different compositions, where one can find combination of very low Ni content with high Cr and low Mn content (631) but also very low Cr content with quite high Ni and middle range of Mn content (303). Generally the results imply that beside Cr no other elements have significant influence on the DBTT values as can be seen from Table 2 and Figures 2–7.

Figure 8 compares the results of Magnetic Barkhausen Noise (represented by RMS) and Charpy Impact Testing (represented by DBTT) obtained on the model steels with reference points set by measuring the standard reference materials as WWER-1000 and JRQ (see Table 3). These two materials differ mostly by Si, Cr and Al content. In the case of JRQ, the content of Si is quite high (about 1.42 mass %) while Cr content is rather low (0.12 mass %). JRQ material contents also limited amount of Al (0.012 mass %). In comparison with JRQ, WWER-1000 contains higher mass % of Cr (around 2.2 mass %) and low amount of Si (around 0.30 mass %). Comparison of the RMS values of WWER-1000 and JRQ materials shows that the RMS measured on WWER-1000 material is higher (6 V) then the one measured on JRQ material (3.7 V). As the WWER-1000 material contains quite high amount of Cr, this result is in good agreement with the previous results obtained on the 12 models steels which are mentioned above. Generally it can be seen that most of the model steels combine high values of RMS with low values of DBTT. When we compare the results of model steels with our reference points, one can see that the reference materials shove higher DBTT's and lower RMS in comparison with our model steels (see Fig. 8 and Tab. 4).

4 CONCLUSIONS

The 12 different kinds of high and low Cr model steels with parametric variation of Ni, Mn and Si were tested by different techniques including electromagnetic one. It is necessary to emphasize that for measurement of Magnetic Barkhausen Noise on KLST samples a frequency of about 10 Hz was used. This low measurements frequency

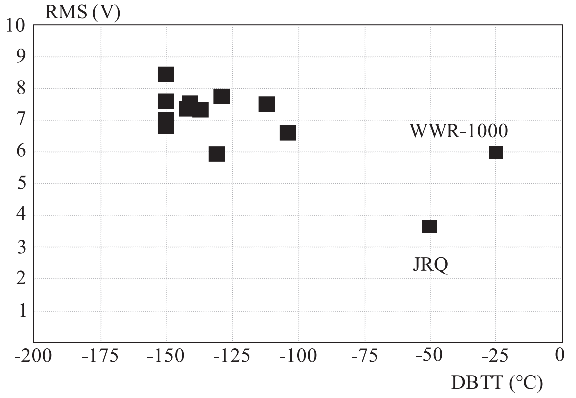

Fig. 8. RMS versus DBTT for 12 model steels and reference materials

was chosen in order to avoid sample vibration and to ensure a good contact and fixation of the sample to the external magnetizing tool and proved to be effective. Results represented by RMS values indicate that an increase of Cr content in model steels leads to an increase of RMS values independently on Mn and Si content. Correlation between the results shows that most of the model steels combine high RMS with high DBTT and are shifted more to the lower DBTT's in comparison with reference materials. JRQ showed high DBTT and lower RMS values in comparison with tested model steels whilst RMS value measured on WWER-1000 sample is twice as higher as those for JRQ. The results are generally as expected and they prove very promising in view of determining the role of chemical elements in properties variation due to the neutron irradiation.

Acknowledgement

This work was partially supported by VEGA 1/0606/09.

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Recieved 31 October 2008

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