



# Mechanical properties and damage behavior of non-magnetic high manganese austenitic steels

H. Takahashi<sup>a,\*</sup>, Y. Shindo<sup>a</sup>, H. Kinoshita<sup>a</sup>, T. Shibayama<sup>a</sup>, S. Ishiyama<sup>b</sup>,  
K. Fukaya<sup>b</sup>, M. Eto<sup>b</sup>, M. Kusuhashi<sup>c</sup>, T. Hatakeyama<sup>c</sup>, I. Sato<sup>c</sup>

<sup>a</sup> Center for Advanced Research of Energy Technology, Hokkaido University, Kita-ku, Kita-13, Nishi-8, Sapporo 060, Japan

<sup>b</sup> Tokai Establishment, Japan Atomic Energy Research Institute, Tokai, Japan

<sup>c</sup> The Japan Steel Works. LTD. Muroran Research Laboratory, Muroran, Japan

## Abstract

Fe–Cr–Mn steels have been considered as materials of structural components for fusion reactor because of their low induced-radio-activity compared with SUS316 stainless steels. It has been expected to develop a non-magnetic steel with a high stability of the austenitic phase and a strong resistance to irradiation environments. For these objectives, a series of the Fe–Cr–Mn steels have been examined by tensile tests and simulation irradiation by electrons. The main alloying compositions of the steels developed are; C:0.02–0.2 wt%, Mn: 15 wt%, Cr: 15–16 wt%, N: 0.2 wt%. These steels were heat-treated at 1323 K for 1 h. The structure of the steels after the heat-treatment was austenite single phase. The yield stress of the steels was 350–450 MPa and the elongation were 55–60%. When the steels of high C and N was electron-irradiated at below 673 K, no voids were nucleated and only small dislocation loops were formed with high density. The austenite phase was also stable during irradiation below 673 K. Thus, newly developed high manganese steels have excellent mechanical properties and high irradiation resistance at relatively low temperature. © 1998 Elsevier Science B.V. All rights reserved.

## 1. Introduction

Manganese steels are candidate materials for structural components of fusion reactors because of their low induced long-term radioactivity compared to that of typical Fe–Cr–Ni austenitic steels [1,2]. However, recent studies on Fe–Cr–Mn steels have indicated that neutron irradiations at high temperature lead to both degradation of the mechanical properties and phase instability caused by manganese loss from the surface of the steel [3,4]. Commercial Fe–Cr–Mn steels have been developed for usage at low-temperature as non-magnetic structural materials. However, Mn based steels used at elevated temperature have not been developed up to now, because of their lesser oxidation resistance, mechanical properties and phase stability [5–8], even though some studies have tried to improve those properties by the addition of alloying elements. For example, the addition

of aluminum can improve tensile properties and oxidation resistance of these steels [9,10]. Recently, as a material for the vacuum vessel of fusion reactor operated at relatively low temperature and under low neutron dose, the Fe–Cr–Mn steels have been considered because of their non-magnetic properties. However, there is not enough data on mechanical properties and phase stability of Fe–Cr–Mn steels under irradiation conditions.

The aims of this study is to find the optimum composition of Fe–Cr–Mn steel for of stability under irradiation at medium temperature between 373 and 673 K and to develop steels with high irradiation resistance and excellent mechanical properties under the conditions where steel will be used in vacuum vessel of fusion reactor.

## 2. Experiments

### 2.1. Materials

(1) Material preparation: Ten steels with different compositions were prepared. The compositions of these

\* Corresponding author. Tel.: +81-11-706 6767; fax: +81 11 757 3537; e-mail: takahasi@ufml.caret.hokudai.ac.jp.

Table 1  
Chemical compositions of Fe–Cr–Mn steels used in this experiment

	Mn	Cr	C	N	Si
VC0	24.5	13.5	0.02	0.20	0.50
VC1	24.5	15.2	0.02	0.20	0.30
VC2	18.5	15.2	0.02	0.20	0.30
VC3	15.5	15.2	0.02	0.20	0.30
VC4	12.5	15.2	0.02	0.20	0.30
VC5	9.5	15.2	0.02	0.20	0.30
VC6	6.5	15.2	0.02	0.20	0.30
VC7	6.5	16.5	0.02	0.20	0.30
VC8	15.5	15.2	0.20	0.20	0.30
VC9	15.5	16.0	0.20	0.20	0.30

steels were given in Table 1. Steels VC1–VC6 are a series of Mn based alloys with 0.02% C–15.2% Cr–0.2% N. VC8 and VC9 steels contain high C levels.

(2) Fabrication techniques: The steels were prepared by the following fabrication process. The steel ingots were melted by a high frequency furnace. After that the ingots were heat-treated at 1423 K for 10 h, and then forged and rolled to plates with 50 mm thickness. After forging and rolling, heat treatment of the plates was further performed at 1323 K  $\times$  1 h followed by quenching in water.

### 2.2. Material characterization

Microstructure and grain size of these steels were observed by optical microscopy. Mechanical properties were examined by tensile and Vickers hardness tests at room temperature. Tensile specimens of 10 mm in diameter with 50 mm gauge were prepared from the plate. Magnetic permeability of the steels was measured for the specimen fractured by tensile test.

### 2.3. Irradiation test

Electron irradiations were performed for VC0, VC3, VC8 and VC9 steels at 1000 KV using a H-1300HVEM at a mean dose rate of  $2 \times 10^{-3}$  dpa  $s^{-1}$  at the temperature range between 473 and 673 K.

## 3. Results

### 3.1. Phase stability of the materials

The microstructure and permeability of as-cast steel were observed to classify the phases after heat-treatments. The results were summarized in Table 2.  $\delta$ ,  $\delta + \gamma$  phase or  $\delta + \gamma + M$  (martensite) structures were observed in VC0–VC7 steels with high permeability. The VC8 and VC9 steels were composed of the  $\gamma$  single phase. In the case of VC1 steel with large amount of  $\delta$

phase and/or other second phase (8.2%), lower permeability was exhibited.

Figs. 1 and 2 show  $\delta$  ferrite formation (and other second phase) and permeability after heat-treatment of the as-casted steels at elevated temperature. Phase transformation to the  $\delta$  and increasing permeability in VC0–VC7 steels were recognized at the temperature of 1423 and 1473 K. This suggests that the transformation of  $\gamma$  to  $\delta$  phase occurs in this temperatures range. Phase stabilities of VC8 and VC9 steels, composed of  $\gamma$  single phase are tested at the temperature range.

Phase stability and permeability of as-forged/rolled, and after heat treatment at 1283–1363 K, are given in Table 3. From these results, it is obvious that  $\delta$  phase in VC0–VC4 steels is not formed and tended to transformation to  $\gamma$  single phase after hot forging and rolling. However, it may be very hard to the achieve phase transformation to  $\gamma$  single phase in VC5–VC7 steels composed of  $\delta$  phase more than 5% or M phase by hot-forging and rolling treatment.

### 3.2. Mechanical properties of the materials

Fig. 3 showed the mechanical properties of the Fe–Cr–Mn steels and SUS316. High strength and higher ductility were recognized for VC0–VC4 and VC8–VC9

Table 2  
Phases of as-casted Fe–Cr–Mn steels after heat treatment

Steels	Phases
VC0	$\gamma + \delta$
VC1	$\gamma + \delta$
VC2	$\gamma + \delta$
VC3	$\gamma + \delta$
VC4	$\gamma + \delta$
VC5	$\gamma + \delta +$ martensite (M)
VC6	$\gamma + \delta +$ martensite (M)
VC7	$\gamma + \delta +$ martensite (M)
VC8	$\gamma$
VC9	$\gamma$

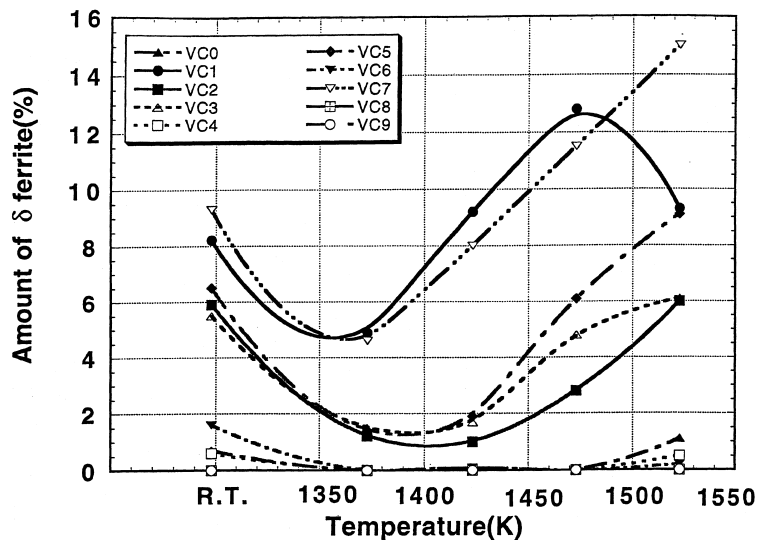


Fig. 1. Amount of  $\delta$ -ferrite after heat-treatment of steels.

steels were compared with SUS316 steel. The tensile strength increased with decreasing manganese content for VC0–VC4 except VC8–VC9 steel with higher carbon content. Fractured specimens showed increasing permeability of the VC2–VC4 steels.

3.3. Irradiation damage

Fig. 4 shows the microstructures of voids formed during electron-irradiation of VC0, VC3, VC8 and VC9 steels at 473, 573 and 673 K to 3 dpa. Voids were observed at the temperature range of 473 and 673 K for VC0, which is a higher Mn-containing steel. In the steel of VC3, no voids were formed at 473 K, and for VC8

voids were nucleated only at 673 K. In the VC9 steel, no voids were observed over the entire temperature range between 473 and 673 K. From these results, it is obvious that void nucleation is retarded with increasing Cr and C contents.

4. Discussion

4.1. Phase stability of the materials

In order to obtain the steels composed of  $\gamma$  single phase, it is important to make clear the effect of C, N, Mn and Cr content on phase stability. The data for the

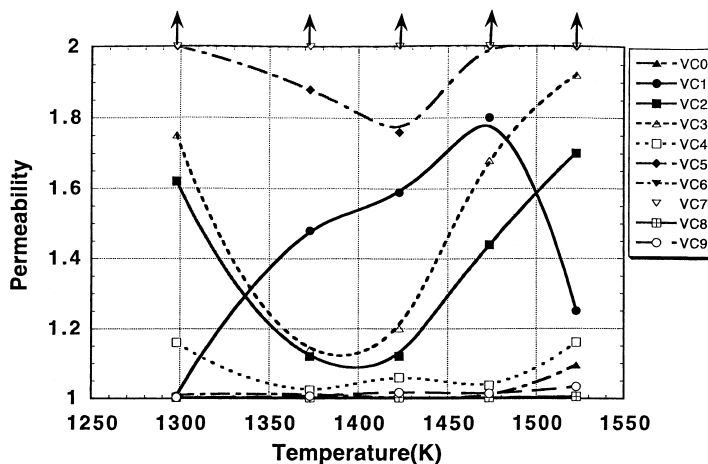


Fig. 2. Permeability of steels after heat-treatment.

Table 3  
Amount of  $\delta$  ferrite (second phase) and permeability of as-forged, as-rolled, and after heat treatment

Steels	As-forged	As-rolled	1283 K	1323 K	1363 K
<i>Amount of <math>\delta</math> ferrite (second phase) (%)</i>					
VC0	0	0	0	0	0
VC2	0.6	0	0	0	0
VC3	0.7	0.1	0	0	0
VC4	0	0	0	0	0
VC8	0	0	0	0	0
VC9	0	0	0	0	0
<i>Permeability</i>					
VC0	1.003	1.002	1.003	1.003	1.003
VC2	1.040	1.018	1.009	1.007	1.015
VC3	1.064	1.019	1.014	1.011	1.019
VC4	1.008	1.004	1.004	1.004	1.003
VC8	–	1.005	1.003	1.003	1.003
VC9	–	1.004	1.003	1.003	1.003

as-casted steels tested were plotted in the Hull diagram [11,12] as shown in Fig. 5. In this figure, data obtained in this experiment can be divided into the  $\gamma$  single phase region and the  $\gamma + \delta$  complex phase region. This diagram indicates that steels with lower  $Ni_{eq}$  and higher  $Cr_{eq}$  value yield large amounts of  $\delta$  phase, whereas the steels of higher  $Ni_{eq}$  value and lower  $Cr_{eq}$  lead to small amount of  $\delta$ . Thus, in order to minimize the formation of  $\delta$  ferrite, it is more effective to decrease the Mn content as far as possible, whereas the content of other alloying elements except Mn appears to be constant. However, it must be noticed that there may be critical

compositions to minimize Mn content, because the martensite phase is formed in lower Mn-containing steels such as VC5–VC7.

After hot forging, the amount of  $\delta$  phase in the VC0, VC2–VC4 steels decreased. In particular, no  $\delta$  phase was formed in VC0 and VC3 steels. On the other hand,  $\delta$  phase still remained in the VC2 and VC3 steels with high permeability. Decreasing permeability in VC2 and VC3 steels was recognized after heat treatment at 1323 K. There was no change in permeability for the rolled VC0, VC4, VC8 and VC9 steels with very lower permeability, even after heat treatment.

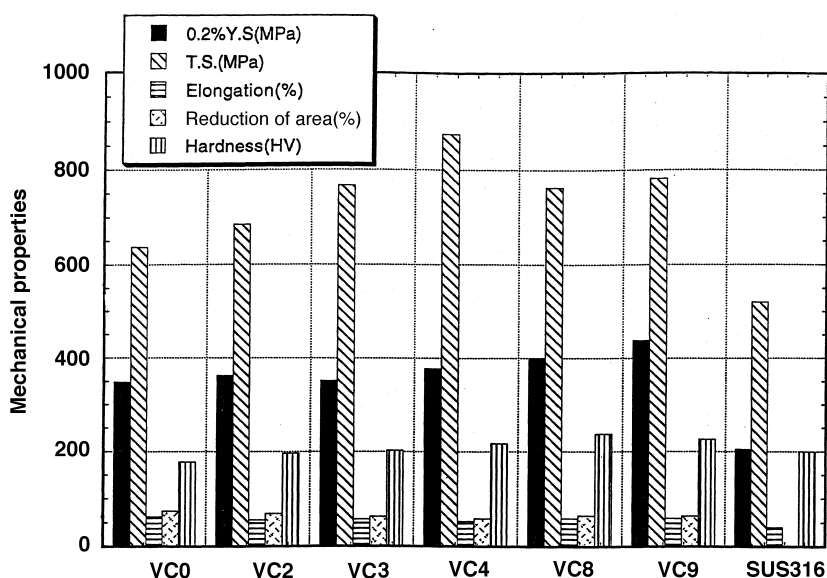


Fig. 3. Mechanical properties of Fe–Cr–Mn Steels and SUS316 at room temperature.

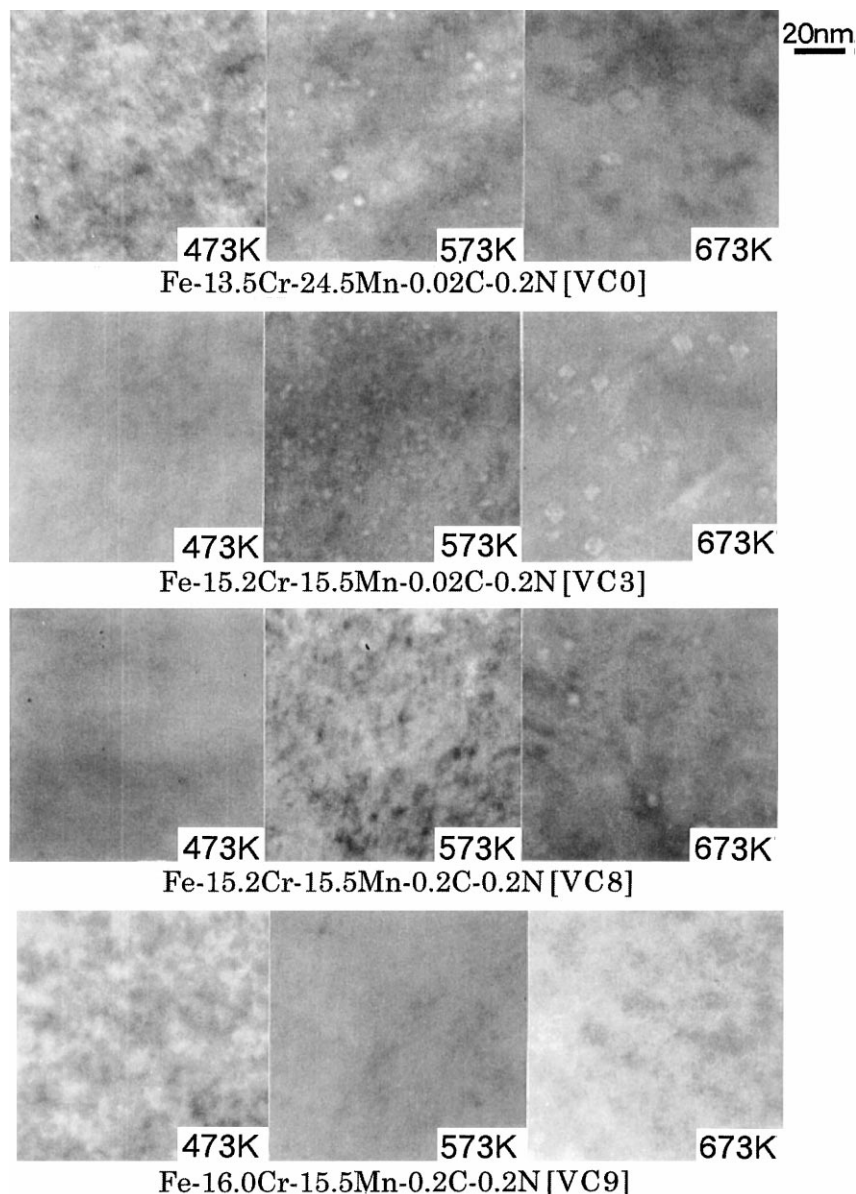


Fig. 4. Microstructures observed during electron-irradiation.

From these results, it can be concluded that a reasonable heat-treatment temperature to obtain non-magnetic  $\gamma$  single phase austenite is 1323 K.

#### 4.2. Characterization of materials

High strengthened steel was obtained in present experiment. It is suggested that the high strength of steels such as VC8 and VC9 was caused by the higher carbon content.

A remarkable improvement of tensile test results was observed in the VC3 and VC4 steels. This is caused by deformation-induced martensite formation, because these steels showed very high permeability after the tensile test. There are well known indices for martensite formation with chemical composition, such as  $Md_{30}$  [3] and  $Ni_{eq}$  value [14]. The relationship between these indices and the permeability obtained in the tensile test is shown in Figs. 6 and 7. It is clear that with increasing  $Ni_{eq}$  or decreasing  $Md_{30}$ , martensite formation

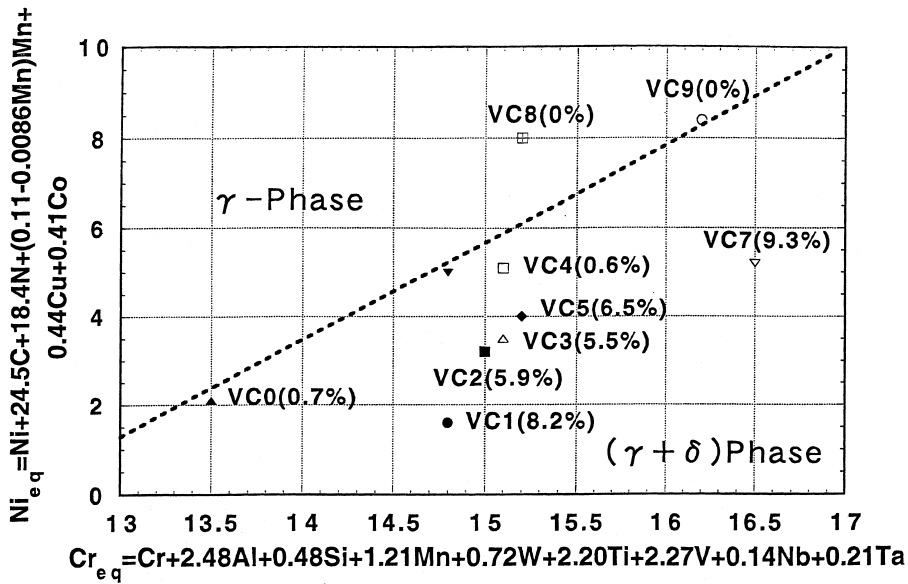


Fig. 5. Classification of austenitic  $\gamma$  and  $(\gamma + \delta)$  phases based on  $Ni_{eq}$  and  $Cr_{eq}$  for Fe–Cr–Mn steels used in this study.

is restrained so that the  $\gamma$  phase is stabilized as proposed by Angle [13] and Hirayama [14]. From the minimum value of  $Ni_{eq}$  or maximum value of  $Md_{30}$  for VC4, this steel is easily transformed to the martensite phase under deformation so that higher yield stress and tensile strength can be reached. Therefore it is suggested that Cr and Mn contribute to  $\gamma$  phase stability, but, have an inverse effect to restrain  $\delta$  ferrite formation. Therefore, further improvement is needed to optimize the composition as a non-magnetic high manganese austenitic steel, even though the steels developed in the present study were relatively stable during irradiation.

#### 4.3. Irradiation damage

As shown in Fig. 4, voids formation was retarded at 473 K with decreasing Mn and increasing Cr for the steel containing 0.02 wt% C and 0.2 wt% N. Furthermore, in the steels with the same levels of Cr and Mn, the temperature to nucleate void shifted to higher temperature side of 673 K with increasing C content. With further increasing of Cr content from 15.2 to 16 wt%, the void formation was restrained at temperature range between 473 and 673 K.

Thus, Cr and C are effective to restrain void nucleation even in Fe–Cr–Mn steels with  $\gamma$  single phase.

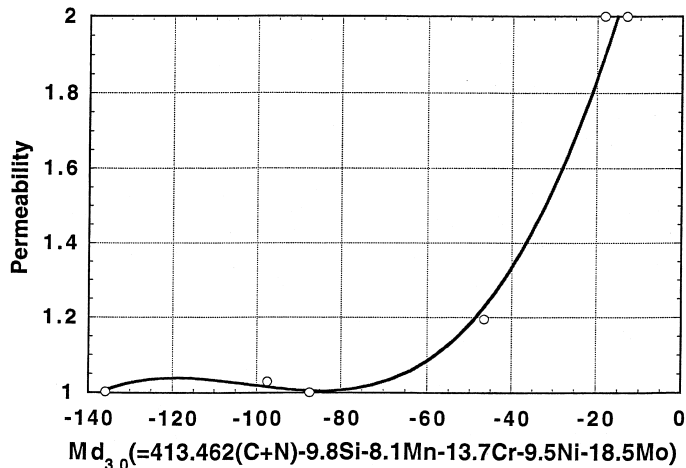


Fig. 6. Relationship between  $Md_{30}$  and Permeability of Fe–Cr–Mn steels used in this study.

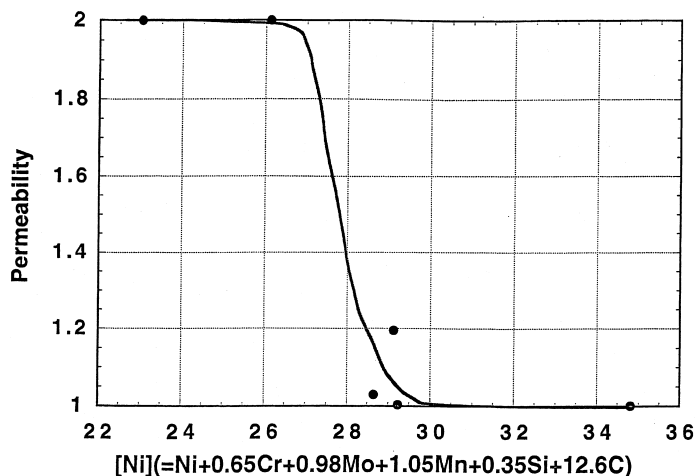


Fig. 7. Relationship between  $Ni_{eq}$  and permeability of Fe–Cr–Mn steels used in this study.

## 5. Conclusions

The results obtained in present study are as follows:

1.  $\gamma$  single phase in the lower Mn containing steels can be obtained by controlling the Cr, C and N content besides heat-treatment conditions.

2. The steels with 15–16 wt%Cr–15.5Mn–(0.02–0.2)wt%C–0.2wt%N, developed in this study gave mechanical properties with the highest strength and largest elongation

3. Void formation was suppressed with increasing Cr and C content in the steels with 15.5 wt%Mn–0.2wt%N. No voids were nucleated in the steel of Fe–16wt%Cr–15.5wt%Mn–0.2wt%N–0, 2wt%C.

## References

- [1] D.G. Doran, H.L. Heinisch, F.M. Mann, *J. Nucl. Mater.* 133/134 (1985) 892.
- [2] F.A. Garner, F. Abe, T. Noda, *J. Nucl. Mater.* 155–157 (1988) 870.
- [3] J.M. McCarthy, F.A. Garner, *J. Nucl. Mater.* 155–157 (1988) 877.
- [4] J.M. McCarthy, F.A. Garner, Reduced activation materials for fusion reactors, ASTM STM 1047, in: R.L. Klueh, D.S. Gelles, M. Okada, N.H. Packa, (Eds.), American Society for Testing and Materials, Philadelphia, PA, 1990, p. 10.
- [5] Y. Okazaki, M. Mochizuki, K. Miyahara, Y. Hosoi, Reduced activation materials for fusion reactors, ASTM STM 1047, in: R.L. Klueh, D.S. Gelles, M. Okada, N.H. Packa, (Eds.) American Society for Testing and Materials, Philadelphia, PA, 1990, p. 80.
- [6] N. Yikata, M. Morinaga, K. Nishiyama, Y. Matsumoto, Y. Murinaga, Y. Murata, H. Ezaki, Reduced activation materials for fusion reactors, ASTM STM 1047, in: R.L. Klueh, D.S. Gelles, M. Okada, N.H. Packa (Eds.), American Society for Testing and Materials, Philadelphia, PA, 1990, p. 30.
- [7] W. Shule, E. Lang, A. Panzarasa, Report EUR 11756, EN, 1990.
- [8] J.I. Cole, D.S. Gelles, J.J. Hoyt, *J. Nucl. Mater.* 191–194 (1992) 657.
- [9] H. Takahashi, S. Ohnuki, in: Proceedings of the High Manganese Austenitic Steels, ASM.Intern., Chicago, 1993, p. 131.
- [10] H. Takahashi, S. Ohnuki, H. Kinoshita, F.A. Garner, Reduced activation materials for fusion reactors, ASTM STM 1047, in: R.L. Klueh, D.S. Gelles, M. Okada, N.H. Packa, (Eds.), American Society for Testing and Materials, Philadelphia, PA, 1990, p. 93.
- [11] F.C. Hull, *Welding J.* 52 (1973) 193.
- [12] R. Frank et al., *Trans. ASM* 47 (1955) 231.
- [13] T. Angel, *J. Iron Steel Inst.* 177 (1954) 165.
- [14] T. Hirayama, M. Ogirima, *J. Jpn. Inst. Met.* 34 (1970) 507.