Review

Recent Research and Development of Reduced Activation Ferrous Materials in Japan

Yuzo HOSOI

Department of Materials Science and Engineering, Faculty of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya, Aichi-ken, 464 Japan.

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The outline of research and development of reduced activation ferrous materials, today in Japan, is reviewed. Reduced activation ferrous materials are considered to be utilized for the first wall materials of fusion reactor. The concept of reduced activation materials is closely related to the waste management and reactor safety. The decay behavior of induced radioactivity shows that the substitution of Mn for Ni and that of W for Mo in ferrous materials are very effective to achieve a reduced level of long-term radioactivity. The efforts are being devoted to the development of ferritic 9Cr-W steels, austenitic Mn-Cr steels and Mn-Cr-W steels as candidate alloys, instead of 9Cr-Mo steels and modified type 316 stainless steels. Some results are presented to the toughness before and after neutron irradiation and creep rupture strength of 9Cr-W steels, and on the characteristics of sigma phase formation, high-temperature strength, and void swelling of austenitic Mn-Cr steels and Mn-Cr-W steels. Preferable chemical compositions for reduced activation ferrous materials are suggested.

KEY WORDS: ferritic 9Cr-W steels; austenitic Mn-Cr steels; induced radioactivity; toughness; high-temperature strength; sigma phase formation.

1. Introduction

The first wall materials of fusion reactor are strongly activated by neutrons with high energy of 14 MeV, produced by D-T reaction.* This induced radioactivity of the materials is considered to be one of the most serious problems from the view-points of reactor safety, operation and maintenance, waste management and environmental concerns for fusion reactor.

The research works of reduced activation materials originated in the early 1980s in U.K. and U.S.A. The studies were mainly focused on the decay characteristics of the radioactive isotopes that make up typical structural materials.¹⁾ The work on reduced activation materials is now extended to radiation damage, structural stability, mechanical properties and alloy design in U.S.A., Europe and Japan.¹⁻⁶⁾

In Japan, the study of low or reduced activation materials had started about 5 years ago in Japan Atomic Energy Research Institute, National Research Institute for Metals, and University Collaborative Research Group under the leadership of A. Miyahara and S. Ishino by sponsorship of the Grant-in-Aid for Fusion Research of the Ministry of Education, Science and Culture of Japan.

The low or reduced activation materials are required from the contact maintenance, waste management, and reactor safety. Some goal of such items was pointed out as follows⁷⁾:

- 1) Contact maintenance—Surface dose rate or γ -ray attains 2.5 mrem/h within several weeks after shutdown.
- 2) Waste management—The activity satisfies the land disposal limits such as natural radioactivity level in 100 years after shutdown or 10CFR61 limits (Part 61 of Title 10 of US Code of Federal Regulations⁸⁾) with respect to land burial of radioactive waste.
- 3) Reactor safety—Minimum BHP (biological hazard potential) values or lowest decay heat.

As far as the contact maintenance, the selection of materials is very limited. U.S. Department of Energy Panel in 1983 suggested that high-purity silicon carbide was only true low activation material.¹⁾ It is clearly understood from the decay characteristics of Si and C being referred from Fig. 1.⁷⁾ Vanadium would be selected in this class. There are, however, several difficulties in producing and fabricating large constructional structure using these materials, and also in controlling the impurity concentration at a level of ppm or less. On the other hand, ferrous materials are the most suitable for the construction of large structure because of many examples of large structure in nuclear plants and numerous data of mechanical, physical, chemical properties, fabricability, weldability and so on. Accordingly, the development of reduced activation ferrous materials is the most practical way for the construction of first wall. In

* The first wall of fusion reactor is the inner wall of huge vacuum vessel facing the plasma where D (deuterium)-T (tritium) fusion reaction occurs; $^2\text{D} + ^3\text{T} = ^4\text{He}$ (3.52 MeV) + n (14.06 MeV).

Fig. 1. The cooling time of first wall materials required for 2.5 mrem·h⁻¹ as a function of neutron fluence.

this sense, it is required to develop the reduced activation ferrous materials which satisfy the land disposal limit, 10CFR61 limit. The Panel mentioned above previously recommended also that materials be developed to help avoid a nuclear waste-disposal problem that would occur when fusion reactor components had to be discarded.¹⁾

This review gives an outline of the trend and research and development of reduced ferrous materials in Japan.

2. Induced Radioactivity of Elements

Induced radioactivity of elements has been calculated by several investigators. One of examples of calculation results of induced radioactivity of elements and their decay behaviors is shown in Fig. 1. This figure shows the cooling time for several elements required for 2.5 mrem·h⁻¹ as a function of the neutron fluence. Si and C show the very fast decay even after irradiation of the neutron fluence of 10 MW·y·m⁻². Chromium requires about 1 year and Fe and W about 10 years for the cooling time, whereas Ni requires about 100 years after irradiation of similar level of neutron fluence. In the case of Mo, the cooling time increases steeply at around 10⁻² MW·y·m⁻², because of the accumulation of long-lived nuclides such as ⁹⁴Nb, ⁹²Nb for Mo.⁷⁾

Fig. 2. Necessary concentration of first wall materials of which activities satisfy 10CFR61 class C limits just after shutdown as a function of neutron fluence.

Fig. 2 shows the acceptable concentration of elements required to satisfy the class C limit of 10CFR61 as a function of the neutron fluence.⁷⁾ It is noted that Fe, Mn, Cr, V, W and Ti are not limited in concentration as first wall material to make class C limit disposal in the operation of 10 MW·y·m⁻². F. M. Mann⁹⁾ proposed the acceptable concentration of elements to make class C limit as follows; Nb<0.00029 %, Mo<0.003 %, Cu<0.12 %, N<0.33 %, Ni<0.91 %, Pb<3.1 %, and Al<3.6 %.

3. Reduction of Radioactivity for Ferrous Materials

Type 316 stainless steel and modified type 316 (PCA and JPCA)* have been considered to be preferable candidate alloys for the first wall material, because of balanced properties for structural material and numerous data of radiation damage. On the other hand, recently, ferritic heat resisting steels, modified 9Cr-Mo* and HT9* steels, have also been selected as candidate materials from the standpoint of superior resistance to void swelling and helium embrittlement compared with austenitic alloys.¹⁰⁾

As mentioned above in previous section for radioactivity of elements, the substitution of Mn for Ni and that of W for Mo in ferrous materials are very effective to achieve a reduced level of long-term radioactivity, taking into consideration of role of alloying

*	Nominal chemical composition (mass%)										
	C	Si	Mn	Cr	Ni	Mo	Ti	V	Nb	W	P
Type 316	0.05	0.5	1.5	18.0	12.0	2.3	—	—	—	—	0.02
PCA	0.05	0.4	1.8	14.0	16.0	2.3	0.24	—	—	—	0.01
JPCA	0.05	0.5	1.8	15.0	16.0	2.3	0.24	—	—	—	0.03
Mod. 9Cr	0.10	0.3	0.5	9.0	—	1.0	—	0.2	0.10	—	—
HT9	0.20	0.3	0.5	11.0	0.5	1.0	—	0.3	0.30	0.5	—
JFMS	0.05	0.7	0.5	10.0	0.9	2.3	—	0.1	0.06	—	—

elements in the materials. It introduces that ferritic 9 to 12Cr-W steels instead of 9 to 12Cr-Mo steels (modified 9Cr-Mo, HT9, or JFMS)* and austenitic high Mn-Cr and high Mn-Cr-W steels instead of Ni-Cr-Mo steels (AISI 316, PCA, or JPCA) are available as preferable candidates for reduced activation ferrous materials. Fig. 3 shows the decay of surface dose rate of induced activity of PCA, 9Cr-2W steel and 30Mn-10Cr steel with cooling time.¹¹⁾ The first gamma-ray intensity was estimated as the intensity after shutdown of operation of 1 MW·m⁻² for 10 years. It is obvious that the decay of induced activity of 9Cr-2W and 30Mn-10Cr steels is much faster than that of PCA which contains Ni and Mo.

Under these conditions, the fundamental studies and the development of ferritic Cr-W steels and austenitic Mn-Cr steels have been promoted as reduced activation ferrous materials for fusion reactor. The important theme to be studied in these materials are toughness, fracture toughness, high-temperature strength, compatibility to environment, and radiation damage for ferritic alloys, and structural stability at high-temperature, high-temperature strength, compatibility to environment and radiation damage for austenitic alloys. Weldability and formability are also investigated for the development of these materials.

Fig. 3. Gamma-ray decay behaviors of PCA, Fe-9Cr-2W and Fe-30Mn-10Cr alloys. (Gamma-ray intensity after shutdown of operation of 1 MW·m⁻² for 10 years)

Fig. 4. Temperature dependence of the Charpy-V-notch absorbed energy of 9Cr-W ferritic steels.

4. Reduced Activation Ferritic Steels

4.1. Change in Toughness by Neutron Irradiation

The decrease in toughness during thermal aging and neutron irradiation is the most serious problem for ferritic 7 to 12Cr-W steel as well as 7 to 12Cr-Mo steel. Fig. 4 shows the effect of neutron irradiation on Charpy impact energy of various 9Cr-2W steels.¹²⁾ The change in DBTT (ductile-brittle transition temperature) of these steels is listed in Table 1. The steel with base composition (9Cr-2W-0.5Mn-0.25V-0.05Ta-0.02Ti-0.03B) shows the best impact properties, the smallest change of DBTT and the lowest DBTT before and after irradiation. The combined addition of small amount of Ti and B was reported to be beneficial in making fine ferrite grain structure.¹²⁾ Rise in DBTT of ferritic steels caused by neutron irradiation is affected by Cr content. As shown in Fig. 5, the lowest DBTT is reached at around 9 % Cr.¹³⁾

4.2. High-temperature Strength and Toughness

Creep rupture strength of 9 Cr steel is increased with increasing the alloying content of W (Fig. 6¹⁴⁾).

Table 1. DBTT of irradiated and unirradiated 9Cr-W ferritic steels and difference of DBTT (ΔT) between unirradiated and irradiated materials.

Specimens	DBTT (K)		ΔT (K)
	Unirradiated	Irradiated	
(1) Base*	148	158	10
(1') No B	167	181	14
(2) 1 Mn	160	193	33
(3) 2 Mn	156	167	11
(4) No Ta	183	193	10
(4') 5.05 Y	183	—	—
(5) 0.1 Ta	161	195	34
(6) 0.05 Ti	159	213	54
(8) 1 W	161	172	11
(8) 7Cr-1W	143	173	30
9Cr-1Mo	169	203	34

* Fe-9Cr-2W-0.5Mn-0.25V-0.05Ta-0.02Ti-0.003B.

* See Table of footnote on p. 918.

Fig. 5. Dependence of the DBTT of several ferritic steels on Cr content before and after neutron irradiation, in comparison with the previous data by H. Kayano *et al.*

Fig. 6. Creep rupture strength of 9Cr-W steels at 873 K.

The addition of W is helpful in making martensite lath stable during creep at around 873 K. Beneficial effect of W to creep rupture strength is also shown in 2.25 to 3 Cr steel. Fig. 7 shows the relation between applied stress and creep rupture time at 873 K for various Cr steels containing 2 % of W.¹⁵⁾ It is demonstrated that the creep rupture strength of 2.25 to 3Cr-2W steel is superior to that of 2.25Cr-1Mo steel.

The addition of W, however, decreases the tough-

Fig. 7. Creep rupture strength of various (2.25-3)Cr-W steels at 873 K.

Fig. 8. Degradation curves of the absorbed energy at 273 K of the 9Cr-W steels, as a function of aging time at 823, 873, 923 and 973 K.

ness of 9 Cr steel. Fig. 8 indicates the change in absorbed energy at 273 K by impact test during aging.¹¹⁾ The increase of W content decreases the absorbed energy more rapidly during aging. The decrease of toughness is mainly caused by precipitation of Laves phase, Fe_2W .^{11,16)} Fig. 9 shows the relation between creep rupture strength and DBTT of 9Cr-W steel.¹⁷⁾ The results suggest that the addition of 2 % of W is preferable in both creep rupture strength and toughness.

A large possibility is suggested for improving creep rupture strength and toughness in 9 Cr steel by substitution of W for Mo, although more extended studies, such as weldability and formability, are needed. A good resistance to void swelling in 9Cr-W steels is also being confirmed.¹⁸⁾ The following alloy compositions

Fig. 9. The relation between DBTT and creep rupture strength of 9Cr-W steels.

Table 2. Proposed materials as preferable reduced activation ferritic steels. (mass%)

Ref.	C	Si	Mn	Cr	W	V	Ta	N
19)	0.12	0.05	0.5	8.5	2.5	0.20	0.12	0.03
20)	0.10	0.10	0.5	8.0	2.0	0.20	0.04	
15)	0.10	<0.10	0.5	9.0	2.0	0.20	0.10	0.05

Heat treatment:

Ref. 19)	1 323 K × 1.8 ks	Normalizing
	1 023–1 073 K × 3.6 ks	Tempering
Ref. 20)	1 313 K × 1.8 ks	Normalizing
	1 013 K × 7.2 ks	Tempering
Ref. 15)	1 323 K × 1.8 ks	Normalizing
	1 048 K × 3.6 ks	Tempering

are recommended as preferable reduced activation ferritic steels.^{15,19,20)} (Table 2)

5. Reduced Activation Austenitic Steels

5.1. σ Phase Formation

In Fe-Cr-Mn alloy, σ phase formation is one of the serious problems and markedly affected by Mn content. Increase in Mn content promotes the σ phase formation in the temperature range from 773 to 973 K as shown in Fig. 10.²¹⁾ It is noted that this effect of Mn is quite different from the effect of Ni which prevents σ phase formation. Chromium is also a detrimental element in forming σ phase. As Cr content increases, σ phase forms more easily (Fig. 11).²²⁾

Effect of Mn and Cr contents on σ phase formation is summarized in the Fe-Cr-Mn ternary system at 923 K obtained by Burgess and Forgeng²³⁾ (Fig. 12²²⁾). In the present case, all data were obtained from aging for 3.6×10^3 ks after cold rolling. It is noted that the region of σ phase formation spreads more widely than that indicated in the phase diagram. In this figure, the predicted $\gamma/(\gamma+\sigma)$ boundary ($\bar{M}d$ line) is shown, which is evaluated using the d -electron concept by Yukawa *et al.*²⁴⁾

○: Non σ phase ●: σ phase formation

Fig. 10. Effect of manganese content, aging time and aging temperature on the formation of σ phase in Fe-16%Cr-Mn alloys.

○: Non σ phase ●: σ phase formation

Fig. 11. Effect of Cr content and aging temperature on σ phase formation in Fe-15Mn-Cr alloys after solution treatment followed by 50 % cold work.

The temperature dependence of $\bar{M}d^*$ for the $\gamma/\gamma+\sigma$ phase boundaries in various ternary alloys is also shown in Fig. 13,²⁵⁾ and they expressed its temperature dependence as follows:

$$\text{Critical } \bar{M}d = 6.25 \times 10^{-5} T + 0.834.$$

They also confirmed that the stable γ phase field can be more accurately presented by the $\bar{B}o^*$ vs. $\bar{M}d$ diagram in Fe-Cr-Mn-Ni alloys as shown in Fig. 14,²⁵⁾ than the Schaeffler diagram.²⁶⁾

C, N and Ni are well known to be very beneficial elements for preventing σ phase formation in stainless steels. A similar effect of these elements was also found in the present alloys. Fig. 15 shows the effect of Ni_{eq}^* value and aging temperature on σ phase formation in Fe-12Cr-15Mn-C-Ni alloys.²²⁾ It is evident that σ phase formation is prevented in the

* $\bar{M}d$ is a parameter relating to the d -orbital level of alloying transition metal. $\bar{B}o$ is a parameter relating to the covalent bond strength between atoms. $\bar{M}d$ and $\bar{B}o$ are the average values of Md and Bo of alloy, defined by

$$\bar{M}d = \sum X_i (Md)_i, \quad \bar{B}o = \sum X_i (Bo)_i, \quad \text{where,} \quad X_i: \text{ the atomic fraction of component } i \text{ in the alloy}$$

$$(Md)_i, (Bo)_i: \text{ the } Md \text{ and } Bo \text{ values for component } i, \text{ respectively.}$$

Refer to Ref. 24) in details.

Fig. 12. Phase diagram of the Fe-Cr-Mn ternary system at 923 K and Ergang and the region of σ phase formation determined by Y. Okazaki *et al.*²²⁾

Fig. 13. Temperature dependence of critical $\overline{M}_d$ for the σ phase in various alloys.

Fig. 14. γ phase field of Fe-Cr-Mn-Ni alloys shown in $\overline{Bo}$ - $\overline{Md}$ diagram.

Fig. 15. Effect of Ni^* equivalent and aging temperature on σ phase formation in Fe-Cr-Mn-C-N-Ni alloys.

Fig. 16. Effect of C and N on the ultimate tensile strength of Fe-12Cr-15Mn-C-N alloys.

temperature range between 773 and 973 K when the Ni^* value is greater than 10. It is mentioned that the addition of 0.2 % C and 0.2 % N is required to prevent σ phase formation in Ni-free Fe-12Cr-15 Mn alloy.

5.2. High-temperature Strength

The effect of C and N on high-temperature tensile strength of 12Cr-15Mn steel at 873 K is shown in Fig. 16.²⁷⁾ C and N are considered to have similar effects on this property. It seems that the combined addition of C and N is very beneficial in increasing high-temperature strength. Fig. 17 shows the change in tensile properties (tensile strength, 0.2 % proof strength and total elongation) with the amounts of C+N in 12Cr-15Mn steel.²⁸⁾ It indicates that comparable tensile strength and elongation with JPCA is obtained when the steel contains 0.4% (C+N). It should be noted that the 12Cr-15Mn-0.2C-0.2N steel has entirely austenitic structure and does not form σ phase.

Fig. 17. Tensile strength, 0.2 % proof stress and total elongation of Fe-12Cr-15Mn-C-N alloy tested at 873 K after solution-treatment and aging at 873 K for 36 ks as a function of C plus N (mass%).

Fig. 18. Relation between applied stress and time to fracture of various Fe-12Cr-15Mn alloys in creep test at 873 K.

Improvement effect of combined addition of C and N was also shown on creep rupture strength in Fig. 18 indicating the relation between applied stress and time to fracture.²⁷⁾ Effect of small amount of V is also demonstrated in this figure. Addition of 0.1 % V slightly improves creep rupture strength but addition of 0.2 % V decreases it. Cold work (about 20 %) is found to be pronouncedly effective in increasing creep rupture strength, as shown in Fig. 18. The strength was reported to be attributable to finely precipitated carbides in the grain interior along slip lines caused by cold work.²⁷⁾

The creep rupture strength is markedly improved by alloying with W. Fig. 19 indicates the effect of W on time to fracture and minimum creep rate of 12Cr-15Mn steel at 873 K under the applied stress of 216 MPa. Minimum creep rate is decreased and time to fracture increased with addition of 2 % of W. Fig. 20 shows the comparison of creep rupture strength of 12Cr-18Mn-2W steel with that of austenitic Cr-Ni stainless steels.¹⁵⁾ It is understood

Fig. 19. Effect of Mn and W on time to fracture and minimum creep rate of 12Cr-15Mn steel.

Fig. 20. Comparison of creep rupture strength for 10⁴ h of 12Cr-18Mn-2W steel (MN-steel) with that of austenitic Cr-Ni stainless steels.

that creep rupture strength of these steels are comparable.

As mentioned above, in the development of austenitic high Mn-Cr steel, the utilization of C and N is necessary for the prevention of σ phase formation and the increase of high-temperature strength. The

alloying of W is also helpful in improving creep rupture strength.

5.3. Void Swelling

Much efforts have been dedicated to make clear the mechanism and fission-fusion correlation of void swelling in pure metals, simple alloys and engineering materials in Japan as well as in U.S.A. and Europe.³⁻⁶⁾ As for reduced activation ferrous materials, however, experimental results of void swelling especially by neutron irradiation are relatively few in Japan. The void swelling characteristics of these materials are now investigated by using FFTF/MOTA (Fast Flux Test Facility, Materials Open Test Assembly) in Pacific Northwest Laboratory, U.S.A. under the MONBUSYO-DOE Collaboration.²⁹⁾

Generally, void swelling of ferritic steels is difficult to occur compared with austenitic steels as shown in Fig. 21.¹⁰⁾ In this section, some results of void swell-

ing of austenitic Fe-Mn-Cr alloys are referred comparing with that of austenitic Fe-Ni-Cr alloys, which include study in U.S.A.

In Fe-Mn-Cr alloys, voids form in significant numbers only in the temperature range 623 to 723 K and are at lower densities than observed in Fe-Ni-Cr alloy as demonstrated in Figs. 22 and 23,³⁰⁾ respectively. In these case, void formation was introduced by electron irradiation using high voltage electron microscope. It is found that void swelling by electron irradiation decreases with addition of W and V (Fig. 24).³¹⁾ Increase amount of Mn is also effective in decreasing neutron-induced swelling of Fe-Mn and Fe-Cr-Mn alloys (Fig. 25).³²⁾ In the neutron-induced swelling, the beneficial effect of Mn and Ni on decrease of swelling is seemed to be comparable as shown in Fig. 26.³²⁾ Void swelling increases with increasing irradiation fluence and the swelling rate is less than 1 %/dpa in Fe-Mn and Fe-Cr-Mn alloys (Fig. 27).³²⁾ A commercial high Mn steel, AMCR 0033 shows much lower swelling than simple Fe-Mn and Fe-Cr-Mn alloys as indicated in Fig. 27. AMCR 0033 contains C and N which form carbides and nitrides in the steel. These dispersed carbides and

Fig. 21. Comparison of the swelling behavior of austenitic with ferritic steels.

Fig. 22. Dose dependence of swelling for Fe-15Cr-20Mn and Fe-15Cr-15Mn(W, V) alloys at various temperatures.

Fig. 23. Dose dependence of swelling for the Fe-15Cr-30Ni alloy at various temperatures.

Fig. 24. Void swelling as a function of irradiation temperature for Fe-Cr-Mn alloys after irradiation to 15 dpa.

Fig. 25. Neutron-induced swelling of Fe-Mn and Fe-Cr-Mn alloys in FFTF at 793 K and 14 dpa as determined by immersion density measurements.

Fig. 26. Comparison of swelling of Fe-Cr-Ni and Fe-Cr-Mn alloys at comparable irradiation conditions.

nitrides increase the void nucleation site, resulting in the decrease of void swelling as a whole. Cold work also increases the void nucleation site and decreases void swelling. Decrease of void swelling by cold work had previously obtained in type 316 stainless steel and many austenitic Fe-Ni-Cr alloys.³³⁾

6. Summary

The outline of research and development of reduced activation ferrous materials, today in Japan, is reviewed. Low or reduced activation materials are used for the first wall material of fusion reactor. Concept of low or reduced activation materials is closely related to the waste management and reactor safety. True low activation materials are considered to be Si, C and SiC. In near-term construction of fusion reactor or fusion reaction facility, however, the utilization of reduced activation ferrous materials is seemed to be the most practical way. It is required for these materials that the land disposal limit, 10CFR61 limit,

Fig. 27. Swelling of simple Fe-Cr-Mn alloys at 723 K in FFTF-MOTA. The swelling of commercial alloy AMCR 0033 is also shown. (AMCR 0033: 10Cr-18Mn-0.7Ni-0.6Si-0.2C-0.06N)

must be satisfied at least. Ferritic 9Cr-W steels and austenitic Mn-Cr steels are now being developed in Japan, which satisfy this limit. In this review, the characteristic features of these materials is presented. However more detailed study and many engineering data are required in order to develop and use these materials as the practical structure. In Japan, data bank system for engineering materials used for fusion reactor is now being developed.³⁴⁾

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