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## METALS AND CERAMICS DIVISION

### ALLOY DEVELOPMENT FOR FAST INDUCED RADIOACTIVITY DECAY FOR FUSION REACTOR APPLICATIONS

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ALLOY DEVELOPMENT FOR FAST INDUCED RADIOACTIVITY DECAY  
FOR FUSION REACTOR APPLICATIONS\*

R. L. Klueh and E. E. Bloom

ABSTRACT

The Cr-Mo ferritic (martensitic) steels and austenitic stainless steels (primarily type 316 and variations on that composition) are the leading candidates for the structural components for future fusion reactors. However, irradiation of such steels in a fusion environment produces long-lived radioactive isotopes. These isotopes lead to difficult radioactive waste disposal problems once the structure is removed from service. Such problems could be reduced by developing steels that contain only elements that produce radioactive isotopes that decay to low levels in a reasonable time (tens of years instead of hundreds or thousands of years). This report discusses the development of such steels by making elemental substitutions in the steels now under consideration. Molybdenum must be replaced in the Cr-Mo steels; nickel and molybdenum both must be replaced in the austenitic stainless steels (the nitrogen concentration must be limited, and niobium, maintained to extremely low levels). Appropriate substitutions are tungsten for molybdenum in the Cr-Mo steels and manganese for nickel in the austenitic stainless steels. Indications are that suitable ferritic steels can be developed, but development of a nickel-free austenitic stainless steel with properties similar to the Cr-Ni stainless steels appears to be much more complex.

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INTRODUCTION

The most serious safety and environmental concerns for fusion reactors involve induced radioactivity in the first-wall and blanket structures. Public safety could be jeopardized by the accidental release of this induced radioactivity, and the biological hazard for plant personnel

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would eliminate the possibility of contact maintenance and repair. Another problem is that of special waste storage of the highly radioactive blanket and first-wall structures after service. All these problems would be alleviated by the use of a low activation structural material. However, as pointed out in a recent report by a U.S. Department of Energy (DOE) panel set up to study this subject, the technology for commercially producing and fabricating material that would meet the low activation criteria is not available and is unlikely to be available soon (high-purity silicon carbide was the only material suggested).<sup>1</sup> Although other solutions must be sought for the safety and maintenance problems, it appears to be possible to simplify the storage of radioactive reactor components by developing a material in which the induced radioactivity decays in a reasonable time to such a level that the waste no longer requires maintenance.

#### RADIOACTIVE WASTE AND STORAGE GUIDELINES

Guidelines (10 CFR Part 61) for the classification and disposal of low-level nuclear wastes have been issued by the U.S. Nuclear Regulatory Commission;<sup>2</sup> these guidelines are summarized in Table 1. The DOE Panel on Low Activation Materials set as a goal the development of reactor materials that fall within Class C, but with the hope to meet Class B criteria.<sup>1</sup> It should be noted that the 10 CFR Part 61 guidelines were developed primarily to treat fission reactor waste and will undoubtedly be extensively revised by the time the first components are discharged from an operating fusion reactor and are ready for disposal. Nevertheless, these guidelines offer a standard to which alloys can be developed for eventual inexpensive near-surface disposal.

The 10 CFR Part 61 guidelines were examined by the DOE panel<sup>1</sup> and more recently by Wiffen and Santoro.<sup>3</sup> On the basis of the guidelines, initial concentration limits were calculated for various common alloying elements for the three waste classes for first-wall and blanket structures ten years after the shutdown of a reactor given a  $9 \text{ MW}\cdot\text{years}/\text{m}^2$  exposure.

Table 1. Nuclear waste classification and storage under proposed 10 CFR Part 61 rules

Waste class	Definition	Disposal
Class A, segregated	Decays to acceptable levels during site occupancy	
Class B, stable	Decays within 100 years to levels not dangerous to public health and safety	Covered to reduce surface radiation to a few percent of background
Class C, intruder	Decays to acceptably safe levels in more than 100 years but less than 500 years	At least 5 m below the surface, with natural or engineered barriers
Waste not meeting Class C intruder waste definition	Decays in more than 500 years	Does not qualify for near-surface disposal; proposed methods will be considered on a case-by-case basis

The limits are given in Table 2. Based on the possibility that a material resulting in Class A waste is quite remote (e.g., impurity-free silicon carbide), vanadium alloys (alloyed with chromium and titanium) offer the possibility for Class B waste (residual impurities would appear to make Class A vanadium alloys impossible). It appears that steels, the subject of this report, will have to be handled as Class C waste.<sup>1,3</sup>

Table 2 shows that, for the induced radioactivity of a steel to decay rapidly enough to qualify for Class C treatment, certain common steel alloying elements must be restricted. In particular, niobium must be eliminated (it is the restriction on niobium that will probably make it impossible ever to meet the Class B criteria for any steel). Concentrations of Ni, Mo, and N must be severely restricted in the steels being considered for first-wall and blanket structure applications. Copper, generally present as an impurity, will also have to be controlled.

Table 2. Initial concentration level restrictions from 10 CFR Part 61 waste disposal rules ten years after shutdown, 9 MW-years/m<sup>2</sup> exposure

Element	Initial concentration limit <sup>a</sup> (at. ppm)		
	Class A	Class B	Class C
N	<i>b</i>	365	3,650
O	250,000	<i>b</i>	10 <sup>6</sup>
Co	30	10 <sup>6</sup>	10 <sup>6</sup>
Cu	12	240	2,400
Fe	350	35,000	10 <sup>6</sup>
Ni	100	2,000	20,000
Mo	365	<i>b</i>	3,650
Mn	5,000	10 <sup>6</sup>	10 <sup>6</sup>
Nb	0.1	<i>b</i>	1
Al, C, Mg, Si, Ti, V	10 <sup>6</sup>	10 <sup>6</sup>	10 <sup>6</sup>

Source: R. W. Conn et al., *Panel Report on Low Activation Materials for Fusion Applications*, UCLA Report PPG-728, University of California at Los Angeles, June 1983.

<sup>a</sup>Limits apply to first-wall region.

<sup>b</sup>Storage in this classification is not defined.

In this report, the development of steels with "fast" induced radioactivity decay (FIRD) characteristics are discussed. *Fast* is a relative term, which for this discussion is taken to mean steels with radioactive decay rapid enough to qualify at least for Class C waste disposal criteria. The term *low activation material* is often used to describe alloys that minimize waste disposal and those that would allow hands-on maintenance. Materials that would allow hands-on maintenance would not only have to have low activation, but would also have to decay to extremely low levels very rapidly (e.g., pure SiC).

The DOE panel called attention to the difficulty of defining low activation materials by defining four types of fusion reactors and materials.<sup>1</sup> It defined standard activation, very low activation, low activation, and reference activation reactors and materials.

A standard activation reactor would be constructed of a standard activation material such as type 316 stainless steel or 12 Cr-1 MoW steel, which does not meet the criteria for shallow land burial of the radioactive waste after reactor shutdown. For such reactors, all maintenance must be performed remotely.

A very low activation reactor would be constructed from a very low activation material that would be disposable according to the Class A criteria of 10 CFR Part 61 and would allow limited hands-on maintenance behind the blanket within 2 d of shutdown.

The low activation reactor would be constructed of materials that met the Class B criteria, and hands-on maintenance would be possible outside the blanket or outside the shield within 2 d of shutdown.

Finally, a reference activation reactor would be constructed of a reference activation material that met Class C criteria; such a material would allow limited hands-on maintenance outside the shield within 2 d of shutdown. The FIRD steels to be discussed here fall into the class of reference activation materials.

The limits on the initial concentrations of Ni, Mo, Cu, and N (Table 2) established by the 10 CFR Part 61 guidelines are mutually exclusive.<sup>3</sup> That is, if any one of the elements is present at the concentration limit, the other elements must be absent. The 1 at. ppm limit for niobium will be the most difficult to meet. Because of this restrictiveness on the niobium, the alloys discussed below will be those for which Mo, Ni, Cu, and N will be kept to a minimum.

For various technological reasons, the austenitic stainless steels and the Cr-Mo ferritic (and martensitic) steels are the leading candidate alloys for fusion reactor structures. The effect of irradiation on such steels has been extensively studied in both the fusion reactor development and the breeder reactor programs. Furthermore, an extensive background of experience is available to draw on for fabricating large and complicated

steel structures for elevated-temperature service. Because of this background of experience with such steels, these materials are the logical choices for modification to develop FIRD alloys.

Common steel alloying elements that can be used in FIRD steels include Mn, Ti, Cr, Si, W, V, Ta, Co, and C. The most important of the elements not available are nickel and molybdenum. Both elements are present in the austenitic stainless steels, and molybdenum is used in the Cr-Mo ferritic steels. If these two types of steels are to be used, substitutions must be found for these two elements. In the following we discuss possibilities for developing FIRD ferritic steels similar to the Cr-Mo steels and FIRD austenitic stainless steels similar to type 316 stainless steel and the austenitic prime candidate alloy (PCA) developed in the fusion program.

#### FERRITIC STEELS

The Cr-Mo ferritic steels are of two types: (1) those that basically contain only Fe, Cr, Mo, and C and (2) those that contain these elements plus one or more of the strong carbide formers Nb, V, Ti, and W\* (ref. 4). An example of the first type is 2 1/4 Cr-1 Mo steel. This steel was an outgrowth of the C-Mo steels, with the chromium added to increase ductility, to decrease the tendency to graphitization, and to provide oxidation and corrosion resistance at elevated temperature. Much of the elevated-temperature strength in this alloy is provided by a dispersion of Mo<sub>2</sub>C (ref. 5); molybdenum also provides solid-solution strengthening. Several other carbides also form,<sup>6</sup> including M<sub>3</sub>C, M<sub>7</sub>C<sub>3</sub>, M<sub>23</sub>C<sub>6</sub>, and M<sub>6</sub>C. After prolonged exposure at elevated temperature, only M<sub>23</sub>C<sub>6</sub> and M<sub>6</sub>C remain. The addition of 0.25% V to 2 1/4 Cr-1 Mo steel has a pronounced effect on the precipitate formed and thus on its elevated-temperature properties.<sup>7</sup> In addition to the molybdenum- and chromium-rich carbides, V<sub>4</sub>C<sub>3</sub> appears. The fine dispersion of this precipitate provides increased creep strength.

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\*These are the elements that give strength to the Cr-Mo steels. The commercial steels generally also contain up to 0.5% Mn, 0.4% Si, and 0.5% Ni plus small amounts of impurities.

The high-chromium Cr-Mo steels have attracted attention for fusion reactor applications.<sup>8-9</sup> Steels with 9 or 12% Cr and 1% Mo are being investigated. Without the addition of a strong carbide-forming element,  $M_{23}C_6$  precipitate forms in these high-chromium steels.<sup>10</sup> When vanadium is added,  $M_{23}C_6$  is still the predominant precipitate and the only one present at equilibrium.<sup>10</sup> However, the addition of vanadium leads to a finer, more stable  $M_{23}C_6$ , which increases the elevated-temperature strength. Small amounts of niobium and titanium added to these steels lead to the formation of niobium- and titanium-rich MC-type carbides in addition to  $M_{23}C_6$  (refs. 10-11). Evidently these alloying elements are not as readily absorbed in the  $M_{23}C_6$  as is vanadium. The formation of a fine dispersion of these precipitates at the service temperature can lead to improved strength, although a reduced ductility can also result. A small amount of tungsten (~0.5 wt %) added to the high-chromium Cr-Mo steels has a minor effect on strength, probably caused by solid-solution effects.<sup>12-13</sup>

#### ALLOY COMPOSITION SELECTION

If the objective in developing a FIRD alloy is the replacement of molybdenum in the Cr-Mo steels with an element that will lead to dispersion strengthening, vanadium would appear to be a logical choice. Just as the Cr-Mo steels evolved from the Fe-Mo-C steels, which develop their strength from molybdenum carbides, a similar progression from Fe-V-C would seem to be possible. Indeed, a 2 1/4 Cr-1.5 V steel has been proposed as a FIRD alloy.<sup>14</sup>

Considerable research has been conducted on Fe-V-C steels, which develop their elevated-temperature strength by the formation of  $V_4C_3$  (refs. 15-17). Two types of heat treatment have been considered for these steels. First, a conventional quench-and-temper or normalize-and-temper heat treatment can be used to form a tempered martensite that contains the  $V_4C_3$ . A second possibility studied in recent years is the use of a direct transformation. In this process, a ferrite that contains fine carbide precipitates and has superior properties to those of a quenched-and-tempered product can be developed. By direct transformation, which is

most easily obtained by an isothermal treatment, a fine interphase precipitate of  $V_4C_3$  is formed rather uniformly throughout the ferrite<sup>16</sup> (an interphase precipitate forms in conjunction with the movement of the austenite-ferrite boundary during transformation). Studies have been made to determine how an interphase precipitate could be developed in a continuous cool (as opposed to an isothermal transformation) from the austenitizing temperature — a probable necessity for a commercial steel.<sup>17</sup> One way to accomplish this is by adding nickel or manganese to the steel; however, nickel should be avoided for a FIRD alloy.

Because any ferritic steel developed for fusion reactor applications should have properties that allow it to be used at temperatures as high as 500 to 550°C, and if possible to 600°C, the alloy will undoubtedly need to contain chromium for oxidation and corrosion resistance. Little information is available, however, on the effect of chromium on the Fe-V-C steels. The major effect of chromium is the appearance of the chromium-rich carbides in the precipitation sequence. The carbides that form will depend on the vanadium and chromium concentrations; an equilibrium structure of  $V_4C_3$  and either  $M_7C_3$  or  $M_{23}C_6$  would be expected for vanadium and chromium concentrations of probable interest (0-1% V and 2-12% Cr) (refs. 18-19). Chromium could also change the precipitate morphology that results during a direct transformation. Instead of a fine interphase precipitate distributed uniformly throughout the microstructure, chromium may promote the formation of a fibrous precipitate, usually consisting of carbide fibers 30 to 50 nm in diameter spaced about 30 to 50 nm apart.<sup>16</sup> (The precipitation sequence for such carbides appears to be similar to the process by which pearlite forms.) The effect of such a precipitate morphology on mechanical properties is not known, although the properties are not expected to be as favorable as are those of a fine interphase precipitate.

Any alloy development program should concentrate on alloys in a normalized-and-tempered or quenched-and-tempered condition (i.e., avoid the direct transformation route, because such a heat treatment would be difficult to apply commercially). If vanadium is to replace molybdenum in Cr-Mo steels, a start should be made with 0.25 and 0.5 wt % V in steels

with 2 to 2.5% Cr (less vanadium is required because the molecular weight of vanadium is about one-half that of molybdenum). Vanadium is sometimes thought to make a steel less weldable, so less than 0.15% C should be used; it may also be of interest to investigate the effect of carbon (0.1 and 0.15% alloys).

Because of the large uncertainty with regard to weldability in the development of Cr-V steels, the Cr-W steels may offer the best possibility for the development of a replacement for Cr-Mo steels. Tungsten is in the same group of the periodic table and displays several similarities to molybdenum when it is used as an alloying element in steels. All indications are that Fe-W-C alloys develop analogous precipitates with similar precipitation sequences to Fe-Mo-C alloys (i.e., both form  $M_2C$  and  $M_6C$  of similar morphology).<sup>16</sup> Tungsten and molybdenum also show similar solid-solution hardening characteristics.<sup>16</sup> One important difference involves the diffusion of the two atoms: the tungsten diffusivity is substantially less than that of molybdenum. This results in a slower developing secondary hardening peak for a tungsten steel; however, the tungsten steel does not overage as rapidly. (Note that the secondary hardening peak for a tungsten steel is not as large as is that for a comparable molybdenum steel.) This similarity of tungsten and molybdenum suggests an initial steel composition of 2 to 2.5% Cr-2% W (2 wt % W is required to obtain an atomic concentration similar to that for 1 wt % Mo).

Table 3 lists ferritic and martensitic steels for a "first cut" in a FIRD alloy development program. These include the Cr-V, Cr-W, and Cr-W-V steels discussed above. The 9 Cr-2 W-0.25 V and 12 Cr-2 W-0.25 V steels are added for comparison with the modified 9 Cr-1 MoVNb and 12 Cr-1 MoVW (HT9) steels under study in the fusion reactor alloy development program. A suitable substitute for the 0.06% Nb in the 9 Cr-1 MoVNb steel may be 0.12% Ta in the 9 Cr-2 W-0.25 V steel. Tantalum, in the same group of the periodic table as niobium, often displays analogous properties to those of niobium. Titanium carbides can also result in dispersion, and, as discussed below, titanium additions may be of interest in later alloy compositions.

Table 3. Possible ferritic steels for fast induced radioactivity decay alloy development program

Alloy	Chemical composition <sup>a</sup> (wt %)			
	Cr	V	W	C
2 1/4 Cr-1/4 V	2-2.5	0.25		0.1-0.15
2 1/4 Cr-1/2 V	2-2.5	0.5		0.1-0.15
2 1/4 Cr-2 W	2-2.5		2	0.1-0.15
2 1/4 Cr-1 W-1/4 V	2-2.5	0.25	1	0.1-0.15
2 1/4 Cr-2 W-1/4 V	2-2.5	0.25	2	0.1-0.15
5 Cr-2 W-1/4 V	5	0.25	2	0.1-0.15
9 Cr-2 W-1/4 V <sup>b</sup>	9	0.25	2	0.1-0.15
12 Cr-2 W-1/4 V	12	0.2	2	0.1-0.15

<sup>a</sup>Balance iron.

<sup>b</sup>An alloy with 0.12% Ta should be considered to complete the analogy with 9 Cr-1 MoVNb steel.

The 9 Cr-1 MoVNb and 12 Cr-1 MoVW steels were chosen for fusion reactor applications because of their excellent swelling resistance in the breeder reactor program. However, the proposed breeder reactor applications involve service conditions considerably different from those envisioned for fusion reactors. Because of these differences, we previously recommended that 2 1/4 Cr-1 Mo steel should be strongly considered for fusion reactor application.<sup>\*4</sup> The reasons for this recommendation included the operating temperatures of proposed fusion reactor designs, possible advantages in welding the low-chromium steel, and conservation of a strategic material.<sup>4</sup> The swelling resistance of 2 1/4 Cr-1 Mo steel was equivalent to that of high-chromium steels when they were compared in ion-irradiation studies.<sup>4</sup>

Chromium in excess of about 2.25% does not affect the elevated-temperature strength, and this amount of chromium provides oxidation resistance adequate for service to about 600°C (ref. 10). Chromium

<sup>\*</sup>At present, the alloy development program for fusion reactors calls for about 5% of the irradiation effects studies to be on 2 1/4 Cr-1 Mo, 60% on 12 Cr-1 MoVW, and 35% on 9 Cr-1 MoVNb.

content will affect the hardenability, but, for fusion reactor applications, in which relatively thin sections are to be used, this effect should not prove important. One of the reasons why the 9 and 12% Cr steels are often assumed to be superior to 2 1/4 Cr-1 Mo steel is that the long-time elevated-temperature strength of the high-chromium Cr-Mo steels containing V, Nb, or Ti are compared with 2 1/4 Cr-1 Mo without any of these strong carbide formers being added. Whenever the elevated-temperature strength of 9 Cr-1 Mo and 12 Cr-1 Mo without vanadium or niobium are compared with 2 1/4 Cr-1 Mo (all with similar microstructures), little difference is observed.<sup>10</sup> At higher temperatures, above 600°C, adequate oxidation resistance will be possible with only the higher chromium steels.

There might well be an advantage for a low-chromium Cr-Mo-V steel because, when the Cr-Mo-V-C diagrams of Smith<sup>18</sup> are consulted for a 1% Mo-0.25% V-0.1% C steel with 2.25% Cr and for the 9 and 12% Cr steels, different carbides are predicted for equilibrium at 700°C. The  $M_4C_3$  ( $V_4C_3$ ) and  $M_7C_3$  carbides are present in the 2.25% Cr steel, but only  $M_{23}C_6$  is present for the two high-chromium steels. The  $M_{23}C_6$  is generally a coarser carbide than are  $M_4C_3$  and  $M_7C_3$ , although, as noted above, vanadium stabilizes a finer precipitate. This possible difference in carbides that occurs with varying chromium concentration does not seem to have been systematically investigated, although the Japanese Steel Works has recently introduced a modified 2 1/4 Cr-1 Mo steel with 0.25% V and 0.02% Ti added.<sup>7</sup>

Although the proposed alloy development scheme summarized in Table 3 appears to emphasize 2 1/4 Cr, this is not the case. The low-chromium composition (2.25%) is used to investigate the effect of vanadium and tungsten. Several alloys with higher chromium are then suggested to determine the effect of chromium. The information thus generated should allow for the determination of the most suitable alloy for any design conditions.

Comparative studies on the 9 Cr-1 MoVNB and 12 Cr-1 MoVW steels being considered for fusion reactor applications have indicated differences in the long-time elevated-temperature strength, with the 9 Cr-1 MoVNB steel the stronger.<sup>20</sup> This difference may be due to the niobium in the 9% Cr steel, because at equilibrium fine MC carbides in addition to  $M_{23}C_6$  are

observed in this steel.<sup>10</sup> Only the  $M_{23}C_6$  is observed in the 12 Cr-1 MoVW steel.<sup>10,18-19</sup> Although niobium must be eliminated in a FIRD steel, tantalum is often analogous to niobium. This is the basis for suggesting a tantalum addition (Table 3).

As noted above, the Japanese Steel Works added titanium to its modified 2 1/4 Cr-1 Mo steel. About 0.02% Ti in steels is known to prevent grain coarsening in the weld heat-affected zone during high-heat-input welding, thus assuring good notch toughness. Such titanium additions may be useful in the FIRD alloy development program. There is also evidence that a low-carbon 2 1/4 Cr-1 Mo steel with about 0.14% Ti has creep properties superior to those of commercial 2 1/4 Cr-1 Mo steel. If the 2 1/4 Cr-2 W and the 2 1/4 Cr-2 W-0.25% V steels should have favorable properties, the addition of titanium to one or both of these compositions should be considered.

#### ALLOY DEVELOPMENT STRATEGY

Any alloy developed for fusion reactor applications must be thoroughly tested in a suitable irradiation environment. However, before detailed irradiation studies are conducted, it is necessary to understand the unirradiated behavior and to compare that behavior with the reference alloys that are currently candidates for fusion reactor applications (i.e., the developmental steel must be compared with 2 1/4 Cr-1 Mo, 9 Cr-1 MoVNb, and 12 Cr-1 MoVW steels). Information will be required on the physical metallurgy (e.g., precipitate type, precipitation kinetics, etc.), fabricability, weldability, elevated-temperature strength, impact properties, compatibility, and other properties. The properties of the FIRD steels must be at least as good as those of commercial Cr-Mo steels. Once alloys with satisfactory unirradiated properties are obtained, detailed irradiation effects studies will be required.

Information on the physical metallurgy of the new steels will come from heat treatment studies to determine the phases developed when quenched, normalized, tempered, and aged. Hardness measurements and optical and electron microscopy will be used to study phase stability. Analysis of carbide extractions may also prove useful.

Fabricability will be determined in the normal melting, casting, and rolling operations necessary to obtain the sheet that will be used to make transmission electron microscopy and mechanical property specimens. A simple weldability test should also be performed; perhaps Varstraint or guide-bend tests could be used for early qualifications.

Elevated-temperature tensile properties of prospective FIRD ferritic steels will be required. Creep properties will also be needed to define upper temperature limits for the alloys. Initially, a comparison of tensile results with the tensile behavior of available steels will provide a relative assessment of the elevated-temperature behavior.

Ferritic steels may be useful only in a temperature window with a lower temperature limit determined by the toughness and impact properties and an upper limit governed by compatibility limits or elevated-temperature strength. The impact properties are affected by irradiation; for example, the ductile-brittle transition temperature of 12 Cr-1 MoVW steel increased by 108°C during irradiation in EBR-II at 419°C to  $1.1 \times 10^{26}$  neutrons/m<sup>2</sup> (ref. 21), accompanied by a decrease in the upper-shelf energy. Any FIRD alloy development program must consider the impact properties of the proposed alloys. These properties must be at least as good as those of present candidate ferritic steels.

Finally, it will be necessary to determine the compatibility of these steels with potential coolant and breeding materials (i.e., water, helium, lithium, lead-lithium alloys, and solid lithium ceramics). Because there is no change in chromium concentration between these steels and the Cr-Mo steels, little difference in compatibility is expected. Nevertheless, comparative tests will be necessary.

#### AUSTENITIC STAINLESS STEELS

Type 316 stainless steel and modifications of that basic alloy composition (e.g., by the adjustment of chromium and nickel contents and the addition of titanium) are the primary austenitic stainless steels under consideration for fusion reactor structural applications.<sup>22</sup> The alloy development approach to be followed here is similar to that followed

for the ferritic steels. We will attempt to develop an austenitic steel with properties similar to those of the PCA, which was developed as a variation of type 316 stainless steel.<sup>22</sup> Of the elements contained in PCA and type 316 stainless steel, nickel and molybdenum are not acceptable in a FIRD alloy. Manganese, like nickel, is an austenite-forming element and has often been used as a replacement for nickel.<sup>23-27</sup> However, the austenite-forming tendency of manganese is considerably less than that of nickel, and the development of an Fe-Cr-Mn stainless steel does not follow by simply replacing nickel with manganese.

Other common austenite-forming alloying elements include C, N, Cu, and Co. The carbon concentration will be maintained at 0.05 to 0.1% to minimize corrosion problems. To meet the 10 CFR Part 61 waste storage criteria for Class C waste, the nitrogen concentration cannot exceed 3650 at. ppm (~0.09 wt %) (ref. 2). Thus, small nitrogen additions may be possible. The decay of radioactive daughter elements produced by the irradiation of copper in a fusion reactor makes it unsuitable for FIRD alloys (Table 2).

Cobalt presents an interesting possibility. As an austenite stabilizer, it is as strong as nickel.<sup>28</sup> Its radioactive decay characteristics are such that it is not forbidden in Class B or Class C waste by 10 CFR Part 61 (ref. 2). However, immediately after irradiation, steels containing cobalt emit high-energy gamma rays that make the steels difficult to handle. Thus, even though cobalt could increase the possibility of developing a FIRD stainless steel, it would make irradiation studies extremely difficult or impossible. For that reason, cobalt will be considered an unsuitable alloy addition for this discussion.

#### ALLOY COMPOSITION SELECTION

The idea of using less expensive manganese to replace nickel in austenitic stainless steels has appeal; it has been investigated quite extensively, mainly in the 1930s and 1940s.<sup>23-27</sup> Although manganese is used to replace some nickel in the 200-series stainless steels, the lower austenite-forming capability of manganese is offset by the addition of up

to 0.25% N. The only Cr-Mn stainless steels that appear to have been used commercially were used in Germany.<sup>23</sup> These were an 8 Cr-18 Mn and an 18 Cr-8 Mn alloy, both containing 0.1% C. The first alloy was entirely austenite (because of the low chromium concentration), but the second contained about 40% ferrite. The low chromium concentration of the first implies relatively low oxidation and corrosion resistance, and the high ferrite content of the second means it will be prone to sigma phase formation; both are reasons for making the alloy unsuitable for elevated-temperature service. Another reason why the development of manganese-stabilized (nickel-free and low-nitrogen) stainless steels has never been actively pursued is that the increased manganese leads to decreased corrosion resistance by sulfuric, hydrochloric, and nitric acids.<sup>29-30</sup>

More recent studies have been published on the effect of manganese on mechanical properties and as an austenite stabilizer.<sup>27,29-33</sup> Manganese lowers the stacking-fault energy and therefore increases the rate of work hardening.<sup>30</sup> This could lead to problems in fabrication. It also lowers the thermal conductivity relative to nickel,<sup>31</sup> a disadvantage for fusion reactor applications. The reduced oxidation resistance of Cr-Mn steels at elevated temperatures should not prove limiting for fusion reactors if the maximum temperature is limited to about 600°C.

Recently, Desforges and Dancoisne investigated a large number of manganese-containing ferrous alloys.<sup>33</sup> Because of the low austenite-stabilizing capacity of manganese, most of the alloys proved to be magnetic (i.e., contained some ferrite or martensite), including an Fe-14% Cr-15% Mn steel. Of the 18 manganese-containing alloys melted and cast, only three were nonmagnetic. One of these contained 5% Ni, another 2% Cu. Because copper is generally considered to be a weaker austenite stabilizer than is manganese, the copper could presumably be replaced by manganese and retain the austenite microstructure. However, this Fe-11% Cr-15% Mn-2% Cu alloy suffered from hot shortness when worked. The only other nonmagnetic alloy was an Fe-8% Cr-2% Al-15% Mn steel, which had reasonably good oxidation resistance to 600°C. It had excellent tensile properties relative to the other steels when tested in the cold-rolled condition.

After a solution anneal at 900 or 1100°C, it was one of the weakest as measured by the 0.2% offset yield stress; the ultimate tensile strength was similar to that for most of the other steels.

Although the early work on the ternary phase diagrams for Fe-Cr-Mn would indicate that a steel containing 15% Cr and 15% Mn would be entirely austenite, but would be near the alpha-gamma phase boundary, little guidance is available when other elements such as carbon are added. A common method used to represent the phases present in Cr-Ni stainless steels is a Schaeffler diagram (Fig. 1), in which the phase fields expected at room temperature are shown in terms of nickel and chromium equivalents.<sup>28</sup> The nickel and chromium equivalents have been determined empirically for the most common alloying elements, including manganese.<sup>28</sup> The equations that apply to Fig. 1 are given by

$$\begin{aligned} \text{Cr equiv} = & (\text{Cr}) + 2(\text{Si}) + 1.5(\text{Mo}) + 5(\text{V}) + 5.5(\text{Al}) \\ & + 1.75(\text{Nb}) + 1.5(\text{Ti}) + 0.75(\text{W}), \end{aligned} \quad (1)$$

$$\begin{aligned} \text{Ni equiv} = & (\text{Ni}) + (\text{Co}) + 0.5(\text{Mn}) + 0.3(\text{Cu}) \\ & + 2.5(\text{N}) + 30(\text{C}), \end{aligned} \quad (2)$$

where ( ) is the concentration expressed in weight percent. According to Eq. (2), manganese is only half as effective as is nickel in stabilizing austenite.

The chromium and nickel equivalents were calculated for several alloy compositions (Table 4), and the position of these steels is shown in Fig. 1. Of interest is the fact that the 15-15 steel (15% Cr-15% Mn-0.1% C) and the 12-15 steel (12% Cr-15% Mn-0.1% C) are in the austenite-plus-martensite field. The phase diagram indicates that such alloys should be entirely austenite.<sup>25-27</sup> A 12-20 steel falls into the austenite-plus martensite region, and a 15-20 steel lies just outside this field in the austenite field. In this instance, the diagram indicates that an increase in chromium from 12 to 15% (with 20% Mn) leads to an austenitic steel.

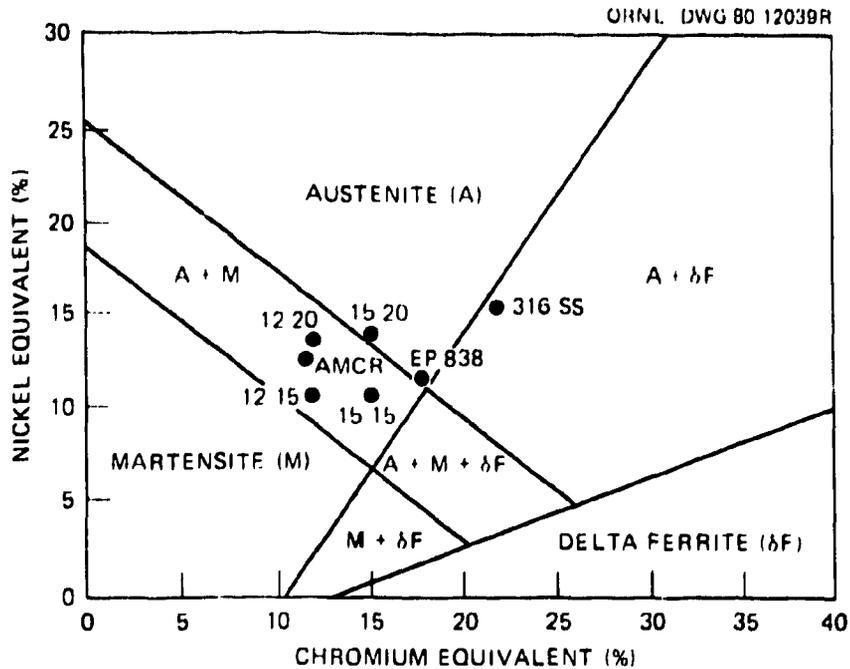


Fig. 1. Schaeffler diagram with points for various steels superimposed. For the double numbers the first number is the weight percent chromium, and the second is the weight percent manganese (e.g., 12-15 is 12% Cr and 15% Mn); all are assumed to have 0.1% C. Alloy AMCR is 10% Cr, 17.5% Mn, and 0.11% C; EP-838 is 11.6% Cr, 13.5% Mn, 4.2% Ni, 0.9% Mo, and 0.02% C; and 316 stainless steel is 17.3% Cr, 1.7% Mn, 12.5% Ni, 2.1% Mo, and 0.06% C.

Table 4. Chromium and nickel equivalents for several commercial and experimental steels

Alloy	Composition <sup>a</sup> (wt %)							Equivalent (wt %)	
	Cr	Mn	Ni	Mo	Si	Al	C	Ni	Cr
316 SS	17.3	1.7	12.4	2.1	0.7		0.06	15.0	21.9
15-15	15	15					0.10	18.0	15.0
12-15	12	15					0.10	18.0	12.0
15-20	15	20					0.10	23.0	15.0
EP-838	11.6	13.5	4.2	0.9	0.4	0.7	0.02	11.6	17.6
AMCR	10.1	17.5	0.10			0.55	0.11	12.2	11.2

<sup>a</sup>Balance iron.

Also shown in Table 4 and Fig. 1 are data for type 316 stainless steel and two experimental steels. The EP-838 is a Russian steel in which much of the nickel from a Cr-Ni-type stainless steel such as type 316 was replaced by manganese. To help maintain austenite, the chromium and molybdenum were reduced from the amounts of these elements in type 316 stainless steel. This steel is in the austenite region. The Cr-Mn steel designated ACMR is a steel being studied by the European communities' fusion program as a possible FIRD steel.<sup>34</sup> Although no information is available for this material, we expect it to be austenitic, but the Schaeffler diagram predicts otherwise. Further difficulties with the diagram are found when the three non-magnetic steels of Desforges and Dancoisne are referred to the diagram.<sup>33</sup> None of them was found to fall in the austenite region (or near a boundary).

These inconsistencies between phase diagrams and experimental results with the Schaeffler diagram indicate that the words of Desforges and Dancoisne are appropriate.<sup>33</sup> They wrote:

A Schaeffler constitution diagram for manganese-containing steels would be of great value in providing designers and welding engineers with the necessary information on weld metal microstructure.

And, we might add, base metal microstructure.

Various studies indicate that the 0.5 multiplier for manganese in Eq. (2) may be an overestimate of manganese's austenite-stabilizing effect.<sup>35</sup> One study indicated that the effect of manganese may be a constant with a value of 0.35 (ref. 35). However, such a low value does not seem reasonable in light of the above discussion. Thus, it appears that much still needs to be learned about the effect of manganese in austenitic steels. The Schaeffler diagram, as now constituted, would appear to be of relatively little help in developing manganese-stabilized austenitic steels.

Molybdenum is present in type 316 stainless steel to increase the corrosion resistance against chlorides and pitting and to improve the high-temperature mechanical properties. It is not known whether tungsten,

which is analogous to molybdenum in ferritic steels, would achieve the same effect as does molybdenum in these steels. However, tungsten is a ferrite former and in a Cr-Mo steel would probably have to replace chromium if a duplex structure were to be avoided.

The strength of the Fe-Cr-Mn steels is of some concern. We have recently determined the strength of the Russian steel designated EP-838 (ref. 36) (the composition is given in Table 4). The tensile properties of this steel in the 20%-cold-worked condition at room temperature and 300°C are similar to those of 20%-cold-worked type 316 stainless steel (Fig. 2). The effect of manganese on the work-hardening capacity is evident, especially in the room-temperature tests. After irradiation to a displacement-damage level of 5 to 8 dpa, the properties of the two steels were also similar (Fig. 2). These two steels also have similar tensile properties in the solution-annealed condition before and after irradiation.<sup>37</sup> From these results it appears that manganese could be used to replace a large portion of the nickel and at the same time to reduce the chromium and molybdenum concentrations but not affect the strength. No information is available on the effect of the reduced amounts of chromium and molybdenum on corrosion resistance.

The major concerns with austenitic steels for fusion reactor applications involve void swelling,<sup>38</sup> helium embrittlement,<sup>39</sup> and compatibility with coolants. The void and dislocation structure that develops during irradiation in the temperature range  $0.35T_m$  to  $0.6T_m$ , where  $T_m$  is the absolute melting point of the steel, can lead to large volume increases, which could not be tolerated in a structural component. The large amounts of transmutation helium that will be generated in a fusion reactor first-wall structure are known to affect void nucleation and growth.<sup>38</sup> In addition, at temperatures greater than  $0.5T_m$ , small amounts of helium and other irradiation-induced segregation at grain boundaries lead to embrittlement.<sup>39</sup>

Methods being tested for the suppression of void swelling are the use of a high dislocation density (cold work) and/or a high density of fine titanium carbide precipitates (MC precipitates) in the microstructures.<sup>22,40</sup> The dislocations and precipitates act as helium collection sites, giving

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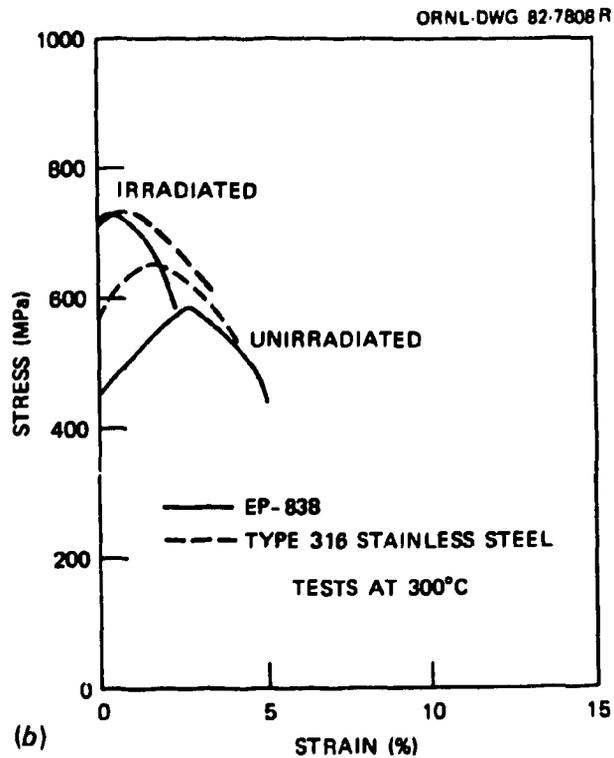
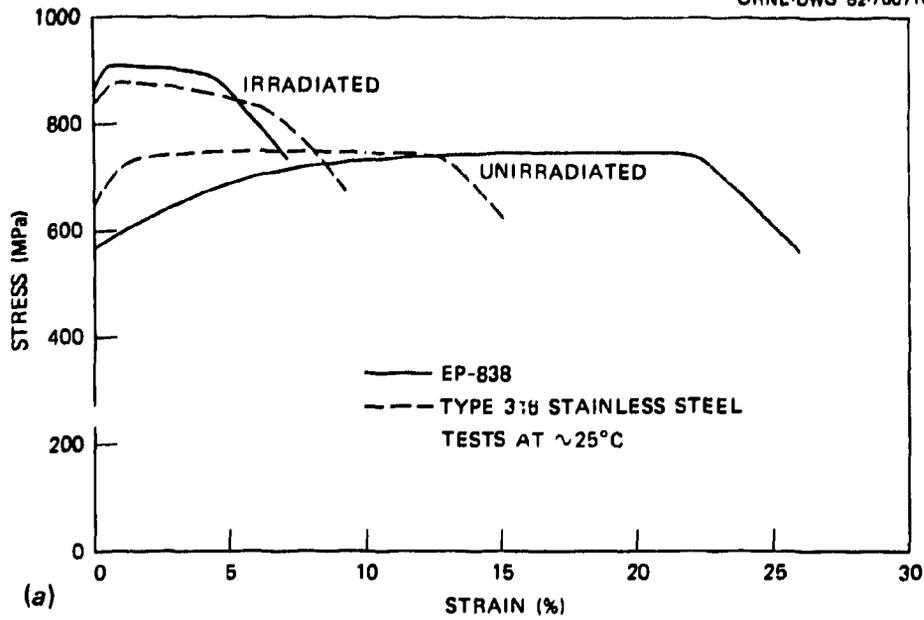


Fig. 2. Engineering stress-strain curves at (a) room temperature and (b) 300°C for unirradiated and irradiated 20%-cold-worked EP-838 and type 316 stainless steels. Irradiation was in HFIR at about 50°C to about 5 to 8 dpa.

rise to a high density of fine cavities, which subsequently act as preferential sinks for the vacancies and interstitials produced during irradiation. The MC on grain boundaries also imparts resistance to embrittlement at elevated temperatures. The application of these principles has led to an austenitic PCA for fusion reactors;<sup>40</sup> this alloy with 14% Cr, 16% Ni, 2% Mo, and 0.25% Ti is being irradiated and tested. In addition to helping control the helium and minimize the swelling, the MC also strengthens by precipitate hardening. The similar use of titanium in a manganese-stabilized austenitic steel would appear to be appropriate for providing swelling resistance and additional strength. Note, however, that titanium is a ferrite stabilizer and that the formation of TiC would eliminate carbon from solution and thus negate its role as an austenite stabilizer.

The above considerations indicate that the development of a FIRD austenitic steel will be more complicated than that of a ferritic steel. The first step in a developmental program must be the determination of a stable composition for a Cr-Mn steel. To obtain an indication of the limits of austenitic phase stability, small heats with the compositions given in Table 5 are proposed. The phase stability of the proposed steels will be determined by optical microscopy following solution-annealing and thermal aging treatments.

Table 5. Possible austenitic stainless steels for fast induced radioactivity decay alloy development program

Alloy	Chemical composition <sup>a</sup> (wt %)			
	Cr	Mn	C	N
15 Cr-15 Mn	15	15	0.05-0.1	<0.01
15 Cr-20 Mn	15	20	0.05-0.1	<0.01
10 Cr-15 Mn	10	15	0.05-0.1	<0.01
10 Cr-20 Mn	10	20	0.05-0.1	<0.01

<sup>a</sup>Balance iron.

After a stable composition is determined, it will be necessary to adjust the composition for swelling resistance, strength, and compatibility with possible breeding and cooling media. Experience with type 316 stainless steel and improvements made when the PCA was developed suggests the addition of Ti, Si, and W (as a substitute for Mo). The development of PCA was aided by the knowledge previously obtained from studies on type 316 stainless steel on processes such as solute segregation during cavity growth and the effect of different precipitates on nucleation and growth of cavities (e.g., the difference between MC and  $M_{23}C_6$ ) (ref. 40). The substitution of manganese for nickel could well alter these processes, thus making detailed studies of the new alloy base system necessary. Note also that Ti, W, and Si are ferrite stabilizers. Even if a phase-stable quaternary alloy is obtained from the initial studies, that stability may be jeopardized by the addition of these elements, and further compositional adjustments and stability studies will be required.

#### ALLOY DEVELOPMENT STRATEGY

Once an austenite-stable alloy composition is identified, the strength of the alloys must be determined in the cold-worked and solution-annealed conditions to get alloys with strengths comparable to those of type 316 stainless steel. This austenite-stable composition will serve as the base for developing a steel with properties similar to PCA. Tensile properties should be adequate for the initial comparisons. Because the life limitation on the austenitic stainless steels is their propensity to swell during irradiation, samples of these alloys should be irradiated to high fluences. Swelling measurements can be used to compare the behavior of the new alloy with type 316 stainless steel and PCA. Transmission electron microscopy on the irradiated steels should be done in an effort to understand the effect of irradiation on phase stability and precipitation processes and the effect these have on swelling.

Information on fabricability will be obtained during the melting, casting, and rolling operations necessary to obtain test material. Simple weldability tests can be conducted on the steels that evolve from the

first series of alloys. Compatibility tests with potential cooling and breeding media (e.g., Li, Pb-Li, and solid lithium ceramics) may prove extremely important for this class of alloys. A large reduction of chromium and elimination of molybdenum may well have a large effect on the compatibility of this class of steel.

#### OTHER AUSTENITIC ALLOYS

Several Japanese steel companies have developed high-manganese, low-chromium austenitic steels. However, most of these either contain nickel or nitrogen above the limits given by 10 CFR Part 61 (ref. 2) and thus do not qualify as FIRD alloys. An exception is a steel by Nippon Kokan, designated NM-1, which contains 20% Mn, 2% Cr, 0.5% C, and 0.5% Si. Of course, this steel is not "stainless" and, as such, does not fit the criteria established above in the discussion on types of alloys to be developed. Nevertheless, this alloy might be used to define another class (low-Cr, high-C, Cr-Mn steel) of FIRD steels to be considered, should those discussed above prove to be inadequate. Indeed, in the alloy development approach suggested above, this class of steels is one of the limits being approached.

#### SUMMARY

If a future fusion reactor could be constructed from a material that develops little or no induced radioactivity during service, a much safer reactor would result, one that would allow hands-on maintenance and minimize radioactive waste disposal problems. No structural materials are currently available to construct such a reactor. However, it appears to be possible to develop alloys that have relative FIRD, which would simplify the radioactive waste disposal problem. An approach to developing such alloys has been presented.

The Cr-Mo ferritic (martensitic) steels and the austenitic stainless steels (primarily type 316) are leading candidate structural materials for fusion reactors. These steels do not qualify as FIRD alloys primarily because of the molybdenum and nickel contents (nitrogen and niobium must

be kept to extremely low concentrations). An alloy development approach has been outlined in which substitutions could be made for these elements in the Cr-Mo steels and austenitic steels yet maintain properties similar to the parent steels. For steels with properties analogous to the Cr-Mo steels, the substitution of tungsten for molybdenum appears to be straightforward. To induce dispersion strengthening, the addition of vanadium is also possible, and titanium and tantalum additions may also be valuable. The production of austenitic steels in which nickel is replaced by manganese may be possible, although this may be much more difficult than the development of a FIRD ferritic steel. This difficulty arises because manganese is not as strong an austenite stabilizer as is nickel.

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