THE DEVELOPMENT OF FERRITIC STEELS FOR FAST INDUCED-RADIOACTIVITY DECAY FOR FUSION REACTOR APPLICATIONS *

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The Cr-Mo ferritic (martensitic) steels are leading candidates for the structural components for future fusion reactors. However, irradiation of such steels in a fusion environment produces long-lived radioactive isotopes that lead to difficult waste disposal problems once the structure is removed from service. One method proposed to alleviate such problems is the development of 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). For such a solution for the Cr-Mo steels, molybdenum must be eliminated. In addition, niobium must be maintained at extremely low levels. Tungsten is proposed as an appropriate substitution for molybdenum, and the procedures for developing Cr-W steels analogous to the Cr-Mo steels are discussed.

1. 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 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 US 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) [1]. 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.

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2. 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 US Nuclear Regulatory Commission [2]; 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 [1]. 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 [1]. 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 MW \cdot yr/m² exposure. 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

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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

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

appears that steels will have to be handled as Class C waste [1].

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.

In this paper, the development of steels with "fast" induced-radio-activity 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 [1]. 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 MoVW 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

Table 2

Initial concentration level restrictions from 10 CFR Part 61 waste disposal rules ten years after shutdown, 9 $MW \cdot yr/m^2$ exposure (from ref. [1])

Element	Initial concentration limit ^a (at. ppm)			
	Class A	Class B	Class C	
N	_ b	365	3650	
0	250 000	_ ^b	10 ⁶	
Co	30	10 ⁶	10 ⁶	
Cu	12	240	2400	
Fe	350	35 000	10 ⁶	
Ni	100	2000	20'000	
Μο	365	_ b	3650	
Mn	5 000	10 ⁶	10 ⁶	
Nb	0.1	_ b	1	
Al, C, Mg, Si, Ti, V	106	10 ⁶	10 ⁶	

^a Limits apply to first-wall region.

^b Storage in this classification is not defined.

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. 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 a 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 or irradiation on such steels has been extensively studied in both the fusion reactor and the breeder reactor development 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 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 a previous paper, the possibility for FIRD austenitic stainless steels similar to type 316 stainless steel and the austenitic prime candidate alloy developed in the fusion program were discussed [3]. In this paper, we will discuss the possibilities for developing FIRD ferritic steels similar to the Cr-Mo steels.

3. FIRD 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* [4]. An example of the first type is $2\frac{1}{4}$ 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 Mo₂C [5]; molybdenum also provides solid-solution strengthening. Several other carbides also form [6], including M₃C, M₇C₃, M₂₃C₆, and M₆C. After prolonged exposure at elevated temperature, only M₂₃C₆ and M₆C remain. The addition of 0.25% V to $2\frac{1}{4}$ Cr-1 Mo steel has a pronounced effect on the precipitate formed and thus on its elevated-temperature properties [7]. In addition to the molybdenumand chromium-rich carbides, V₄C₃ appears. The fine dispersion of this precipitate provides increased creep strength.

The high-chromium Cr-Mo steels have attracted attention for fusion reactor applications [8,9]. 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 [10]. When vanadium is added, $M_{23}C_6$ is still the predominant precipitate and the only one present at equilibrium [10]. However, the addition of vanadium leads to a finer, more stable M₂₃C₆, which increases the elevatedtemperature 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 M₂₃C₆ [10,11]. Evidently these alloying elements are not as readily absorbed in the M23C6 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 [12,13].

3.1. 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

* These are the elements that give strength to the Cr-Mo steels through carbide formation. The commercial steels generally also contain up to 0.5% Mn, 0.4% Si, and 0.5 Ni plus small amounts of impurities. 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 $2\frac{1}{4}$ Cr-1.5 V steel has been proposed as a FIRD alloy [14].

Considerable research has been conducted on Fe-V-C steels, which develop their elevated-temperature strength by the formation of V_4C_3 [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 V₄C₃ is formed rather uniformly throughout the ferrite [16] (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 [17]. 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 chromiumrich carbides in the precipitation sequence. The carbides that form will depend on the vanadium and chromium concentrations; an equilibrium structure of V₄C₃ 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) [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 [16]. (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 quenchedand-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 can affect the weldability of a steel, 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 M₂C and M₆C of similar morphology [16]). Tungsten and molybdenum also show similar solid-solution hardening characteristics [16]. 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 9 Cr-1 MoVNb and 12 Cr-1 MoVW 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 strengthening, and, as discussed below, titanium ad-

Alloy	Chemical composition ^a (wt%)				
	Cr	V	w	C	
$\frac{1}{2\frac{1}{4} \operatorname{Cr} - \frac{1}{4} \operatorname{V}}$	2-2.5	0.25		0.1-0.15	
$2\frac{1}{4}$ Cr $-\frac{1}{2}$ V	2-2.5	0.5		0.1-0.15	
$2\frac{1}{4}$ Cr-2 W	2-2.5		2	0.1-0.15	
$2\frac{1}{4}$ Cr-1 W- $\frac{1}{4}$ V	2-2.5	0.25	1	0.1-0.15	
$2\frac{1}{4}$ Cr-2 W- $\frac{1}{4}$ V	2-2.5	0.25	2	0.1-0.15	
$5 Cr - 2 W - \frac{1}{4} V$	5	0.25	2	0.1-0.15	
9 Cr-2 W $-\frac{1}{4}$ V ^b	9	0.25	2	0.1-0.15	
$12 \text{ Cr} - 2 \text{ W} - \frac{1}{4} \text{ V}$	12	0.2	2	0.1-0.15	

 Table 3

 Possible ferritic steels for fast induced radioactivity decay alloy development program

^a Balance iron.

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

ditions may be of interest in later alloys compositions.

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 $2\frac{1}{4}$ Cr-1 Mo steel should be strongly considered for fusion reactor applications [4]. * 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 [4]. The swelling resistance of $2\frac{1}{4}$ Cr-1 Mo steel was equivalent to that of high-chromium steels when they were compared in ion-irradiation studies [4].

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 [10]. Chromium 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 $2\frac{1}{4}$ Cr-1 Mo steels is that the long-time elevated-temperature strength of the high-chromium Cr-Mo steels containing V, Nb, or Ti are compared with $2\frac{1}{4}$ 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 $2\frac{1}{4}$ Cr-1 Mo (all with similar microstructures), little difference is observed [10]. 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 [18] 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 M₄C₃ (V₄C₃) and M₇C₃ carbides are present in the 2.25% Cr steel, but only M₂₃C₆ is present for the two high-chromium steels. The M₂₃C₆ is generally a coarser carbide than are M₄C₃ and M₇C₃, 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 Japanèse Steel Works has recently introduced a modified $2\frac{1}{4}$ Cr-1 Mo steel with 0.25 V and 0.02% Ti added [7].

Although the proposed alloy development scheme summarized in table 3 appears to emphasize $2\frac{1}{4}$ 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

^{*} At present, the alloy development program for fusion reactors calls for about 5% of the irradiation effects studies to be on 2¹/₄ Cr-1 Mo, 35% on 9 Cr-1 MoVNb, and 60% on 12 Cr-1 MoVW.

steel the stronger [20]. 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 [10]. Only the $M_{23}C_6$ is observed in the 12 Cr-1 MoVW steel [10,18,19]. 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 $2\frac{1}{4}$ 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 $2\frac{1}{4}$ Cr-1 Mo steel with about 0.14% Ti has creep properties superior to those of commercial $2\frac{1}{4}$ Cr-1 Mo steel. If the $2\frac{1}{4}$ Cr-2 W and $2\frac{1}{4}$ 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.

3.2. 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 $2\frac{1}{4}$ 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 Varistraint 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 1.1×10^{26} n/m² [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.

4. Summary

It appears to be possible to develop Cr-Mo steels that have relatively fast induced-radioactivity decay (FIRD) characteristics, which would simplify the radioactive waste disposal problem for first wall and blanket structures of fusion reactors after shutdown. An approach to developing such steels has been presented.

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 possible; titanium and tantalum additions may also be valuable.

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