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Impurity effects on reduced-activation ferritic steels developed for fusion applications

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Abstract

Reduced-activation steels are being developed for fusion applications by restricting alloying elements that produce long-lived radioactive isotopes when irradiated in the fusion neutron environment. Another source of long-lived isotopes is the impurities in the steel. To examine this, three heats of reduced-activation martensitic steel were analyzed by inductively coupled plasma mass spectrometry for low-level impurities that compromise the reduced-activation characteristics: a 5-ton heat of modified F82H (F82H-Mod) for which an effort was made during production to reduce detrimental impurities, a 1-ton heat of JLF-1, and an 18-kg heat of ORNL 9Cr–2WVTa. Specimens from commercial heats of modified 9Cr–1Mo and Sandvik HT9 were also analyzed. The objective was to determine the difference in the impurity levels in the F82H-Mod and steels for which less effort was used to ensure purity. Silver, molybdenum, and niobium were found to be the tramp impurities of most importance. The F82H-Mod had the lowest levels, but in some cases the levels were not much different from the other heats. The impurity levels in the F82H-Mod produced with present technology did not achieve the low-activation limits for either shallow land burial or recycling. The results indicate the progress that has been made and what still must be done before the reduced-activation criteria can be achieved. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Development of low- or reduced-activation materials for fusion has focused on the issue of radioactive waste disposal [1] or recycling [2] of materials from fusion power plant components after they have reached the end of their service lifetime. The objective has been to eliminate or minimize those elements from an alloy that would produce long-lived radioactive isotopes during irradiation in a fusion neutron spectrum. Emphasis in the development process has generally centered on eliminating Nb, Cu, Ni, Mo, and N, widely used alloying elements, with niobium usually receiving the most attention because of the very low levels (<1 wppm) that

niobium cannot exceed if the steel is to meet low-activation criteria.

Besides the elements listed above, Butterworth et al. [2–4] have pointed out that there are various other elements that must be restricted to extremely low levels. Such elements could appear in the materials as tramp impurities and include Ag, Ho, Bi, Co, Sm, Lu, Dy, Gd, and Cd. Murphy and Butterworth [4] calculated the maximum amount of these elements allowed for recycling based on the assumption that “the concentrations of impurity elements were restricted to levels that would allow attainment of a surface γ dose rate not exceeding the ‘hands-on’ dose rate limit of 25 $\mu\text{Sv h}^{-1}$ at a 100 y cooling time for material subjected to a first wall neutron fluence of 12 MW y m^{-2} ”. This limit was suggested as a ‘target value’ [4].

Since the Murphy and Butterworth work [4], an updated library of activation cross-sections, FENDL/A-2.0, was developed as part of the fusion evaluated nuclear data library (FENDL) under the coordination

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of the International Atomic Energy Agency [5–7]. These new cross-sections were used previously to calculate the concentration limits of natural elements in low-activation fusion materials [8], and they will be used in this paper to compare the values obtained by analysis of selected steels.

Activation calculations to determine the decay characteristics of radioactive isotopes produced during irradiation of a material in a fusion environment have often ignored the tramp impurities listed above. In this work, several of these elements were determined in selected steels by analytical techniques to establish a base concentration for activation calculations and also to give an indication of what elements may need to be considered when processes are developed for maximizing reduced-activation characteristics for potential structural materials. A similar procedure was used previously to explore impurities in V–Cr–Ti alloys [9]. The results of these studies should be helpful to fusion design studies, such as the ARIES project.

2. Activation calculation procedure

For their calculations, Murphy and Butterworth [4] chose to apply the limiting criterion to each element individually and disregard additive effects. However, they pointed out that additive effects could be important for real materials. Under those conditions, the concentration of an individual element must be less than the limit for that element, and the amount allowed for the material to meet the guidelines for shallow land burial or recycle will depend on all the non-reduced-activation elements present. For n restricted elements, shallow land burial will be allowed if the waste disposal rating (WDR) is

$$\text{WDR} = \sum_{i=1}^n \frac{c_i}{c_{iW_{\max}}} \leq 1, \quad (1)$$

where c_i is the concentration of the i th element present in the alloy and $c_{iW_{\max}}$ is the maximum allowable concentration for that element as determined by the guideline for shallow land burial.

A similar materials recycling rating (MRR) can be defined as

$$\text{MRR} = \sum_{i=1}^n \frac{c_i}{c_{iR_{\max}}} \leq 1, \quad (2)$$

where c_i is the concentration of the i th element present in the alloy and $c_{iR_{\max}}$ is the maximum allowable concentration for that element as determined by the guideline for recycling.

The waste disposal limit calculation was based on the criteria of the guidelines (10 CFR Part 61) for Class C waste issued by the Nuclear Regulatory Commission

[10]. For the recycling calculation, the hands-on value of $25 \mu\text{Sv h}^{-1}$ was used for a 20 MW y m^{-2} exposure [4].

3. Experimental procedure

Inductively coupled plasma mass spectrometry (ICPMS) was used to determine the extremely low levels of impurities in several reduced-activation and conventional steels that have been studied in the fusion program.

The steels analyzed by ICPMS included: (1) a 5-ton heat of modified F82H (F82H-Mod), a nominal Fe–7.5Cr–2W–0.2V–0.02Ta–0.1C steel (all compositions are in wt%) that was purchased by the Japan Atomic Energy Research Institute, Tokai, Japan, from NKK Corporation, Kawasaki, Japan, for use in a collaborative test program by investigators in Japan, the European Union, and the United States coordinated by a committee of the International Energy Agency (IEA) to establish the feasibility of using ferritic steels for fusion; (2) a 1-ton heat of JLF-1, a nominal Fe–9Cr–2W–0.2V–0.09Ta–0.02N–0.1C reduced-activation steel (also used in the IEA collaboration) produced in Japan by Nippon Steel Corporation; (3) an experimental 18-kg heat of the ORNL 9Cr–2WV Ta steel, a nominal Fe–9Cr–2W–0.25V–0.07Ta–0.01C steel produced by Combustion Engineering, Chattanooga, TN; (4) a commercial heat of modified 9Cr–1Mo, a nominal 9Cr–1Mo–0.2V–0.07Nb–0.06N–0.1C steel; and (5) a commercial heat of Sandvik HT9, a nominal 12Cr–1Mo–0.3V–0.5W–0.5Ni–0.2C steel.

The metal samples for the ICPMS were dissolved in a mixture of HNO_3 , HF, and H_2O_2 . A reagent blank was prepared with the sample. A semiquantitative scan was conducted of the samples, and the spectra were examined for elements of interest and any other elements that were present. Before analyzing the sample, the instrument was checked for mass calibration and resolution. All elements reported were obtained by quantitative analysis using a blank 10 and 60 ppb standard for all the elements in a calibration curve. A calibration verification consisting of an EPA QC standard and a multi-element SPEX standard was analyzed to validate the calibration. All elements in the verification standard were within 10% of the calibration standards. A tenfold dilution was made on the dissolved sample that was analyzed, and the reagent blank was subtracted and calculated with an appropriate dilution factor applied. A portion of the diluted sample was spiked with 20 ppb of all elements reported. Spike recoveries were within 10%, which is the accuracy reported for the technique. Two readings were made and the precision for the elements for the first specimen is given in Table 1.

Table 1
Precision for elements analyzed by inductively coupled plasma mass spectrometry (wppb)

Element	Concentration	Element	Concentration	Element	Concentration
Li	0.01	Cd	0.23	Ta	0.3
Zr	0.67	Co	0.43	Er	1.5
Cr	0.11	Fe	0.65	Tb	0.2
Al	0.4	Ir	0.6	Ho	0.4
Ni	1.2	Nb	0.7	Y	2.0
Ag	0.4	Mo	1.2	Hf	0.14
B	1.8	Pd	0.04	W	0.5
Bi	0.03	Sn	0.5	Eu	0.14

4. Results

The following elemental impurities that must be limited for reduced-activation consideration were determined by ICPMS: Ag, Bi, Cd, Co, Dy, Er, Eu, Ho, Ir, Mo, Nb, Ni, Os, Tb, and U. Results for two specimens of the F82H-Mod are given in Table 2. Also shown in the table are results for the JLF-1, ORNL 9Cr–2WVTa, modified 9Cr–1Mo, and HT9 steels.

In an effort to understand what may be possible in the future, samples of ‘high-purity’ iron, chromium, and tungsten were purchased from Alpha Aesar Company and analyzed. These included two iron rods labeled 99.99% and 99.9985% purity, chromium labeled 99.995% purity and tungsten rod and powder labeled 99.96% and 99.999% purity, respectively. A piece of iron from a research program to obtain high-purity iron by ‘selective prepurification and oxidation zone refining’ [11] was also analyzed. All but the chromium was ana-

lyzed by ICPMS; the chromium was analyzed for another study [9] by glow discharge mass spectroscopy.

Concentration of the impurities in these materials are given in Table 3. High-purity vanadium was analyzed in previous work [9], but because of the small amount of vanadium present in the steels (0.2–0.25%) and the low-level of impurities found in that analysis, the analysis is not given here.

5. Discussion

Table 2 indicates that with the exception of two elements, cobalt and nickel, the concentrations of the individual impurity elements in the F82H-Mod are lower or as low as in any of the other five steels. Nickel and cobalt are lowest in the JLF-1 steel. These results provide an indication of what may be possible for reduced-activation steels. They can also serve as the starting

Table 2
Chemical composition of deleterious impurity elements in reduced-activation of ferritic steels determined by inductively coupled plasma mass spectrometry (wppm)

Element	F82H-Mod	JLF-1	ORNL 9Cr–2WVTa	Modified 9Cr–1Mo	HT9
Ag	<0.05	<0.1	0.21	0.16	0.23
Bi	<0.2	<0.05	<0.1	<0.05	<0.1
Cd	<0.4	<0.05	<0.05	<0.05	3.3
Co	28	16	7.6	34	58
Ir	<0.02	<0.05	<0.05	<0.05	<0.05
Mo	21	19	20	70	— ^a
Nb	3.3	2.4	4.3	4	— ^a
Ni	NA ^b	474	13	402	1251 ^a
Os	NA	<0.05	<0.05	<0.02	<0.02
Pd	NA	<0.05	<0.05	0.18	0.27
Dy	NA	<0.05	<0.05	<0.05	<0.05
Er	NA	<0.05	<0.05	<0.05	<0.05
Eu	<0.02	<0.05	<0.05	<0.05	<0.05
Ho	<0.05	<0.05	<0.05	<0.05	<0.05
Tb	<0.02	NA	<0.05	NA	<0.05
U	<0.003	<0.05	<0.05	0.6	0.12
Al	NA	14	26	170	<0.1
Cu	NA	100	100	300	1000

^a Element is part of the specified composition.

^b Not analyzed.

Table 3
Chemical composition of deleterious impurity elements in high-purity elements (wppm)^{a,b}

Element	Iron			Chromium 99.995%	Tungsten	
	99.99%	99.9985%	OxZR		99.99%	99.999%
Ag	<0.2	0.2	4.4	0.85	<0.2	<0.2
Bi	6	<0.2	<0.2	<0.12 ^c	0.95	3.4
Cd	1	0.3	<0.5	<0.3	<0.2	0.5
Co	32	<0.2	<0.2	<0.012	<0.2	<0.2
Ir	<0.2	<0.4	<0.2	<44	<0.2	<0.2
Mo	1.4	0.85	<0.5	0.023	1.4	<0.2
Nb	1.2	4.1	<0.2	<20 ^c	<0.2	6.8
Ni	21.9	5.6	<0.2	<0.22	<0.2	0.68
Os	<0.2	<0.2	<0.2	<0.04	<0.2	<0.2
Pd	<0.2	<0.2	<0.5	<0.07 ^c	<0.2	<0.2
Dy	<0.2	<0.2	<0.2	<0.04	<0.2	<0.2
Er	<0.2	<0.2	<0.2	<0.035 ^c	6	<0.2
Eu	<0.2	<0.2	<0.2	<0.006	<0.2	<0.2
Ho	<0.2	<0.2	<0.2	<0.003	<0.2	<0.2
Tb	<0.2	<0.2	<0.2	<0.003	<0.2	<0.2
U	0.46	0.3	<0.3	<0.009	<0.2	<0.2
Al	1800	<1	<0.5	<2 ^c	<1	2000
Cu	62	49	4.9	<0.27 ^c	<0.2	<0.2

^a All high-purity elements but the OxZR were obtained from Alpha Aesar; the purity designations are those given by the company. The OxZR is oxide zone-refined iron obtained from Dr B.F. Oliver (see Ref. [11] for information on the purification technique).

^b Except for the chromium, concentrations were determined by inductively coupled plasma mass spectrometry; the chromium was analyzed by glow discharge mass spectroscopy.

^c Interference limited the determination.

point for determining how these steels can be processed to the purity levels required. That is, a detailed analysis of the materials processing techniques used for these heats could indicate reasons for the differences and how the impurities might be further reduced.

For the F82H-Mod heat produced by NKK Corporation, an effort was made to minimize niobium in the steel, but no special effort was made to reduce any of the other elements that were not specified (i.e., Ag, Cd, Co, etc.). High purity iron ingots were produced using a converter, after which only ingots containing below 0.5 ppm Nb were selected for the heat [12]. Clean materials were also selected for the other alloying additions, and vacuum-induction melting (VIM) was used. This processing procedure resulted in this steel having the lowest silver and niobium content for any of the steels in Table 2.

Despite the chosen process to reduce niobium content, the concentration in the F82H-Mod was only slightly lower than for JLF-1 and 9Cr–2WVTa. The exact procedure used for choosing the melt stock for the JLF-1 is not known, but VIM was used for melting. The ORNL 9Cr–2WVTa was a small 18-kg experimental heat of steel made using off-the-shelf melt stock that was air melted and then electro-slag remelted. Molybdenum was slightly lower in the F82H-Mod and the JLF-1 steel than the 9Cr–2WVTa, but only by a factor of 3. The Sandvik HT9 and modified 9Cr–1Mo steels were large commercial heats, probably made with scrap. The

niobium concentration was an order of magnitude higher in the HT9 than for the reduced-activation steels (niobium is part of the specified composition of modified 9Cr–1Mo steel).

As pointed out, the F82H-Mod had the highest nickel and cobalt content. These elements go together; that is, most of the cobalt is probably introduced as an impurity in the nickel. Even electrolytic nickel (described as 99.95% Ni + Co), which is also known as ‘pure nickel’, contains 0.3–0.5% cobalt. The data in Table 2 indicate a correlation between the cobalt and nickel in the steels. Nickel (0.5%) is added to the HT9, and this steel also has the highest cobalt (0.04%) content. Of interest is the low level of cobalt in the JLF-1 and the relatively high level in the F82H-Mod and 9Cr–2WVTa. Since no nickel was added to any of these steels, tracing the origin of the nickel should indicate part of the solution to reducing cobalt in future heats, should that be necessary.

These results provide a starting point for discussing the present status of reduced-activation ferritic/martensitic steels and the steps required to reach the goal of a steel that meets the criteria for shallow land burial and recycling. In Table 4, the waste disposal limit (WDL) and the materials recycle limit (MRL) are given as calculated using FENDL/A-2.0 [5–7] for the elements that need to be restricted for a fusion system after 4 years of operation at 5 MW m⁻², an integrated wall loading of 20 MW y m⁻². In all cases a range of values is given for the WDL and MRL. The low number is for the first wall

Table 4

Limits of deleterious impurity elements for shallow land burial and recycle compared with the concentration of these elements in present and future reduced-activation steels (wppm)^a

Element	Waste disposal limit ^b	Materials recycle limit ^b	F82H-Mod	Present	Future
Ni	15–38%	87–470 (1.6–4.3%) ^c	474	13	0.1
Mo	31–37	3.6–20 (4.1–23)	21	5	1
Ag	1.2–2.7	0.012–0.026 (0.017–0.036)	<0.1	<0.05	<0.005
Bi	22–109	0.017–0.41 (0.48–3.2)	<0.2	<0.05	<0.01
Cd	0.14–1.7%	2.1–24 (2.9–29)	<0.4	<0.05	<0.05
Co	19% – no limit	2.3–14 (0.53–18%)	28	8	<0.02
Ir	22–29	0.21–0.27 (0.37–0.48)	<0.05	<0.05	<0.05
Nb	2.4–3.5	0.055–0.08 (0.055–0.08)	3	0.5	<0.02
Os	560–3100	5.2–29 (9.3–52)	<0.05	<0.05	<0.02
Pd	110–1700	0.17–2.5 (0.23–3.5)	<0.05	<0.05	<0.05
Dy	4.6–140	0.095–2.0 (0.11–2.8)	<0.05	<0.05	<0.05
Er	28–285	0.73–6.5 (0.82–7.3)	<0.05	<0.05	<0.05
Eu	1.3–1.4%	0.016–0.0054 (2.4–1.3)	<0.05	<0.02	0.01
Ho	0.7–1.2	0.017–0.028 (0.019–0.032)	<0.05	<0.05	0.01
Tb	1.9–5	0.0046–0.012 (0.0099–0.026)	<0.02	<0.02	<0.001
Al	660–3900	13–79 (13–79)	140	30	0.5
Cu	73% – no limit	160 – no limit (20% – no limit)	10	10	<5

^a Cooling time for waste disposal and hands-on materials recycling 100 y after a 20 MW y m⁻² exposure.

^b The low number of the two is for the first wall alone, and the high number is for the blanket average; concentrations are in wppm (no units given) wt% (units given).

^c Concentration limits given in parentheses are for cooling time of 300 y.

alone, and the high number is for the blanket average. Values are for a cooling time of 100 y, except for the MRL values in parentheses, which are for a 300 y cooling period. The WDL and MRL values are the $c_{iW_{max}}$ of Eq. (1) used to calculate WDR and the $c_{iR_{max}}$ of Eq. (2) for MRR.

The fourth column of Table 4 gives the concentration of restricted elements for the F82H-Mod (the highest of the two values in Table 2 was chosen). A WDR value of 2.2–1.5 was calculated with Eq. (1) for the low and high WDL values, indicating that more of the restricted elements need to be removed from F82H-Mod before the steel will meet reduced-activation criteria for shallow land burial. Calculated WDR and MRR values are given in Table 5.

The concentrations in the column labeled ‘Present’ in Tables 4 and 5 are the lowest values that have been measured in different steels (e.g., the low values measured for F82H-Mod, the Ni and Co in JLF-1, Os in ORNL 9Cr–2WVTa, etc.). These values should be achievable at present with a relatively modest effort by analyzing the techniques used to achieve the low levels in

the different steels. The WDRs calculated using these concentrations is 0.51–0.28 (Table 5). Thus, such steel would meet the criteria for shallow land burial.

The final column in Tables 4 and 5 labeled ‘Future’ gives estimated concentration values that should be possible to achieve in the future with the proper choice of techniques to produce ‘pure’ melt stock. These estimated values were derived by using the chemical analyses of the ‘high-purity’ Fe, Cr, and W of Table 3 and the special iron obtained by oxidation zone refining (OxZR in Table 3) after prepurification [11]. The OxZR iron contained <0.2 ppm Nb, the lowest value of this element in any of the iron analyzed. The lowest value in commercial high purity iron was 1.2 ppm. Calculated WDR values for the Future concentrations are 0.09–0.04 (Table 5). Note that the concentration values given in Table 4 for Ag, Bi, Co, Nb, Eu, Ho, Tb have not been analyzed in the steels or the pure materials. The lowest values obtained for these elements have been at the limit of detection for the analytical chemistry techniques used. Lower estimates for these elements were made and are given in Table 4.

Table 5

Waste disposal rating (WDR) and materials recycle rating (MRR) for reduced-activation ferritic/martensitic steels^a

Rating	F82H-Mod	Present	Future
WDR (100 y)	2.1–1.2	0.51–0.28	0.09–0.04
MRR (100 y)	120.2–60.6	33.1–16.8	4.6–3.3
MRR (300 y)	82.2–45.5	21.1–10.7	2.9–1.25

^a WDR < 1 and MRR < 1 to meet criteria for shallow land burial and recycling, respectively.

Table 6
Estimated concentration of typical elements in reduced-activation steel (wt%)^a

Element	Waste disposal limit ^b	Materials recycle limit ^{b,c}	F82H-Mod
C	No limit	No limit	0.1
Si	No limit	No limit	0.1
Mn	No limit	No limit	0.2
P	No limit	No limit	0.008
S	No limit	No limit	0.002
Cr	No limit	No limit	7.5
W	41–no limit	0.11–0.37 (1.8–19)	2
V	No limit	No limit	0.2
B	No limit	No limit	0.002
N	0.18–0.36	No limit	0.006
Ti	No limit	0.14–1.4 (9.3–no limit)	0.001
Ta	No limit	0.026–1.0 (2.2–7.8)	0.02

^a Cooling time for waste disposal and hands-on materials recycling 100 y after a 20 MW y m⁻² exposure.

^b The low number of the two is for the first wall alone, and the high number is for the blanket average.

^c Concentration limits given in parentheses are for cooling time of 300 y.

Despite the low WDR values for the Future reduced-activation steel, the steel would not meet the criteria for recycling because the MRR is 4.6–3.3 after a 100 y cooling period (Table 5). For a 300 y cooling period, the MRR drops to 2.9–1.25, still above the value required for ‘hands-on’ recycling. This applies to the 20 MW y m⁻² integrated first wall neutron exposure. It could be hands-on recycled after 100 years if the neutron exposure were reduced to 6 MW y m⁻² or after a 300 y cooling period if the exposure were reduced to 16 MW y m⁻². The 300 y cooling period is probably impractical, but it was included to serve as an indication of the magnitude of the problem posed by hands-on recycling.

Much more work and probably considerably more expense will be required to reach the levels required for materials recycling based on a hands-on dose-rate limit of 25 μSv h⁻¹ at a 100 y cooling time. Even a 300 y cooling time would not allow these criteria to be reached easily for a 20 MW y m⁻² neutron exposure. Indeed, it would probably take considerable effort and expense just to determine if those criteria for recycling could ever be met for the compositions in question.¹

Another problem for recycling Fe–Cr–W–V–Ta steel appears when the WDL and MRL values are examined for the major alloying elements (Table 6). Although none of these elements will affect the WDL values calculated from the impurity concentrations (Table 5), there will be an effect of tungsten and tantalum on the MRL. Obviously, if reduced-activation steel were ever to be recycled under the criteria used for the present cal-

culations, then they would essentially have to be free of tungsten. Tantalum would probably also need to be restricted, depending on the levels of other restricted elements. This means that new steels need to be developed for the recycling option.

6. Conclusions

Chemical analysis of potential reduced-activation ferritic/martensitic steels produced by present technology were analyzed to provide an indication of the level of tramp impurity elements that compromise the reduced-activation characteristics of the steel. The results for the steels indicate that progress has been made in reducing the level of detrimental impurities for two large heats of reduced-activation steel. Silver, niobium, and molybdenum proved to be the most important of the restricted elements, and the steels that have been produced up to now do not meet the criteria for low-activation for shallow land burial of nuclear waste made up of these steels after service to an integrated wall loading of 20 MW y m⁻² followed by a 100 y cooling period. However, it appears that reduced-activation steels could be produced with these detrimental impurity elements at levels low enough to meet the present criteria for shallow land burial. If instead of shallow land burial, it is desired to recycle the steel with a hands-on dose rate limit of 25 μSv h⁻¹ after a 100 y cooling time, then it appears that considerable research and expense will be required to develop processes that will allow the production of steel that will meet these criteria. These conclusions for the reduced-activation steels are the same as those previously reached for V–Cr–Ti alloys [9].

There has probably never been a requirement for a structural material to be processed to reduce specified impurity levels as low as those required to meet the re-

¹ Remote recycling could be another alternative – a very expensive one – that will need to be explored. For example, remote recycling with a 10 mSv h⁻¹ dose rate limit would allow a 1000 fold increase in the concentration limits.

duced-activation criteria. Even though the materials will not be needed for some time, an effort should be mounted to determine the means to achieve the desired purity levels, especially if recycling is to be pursued. Such an effort should enlist support from industrial materials processors to examine techniques used in the past to produce high-purity materials and determine ways that these techniques might be combined with the latest technology for future application. The effort should not be delayed if reduced-activation materials are to be available when fusion power production is ready to begin operation.

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