

## **IMPURITY CONTENT OF REDUCED-ACTIVATION FERRITIC STEELS AND A VANADIUM ALLOY R. L. Klueh, M. L. Grossbeck, and E. E. Bloom (Oak Ridge National Laboratory)**

### **OBJECTIVE**

This work was conducted to obtain a benchmark for the chemical concentrations of important impurity elements in a reduced-activation ferritic steel and a vanadium alloy produced by present commercial technology and to provide a consistent set of concentration values for use in activation calculations.

### **SUMMARY**

Inductively coupled plasma mass spectrometry was used to analyze a reduced-activation ferritic/martensitic steel and a vanadium alloy for low-level impurities that would compromise the reduced-activation characteristics of these materials. The ferritic steel was from the 5-ton IEA heat of modified F82H, and the vanadium alloy was from a 500-kg heat of V-4Cr-4Ti. To compare techniques for analysis of low concentrations of impurities, the vanadium alloy was also examined by glow discharge mass spectrometry. Two other reduced-activation steels and two commercial ferritic steels were also analyzed to determine the difference in the level of the detrimental impurities in the IEA heat and steels for which no extra effort was made to restrict some of the tramp impurities. Silver, cobalt, molybdenum, and niobium proved to be the tramp impurities of most importance. The levels observed in these two materials produced with present technology exceeded the limits for low activation for either shallow land burial or recycling. The chemical analyses provide a benchmark for the improvement in production technology required to achieve reduced activation; they also provide a set of concentrations for calculating decay characteristics for reduced-activation materials. The results indicate the progress that has been made and give an indication of what must still be done before the reduced-activation criteria can be achieved.

### **PROGRESS AND STATUS**

#### **Introduction**

Development of low- or reduced-activation materials for fusion has focussed on the issue of radioactive waste disposal or recycle; the objective has been to eliminate or minimize alloying elements that would produce long-lived radioactive isotopes during irradiation in a fusion neutron spectrum. Emphasis in the development process has centered on eliminating Nb, Cu, Ni, Mo, and N, widely used alloying elements, with niobium often receiving much of the attention because of the very low concentrations (<1 wppm) that will be required to meet criteria for shallow land burial or recycling, should those methods be chosen to handle the waste materials from a fusion power plant component after it has reached the end of its service lifetime.

Besides the elements listed above, Butterworth and his co-workers [1-3] 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. Table 1, which is taken from Murphy and Butterworth (M&B) [3], gives a list of elements and the limits that must be achieved to keep from compromising the reduced-activation criteria. These maxima were calculated so that "the concentrations of impurity elements were restricted to levels that would allow attainment of a surface  $\gamma$  dose rate not exceeding the "hands-on" dose rate limit of  $25 \mu\text{Svhr}^{-1}$  at a 100 y cooling time for material subjected to a first wall neutron fluence of  $12 \text{ MWym}^{-2}$ . This limit was suggested by M&B [3] as a "target value," and the impurity concentrations that are required to achieve this level will be used in this report for comparison with the values found in the materials analyzed. It needs to be emphasized that the M&B criteria have no "official" status relative to what might be required in the future (when fusion power plants are built or when they are decommissioned) for recycling or shallow land burial (values for recycle are expected to be lower than for shallow land burial, for which limits will probably be higher than the "hands-on" level).

Activation calculations to determine the decay characteristics of radioactive products produced during irradiation of a material in a fusion environment have often ignored some of the elements listed in Table 1. In this report, several of these elements have been determined 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.

M&B chose to apply the 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 the individual elements must be less than those given in Table 1. In particular, for  $n$  restricted elements, the total concentration of all such impurity elements,  $C$ , must be such that

$$C = \sum_{i=1}^n \frac{c_i}{c_{imax}} \leq 1$$

where  $c_i$  is the concentration of the element present in the alloy and  $c_{imax}$  is the maximum allowable concentration for that element.

Table 1. Maximum permitted concentrations of radiologically critical elements to achieve a "hands-on" dose rate limit after 100 y of cooling after a neutron fluence of  $12\text{MWy m}^{-2}$  (after reference2)

Element	Concentration (wppm)	Element	Concentration (wppm)
Ag	0.011	U	1
Ho	0.067	Gd	4.1
Bi	0.14	Cd	5.0
Co	0.16	Ba	16
Nb	0.18	Mo	18
Sm	0.30	K	76
Lu	0.33	Al	170
Dy	0.62	Cl	8500

### Experimental Procedure

A heat of ferritic/martensitic steel and a vanadium alloy were analyzed by inductively coupled plasma mass spectrometry (ICPMS). The steel specimen was taken from a 5-ton heat of modified F82H, an 8Cr-2WVTa steel that was purchased by the Japan Atomic Energy Research Institute from NKK Corporation, Kawasaki, Japan, for use in a collaborative test program by investigators in the U.S., Japan, and the European Union coordinated by the International Energy Agency (IEA) to establish the feasibility of using ferritic steels for fusion applications. Details of the melting and processing of this IEA Mod F82H steel will be published in the future. During the production of this heat, an effort was made to minimize niobium in the steel, but not any of the other elements that are not specified (i.e., Ag, Cd, Co, etc). Table 2 gives the vendor analysis of the steel. The vanadium alloy was from a 500-kg heat of V-4Cr-4Ti alloy obtained for the fusion program. This heat was specified and purchased on a "best-effort" basis from Teledyne Wah Chang, Albany, Oregon, by Argonne National Laboratory [4]; the vendor analysis is given in Table 2. Because of time considerations in delivering the 500-kg heat, Teledyne Wah

Chang used available starting materials to produce the alloy. Teledyne Wah Chang personnel believe that tramp impurities, especially molybdenum and niobium, can be lowered substantially by proper choice of starting materials. However, the niobium values of this heat seem to be typical of those obtained for vanadium alloys produced up until now. Molybdenum values are generally lower, often reported as <100 ppm. Recently, a 1200-kg ingot was produced with specification of <50 ppm for molybdenum and niobium [5]. The molybdenum specification was met (<50 ppm reported), but the niobium was measured at 140, 91, and 87 ppm at the top, middle, and bottom of the ingot [5].

To determine the variation in impurities in current fusion-program steels (reduced-activation and commercial steels), several other steels were analyzed by ICPMS. These included specimens taken from: 1) a 1-ton heat of JLF-1, a 9Cr-2WVTa steel produced in Japan by Nippon Steel Corporation; 2) an experimental 25 kg heat of a 9Cr-2WVTa steel produced by Combustion Engineering for ORNL with no effort to minimize impurities; 3) a commercial heat of modified 9Cr-1Mo (9Cr-1MoVNb) steel; and 4) a commercial heat of Sandvik HT9 (12Cr-1MoVW).

Table 2. Vendor analysis of the ingot of IEA heat of modified F82H steel and V-4Cr-4Ti alloy (wt. pct)

Element	Mod F82H	V-4Cr-4Ti	Element	Mod F82H	V-4Cr-4Ti
C	0.09	0.008	V	0.16	Balance
Si	0.11	0.078	Nb	0.0001	0.006
Mn	0.16		B	0.002	<0.0005
P	0.002	<0.003	N	0.006	0.0085
S	0.002	<0.001	Al	0.003	<0.001
Cu	0.01	<0.005	Co	0.005	
Ni	0.02		Ti	0.01	3.9
Cr	7.72	3.8	Ta	0.02	
Mo	0.003	0.031	W	1.97	
Fe	Balance	0.022	O		0.031

To examine analytical chemistry techniques for low-level concentrations, the V-4Cr-4Ti was also analyzed by glow discharge mass spectrometry (GDMS) by two different vendors. Therefore the results for the different techniques can be compared, along with the variations in the same technique when conducted by different vendors.

The metal samples for the ICPMS were dissolved in a mixture of HNO<sub>3</sub>, HF, and H<sub>2</sub>O<sub>2</sub>. 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 ppb and 60 ppb standard for all the elements in a calibration curve. A calibration verification combined of an EPA QC standard and a multi-element SPEX standard was analyzed to validate the calibration standards. All elements in the verification standard were within 10% of the calibration standards. A ten-fold 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 are given in Table 3.

Table 3. Precision for elements analyzed by inductively coupled plasma mass spectrometry (wppb)

Element	Concentration (ppb)	Element	Concentration (ppb)	Element	Concentration (ppb)
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

The GDMS analyses were conducted in two runs on the same sample on different days. Since GDMS is a sputtering process, the material for the first run was taken closer to the surface of the sample than the second run. The relative sensitivity factor (RSF) for the elements was taken as the standard for iron, which was then normalized for vanadium. This limits the accuracy of the analysis to the accuracy of the standard iron RSF, some of which may not be correct. Thus a high accuracy could not be guaranteed, and a calibration with vanadium standards is required for high accuracy analyses.

### Results

The following elemental impurities 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 V-4Cr-4Ti and the IEA Mod F82H are given in Table 4.

For the ferritic steel, Co (28 and 16 wppm observed vs. the 0.16 wppm limit) and Nb (3.3 wppm and 2.4 vs. 0.18 wppm) were over the B&M limit, and Mo was at the limit (21 and 19 wppm vs. 18 wppm). The limit of detection for ICPMS for the Ag and the Bi were just above the permitted limit, indicating that these two elements may or may not be above the B&M limit. A more sensitive technique is required to determine this.

The elements definitely above the B&M limits for the V-4Cr-4Ti alloy include Ag (0.86 and 0.6 wppm vs. 0.011), Co (4.2 and 0.2 wppm vs. 0.16 wppm), Mo (370 and 329 wppm vs. 18 wppm), and Nb (32 and 64 wppm vs. 0.18 wppm). The Ho and Tb may or may not be above the limit, but a more sensitive technique is required for this to be determined.

There were some discrepancies as well as agreement for the elements analyzed by the ICPMS and GDMS techniques and between the GDMS analyses of the two vendors (Table 5). The major discrepancies appeared to be between ICPMS and GDMS Vendor 1 for Ag, Bi, and Cd. The GDMS Vendor 1 found less of these elements than were found by ICPMS or by Vendor 2. The ICPMS results for the different reduced-activation and commercial steels showed a range of differences in several of the impurity elements, with the major differences being in Ag, Cd, Co, Mo, Ni, Pd, and U (Table 6).

Table 4. Chemical composition from two measurements of important impurity elements for a heat of V-4Cr-4Ti and a reduced-activation martensitic steel determined by inductively coupled plasma mass spectrometry (wppm)

Elements	V-4Cr-4Ti		IEA F82H	
Ag	0.86	0.6	<0.05	<0.1
Bi	<0.05	0.46	<0.2	<0.05
Cd	1.6	0.3*	<0.4	<0.05
Co	4.2	0.2*	28	16
Ir	<0.5	<0.05	<0.02	<0.05
Mo	370	329	21	19
Nb	32	64	3.3	2.4
Ni		11		474
Os		<0.05		<0.05
Pd		0.2		<0.05
Dy		<0.05		<0.05
Er		0.1*		<0.05
Eu	<0.2	<0.05	<0.02	<0.05
Ho	<0.2	<0.05	<0.05	<0.05
Tb	<0.2	<0.05	<0.02	
U	0.13	0.21	<0.003	<0.05

\* At or near the limit of detection

### Discussion

The results of the ICPMS analyses (Table 4) indicate that there are several impurities in the V-4Cr-4Ti and the IEA Mod F82H steel that are above the "hands-on" level defined by M&B [3]. These include Ag, Co, Mo, and Nb for the V-4Cr-4Ti and Co and Nb for the IEA Mod F82H steel. These results for the V-4Cr-4Ti are similar to those

of M&B, who studied the production of high-purity V, Cr, and Ti and wrote [3], "Of the three metals studied, vanadium and its compounds exhibited the highest levels of contamination. Mo, Nb, Co and Ag were frequently found near or above the concentration limits." These authors described additional purification steps that might be required to remove these impurities.

The ICPMS results for the different steels in Table 6 provide an indication of what may be possible for reduced-activation steels without special purification. They can also serve as the starting point for determining how these steels can be processed to the 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.

Table 5. Chemical composition of impurity elements for V-4Cr-4Ti alloy determined by ICPMS and GDMS (wppm)

Element	ICPMS		GDMS Vendor 1		GDMS Vendor 2	
Ag	0.86	0.6	0.11	0.045	<0.3	<0.6
Bi	<0.05	0.46	<0.0075	<0.0061	<2	<<134
Cd	1.6	0.3*	<0.19	≤0.13	1	1
Co	4.2	0.2*	0.31	0.28	≤0.22	≤0.21
Ir	<0.5	<0.05	<0.003	<0.003	1.5	<0.1
Mo	370	329	310	320	325	350
Nb	32	64	<59	<63	70	78
Ni		11	9.5	9.7	7	7
Os		<0.05	<0.009	<0.0072	<0.004	<0.002
Pd		0.2	<0.13	0.14	0.4	0.3
Dy		<0.05			<0.002	N/D
Er		0.1*			<0.08	<0.3
Eu	<0.2	<0.05			<0.035	<0.009
Ho	<0.2	<0.05			<0.008	<0.016
Tb	<0.2	<0.05			N/D	<0.004
U	0.13	0.21	0.085	0.082	<0.03	<0.003

N/D Not detected

\* At or near the limit of detection

Special purification techniques were used by NKK Corporation to obtain the iron used to make the IEA Mod F82H, along with the selection of clean materials for the other alloying additions. This choice of materials resulted in this steel having the lowest silver content. This approach was used in the hope that it would reduce the niobium to extremely low levels, and a low level was achieved compared to the commercial HT9 (Table 6); however, the concentration of niobium for this steel was only slightly lower than that for JLF-1 and 9Cr-2WVTa, for which no such precautions were taken. The exact procedure used for choosing the melt stock for the JLF-1 is not known, but the ORNL 9Cr-2WVTa was an experimental steel made by Combustion Engineering, Chattanooga, TN, using off-the-shelf melt stock to make the small heat. The Sandvik HT9, which was a large commercial heat, probably was made with the use of scrap, and the niobium concentration was an order of magnitude higher than for the reduced-activation steels. Molybdenum was slightly lower in the IEA Mod F82H and the JLF-1 steel than the 9Cr-2WVTa, but only by a factor of 3. Most of the cobalt must come from the nickel. Even electrolytic nickel (99.95% Ni), which is also known as "pure nickel," contains 0.3-0.5% cobalt. The data in Table 6 indicate a correlation between the cobalt and nickel in the steels. Of interest is the low level of cobalt in the JLF-1 and the relatively high level in the IEA Mod F82H 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 of what will be required to reduce cobalt in future heats.

Table 6. Chemical composition of important impurity elements for reduced activation of ferritic steels determined by inductively coupled plasma mass spectrometry (wppm)

Element	ORNL					
	IEA Mod F82H	JLF-1	9Cr-2WVTa	Mod 9Cr	HT9	
Ag	<0.05	<0.1	0.21	0.16*	0.23	1.3
Bi	<0.2	<0.05	<0.1	<0.05	<0.1	<0.1
Cd	<0.4	<0.05	<0.05	<0.05	3.3	5.1
Co	28	16	7.6	34	58	393
Ir	<0.02	<0.05	<0.05	<0.05	<0.05	<0.05
Mo	21	19	20	70	**	**
Nb	3.3	2.4	4.3	4	**	23
Ni		474	13	402	1251**	5692**
Os		<0.05	<0.05	<0.02	<0.02	<0.05
Pd		<0.05	<0.05	0.18*	0.27	0.4
Dy		<0.05	<0.05	<0.05	<0.05	<0.05
Er		<0.05	<0.05	<0.05	<0.05	<0.05
Eu	<0.02	<0.05	<0.05	<0.05	<0.05	<0.05
Ho	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Tb	<0.02		<0.05		<0.05	<0.05
U	<0.003	<0.05	<0.05	0.6	0.12	<0.05

\* At or near the limit of detection

\*\* Element is part of the specified composition

As stated above, it is believed that with the proper choice of starting materials the molybdenum and niobium can be reduced substantially from the values in Table 5. However, the 500-kg heat is one of the largest vanadium-alloy heats ever produced, and so the results for this heat and the information on the IEA Mod F82H provide a "starting point" for discussing the present status of potential reduced-activation materials and the steps required to reach the goal of a reduced-activation material.

Recently, calculations have been made to predict decay characteristics of fusion first wall and blanket structures after service operation [6,7]. Various assumptions were used for the concentrations of the impurities. Ehrlich et al. [6] considered the radioactivity decay after a neutron wall loading of 5 MW/m<sup>2</sup> for 2.5 years, which gives an integral wall loading of 12.5 MWym<sup>2</sup>. For the pure materials, V, Ti, and Cr had the best decay characteristics, followed by iron. Without impurities, V-Cr-Ti alloys have radioactivity decay properties superior to any other potential reduced-activation structural material, including silicon carbide, often considered the best low-activation material. When the calculations were made for the vanadium alloy with "realistic" impurity concentrations, Ehrlich et al. [6] found that, "a strong increase of long-term activation by several orders of magnitude can be caused by impurities."

Of the impurities discussed in the present report, Ehrlich et al. [6] assumed values for Ag, Co, Nb, and Mo of 0.03, 0.02, 0.01, and 0.15 wppm, respectively. These values are respectively about 30, 200, 3000, and 2500 times less than the values observed for the vanadium alloy analyzed by ICPMS (Table 4). Ehrlich et al. chose those impurity concentrations because these values were estimated by Johnson Matthey as concentrations that could be achieved in vanadium alloys by special techniques, although these limits would be difficult and expensive to reach [6].

Ehrlich et al. [6] also examined reduced-activation ferritic steels and made calculations for existing steels and for an optimized steel, where the impurity levels are assumed to be those that should be achievable under conditions where special efforts are made to reduce impurity levels. The interesting observation that came out of the comparison of the optimized ferritic steel and the V-Cr-Ti alloy with impurities at the level that should be achievable was the conclusion by Ehrlich et al. [6] that a "realistic assumption of technically achievable amounts of tramp elements leads nearly to the same level of activation [for the vanadium alloy] as for the optimized ferritic-martensitic steels."

Attaya and Smith [7] performed radioactivity decay calculations for a V-4Cr-4Ti alloy and the ORNL 9Cr-2WVTa steel after exposure to a  $5\text{MWm}^{-2}$  neutron wall loading and 5 years of operation. The vanadium alloy was found superior to the 9Cr-2WVTa steel with and without the impurities. Impurity levels were assumed to be those typical for present technology. For the V-4Cr-4Ti, it was stated that [7], "impurity content is based on chemical analyses of different heats of similar alloys." Impurities had a large effect on the behavior of the vanadium alloy, but a relatively small effect on the ferritic steel. Attaya and Smith assumed that Ag, Co, Nb, and Mo were present in the V-4Cr-4Ti at levels of 0, 0, 1, and 4 wppm, respectively. These are low values for niobium and molybdenum based on the values of Table 4 and those reported for vanadium alloys produced up to the present; likewise, the values for silver and cobalt are low for those measured in the 500-kg heat. In the case of ferritic steel, Attaya and Smith [7] stated that the "impurities of the ORNL 9Cr-2WVTa ferritic steel are assumed as that of the modified HT-9" [7], which appears to be a hypothetical steel postulated for a previous calculation [8]. The concentrations for Ag, Co, Nb, and Mo were assumed to be 0.9, 50, 1.1, and 2.7 wppm, respectively. In this case, the Nb and Mo were below what was observed in the IEA Mod F82H, and the Ag and Co were substantially above what was observed in the present analyses. Since Attaya and Smith [7] found that impurities had a large effect on the decay characteristics of the vanadium alloy but much less relative effect on the ferritic steel, the additional impurities found in the results given in Table 4 would cause the vanadium alloy decay curve to approach the curve for the ferritic steel.

If it is assumed that the impurity levels measured by ICPMS (Table 4) are representative of today's processing practice for vanadium alloys and reduced-activation ferritic steels, calculations similar to those of Ehrlich et al. [6] and Attaya and Smith [7] could be used to qualitatively estimate the relative reduced-activation status of vanadium alloys and ferritic steels today and where the materials will stand once they are optimized with respect to deleterious impurities. Although the calculations need to be performed, it appears that if the Ag, Co, Nb, and Mo impurities detected in the present study (or appropriate values from other heats in the case of molybdenum, which appears extra high in the 500-kg heat) were used for the calculations, values that were not used by either Ehrlich et al. [6] or Attaya and Smith [7], the differences between the two materials would not be extremely large, based on the larger amounts of Nb, Mo, and Ag present in the vanadium alloy and the greater effect of impurities in the vanadium alloys than the ferritic/martensitic steel. Then, the question becomes: Can the optimized impurity values of Ehrlich et al. be achieved? If both can be achieved, then according to Ehrlich et al. [6], "the actual advantage of vanadium-chromium-titanium alloys in comparison to the optimized ferritic-martensitic steels diminishes to about one order of magnitude with regard to the long-term radioactivity."

## Conclusions

Chemical analysis of potential reduced-activation ferritic steels and a V-4Cr-4Ti alloy produced by present technology provides an indication of the status of some of the important impurity elements in the steels and gives an indication of the precision of the analytical chemistry methods. Several detrimental impurities are present that

would compromise the reduced-activation characteristics of these materials. Nevertheless, the results for the steels indicate that progress has been made in reducing the level of these impurities for two large heats of reduced-activation steel. Likewise, information exists that processes can be employed that will lower the impurity levels of vanadium alloys. There has probably never before been a requirement for a structural material to be processed to have specified impurity levels as low as those that will be required to meet the reduced-activation criteria. Even though the materials will not be needed for some time, it would appear that an effort should be mounted in the near future to determine the means to achieve the desired purity levels. As a start, an effort should be made to determine how low the impurity levels can be reduced in a large vanadium heat by selecting the starting materials with low impurity concentrations. In the case of the ferritic/martensitic steels, a study needs to be conducted to determine the origin of the various impurities to determine what starting materials will need to be purified to reach the desired goal. Such an effort should enlist support from industrial materials processors to determine techniques used in the past and how they 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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