EFFECT OF SMALL AMOUNTS OF RHENIUM AND OSMIUM ON MECHANICAL PROPERTIES OF A 9Cr-2W-0.25V-0.07Ta-0.1C STEEL—R. L. Klueh, D. J. Alexander, and M. A. Sokolov (Oak Ridge National Laboratory)

OBJECTIVE

The experiments in this work were meant to determine the effect of small amounts of rhenium and osmium on the mechanical properties of a 9Cr-2W-0.25V-0.07%a-0.1C steel. These effects could become important because tungsten is transmuted to rhenium and osmium when irradiated with neutrons in a fission or fusion reactor.

SUMMARY

The nuclear transmutation of tungsten to rhenium and osmium in a tungsten-containing steel irradiated in a fission or fusion reactor could change substantially the chemical composition of the steel. To determine the possible consequences of such changes on mechanical properties, tensile and Charpy impact properties were determined on five 9Cr-2W-0.25V-0.07Ta-0.1C steels that contained different amounts of rhenium, osmium, and tungsten. The mechanical properties changes due to these changes in composition were relatively minor. Observations were also made on the effect of carbon concentration. The effect of carbon on tensile behavior was relatively minor, but there was a large effect on Charpy properties. The steels showed relatively little effect of tempering temperature on the Charpy transition temperature, which was tentatively attributed to the silicon and/or manganese concentration.

PROGRESS AND STATUS

Introduction

Irradiation of the first wall and blanket structure of a fusion power plant by neutrons from the fusion reaction will induce the transmutation of constituent elements of the structural material, which will result in the replacement of the transmuted atom with one solid and one gas (helium or hydrogen) atom in the matrix of the material. The production of the solid radioactive transmutants are the impetus for the development of reduced-activation materials designed to ameliorate the radioactive waste disposal of components of a fusion power plant after the service lifetime [1]. Efforts have been made to determine the effect of the gaseous helium and hydrogen formed this way on the mechanical properties of the material. However, it has been assumed that the solid transmutants will have little effect on the mechanical properties, since only small amounts of such elements are expected to form and only small amounts of the elements of the structural material will be transmuted.

Recently, Greenwood and Garner [2] pointed out that significant amounts of transmutants can be produced when certain materials are irradiated in the fission reactors being used in the United States Department of Energy Fusion Reactor Materials Program. They concluded that the effect is most acute for certain elements irradiated in the High Flux Isotope Reactor (HFIR) [2], because of the thermal neutrons present in the mixed-spectrum of this reactor. The HFIR is the principle fission reactor used in the United States Fusion Materials Program. Elements of concern to Greenwood and Garner included molybdenum, tungsten, vanadium, and rhenium.

Tungsten is important because it has been used in the new reduced-activation steels as a replacement for molybdenum [1]. Steels with 9 % Cr-2% W are the leading reduced-activation steels under consideration (compositions are in wt. % unless otherwise stated). Figure 1, taken from Greenwood and Garner [2], shows that a considerable portion of the tungsten could be burned out of the steel during irradiation in the Fast Flux Test Facility (FFTF) and HFIR (two fission reactors), and the conceptual fusion power plant, STARFIRE. Tungsten transmutes to rhenium [2], and then much of the rhenium transmutes to osmium. This change in composition for a steel with 2% W could significantly affect the tungsten composition of the steel, and thus, it could conceivably affect the mechanical properties. The

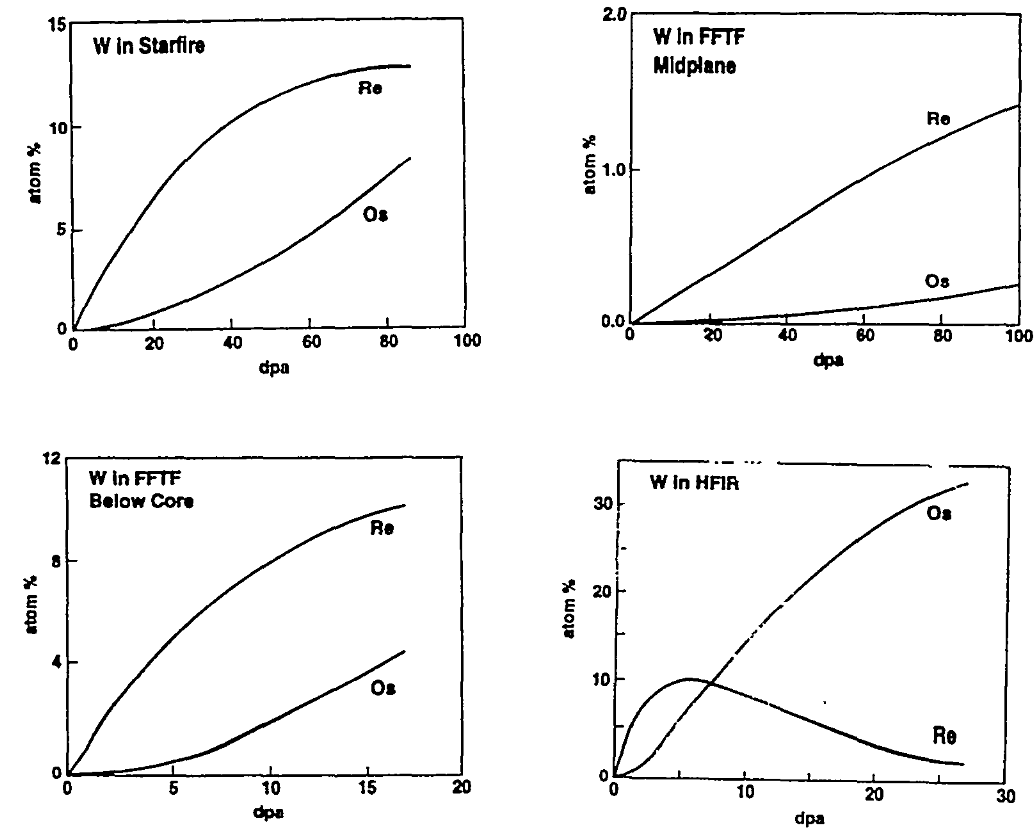

Figure 1. The transmutation of tungsten to rhenium and osmium in the conceptual fusion reactor Starfire, in two positions of the Fast Flux Test Facility (FFTF), and in the High Flux Isotope Reactor (HFIR). Taken from Greenwood and Garner [2].

largest effect occurs for HFIR (Fig. 1). Therefore, if the mechanical properties are affected by the change in composition, the properties could differ after irradiation in HFIR and irradiation in a fast reactor, such as FFTF [2], or irradiation in a fusion power plant.

Tensile and Charpy impact properties were measured on steels of nominal composition 9% Cr-2% W-0.25% V-0.07 % Ta-0.1% C (9Cr-2WVTa) with and without the addition of rhenium and osmium to determine if these elements have a significant effect on the mechanical properties. The 9Cr-2WVTa steel was used as the base because this is a reduced-activation steel with excellent properties in the normalized-and-tempered condition [3-5] and after irradiation [6].

Experimental Procedure

Small 450-g vacuum arc-melted heats of 9Cr-2WVTa steel and this composition with various levels of rhenium and osmium were made. Compositions of the experimental steels are given in Table 1.

Rhenium and osmium were added to the basic 9Cr-2WVTa composition with the objective of producing steels with 0.2 Re and 0.2 Os (ReOs-1), 0.1 Re and 0.6 Os (ReOs-2), and a third steel with the latter combination of rhenium and osmium, but with less tungsten to account for the tungsten that is transmuted during irradiation (ReOs-3). The first attempt to produce ReOs-3 resulted in an alloy with twice the desired carbon (ReOs-4), and although this high carbon was beyond that for the 9Cr-2WVTa,

Table 1. Chemical composition of steels (wt. %)

Element	9Cr-2WVTa	ReOs-1	ReOs-2	ReOs-3	ReOs-4
C	0.081	0.077	0.078	0.060	0.20
Mn	0.01	0.01	0.01	0.02	0.01
P	0.007	0.004	0.006	0.012	0.003
S	0.006	0.005	0.006	0.005	0.007
Si	0.09	0.20	0.10	0.06	0.01
Cr	8.96	8.76	8.76	8.72	8.76
V	0.20	0.21	0.20	0.23	0.21
Ta	0.06	0.06	0.08	0.06	0.07
W	2.17	2.29	2.26	1.47	1.58
Os		0.25	0.79	0.76	0.84
Re		0.20	0.07	0.11	0.14
Fe	Balance	Balance	Balance	Balance	Balance

the steel was still included in the tests as it provided an opportunity to examine the effect of carbon on the mechanical properties on this type of steel.

Chemical analyses of the heats of steel indicated that the rhenium and osmium values achieved were close to those desired; the Os of the high-Os steels was measured as ≈0.8% instead of the 0.6% desired (Table 1). Further, the objective for ReOs-3 was to reduce the 2% W in proportion to the amount of rhenium and osmium added, resulting in a 1.25% W steel. However, because the tungsten was high in the other steels (closer to 2.25% instead of 2%), ReOs-3 and ReOs-4 contained ≈1.5% W. The five steels will be referred to as 9Cr-2WVTa, 9Cr-2WVTa-0.2Re-0.2Os (ReOs-1), 9Cr-2WVTa-0.1Re-0.8Os (ReOs-2), 9Cr-1.5WVTa-0.1Re-0.8Os-0.1 (ReOs-3), and 9Cr-1.5WVTa-0.1Re-0.8Os-0.2C (ReOs-4).

The 9Cr-2WVTa was meant to be a reproduction of a larger heat (18 kg) of this composition produced for the original work to develop the reduced-activation steels and for which a range of data have been obtained (Table 2) [3-5]. The small 450-g heat of 9Cr-2WVTa was used as the control for this experiment in order to compare steels made by the same process. The nominal composition of the large heat for Cr, W, V, and Ta, the primary alloying elements, was achieved in the small heat. However, the silicon and manganese contents of the 18-kg heat were adjusted to ≈0.2% Si and ≈0.45% Mn [3], which are typical compositions for these elements when such steels are produced by a commercial vendor. The small heats for the present study were made from the individual elements and contained less manganese and, in most cases, less silicon: the manganese level was 0.01-0.02%, and silicon varied from 0.01 to 0.2% (Table 1). Carbon concentration was also different: in the small experimental steels, analyses indicated that it varied from 0.06% for ReOs-3 to ≈0.08% for the other three heats, compared to 0.11% in the large heat. The ReOs-4 was analyzed as containing 0.2% C. For the general discussion of the steels, they will be referred to as containing ≈0.1 and ≈0.2% C.

Half of each 12.7 x 25.4 x 127 mm ingot was hot rolled to a thickness of 6.4 mm and half to a thickness of 0.76 mm. Mechanical properties tests were made on normalized-and-tempered steel. The steels

Table 2. Chemical composition of different heats of 9Cr-2WVTa steels

Element	450-g Heat	18-kg Heat
C	0.081	0.11
Mn	0.01	0.44
P	0.007	0.015
S	0.006	0.008
Si	0.09	0.21
Cr	8.96	8.90
V	0.20	0.23
Ta	0.06	0.06
W	2.17	2.01
Fe	Balance	Balance

were austenitized for 0.5 h at 1050°C in a helium atmosphere, after which they were quickly cooled in flowing helium. Specimens were tested in two tempered conditions: 1 h at 700°C and 1 h at 750°C.

Tensile specimens 44.5-mm long with a reduced gage section of 20.3 x 1.52 x 0.76 mm were machined from the 0.76-mm sheet with gage lengths parallel to the rolling direction. The specimens were heat treated after machining. Tensile tests were conducted over the range room temperature to 600°C in vacuum on a 44-kN Instron universal testing machine at a nominal strain rate of $\approx 4 \times 10^{-4}$ s^{-1}.

One-third-size Charpy specimens 3.3 x 3.3 x 25.4 mm with a 0.51-mm-deep 30° V-notch and a 0.05- to 0.08-mm-root radius were machined from the normalized 6.4-mm plate along the rolling direction with the notch transverse to the rolling direction. Specimens were tempered after machining. Charpy tests were carried out in a pendulum-type impact machine specially modified to accommodate subsize specimens [7]. The absorbed energy values were fitted with a hyperbolic tangent function to permit the upper-shelf energy (USE) and ductile-brittle transition temperature (DBTT) to be evaluated. The DBTT was determined at the energy midway between the upper- and lower-shelf energies. Note that for these miniature specimens different DBTT and USE values are obtained than for full-size specimens. However, it has been shown that a low transition temperature for miniature specimens translates to a low value for full-size specimens [8,9]. A correlation likewise exists for the USE [8,9].

Results

Metallography and Microhardness

The steels were examined by optical microscopy. All of the microstructures were 100% tempered martensite. There was some variation in the estimated prior-austenite grain size, determined by comparing the microstructure with ASTM Grain Size charts. The three steels with 2% W and different amounts of Re and Os had similar grain sizes (Table 3), while the two steels with 1.5% W had different values: the 1.5% W steel containing ≈0.1% C (ReOs-3) had the largest grain size of all five steels, and the 1.5% W steel with 0.2% C (ReOs-4) had the smallest grain size of the steels.

Table 3. Microhardness and prior austenitized grain size of steels

Steel	Vickers Hardness (average of 5 readings)	Prior Austenite Grain Size mm (ASTM No.)
ReOs-0	253.8	0.016 (9)
ReOs-1	235.0	0.015 (9.25)
ReOs-2	254.6	0.016 (9)
ReOs-3	246.8	0.0205 (8.25)
ReOs-4	262.7	0.0095 (10.25)

Hardnesses showed a relatively small variation (Table 3), with the 2% W steel with 0.2% Re and 0.2% Os (ReOs-1) having the lowest hardness. There was less variation among the other steels. The 1.5% W steel containing 0.2% C had highest hardness.

Tensile Behavior

There was considerable variation in the strength (Figs. 2 and 4) and ductility (Figs. 3 and 5). The amount of variation for the yield stress (YS) [Figs. 2(a) and 4(a)] was greatest for the room temperature tests and least at 600°C. Variability was less for the ultimate tensile strength (UTS) of the different steels [Figs. 2(b) and 4(b)] than for the YS, but again, the variation for the UTS was greatest at the lowest temperatures. At most test temperatures below 600°C, the YS and UTS of the 9Cr-2WVTa-0.2Re-0.2Os steel (ReOs-1) was the smallest. The steels with 1.5% W, 0.8% Os and 0.1% Re with 0.1% C (ReOs-3) and 0.2% C (ReOs-4) were near the strongest of all the steels below 600°C. There was not much difference between the YS of those two steels below 600°C, but the UTS of the steel with 0.2% C (ReOs-4) was generally the highest of these two steels as well as of the other steels below 600°C.

The variation in ductility—uniform elongation [Figs. 3(a) and 5(a)] and total elongation [Figs. 3(b) and 5(b)]— was also quite wide for the steels tempered at both 700 (Fig. 3) and 750°C (Fig. 5). The relative ductilities of the steels were not always inverse to the strength, as might be expected. For example, the

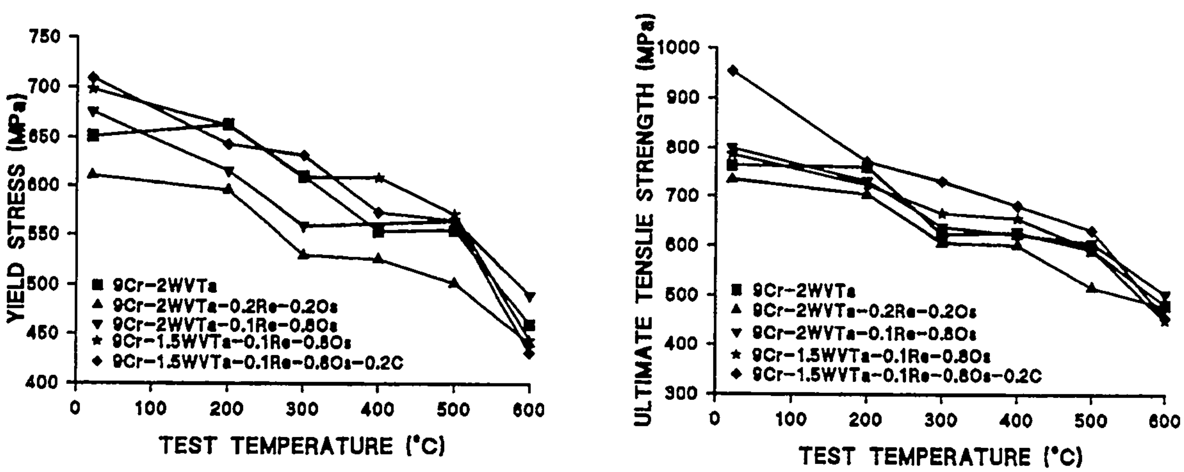

Figure 2. The yield stress and ultimate tensile strength of the 9Cr-2WVTa steels containing varying amounts of rhenium and osmium after normalizing and tempering 1 h at 700°C.

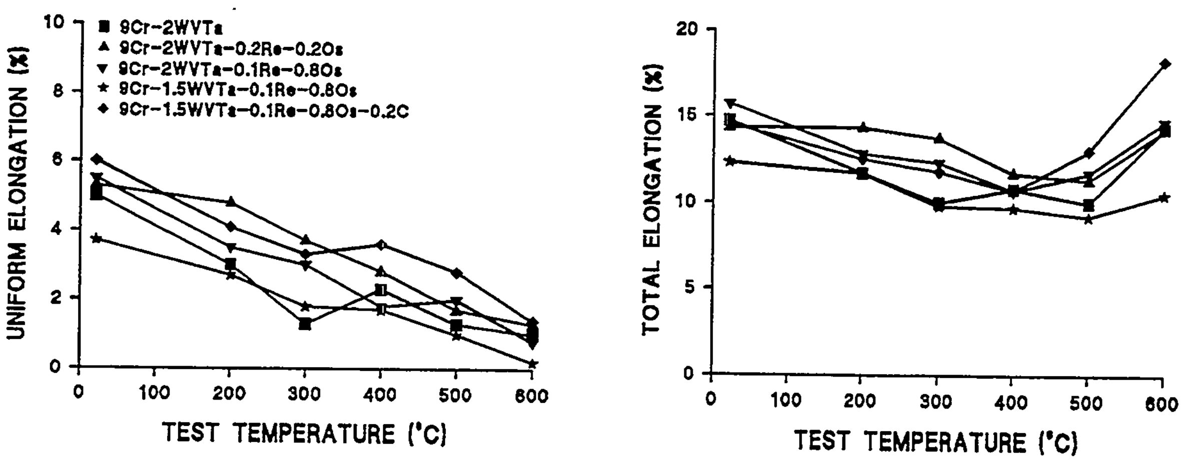

Figure 3. The uniform and total elongation of the 9Cr-2WVTa steels containing varying amounts of rhenium and osmium after normalizing and tempering 1 h at 700°C.

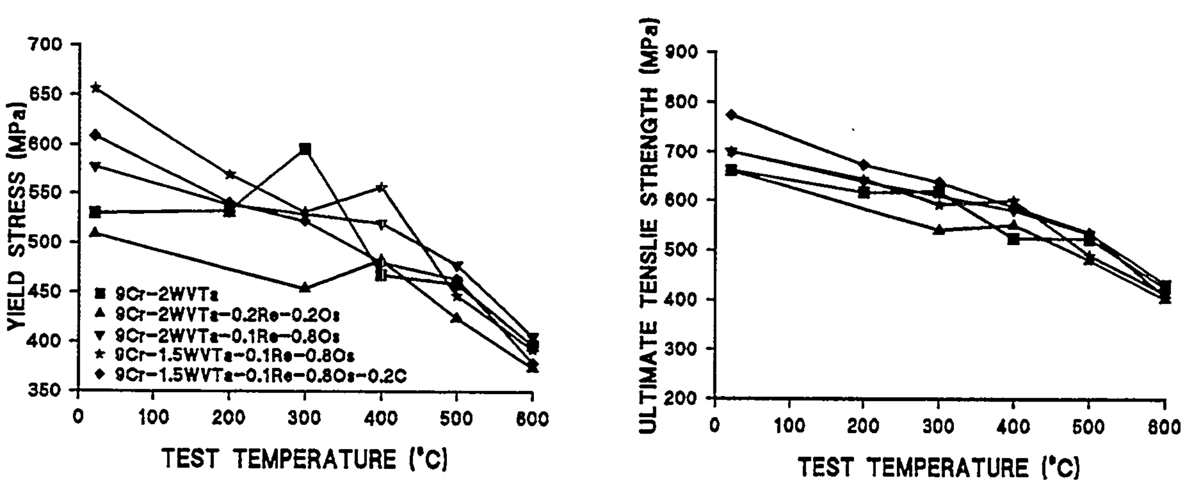

Figure 4. The yield stress and ultimate tensile strength of the 9Cr-2WVTa steels containing varying amounts of rhenium and osmium after normalizing and tempering 1 h at 750°C.

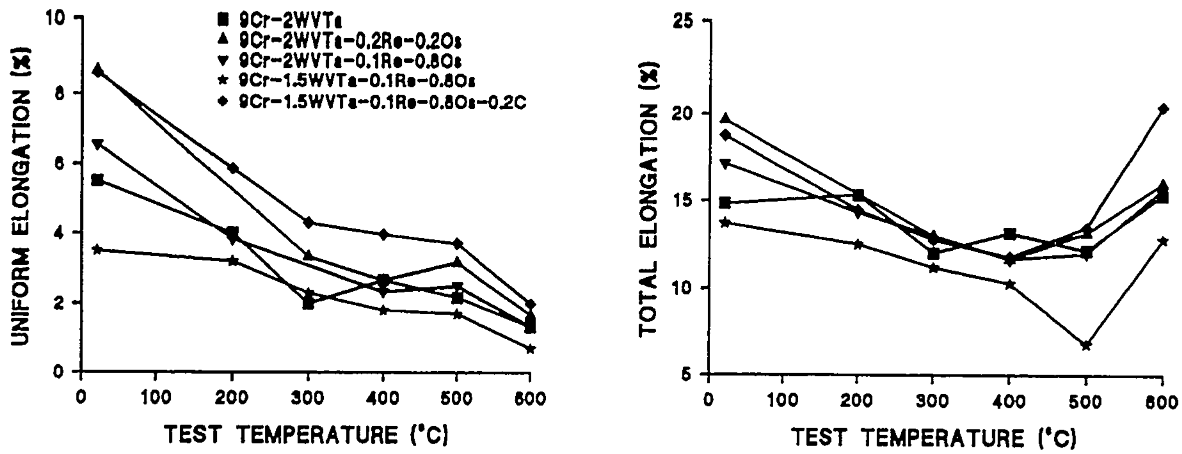

Figure 5. The uniform and total elongation of the 9Cr-2WVTa steels containing varying amounts of rhenium and osmium after normalizing and tempering 1 h at 750°C.

1.5% W steel with 0.1% C (ReOs-3) generally had the lowest uniform and total elongation, while the steel with 0.2% C (ReOs-4) often had the highest ductility, even though these were two of the strongest steels. The total elongation of all the steels increased with temperature above ≈400°C, with the largest change occurring for the steel with 0.2% C.

Yield stress results for the small 450-g heat and the large 18-kg heat of 9Cr-2WVTa steel at room temperature and 600°C are compared in Table 4. The large heat was substantially stronger than the small heat.

Table 4. A comparison of properties of the 450-g and 18-kg heats of 9Cr-2WVTa steel

Heat Size	Tempering Temperature (°C)	YS (MPa)		UTS, MPa		Charpy	
		RT	600°C	RT	600°C	TT(°C)	USE (J)
450-g	1 h at 700°C	651	460	764	481	-103	13.7
	1 h at 750°C	530	397	661	425	-120	15.4
18-kg	1 h at 700°C	823	651	942	696	-43	7.5
	1 h at 750°C	645	489	774	526	-88	11.2

Charpy Impact Properties

The transition temperature (DBTT) and upper-shelf energy (USE) for the steels tempered at 700 and 750°C are given in Table 5 and Fig. 6.

As expected, the USE of all five steels is higher after the 750°C temper than after the 700°C temper [Fig. 6(b)]. For the four steels with ≈0.1% C, there was relatively little difference in DBTT, all of which were lower than for the steel with 0.2% C (Figs. 7 and 8), which was also expected. The lower DBTT after the 750°C temper than after the 700°C temper observed for the 9Cr-2WVTa and the 9Cr-2WVTa-0.1Re-0.8Os-0.1C steels was the expected behavior, since the strength decreases with tempering temperature. What was not expected was the relatively small difference in DBTT for the other three steels after the different tempering treatments. These steels showed essentially no effect of tempering temperature on the DBTT (Table 2 and Fig. 6). For some of the steels (ReOs-3 and ReOs-4), the measured values after tempering at 700°C were lower than after tempering at 750°C.

Table 5. Charpy impact properties of steels

Steel	Tempering Temperature (°C)	Transition Temperature (°C)	Upper-Shelf Energy (J)
9Cr-2WVTa	1 h at 700°C 1 h at 750°C	-103 -120	13.7 15.4
9Cr-2WVTa-0.2Re-0.2Os-0.1C	1 h at 700°C 1 h at 750°C	-103 -105	12.7 14.2
9Cr-2WVTa-0.1Re-0.8Os-0.1C	1 h at 700°C 1 h at 750°C	-102 -133	13.3 14.3
9Cr-1.5WVTa-0.1Re-0.8Os-0.1C	1 h at 700°C 1 h at 750°C	-114 -113	12.6 13.8
9Cr-1.5WVTa-0.1Re-0.8Os-0.2C	1 h at 700°C 1 h at 750°C	-84 -78	8.6 9.4

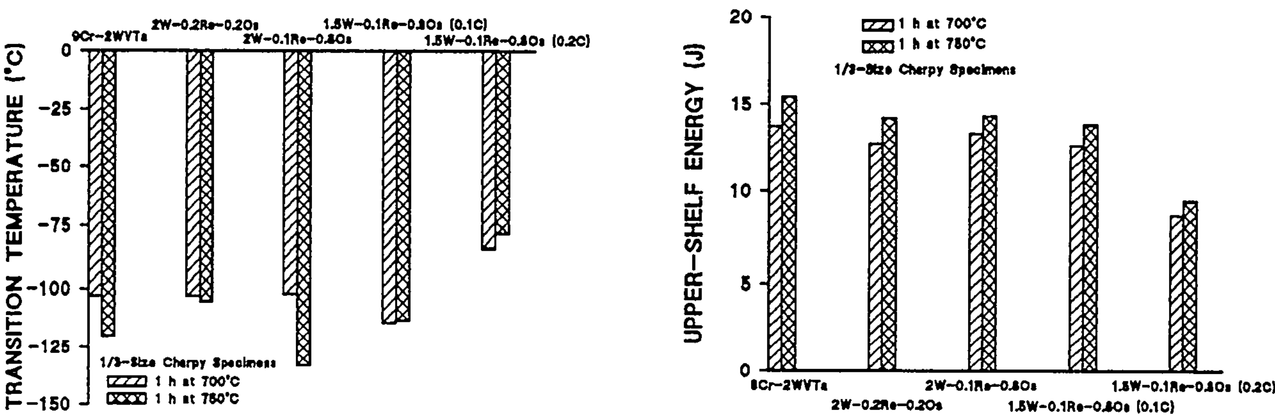

Figure 6. The Charpy transition temperature and upper-shelf energy of the 9Cr-2WVTa steels containing varying amounts of rhenium and osmium after normalizing and tempering 1 h at 700°C and 1 h at 750°C.

The 450-g heat of 9Cr-2WVTa steel used in this experiment had a lower DBTT and a relatively smaller difference after tempering at 700 and 750°C than the 18-kg heat tested previously (Table 4). The DBTTs of the large heat after tempering at 700 and 750°C were -43 and -88°C, respectively, compared to -103 and -120°C, respectively, for the small heat. The small heat also had a significantly higher USE in each case (Table 4).

Discussion

During irradiation in HFIR, tungsten transmutes to rhenium, which subsequently transmutes to osmium (Fig. 1). The objective of these experiments was to determine the possible effect rhenium and osmium and the substitution of rhenium and osmium for tungsten could have on the mechanical properties of the 9Cr-2WVTa steel. Such compositional changes occur when tungsten is transmuted to rhenium and osmium when the steel is irradiated in a reactor, such as HFIR (and in a future fusion reactor). After tungsten is irradiated to ≈25 dpa in HFIR, ≈32% will be transmuted to ≈29% Os and ≈3 % Re. For the 2% W in the 9Cr-2WVTa steel, this means ≈0.58% Os and ≈0.06% Re form, thus reducing the tungsten composition by about this amount. Steels containing 0.6% Os and 0.1% Re were proposed. The actual alloys produced contained somewhat more osmium. In addition, the tungsten was somewhat higher than desired (Table 1). Of course, the mechanical properties after irradiation will also be affected by radiation damage. Nevertheless, the results provide information on the effect of osmium and rhenium on the properties of the 9Cr-2WVTa steel.

The objective of testing the two Re-Os alloys with 2% W (9Cr-2WVTa-0.2Re-0.2Os and 9Cr-2WVTa-0.1Re-0.8Os) was to determine the effect of the rhenium and osmium on the steel (Figs. 2-5). The steel containing 0.2% Re and 0.2% Os was generally the weakest of the five steels. The 9Cr-2WVTa and 9Cr-2WVTa-0.1Re-0.8Os steels had similar strengths and ductility that were more in line with those of the two 1.5% W steels.

The Charpy properties of the three 2% W steels were quite similar (Fig. 6), with the only unusual observation being that there was essentially no difference in the DBTT of the 0.2Re-0.2Os steel after the 700 and 750°C tempers. For all three steels, the USE after the 750°C temper was greater than after the 700°C temper.

When the tungsten concentration was reduced from ≈2.2% to ≈1.5% with no change in carbon, the 1.5% W steel was generally the strongest of these steels—at least below 600°C. The stronger 1.5% W steel with ≈0.1% C generally had the lowest uniform and total elongation of the five steels. However, the Charpy impact properties of the steel with 1.5% W were similar to those for the 2% W steels. Although the 1.5% W steel with ≈0.1% C had a similar strength to the 1.5% W steel with ≈0.2% C, the steel with 0.2% C was close to having the highest ductility of the five steels. A possible explanation is that the higher carbon content means that not all carbides are dissolved during austenitization, which could cause the smaller prior austenite grain size that can probably affect ductility. Whether undissolved carbides remained during austenitization needs to be verified by transmission electron microcopy (TEM).

Charpy properties of the 1.5% W steels reflected the carbon content. The steel with the highest carbon had the highest DBTT and the lowest USE of all five steels (Fig. 6); this occurred despite the high-carbon steel having the smallest prior austenite grain size and not necessarily always being the strongest of the steels. This was probably due to larger carbides in this steel, although that needs to be established by TEM.

Tempering temperature apparently had no effect on the DBTT of either of the 1.5% W steels, just as there was no effect on the 0.2Re-0.2Os steel, although for the 1.5% W steels, the DBTT was slightly higher after the 750°C temper than the 700°C temper, which is contrary to expectations. The reason for this lack of effect of tempering temperature is not known. In all cases, these steels did have the expected effect of tempering temperature on USE (i.e., the USE was always higher for the higher tempering temperature).

Basically, the results of these tests give little indication that the tensile and Charpy properties will be affected significantly by the amounts of rhenium and osmium estimated to form in a 9Cr-2WVTa steel irradiated to ≈25 dpa in HFIR. Likewise, the reduction of tungsten that would accompany the increase in rhenium and osmium also appears to have little effect—at least in the presence of the additional Re and Os. Under the influence of irradiation at temperatures below ≈400°C where irradiation hardening is expected, the effect of the compositional changes would probably be of even less significance.

The largest effect of composition on mechanical properties involved the apparent effect of carbon concentration on the Charpy properties (Figs. 6-8). Although there was relatively little difference in the strength of the 1.5% W steels with ≈0.1 and 0.2% C, there was a substantial difference in the Charpy behavior. Carbon can play a role on the properties through its effect on the strength and the carbide morphology. Since there was relatively little difference in strength for the two steels, the difference in the carbides in the two steels must be the cause, especially since the steel with 0.2%C had a much smaller prior austenite grain size than the steel with ≈0.1% C. TEM is required to verify these suggestions.

The difference in properties noted between the small (450-g) heat of steel (0.08% C) produced for this experiment and the larger (18-kg) heat (0.11% C) previously studied [3-6] (Table 4) may also be at least partially due to the difference in carbon concentration. As Table 4 indicates, the large heat is stronger and has a lower DBTT and higher USE. Without further experiments, it is not possible to determine if the difference in properties for these two steels is attributable to the carbon, but based on the small difference in the strength of the 1.5% W steels with ≈0.1% C (measured as 0.06%) and ≈0.2% C, there would appear to be other reasons for the differences. That is, the DBTTs of the 1.5% W steels with ≈0.1 and ≈0.2% C after tempering at 700°C were -114 and -84°C, respectively, and after tempering at 750°C they were -113 and 78°C, respectively. This compares with the 450-g heat of 9Cr-2WVTa steel that had DBTTs of -103 and -120°C after tempering at 700 and 750°C, respectively, compared to the DBTTs of the 18-kg heat that were -43 and -88°C, respectively. The relative difference for the latter steels seems to be somewhat larger than for that attributed to carbon for the 1.5% W steels, especially when it is considered that there is much less difference in carbon for the 450-g and 18-kg heats of 9Cr-2WVTa steels.

As pointed out in the previous section, the 9Cr-2WVTa steels also contain different amounts of manganese and silicon; both manganese and silicon are thought to strengthen by solid solution hardening [10]. If this is the case here, then the increased strength and reduced Charpy properties of the large heat might also be partially attributed to the higher silicon and manganese in the 18-kg heat, which contains about 40 times more manganese (0.44 vs. 0.01) and twice as much silicon (0.21 vs. 0.09). Again, further work is required to make a clear determination on the cause for the differences that have been observed. Should manganese be the cause, that could be important because fairly large amounts of manganese are expected to form in such a steel by transmutation in a magnetically confined fusion reactor [11].

When reasons for the observations of little or no change in DBTT with tempering temperature for the steels of this experiment (Table 5) compared to the previous experiment are considered, manganese and silicon concentration differences for the heats of this experiment and heats used previously appear to be the only possible reasons that can be cited. Further work is required to elucidate a relationship to the composition. If there is such a relationship, it might be possible to exploit it in the development of such steels.

Summary and Conclusions

Tensile and Charpy impact properties were determined for the following five steels: 9Cr-2WVTa, 9Cr-2WVTa with additions of ≈0.2% Re and ≈0.2% Os, 9Cr-2WVTa with additions of≈0.1%Re and ≈0.8% Os, one 9Cr-1.5WVTa with additions of ≈0.1Re and ≈0.8Os, and a 9Cr-1.5WVTa with additions of ≈0.1Re, ≈0.8Os, and ≈0.2% C. All but the last steel contained ≈0.1% C. There were only minor variations in the tensile properties due to the addition of the rhenium and osmium to the 9Cr-2WVTa or the simultaneously reduction of the tungsten content to 1.5% and the addition of rhenium and osmium. The change in carbon concentration had the major effect on the Charpy impact properties by causing an increase in the transition temperature and a reduction in the USE. For most of the steels, there was little difference in the transition temperature after tempering at 700 and 750°C, much less than for a heat of 9Cr-2WVTa tested previously. A smaller amount of silicon and manganese in the steels used in the present experiments may be the cause for these differences, although that still needs to be verified.

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