OXIDE DISPERSION-STRENGTHENED STEELS: A COMPARISON OF EXPERIMENTAL AND COMMERCIAL STEELS—R. L. Klueh, J. P. Shingledecker, R. W. Swindeman, and D. T. Hoelzer (Oak Ridge National Laboratory)

OBJECTIVE

This work is carried out to develop an understanding of the mechanical properties and microstructures of experimental and commercial oxide dispersion-strengthened steels that are considered possible candidate materials for fusion applications.

SUMMARY

Oxide dispersion-strengthened (ODS) steels are being developed and investigated for nuclear fission and nuclear fusion applications in Japan, Europe, and the United States. In addition, commercial ODS products are available and have been used in niche applications. Microstructural and mechanical properties studies have been conducted at Oak Ridge National Laboratory and elsewhere on various commercial and experimental ODS steels. Tensile and creep properties have been obtained and collected from literature and commercial sources. These data are compared to show the differences and similarities of different ODS steels, and observations are explained in terms of the microstructures of the steels.

PROGRESS AND STATUS

Introduction

If the conventional high-chromium ferritic/martensitic steels, such as modified 9Cr-1Mo and Sandvik HT9, or the reduced-activation steels, such as F82H, ORNL 9Cr-2WVTa, EUROFER, and JLF-1, were used for a fusion power plant first wall and blanket structure, the upper operating temperature would be limited to 550-600°C.

One way suggested to increase this limit to higher temperatures and maintain the advantages inherent in ferritic/martensitic steels (i.e. high thermal conductivity and low swelling) is to use oxide dispersion-strengthened (ODS) steels. Elevated temperature strength in these steels is obtained through microstructures that contain a high density of small Y_2O_3 and/or TiO_2 particles dispersed in a ferrite matrix.

ODS steels are being developed and investigated for nuclear fission and fusion applications in Japan [1,2], Europe [3,4], and the United States [5]. Some commercial ODS products are available and are being used in limited quantities; commercial alloys include MA 956 and PM 2000 from Special Metals Corporation in the United States and Metallwerk Plansee GmbH in Germany, respectively. Microstructural and mechanical properties studies have been conducted at Oak Ridge National Laboratory (ORNL) on various commercial and experimental ODS steels. Results obtained at ORNL and elsewhere will be presented, compared, and discussed to show the differences and similarities of the different steels.

In previous work at ORNL, the microstructures and tensile and creep properties of two experimental ODS steels with nominal compositions of Fe-12Cr-0.25Y_2O_3 (designated 12Y1), manufactured by Sumitomo Metal Industries Ltd., and Fe-12Cr-2.5W-0.4Ti-0.25Y_2O_3 (designated 12YWT), manufactured by Kobe Special Tube Co. Ltd., were investigated [6]. Optical microscopy, transmission electron microscopy (TEM) [7], and atom probe field ion microscopy [8,9] studies indicated that the 12Y1 microstructures were very different from those of 12YWT (Fig. 1). For 12Y1 [Fig. 1(a)], particles were estimated to be 10-40 nm in diameter at a number density of 10^{20}-10^{21} m^{-3}; the dislocation density was estimated at ≈10^{15} m^{-2}. Diffraction studies indicated the particles were essentially pure, crystalline Y_2O_3. For 12YWT [Fig. 1(b)], dislocation density, particle size, and particle number density were estimated at 10^{15}-10^{16} m^{-2}, 3-5 nm diameter, and 1-2 x 10^{23} m^{-3}, respectively. For this alloy, three-dimensional atom probe analysis revealed

compositionally distinct nano-sized clusters enriched in Y, Ti, and O, slightly enriched in Cr, and slightly depleted in Fe and W.

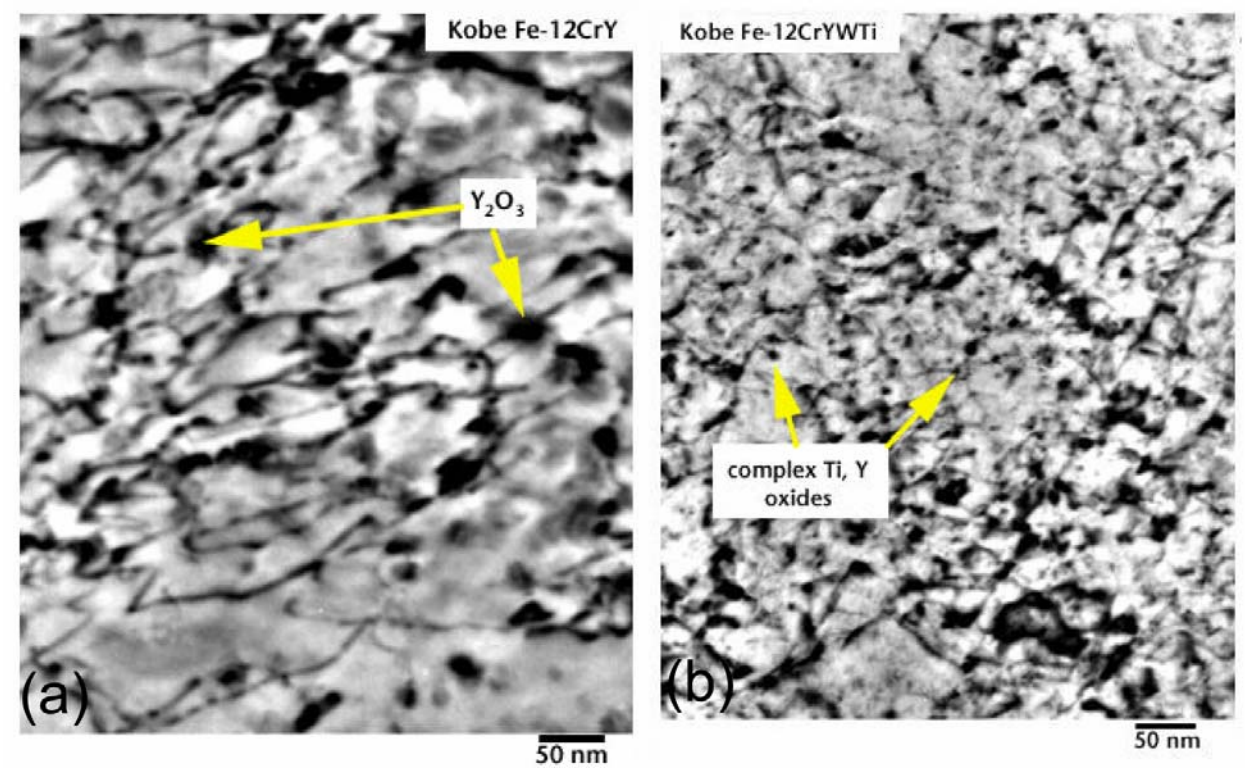

Fig. 1. Transmission electron micrographs of experimental ODS steels (a) 12Y1 (Fe-12Cr-0.25Y_2O_3) and (b) 12YWT (Fe-12Cr-2.5W-0.4Ti-0.25Y_2O_3).

These differences in microstructure were obviously the origin of large differences in tensile and creep properties [6]. Room-temperature yield stress of 12YWT was about 20% greater than 12Y1, but at 900ºC, the 12YWT was about 2.5 times as strong. This large difference at the highest temperatures translated into a much higher creep strength for the 12YWT [6].

Mechanical properties of three other ODS steels have been determined: MA 956, MA957, and PM 2000. In this paper, tensile and creep properties of these commercial steels will be presented and compared with those of the experimental steels. The implications of the mechanical property results from the commercial steels will be discussed in terms of microstructural observations on the steels.

Experimental Procedure

Table 1 gives chemical compositions of the ODS steels to be discussed. Experimental alloys 12Y1 and 12YWT were produced in Japan by Sumitomo Industries and Kobe Special Tube, respectively. PM 2000 is a commercial product of Metallwerk Plansee GMbH of Germany. The MA 957 was manufactured by INCO Metals, and MA956 is a product of Special Metals Corporation.

Tensile and creep tests were conducted at ORNL on the 12Y1 and 12YWT [6]. Creep tests were conducted at ORNL on MA 957[10] and PM 2000 [11]. Creep and tensile data for the MA 956 [12] and MA 957 [13,14] and tensile data for PM 2000 [15] were taken from the literature or vendor data sheets. All tests are for specimens taken in the worked direction—the high-strength direction.

Different product forms for the different alloys were used in the creep and tensile tests at ORNL. Specimens of 12Y1 and 12YWT were from 2-mm sheet; the MA 957 specimens were from a tube 65-mm OD, 25-mm wall thickness, and the PM 2000 specimens were from 0.08- and 0.13-mm sheet.

Table 1. Chemical compositions of ODS steels

Element*	12Y1	12YWT	MA 956	MA 957	PM 2000
C	0.045	0.050	0.03	0.030	0.01
Mn	0.04	0.60	0.06	0.09	0.11
P	<0.001	0.019	0.008	0.007	<0.002
S	0.002	0.005	0.005	0.006	0.0021
Si	0.03	0.18	0.05	0.04	0.04
Ni	0.24	0.27	0.11	0.13	0.01
Cr	12.85	12.58	21.7	13.7	18.92
Mo	0.03	0.02	<0.05	0.30	0.01
V	0.007	0.002			
Ti	0.003	0.35	0.33	0.98	0.45
Co	0.005	0.02	0.03		0.01
Cu	0.01	0.02			0.01
Al	0.007		5.77	0.03	5.10
B	0.004			0.0009	<0.0003
W	<0.01	2.44			0.04
Zr	0.003				<0.01
N	0.017	0. 0.014	0.029	0.044	0.0028
O	0.15	0.16	0.21	0.21	0.25
Y	0.20	0.16	0.38	0.28	0.37

*Balance iron

Results

Tensile behavior

The behavior of the yield stress and ultimate tensile strength of the experimental 12Y1 and 12YWT steels over the range room temperature to 900ºC is shown in Fig. 2, along with data for a non-ODS reduced activation steel, ORNL 9Cr-2WVTa [16]. The obvious strength advantage of 12YWT is evident in the figure.

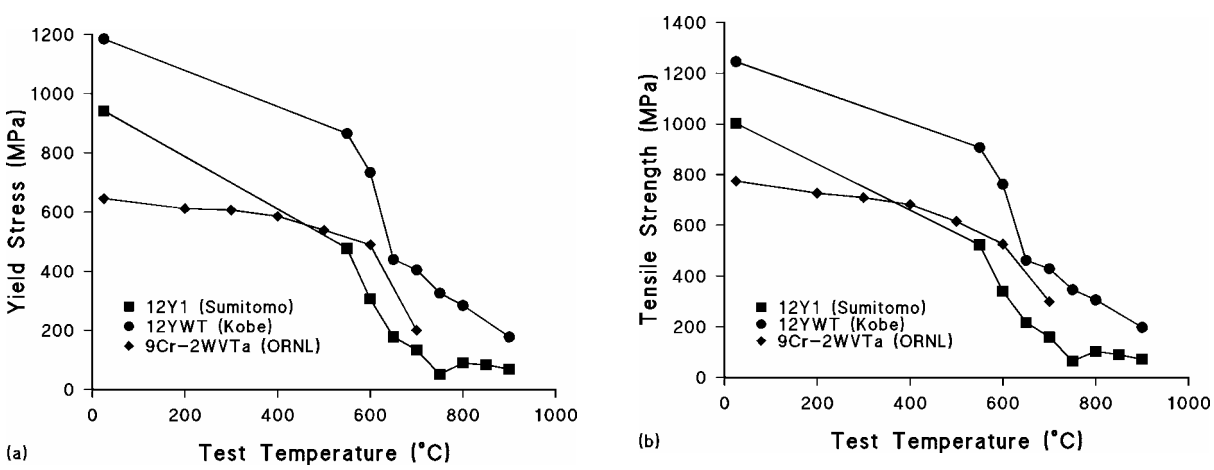

Fig. 2. (a) Yield stress and (b) ultimate tensile strength as a function of test temperature for the experimental ODS steels 12Y1 and 12YWT compared to the non-ODS reduced-activation steel ORNL 9Cr-2WVTa.

Of the commercial ODS steels, the MA 957 had the highest yield stress and ultimate tensile strength with the MA 956 having the lowest values (Fig. 3). In fact, up to 700ºC, the yield stress of the MA956 was less

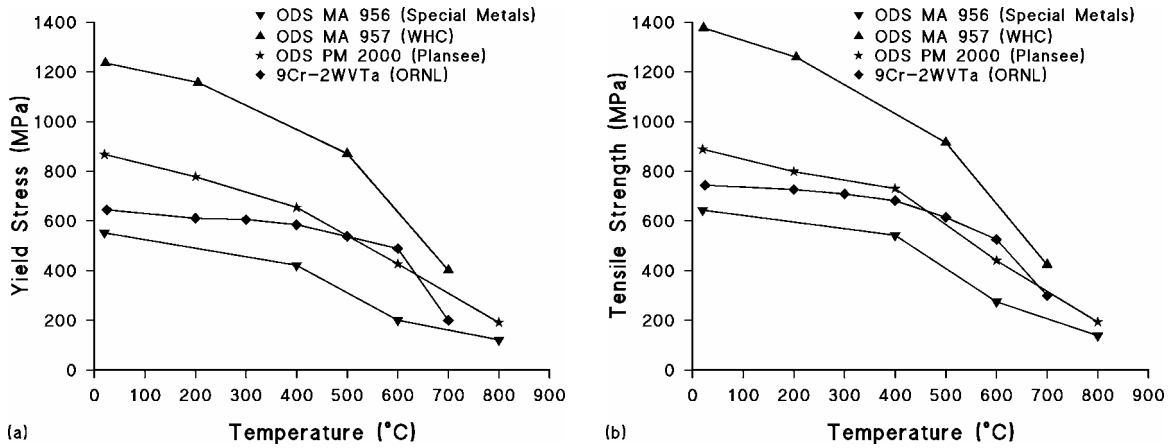

Fig. 3. A comparison of the (a) yield stress and (b) ultimate tensile strength as a function of test temperature for the three commercial ODS steels and the non-ODS reduced-activation steel ORNL 9Cr-2WVTa.

than that of 9Cr-2WVTa— a non-ODS martensitic steel. The strength of PM 2000 fell between the values for the MA 957 and MA 956.

When the commercial steels are compared with the experimental steels (Fig. 4), the MA957 and 12YWT, the strongest of the commercial and experimental steels, respectively, have similar strengths. The 12Y1 is similar to PM 2000 at low temperatures, but it then approaches the values for MA 956 at the higher temperatures. All steels appear to approach a common low strength value as the temperature is increased to 900ºC.

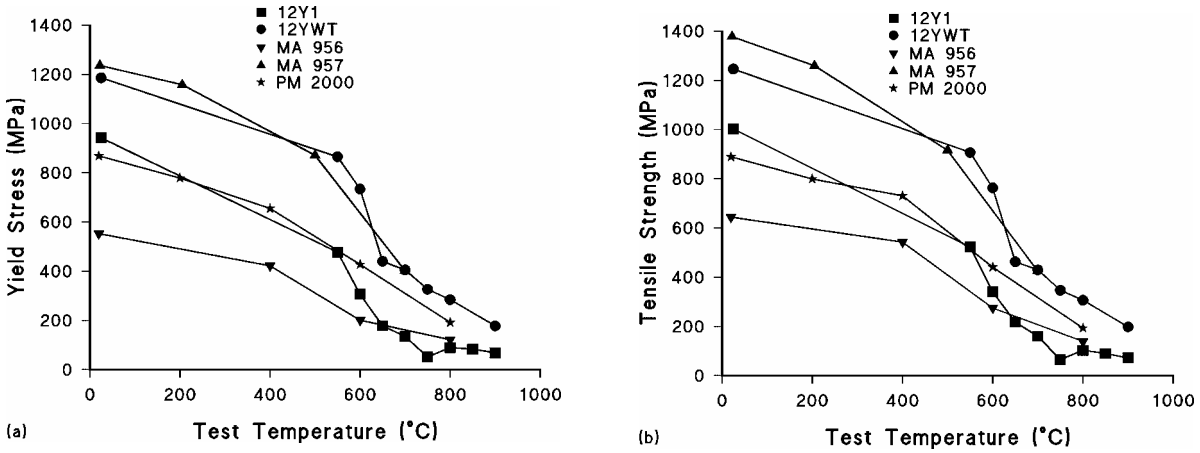

Fig. 4. A comparison of (a) yield stress and (b) ultimate tensile strength as a function of test temperature of the experimental and commercial ODS steels

Minor differences were observed in the total elongations of the experimental and commercial steels (Fig. 5). The 12Y1 and 12YWT had the lowest values at room temperature, and 12Y1 had the highest values.

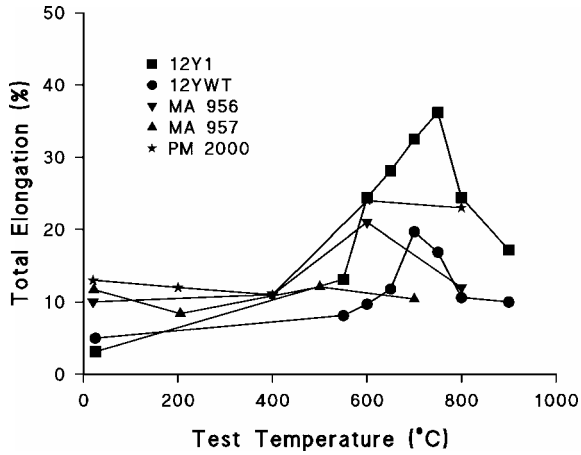

Fig. 5. A comparison of the total elongation as a function of test temperature of the experimental and commercial ODS steels between 600 to 800ºC. Otherwise, the elongations of the commercial steels and the 12YWT were comparable.

Creep-rupture Behavior

Creep-rupture behavior was compared using a Larson-Miller Parameter (LMP) with a constant of 25, which was used for MA 956 and MA 957 previously [5]. A comparison of the experimental steels (Fig. 6) showed the superiority of the 12YWT to the 12Y1, in agreement with the yield stress behavior. Also shown on Fig. 6 are data for a conventional 9Cr-WMoVNb steel (a commercial steel designated NF616 or Grade 92). The creep behavior of the 12Y1 was similar to that of the conventional steel.

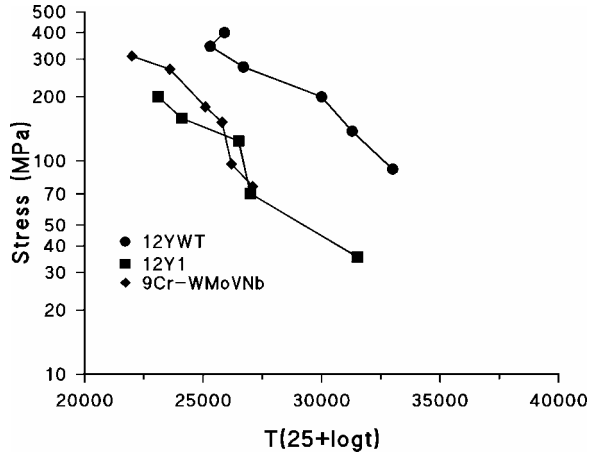

Fig. 6. Rupture stress vs. Larson-Miller parameter for the experimental ODS steels 12Y1 and 12YWT and a conventional non-ODS steel NF616 (9Cr-WmoVNb).

Comparison of the creep of the experimental ODS steels with the strongest (highest yield stress) commercial ODS steel MA 957, which was creep tested in this investigation, and MA 956, the weakest commercial ODS steel, indicates that the MA 956 and 12YWT have similar properties at high LMP (Fig. 7). Over the range where the LMP data for MA 957 and MA 956 overlap, it appears that they approach similar values, indicating an eventual convergence of the data for MA 957 and 12YWT, although that still needs to be demonstrated experimentally.

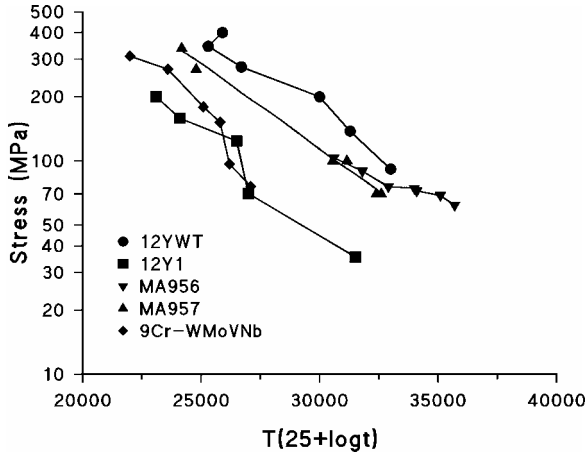

Fig. 7. Rupture stress vs. Larson-Miller parameter for the commercial steels MA 956 and MA957 compared to the experimental ODS steels and the non-ODS steel.

A comparison of the LMP values for PM 2000 with the experimental steels (Fig. 8) indicates that the creep-rupture strength of this steel is as good as that of 12YWT for the conditions tested. For the different sheet thicknesses, the thinner 0.08-mm sheet had better properties than the 0.13-mm sheet.

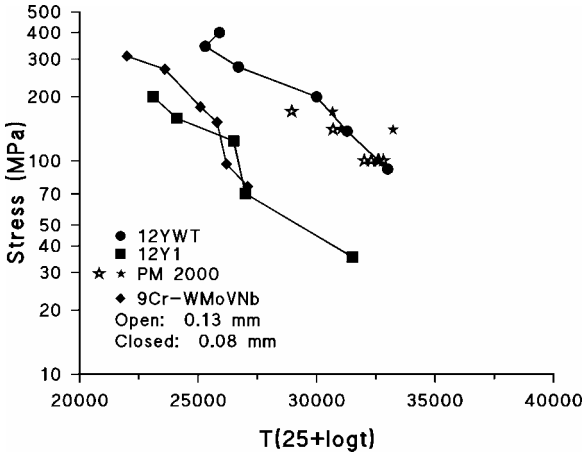

Fig. 8. Rupture stress vs. Larson-Miller parameter for the commercial steels PM 2000 compared to the experimental ODS steels and the non-ODS steel.

When the LMP for all the materials are compared (Fig. 9), it appears that all of the ODS steels except 12Y1 approach similar values for high LMP (low-stress and/or high temperature tests). This occurs despite the quite different yield stress values for the different steels.

Microstructures

Optical microscopy, TEM, and atom probe studies have been conducted on the 12Y1, 12YWT [7-9], and MA957 [17] with particular emphasis on the fine oxide particles that are responsible for the strength of the ODS alloys. In this study, microstructural features that are not part of the oxide dispersion and may therefore limit the strength and ductility have been conducted on the MA 957, and these will be discussed here.

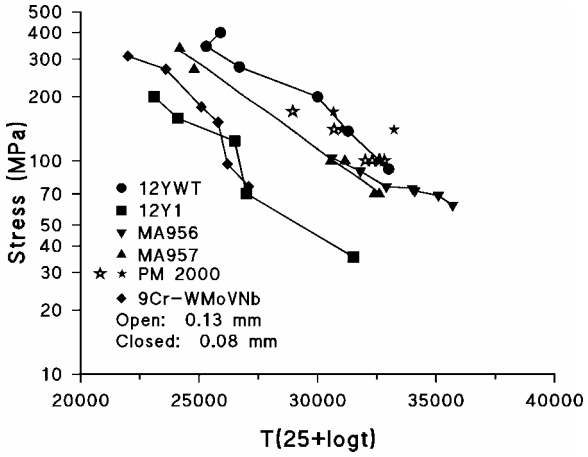

Fig. 9. Rupture stress vs. Larson-Miller parameter for all the commercial and experimental ODS steels and the non-ODS steel.

Optical microscopy of the MA 957 revealed large second-phase particles that often appeared as relatively large "stringers" (Fig. 10) aligned along the rolling direction. A failed creep-rupture specimen was examined, and the failure appeared to be associated with the stringer material (Fig. 11). That is, it appeared that cavities formed in conjunction with the particles of stringer material and other extraneous particles not in the stringers, and these cavities linked up perpendicular to the worked direction (parallel to the eventual fracture surface).

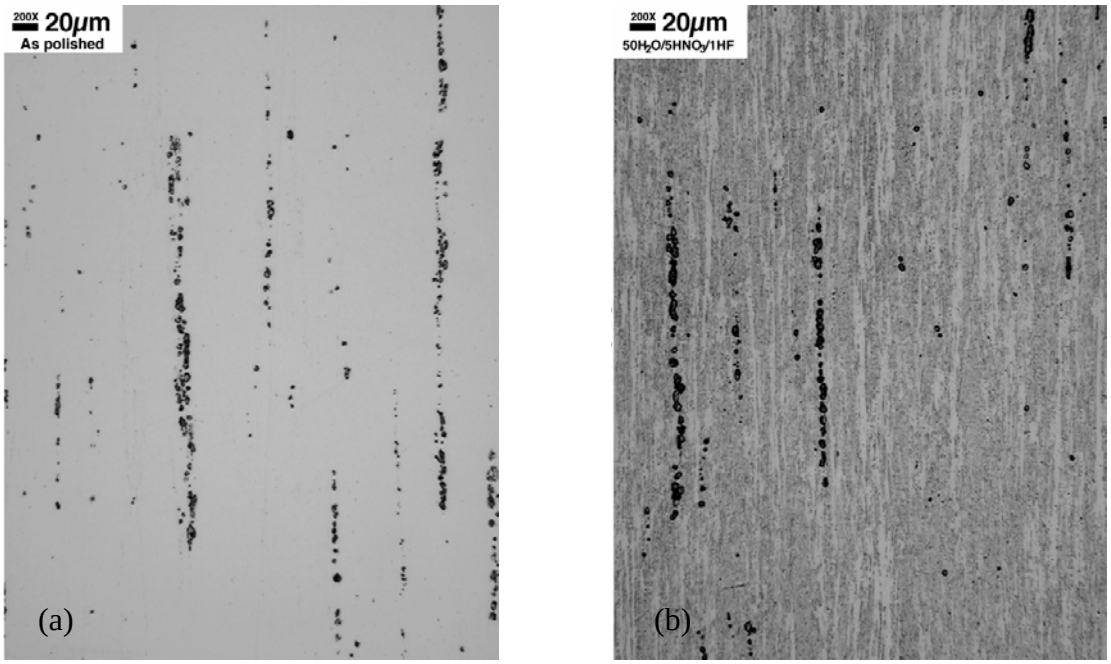

Fig. 10. Optical microstructure of MA 957 ODS steel (a) as polished and (b) etched.

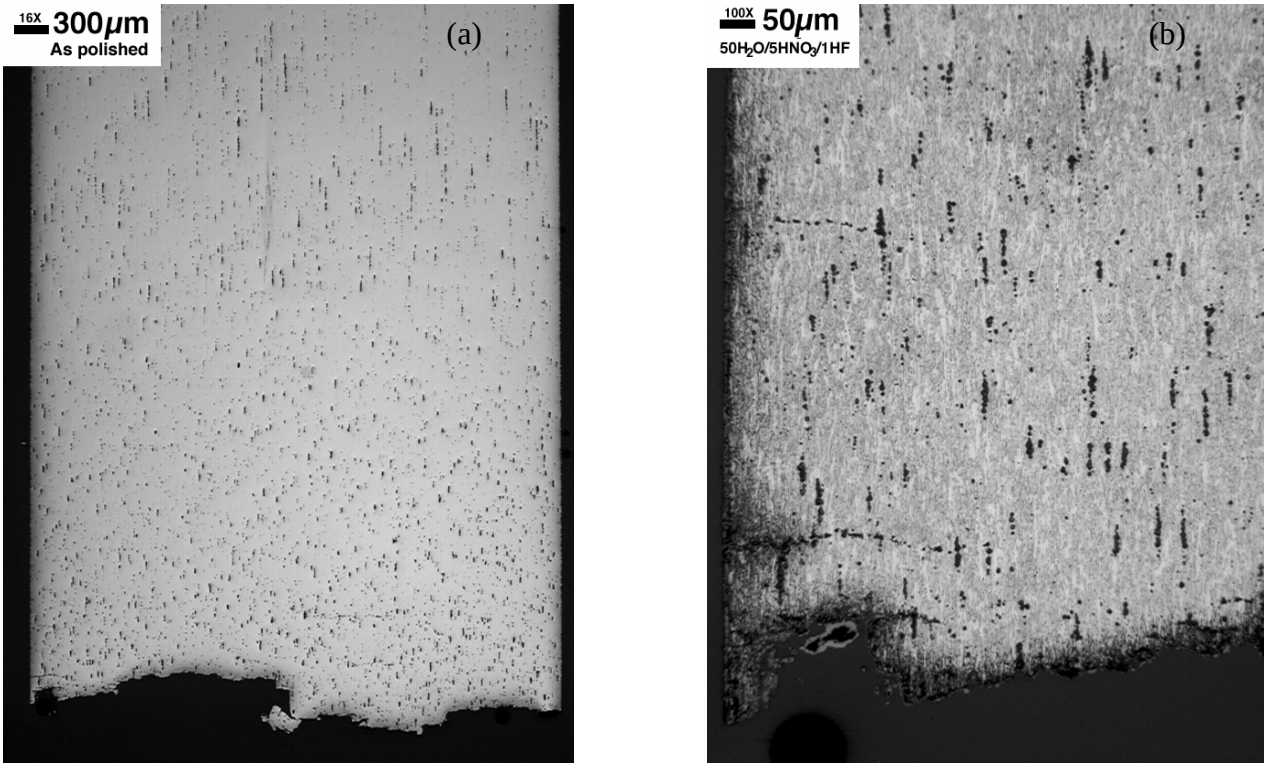

Fig. 11. Optical micrographs of fractured creep-rupture specimen of MA 957 tested at 100 MPa at 900ºC that ruptured in 36.7 h at two different magnifications and (a) unetched and (b) etched.

To determine the nature of the particles in the stringers and other extraneous particles (not the fine distribution of oxide particles that strengthen ODS steels), the gage section of the creep-rupture specimen shown in Fig. 11 was examined by scanning electron microscopy (SEM) with a field emission gun (FEG) using backscattered and secondary electrons (Fig. 12). Such large particles are expected to be deleterious to the mechanical properties.

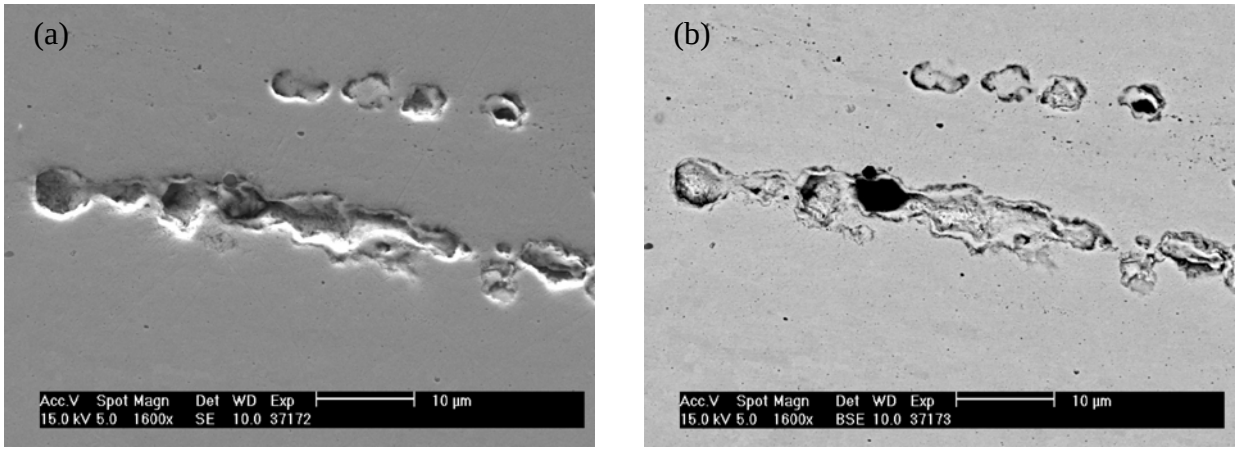

Fig. 12. (a) Secondary electron image and (b) backscattered electron image (compositional contrast) (right) of “stringers” in gage length of MA 957 creep specimen.

As expected, the primary elements identified in the matrix of the MA 957 were Fe, Cr, and Ti. The extraneous particles in and out of the stringers were rich in several different elements. Many particles in the stringers were carbon rich (Fig. 13); the presence of carbon is probably the result of mill contamination during the mechanical alloying process. Small precipitates throughout the microstructure were found to be aluminum rich (Fig. 13); aluminum is probably also from mill contamination, since the specification for MA 957 does not contain aluminum.

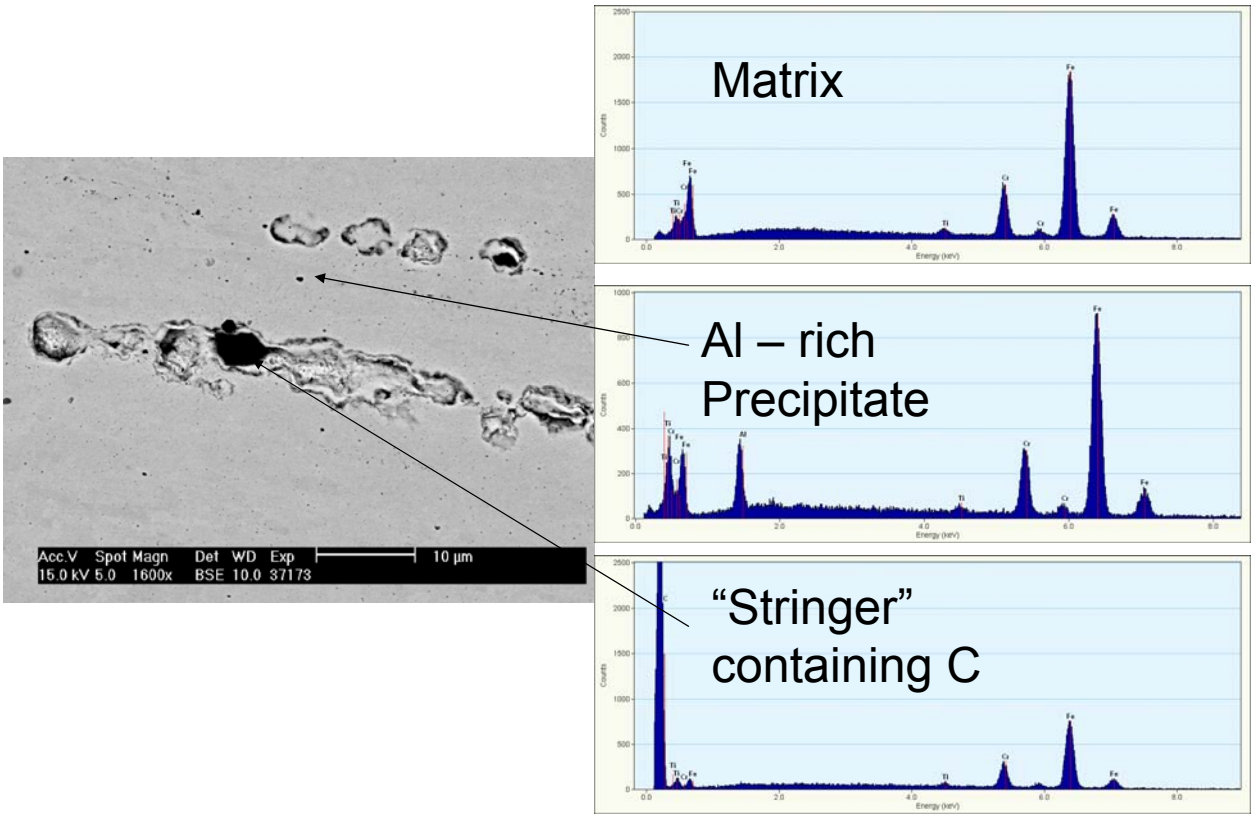

Fig. 13. Results of backscattered image analyses that show high-carbon and aluminum-rich particles present in the large particles present in the ODS steel MA 957.

Examination of a region near the creep failure indicated the presence of segregation that links up regions between stringers (Fig. 14) and probably leads to the relatively brittle creep failure shown in Fig. 11. The segregation, especially in the stringers, is rich in chromium, titanium, and carbon, which probably indicates the presence of carbide particles. These particles in the stringers were joined by "trails" of segregation that were found to be chromium rich. There were also indications of cavitation associated with areas of the segregation, indicating that it could be the joining of such cavitation that leads to the creep failure.

Discussion

Development of ODS steels for fast reactor cladding began in Belgium in the late 1960s [18], and work for that application has continued [19,20]. The main problem that kept the steels from being used for that application is anisotropy in properties that results from the fabrication process. Fabrication involves mixing the metal alloy powders with oxide (Y_2O_3) powders and mechanical alloying the powders, followed by compaction, and extrusion at elevated temperatures. The steels have superior creep strength in the longitudinal (extrusion) direction (properties most often studied), but, in the case of tubes, not in the biaxial direction. The problem with anisotropy still exists in more recent steels being developed [19,21],

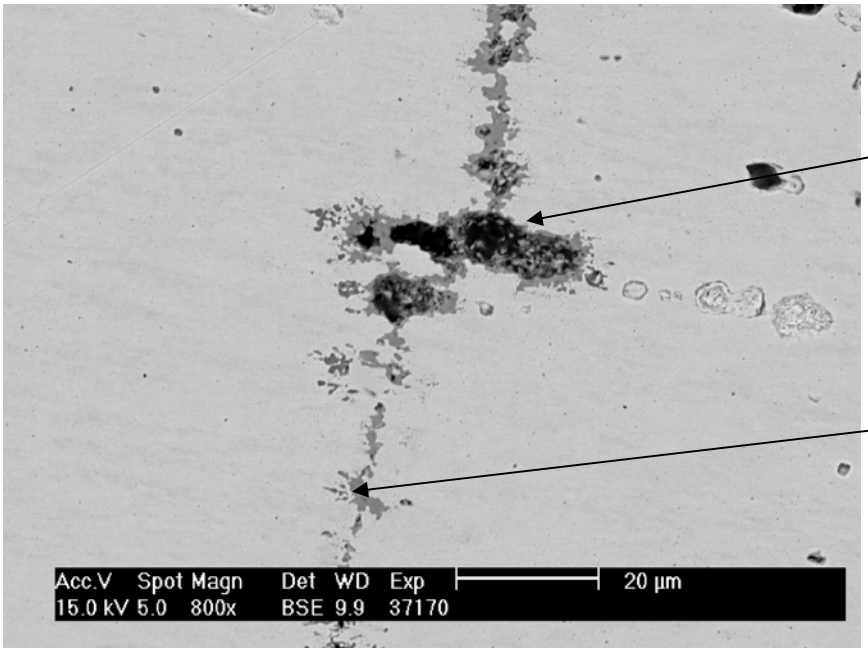

Fig. 14. Backscatter image showing that segregation occurs perpendicular to the worked direction in the region of the creep fracture.

although there have been advances in reducing it [22]. Anisotropy is present in the steels discussed in this paper, although that subject will not be discussed further. The objective of these studies was to compare strength properties in the strong (longitudinal) direction to determine similarities and differences among the different commercial and experimental steels that have been studied recently.

Initial TEM examination at ORNL of the experimental Fe-12.3Cr-3W-0.39Ti-0.25Y_2O_3 (12YWT) and an Fe-12.4Cr-0.25 Y_2O_3 (12Y1) ODS steels produced by Kobe Steel and Sumitomo Tubes, respectively, indicated that the 12YWT contained 3-5-nm diameter particles at a number density of 1-2x10^{23} m^{-3}, whereas the 12Y1 contained 10-40 nm particles at a number density of 10^{20}-10^{21} m^{-3} (Fig. 1) [7]. Electron diffraction indicated that particles in the 12Y1 were essentially pure Y_2O_3. Because of the size of the particles in 12CrYWT, the three-dimensional atom probe (3-DAP) was used to determine that the small particles were primarily atom clusters enriched in Y, Ti, and O [8,9]. From this [8,9] and previous work [23], it was concluded that the original Y_2O_3 particles dissolved during the mechanical alloying, and new clusters formed during subsequent processing (extrusion, etc.). This dissolution and subsequent reformation was obvious, since the atom probe indicated the concentration of titanium in the clusters was higher than the concentration of yttrium [8,9].

The difference in the microstructures of 12Y1 and 12YWT was reflected in the difference in tensile (Fig. 2) and creep (Fig. 6) strengths, with the 12YWT having much higher tensile and creep strengths than 12Y1. The yield stress of the 12YWT was also significantly higher than that of the commercial MA 956 and PM 2000, and it was comparable to that of MA 957 (Fig. 4).

The difference in creep properties of the commercial steels and 12YWT did not seem to follow from the relative strengths observed in the tensile tests. In particular, although 12YWT had the highest yield stress and ultimate tensile strength of the experimental steels and all the commercial steels but MA 957 (Fig. 4), creep-rupture properties based on the LMP comparison indicated that in the limit of low stresses and/or long rupture times, the commercial steels and the 12YWT approach a common strength level (Fig. 9).

The MA 956 had yield stress and ultimate tensile strength values from room temperature to 800ºC that were much less than those for 12YWT. Nevertheless, the LMP values indicated that for long-time tests and/or high-temperature creep tests, the values for MA 956 approach those for 12YWT.

Similarly, the yield stress and ultimate tensile strength of PM 2000 were less than for 12YWT, but the long-time, low-stress creep properties of the PM 2000 0.08-and 0.13-mm sheet specimens were as good as those for the 12YWT based on LMP (Fig. 8). Note that the tensile properties in Fig. 4 for PM 2000 are from vendor data on bar product. Vendor data for ultimate tensile strength (no yield stress data were given) for 1-8-mm sheet showed the sheet to be weaker than the bar [15].

Finally, the MA957 and 12YWT had equivalent yield stresses and ultimate tensile strengths, but the a comparison of the LMP indicated that for the completed creep tests for MA 957, there was an advantage for 12YWT (Fig. 7). However, just as for the other commercial products, at high LMP, the values for MA 957 approached those for MA 956, which appear to converge with those for 12YWT.

These results indicate that, with the exception of 12Y1, the creep-rupture properties of the other four steels appear to approach a similar level with decreasing stress and/or increasing test temperature. As stated above, the excellent creep strength of the 12YWT was attributed to the fine distribution of particles (atom clusters) observed by atom probe studies in 12YWT and not 12Y1 [7-9].

Recent atom probe studies on the MA 957 indicated the presence of 2-nm-diameter particles similar to those in 12YWT [17]. Just as in 12YWT, the particles were rich in Ti, Y, and O. These results indicate that the same dissolution of the Y_2O_3 that occurred during mechanical alloying in Y12WT [8,23] must also occur during the mechanical alloying of MA 957. Since the creep properties of these steels depend on the distribution of oxide particles, the convergence of rupture properties at long times would be expected, given the similarity of the microstructures. Given the convergence of creep-rupture properties of MA 956, MA 957, PM 2000, and 12YWT at long rupture time and/or high temperatures (Fig. 9), one explanation is that the MA 956 and PM 2000 also contain the high density of small particles found in MA 957 and 12YWT.

Generally, relative creep strengths are comparable to the relative strengths observed in a tensile test. This raises a problem with the explanation that the similarity of creep behavior of the 12YWT and the commercial steels is caused by similar nano-sized particles, because it does not explain the difference in tensile properties of the different steels. That is, it would be expected that the high strength in the tensile tests of 12YWT and MA 957 would also depend on the presence of the small particles.

From TEM and atom-probe observations on the 12Y1 and 12YWT, it appeared that the major microstructural difference in the two was the absence of the small nano-sized clusters in 12Y1[7-9]. This difference could be used explain why 12Y1 has inferior tensile and creep properties compared to 12YWT. The observation of the nano-sized clusters in the MA 957 [17] also explains the similarity in tensile and creep properties of this steel and 12YWT. Therefore, if all the commercial steels contain the small nano-sized clusters postulated above and tensile and creep properties are determined by these particles, then all the commercial steels should have similar tensile properties, which is not the case.

Another possibility is that the lower tensile strength properties of the MA 956 and PM 2000 result from the absence of nano-sized particles in the latter two steels. This appears unlikely, given the observations on the much lower creep strength of the 12Y1, which did not contain the nano-sized particles. If it were true, however, the conclusion would be that the creep-rupture properties do not depend on the nano-sized particles, but on another dimension of particles. Obviously, TEM and atom probe studies of MA 956 and PM 2000 are required.

It appeared that the yield stress and ultimate tensile strength for all the steels converged at the highest test temperatures, although the 12Y1 had the lowest values at the highest temperatures (Fig. 4). Convergence for this steel appeared to be at a much higher temperature than for the other steels. Given this observation and the observation on the inhomogenity of the microstructure of the MA 957 (Figs. 11-14), another explanation can be suggested. Such inhomogeneous regions may affect the strength

negatively at low tensile test temperatures, and it is the difference in the relative amounts of such regions in the different steels that plays a role in limiting the strength in a tensile test at temperatures up to 800-900ºC, where the tensile properties begin to converge. Optical microscopy indicated that such inhomgeneous regions exist in all of the experimental and commercial steels tested. Another possibility is the effect of an inhomogeneous grain size, which is generally observed in the microstructures of these steels. Detailed SEM and optical quantitative microscopy along with TEM is required to determine if these explanations can be correlated with the mechanical properties observations.

Summary and Conclusions

Tensile and creep-rupture data for two experimental ODS steels and three commercial ODS steels were collected and compared. The experimental steels are: 12Y1 (nominally Fe-12Cr-0.25Y_2O_3) and 12YWT (Fe-12Cr-3W-0.4Ti-0.25Y_2O_3); the commercial steels are: MA 956 (Fe-20Cr-4.5Al-0.33Ti-0.5 Y_2O_3), MA 957 (Fe-14Cr-0.3Mo-1Ti-0.25Y_2O_3), and PM 2000 (Fe-19Cr-5.5Al-0.5Ti-0.5 Y_2O_3).

There was a significant variation in the yield stress and ultimate tensile strength of the different steels tested at room temperature to 800ºC. The 12YWT was much stronger than the 12Y1. Of the commercial steels, the MA 957 was the strongest with strength properties similar to the 12YWT. The MA 956 was the weakest of the commercial steels, with strengths below that of the 12Y1 below 650ºC, and somewhat stronger at higher temperatures. The PM 2000 had a strength between that of MA 956 and MA 957; up to ≈550ºC, the strength of PM 2000 was comparable to that of 12Y1, but at higher temperatures, PM 2000 was substantially stronger. Strength properties appeared to merge for extrapolations above ≈900ºC.

Creep-rupture properties were compared using Larson-Miller parameters. Although there were differences between the 12YWT and the three commercial steels, at high Larson-Miller parameter values (high-temperature, long rupture time tests) there appeared to be a convergence of properties. Values for these four steels were greater than those for 12Y1, which gave no indication of converging to the same value.

The reason for the similar tensile and creep behavior of the MA 957 and 12YWT was concluded to be the distribution of nano-sized particles rich in Y, Ti, and O that have been observed by TEM and atom probe studies on 12YWT and MA 957. In contrast, such studies found much larger Y_2O_3 particles in the weaker 12Y1, explaining why this steel had inferior tensile and creep properties. However, a quandary exists when it comes to explaining the apparent excellent long-term creep properties of MA 956 and PM 2000, despite these steels having significantly lower tensile properties than 12YWT and MA 957.

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