TRANSVERSE AND LONGITUDINAL TENSILE PROPERTIES AT 760\(^{\circ}\) C OF SEVERAL OXIDE DISPERSION STRENGTHENED NICKEL-BASE ALLOYS

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STRENGTHENED NICKEL-BASE ALLOYS

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SUMMARY

The transverse and longitudinal tensile properties of the oxide dispersion strengthened nickel-base alloys MA-753, MA-754, MA-755E, and MA-6000E were determined at 760°C. MA-753 and MA-754 have small amounts of gamma prime and strength levels suitable for turbine vane applications, while MA-755E and MA-6000E are highly alloyed, gamma prime strengthened superalloys with strengths typical of turbine blade materials. These alloys were produced by mechanical alloying and extrusion. In addition MA-755E and MA-6000E were also directionally recrystallized. Resultant grain aspect ratios varied from 1:1 to over 20:1. Longitudinal tensile strengths ranged from 285 to 1175 MPa, while longitudinal elongations were in excess of 4 percent for all alloys. Transverse tensile strengths were comparable to longitudinal strengths, but transverse ductility levels were generally less than 2 percent elongation. Tensile and yield strengths increased with increasing strain rate over the range 0.001 to 0.05 per second. Ductility in both orientations was not strain rate sensitive but could be related to grain size and grain aspect ratio. The fracture mode of most of these ODS alloys changed from transgranular for longitudinally oriented specimens to intergranular for transversely oriented specimens. Processing history influenced the ductility and fracture mode of MA-753. The transverse ductility of MA-6000E was improved from 0.8 to 1.8 percent elongation by hot rolling. However, the transverse ductility of most ODS alloys tested was lower than that (6 percent) of a currently used turbine blade alloy, DS MAR M-200 + Hf, also tested for comparison.
INTRODUCTION

Advanced blade and vane materials for air breathing engines are likely to have directional microstructures. Such materials include directionally solidified (DS) eutectics, directionally recrystallized oxide dispersion strengthened (ODS) superalloys and fiber reinforced composites. Some attention has recently been given to off-axis mechanical properties of these materials, such as shear strength and transverse ductility. Transverse ductility near $760^\circ$ C is particularly important because it influences thermal fatigue life which is critical for economical commercial engine applications (1).

For example, a recent investigation (2) has shown that transverse tensile ductility of the $\gamma/\gamma'$ - $\delta$ DS eutectic is only 0.5 percent elongation at $760^\circ$ C. This low ductility is one of the reasons why the $\gamma/\gamma'$ - $\delta$ eutectic is no longer being developed for advanced commercial turbine blade applications.

The objective of this study was to determine the transverse and longitudinal tensile properties of several ODS alloys. The nickel-base alloys studied in this program were MA-753 (3, 4), MA-754 (5, 6), MA-755E (6), MA-6000E (7, 8), and for a baseline, the currently used directionally solidified MAR M-200 plus Hf (1). Tensile tests were conducted at $760^\circ$ C over the range of strain rates 0.001 to 0.05 per second. Strain rate sensitivity was evaluated in order to compare with previously reported results for MA-753 (3). After determination of mechanical properties, selected specimens were examined by optical, electron replica and scanning electron microscopy, to correlate mechanical properties with microstructural and fractography features.

EXPERIMENTAL PROCEDURE

The compositions of all alloys evaluated are listed in table I. MA-753 contains 4.2 weight percent (Al + Ti) and 15 volume percent gamma prime. MA-754 contains 0.8 weight percent (Al + Ti) and has essentially no gamma prime. MA-755E and MA-6000E contain 7.5 and 7.0 weight percent (Al + Ti), respectively, and each contains approximately 55 volume percent gamma prime. Both alloys have significant amounts of W, Mo, Ta, and Zr. The bar sizes and processing history of alloys available for this program, and the specimen lengths and orientations tested are listed in table II. The tensile specimens are designated regular (50.8 mm in length), and miniature (12.7 mm), see figure 1. Note that the thread dimensions are given in inches because no exact metric equivalent exists.

Specimens were tested in air at $760^\circ$ C at strain rates of 0.001, 0.005, 0.01, 0.05 per second. Insufficient quantity or size of some materials did not permit testing at both specimen sizes or at all strain rates; for example, no longitudinal tests of
MA-755E and DS MAR M-200 + Hf were conducted because of lack of material. For specimens with transverse tensile fracture strain less than 2 percent, strip chart load-displacement curves were used to determine ductility. For the more ductile specimens the standard specimen "fit-back" method was used.

Ultimate tensile strength, yield strength, elongation and reduction of area were determined for all specimens. All data plotted in subsequent figures represent the average value of at least three separate test specimens. Tensile tested specimens representative of both orientations were examined by optical, replica electron and scanning electron microscopy. Figure 2 illustrates specimen orientations and directions of metallographic examination.

RESULTS AND DISCUSSION

A. Tensile Strength

The tensile and yield strength results at 760°C for both orientations are plotted on a log stress versus log strain rate basis in figures 3 and 4, respectively. For both ultimate and yield strengths, the ODS alloys exhibit a strain rate sensitivity exponent (slope) of approximately 20, with the exception of MA-754 which has an exponent of 8. These exponents are greater than those exhibited by commercial superalloys, but similar to a previous investigation (3) of MA-753. These results were determined with miniature specimens oriented in both the longitudinal and transverse orientations of the ODS alloys MA-754, MA-753, and MA-6000E. MA-755E and DS MAR M-200 + Hf were tested only in the transverse orientation. The longitudinal and transverse tensile test results for the regular size specimens for MA-754 and MA-753 are not plotted because the results are almost identical to those presented for the miniature specimens. Therefore, since no specimen size effect was determined for tensile or yield strength for these alloys, it was assumed that the use of miniature specimens in this investigation for the evaluation of MA-755E and MA-6000E gave valid tensile and yield strength test data.

The longitudinal tensile strength of MA-754 and longitudinal yield strength of MA-6000E bar stock are slightly greater than transverse strengths at all strain rates, see figures 3 and 4. The transverse ultimate and yield strengths of the other alloys are generally equivalent or slightly greater than the longitudinal strengths at all strain rates. Such behavior is probably due to grain size and grain aspect ratios, as will be discussed subsequently in the section entitled Microscopy.
These tensile results are also consistent with alloy composition. MA-754 is a relatively simple solid solution strengthened alloy and has the lowest strength, while MA-755E and MA-6000E have large amounts of gamma prime and refractory metal additions resulting in higher strength levels. The results for the directionally solidified MAR M-200 + Hf indicate that its transverse ultimate (875 MPa (127 ksi)) and yield (779 MPa (113 ksi)) strengths are approximately equal to the transverse strengths of MA-755E at a strain rate of 0.01 per second. Limited transverse testing performed on DS MAR M-200 + Hf did not indicate strain rate sensitivity for either ultimate or yield strengths.

MA-6000E (15 mm diameter bar and 12×25 mm shape) was evaluated in two product forms. The tensile results shown in figures 3 and 4 indicate that the hot rolled shape was stronger than the as-extruded bar. Both product forms of MA-6000E received the same total reduction in area (20/1) at 1038°C prior to gradient annealing. The 12×25 mm hot rolled shape was extruded approximately 10/1 and then hot rolled approximately 2/1, or 50 percent reduction in thickness, while the 15.2 mm diameter bar was extruded directly to 20/1 RA. However, these two materials had considerable differences in grain size and grain aspect ratios. These differences will be more fully discussed in the section entitled Microscopy.

B. Tensile Ductility

Longitudinal ductility. - The longitudinal tensile ductility results at 760°C are presented in figure 5 for the three ODS alloys tested. Insufficient quantity of MA-755E and DS MAR M-200 + Hf prevented testing in the longitudinal direction. There appeared to be indications that the elongation of MA-753 and 754 was sensitive to test strain rate. However, since the strain rate range was limited (0.005 to 0.05 per second) the effect is not conclusive. Therefore, only data at a strain rate of 0.05 per second are presented in figure 5.

The 44 mm diameter bar of MA-753 was slightly more ductile than the 58 mm diameter bar. Only a slight specimen size effect was noted for MA-753. For MA-6000E the hot rolled shape had a reduction in area of 20 percent while the as-extruded bar had a reduction in area of 12 percent. MA-754 has excellent ductility, approximately 70 percent reduction in area. Although no specimen size effect was determined for reduction in area of MA-754, the elongation of miniature specimens was 21 percent while the elongation of the regular size specimen was only 10 percent. Such an effect may be due to fewer grains and grain boundaries present in the gage section of miniature size specimens.
As mentioned previously, neither MA-755E nor DS MAR M-200 + Hf were tested in the longitudinal orientation. However, 7 percent elongation has been reported for DS MAR M-200 + Hf (2). In general, the three ODS alloys tested have ductility in the longitudinal direction comparable to that of DS MAR M-200 + Hf.

Transverse ductility. - The transverse ductility results at 760°C are presented in figure 6. There was no indication that transverse ductility was sensitive to strain rate, so only the results at 0.05 per second are presented. Note that the data for both MA-754 and DS MAR M-200 + Hf are plotted on a scale 10 times that of the other three ODS alloys.

Once again the 44 mm diameter bar of MA-753 was slightly more ductile than the 58 mm diameter bar. Regular size specimens taken from the 44 mm diameter bar were considerably less ductile than miniature specimens.

The hot rolled shape (1.8 percent elongation) of MA-6000E was significantly more ductile than the as-extruded bar (0.8 percent elongation). These results obtained with miniature size specimens are comparable to independently determined values of ductility obtained with conventional size specimens of MA-6000E in both as-extruded (7) and hot rolled forms (8). The transverse ductility of MA-755E was 0.3 percent elongation and 1.8 percent reduction in area. Both MA-754 and DS MAR M-200 + Hf had excellent transverse ductility. MA-754 had 11 percent elongation and 35 percent reduction in area. DS MAR M-200 + Hf had 6 percent elongation and 20 percent reduction in area.

C. Microscopy

Longitudinal specimens. - Micrographs of representative specimens of the alloys tested in the longitudinal orientation are shown in figure 7. Specimens of MA-753 from both the 58 mm and 44 mm diameter bars exhibited primarily transgranular fracture with subsurface cracking present throughout the cross section. Both size bars have a diversity of grain size and grain aspect ratios (GAR). The grain size of the 58 mm diameter bar ranges from 10 to 150 microns and GARs from 1:1 to 10:1. The 44 mm diameter bar has a range of grain sizes from 50 to 150 microns and GARs from 5:1 to 20:1.

These data are summarized for all alloys in table III, which also lists information on inclusions and transverse elongation of all alloys. This variability of grain size and shape of MA-753 could obviously result in significant differences in microstructures of specimens machined from varying locations within each bar. Such
microstructural variabilities will be related to transverse ductility results later in this section.

MA-754 fractured primarily in a transgranular mode with some cracking extending into the gage section. The grain size (50 to 250 microns) and grain aspect ratio of 5 1 to 10 1 for the MA-754 hot rolled shape show slightly more uniformity than for MA-753.

The specimen shown of the MA-6000E hot rolled shape has essentially one grain in the specimen gage diameter and failure occurred transgranularly. Both the MA-6000E as-extruded bar and hot rolled shape have large uniform grains (100 to 200 microns) with aspect ratios over 20.1, see table III. All the photomicrographs of MA-6000E are devoid of structure at this (75X) magnification and hence, the one specimen for the MA-6000E hot rolled shape is representative of both forms of this alloy.

Transverse specimens. - Photomicrographs of transversely oriented tensile specimens of all alloys tested are shown in figures 8 through 17. The orientation of metallographic examinations with respect to specimen orientations were presented in figure 2. In each figure the top photographs are optical micrographs taken at 75X on the left and 500X on the right. The bottom left is an electron replica micrograph at 10 000X, showing in all cases the edge of the fracture surface at the top of the photograph. On the bottom right in all figures is a scanning electron microscope fractograph at 1000X (3000X for fig. 8 only) of the actual fracture surface of the transverse specimen, taken at a 45 degree tilt angle at 25 kV.

MA-753 is shown in figures 8 through 12 after testing at strain rates of 0.001 and 0.005 for the 58 mm diameter bar (figs. 8 and 9) and strain rates 0.005, 0.01, and 0.05 for the 44 mm diameter bar (figs. 10 to 12). Although all five transverse specimens failed primarily intergranularly, a diversity of grain sizes, aspect ratios, inclusion sizes and distribution and fracture initiation sites was noted. The grain size and aspect ratio varies from small equiaxed grains shown particularly in figure 9, to large elongated grains spanning the entire specimen width, as shown in figure 12. As discussed above in the section entitled Tensile Ductility, the 44 mm diameter bar was more ductile than the 58 mm diameter bar. In addition, within the 44 mm diameter bar, it was apparent that the large grain aspect portion shown in figure 12 (1.6 percent transverse elongation) was more ductile than the material shown in figure 10 (0.7 percent transverse elongation). Hence, there appears to be a relationship between increased ductility and large grain size and aspect ratio.
In addition, large variations in grain boundary carbide/oxide/nitride inclusion sizes and concentrations and their effect on fracture initiation sites were observed in the MA-753 specimens presented in figures 8 through 12, see also table III. MA-753 had the highest oxygen (0.92 percent) and nitrogen (0.14 percent) content of all the alloys tested. These gas contents were probably responsible for numerous inclusions which varied in size and number among the MA-753 specimens tested. These high gas and inclusion contents may have contributed to the extremely low ductility levels exhibited by MA-753. These high oxide or nitride contents are associated with the original powder particle surfaces and become deleterious, non-soluble grain boundary inclusions during processing and heat treatment. Intergranular fracture appeared to initiate at coalesced voids in figures 8, 10 and 11 and at large clusters of inclusions of oxides, carbides or nitrides as shown in figures 9 and 12. Such a hypothesis seems reasonable because the balance of the fracture surface was devoid of coalesced voids or clusters of inclusions. Coalesced voids believed to have been formed by localized micro-deformation are particularly evident in the SEM micrographs in figures 8 and 11. The voids are approximately 0.5 to 1 micron in diameter and the distance between lines of voids is 1.5 to 2 microns. These voids are most likely associated with the 1 micron diameter inclusions shown in the replicas.

The MA-754 alloy fractured primarily in an intergranular mode with fracture appearing to be associated with large voids, as shown in figure 13. This alloy was the most ductile of all the ODS alloys tested. Transverse ductility at a strain rate of 0.01 per second of this specimen was 24 percent reduction in area and greater than 6 percent elongation. MA-754 was also the lowest strength alloy having the lowest concentration of gamma prime. Intergranular cracking was also prevalent throughout the test section in both longitudinal and transverse specimens. This alloy generally had a large grain size and grain aspect ratio.

The large volume percent of blocky gamma prime precipitates in MA-755E masks the grain boundaries, see figure 14. However, the grain aspect ratio for this material was over 20:1, as determined from longitudinal photomicrographs of the grip section of the transverse specimen. Fracture appears to be intergranular, initiating at carbides or oxides, not shown in the figures.

Figures 15 and 16 show MA-6000E specimens of hot rolled shape and as-extruded bar stock, respectively. The hot rolled shape in figure 15 failed intergranularly with some transgranular failure as shown in the upper right photomicrograph. The specimen orientation in the top photomicrograph shows the short transverse direction oriented vertically and the long transverse direction oriented horizontally, see figure 2. Hot rolling slightly flattened the original as-extruded grains. Fracture
appeared to initiate at random holes probably associated with subsurface carbides or oxides, with severe cracking in the bottom of each hole. The MA-6000E as-extruded bar, shown in figure 16, exhibited a transgranular failure mode which initiated at oxide/carbide inclusions.

Directionally solidified MAR M-200 + Hf exhibited a transgranular fracture mode, see figure 17. Fracture appears to initiate at casting porosity and at large carbides.

CONCLUDING REMARKS

Perhaps the most significant observation of this investigation was the microstructural variability noted for both bars of MA-753, see table III. Specifically, MA-753 was evaluated in two different final bar sizes, 44 and 58 mm diameter. Both of these bars had been hot rolled from much larger (≥200 mm) diameter as-extruded billets. Since the total reduction of these bars was almost equivalent, the variations in observed distribution of inclusions, grain sizes and grain aspect ratios were unexpected. Furthermore, the observation that significant microstructural variations were observed within each bar of this alloy suggests the alloy's apparent sensitivity to slight variations in powder processing, working and heat treating schedules.

Likewise the MA-6000E alloy appears sensitive to such processing variables. The two product forms of this alloy received the same total amount of deformation and a common heat treatment. However, it was apparent from the final ductility that the actual hot working and heat treating sequence was critical for developing the subsequent microstructures.

In conclusion, it appears that larger grain sizes and larger grain aspect ratios in a given alloy result in improved transverse ductility. Additional support for this statement has been reported. Cairns et al (4) significantly improved longitudinal ductility and rupture life of MA-753 by directional zone recrystallization rather than the isothermal heat treatment used for the material in this investigation. Presumably this would also benefit transverse properties. Kim and Merrick (8) also improved the ductility and rupture life of MA-6000E by both thermal mechanical processing and by use of directional zone recrystallization. Transverse ductility was increased from 0.8 percent elongation for as-extruded bar to 3.5 percent elongation for the long transverse direction of the hot rolled shape. The grain aspect ratio was observed to be over 20:1.

Proper consideration of process variables and resultant effects on alloy microstructures are considered essential to the eventual use of these materials for advanced turbine engine applications.
SUMMARY OF RESULTS

The transverse and longitudinal tensile properties at 760°C were determined for the oxide dispersion strengthened nickel-base alloys MA-753, MA-754, MA-755E, and MA-6000E. DS MAR M-200 + Hf was also tested for comparison.

The following results were obtained for the ODS alloys:

1. Longitudinal tensile strengths ranged from 285 to 1175 MPa. Elongations for all alloys were in excess of 4 percent. The highly alloyed gamma prime strengthened alloy MA-6000E had the greatest tensile strength, while the solid solution strengthened alloy MA-754 had the lowest strength.

2. Transverse tensile and yield strengths were comparable to longitudinal strengths, but transverse ductility levels were generally less than 2 percent elongation.

3. Tensile and yield strengths in both longitudinal and transverse orientations exhibited strain rate sensitivity over the range from 0.001 to 0.05 per second. Ductility was not strain rate sensitive but could be related to grain size and grain aspect ratio, especially in MA-753.

4. The fracture mode of most of the ODS alloys generally changed from transgranular for the longitudinally oriented specimens to intergranular for the transversely oriented specimens. For both orientations fracture appeared to initiate at carbide/oxide inclusions or voids.

5. Processing history and inclusion concentrations influenced the fracture mode and ductility of MA-753. The transverse ductility in the short transverse direction of MA-6000E was improved from 0.8 to 1.8 percent elongation by hot rolling.

6. The transverse ductility of most ODS alloys tested was lower than that of currently used turbine blade alloys. For example, the transverse elongation of DS MAR M-200 + Hf was 6 percent compared to less than 2 percent elongation for all the ODS alloys, except MA-753 which had 10 percent elongation.

REFERENCES

TABLE I. - NOMINAL COMPOSITION OF ALLOYS (wt. percent)\textsuperscript{a}

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cr</th>
<th>Al</th>
<th>Ti</th>
<th>W</th>
<th>Mo</th>
<th>C</th>
<th>N</th>
<th>O</th>
<th>(\gamma') (V/O)</th>
<th>Other</th>
<th>(\gamma_2)O\textsubscript{3}</th>
<th>Total</th>
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</thead>
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<td>MA-753</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>22.3</td>
<td>1.6</td>
<td>2.3</td>
<td>0.05</td>
<td>0.14</td>
<td>0.92</td>
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<td></td>
<td>0.04</td>
<td>0.15</td>
<td>0.92</td>
<td>1.3</td>
<td>0.7 Zr</td>
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<td>44 diam</td>
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<td>1.6</td>
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<td></td>
<td></td>
<td>0.04</td>
<td>0.15</td>
<td>0.92</td>
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<td>0.7 Zr</td>
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<td>0.3</td>
<td>0.5</td>
<td></td>
<td></td>
<td>0.06</td>
<td>0.11</td>
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<td>0.6</td>
<td>0.11</td>
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<td>4.5</td>
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<td>1.1</td>
<td>2.5 Ta, 0.15 Zr</td>
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<td>15</td>
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<td>0.10</td>
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<td>2.5 Ta, 0.15 Zr</td>
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<td>DS MAR</td>
<td>9</td>
<td>5</td>
<td>2</td>
<td>12.5</td>
<td>4</td>
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<td>10 Co, 1.6 Cb</td>
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<td>5</td>
<td>2</td>
<td>12.5</td>
<td>4</td>
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\textsuperscript{a}Nominal except for C, N, and O, which were analyzed at LeRC.

\textsuperscript{b}Experimental alloys, other alloys in this table are commercially available.
<table>
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<tr>
<th>Alloy</th>
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<th>Heat treatment</th>
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<td>58 diam</td>
<td>Extruded + hot rolled 1080°C/7 hr</td>
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<td></td>
<td></td>
<td>Extruded + hot rolled 704°C/16 hr</td>
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</tr>
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<td>31×80 shape</td>
<td>Extruded + hot rolled 1316°C/0.5 hr</td>
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<td></td>
<td></td>
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<tr>
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<td>13 diam</td>
<td>Extruded at 1038°C (20/1) 1332°C/0.5 hr</td>
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### TABLE III. - SUMMARY OF MICROSTRUCTURAL FEATURES AND AVERAGE TRANSVERSE DUCTILITY

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<thead>
<tr>
<th>Alloy</th>
<th>Bar size mm</th>
<th>Microstructure</th>
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<td></td>
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<td>Grain size μm</td>
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<td>MA-753</td>
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<td>10-150</td>
<td>Carbides, Nitrides, Large Oxides</td>
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<td>M-200 + Hf</td>
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<sup>a</sup>Inclusion concentration

Large, Medium

Based on qualitative analysis of photomicrographs shown in figures 7 to 17

Low
Figure 1 - Tensile specimens

Figure 2. - Specimen orientations and direction of transverse metallographic evaluation (→).
Figure 3 - Ultimate tensile strength of ODS alloys and DS MAR M-200 + Hf at 760°C.

Figure 4 - Yield strength of ODS alloys and DS MAR M-200 + Hf at 760°C.
Figure 5 - Longitudinal tensile ductility at 760°C and 0.05 sec^{-1} strain rate

Figure 6 - Transverse tensile ductility at 760°C and 0.05 sec^{-1} strain rate
Figure 7. - Micrographs of longitudinal specimens.

Figure 8. - Microstructure of transverse specimen of MA-753, 58 mm diameter bar (0.6 percent C).
Figure 9. - Microstructure of transverse specimen of MA-753, 58 mm diameter bar (0.2 percent C).

Figure 10. - Microstructure of transverse specimen of MA-753, 44 mm diameter bar (0.7 percent C).
Figure 11. - Microstructure of transverse specimen of MA-753, 44 mm diameter bar (1.2 percent ε).

Figure 12. - Microstructure of transverse specimen of MA-753, 44 mm diameter bar (1.6 percent ε).
Figure 13. - Microstructure of transverse specimen of MA-754 (6.6 percent Cr).

Figure 14. - Microstructure of transverse specimen of MA-755E (0.2 percent Cr).
Figure 15. Microstructure of transverse specimen of MA-6000E, hot rolled shape (1.8 percent c).

Figure 16. Microstructure of transverse specimen of MA-6000E, as-extruded bar (0.5 percent c).
Figure 17. - Microstructure of transverse specimen of DS MAR M-200 + Hf (2.5 percent).
The transverse and longitudinal tensile properties of the oxide dispersion strengthened nickel-base alloys MA-753, MA-754, MA-755E, and MA-6000E were determined at 760°C. Transverse tensile strengths were comparable to longitudinal strengths. Transverse ductility levels generally were less than two percent elongation. Both tensile and yield strengths increased with increasing strain rate over the range 0.001 to 0.05 per second. Ductility was not strain rate sensitive, but related to grain size and grain aspect ratio. The fracture mode of most alloys changed from transgranular for longitudinally oriented specimens to intergranular for transverse specimens. Transverse properties of DS MAR M-200 + Hf were also determined for comparison.
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