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Irradiation creep and swelling from 400 to 600 °C of the oxide dispersion strengthened ferritic alloy MA957

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Abstract

An irradiation creep and swelling study was performed on the Y_2O_3 -strengthened ODS ferritic steel MA957. Pressurized tubes were irradiated in the Fast Flux Test Facility (FFTF) to doses ranging from 40 to 110 dpa at temperatures ranging from 400 to 600 °C. None of the stress-free tubes exhibited any evidence of swelling as determined by diameter change measurements. With a few exceptions, the irradiation creep behavior is similar to that of conventional ferritic-martensitic steels. Calculated creep compliance values are equal to those of HT9 irradiated within the same temperature range, except at 600 °C where the creep rate of MA957 is about one-half the value for HT9. The magnitude of the creep transient for MA957 is comparable to HT9, again except at 600 °C where the transient is much lower for MA957. © 2004 Elsevier B.V. All rights reserved.

1. Introduction

Oxide dispersion strengthened (ODS) ferritic steels have been identified as potential candidates for higher temperature applications in fusion reactor first wall structures because of their excellent thermal creep resistance [1]. One such ODS steel that was under development is the ODS steel MA957. This steel was developed by the International Nickel Company (INCO). MA957 is a 14Cr ferritic steel strengthened with a fine dispersion of ~ 5 nm yttrium oxide particles. Presented here are irradiation creep data on MA957 for irradiation temperatures from 400 to 600 °C and doses up to 110 dpa. Data from pressurized tubes constructed by two different methods are presented. The data are presented along side HT9 irradiation creep data for comparison. This study is an extension of an MA957 mechanical properties project that was undertaken at Pacific Northwest National Laboratory [2].

2. Experimental

Heat DBB0111 and Heat DBB0122 of MA957 were utilized. The nominal composition of MA957 is 14Cr– 0.9Ti–0.3Mo–0.25Y₂O₃. The compositions of Heat DBB0111 and Heat DBB0122 are shown in Table 1. They are nominally identical in composition. Three sets of independent compositional certifications were performed on the MA957 material, and among the certifications, there were two distinct sets of compositions observed for O, Mn, and P. The reason for the two distinct sets is not known. Some aluminum was also present as alumina stringers that formed by contamination of the powders.

From hot extruded powders, rod stock was fabricated by a combination of hot-working and coldworking. From this rod stock, tubing was made by two different methods. Rod stock from Heat DBB0111 was swaged and annealed in several steps down to 6.73 mm diameter, and 5.84 mm OD \times 5.08 mm ID tubing was gun drilled from this rod. Rod stock from Heat DBB0122 was drilled, and then by a combination of rod drawing and annealing, was taken to the final dimensions of 6.86 mm OD \times 5.74 mm ID. 28.1 mm long (including endcaps) pressurized tubes were fabricated

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| Table 1 | | | | | | | | | | | | | |
|-------------|-----|------|-----------|----|-----|-------|-----|-----|--------|------|----|------|-------|
| Composition | and | heat | treatment | of | the | MA957 | and | HT9 | alloys | used | in | this | study |

| - | | | | | | | | | | | | | |
|---|--------------------|------|------|------|------|------|------|------|------|-----|-------|------|-------------|
| | Alloy | Ni | Cr | Mn | Мо | Si | С | V | W | 0 | Р | Ti | $Y(Y_2O_3)$ |
| | MA957 ^a | 0.13 | 13.9 | (a) | 0.30 | 0.05 | 0.01 | _ | _ | (b) | (c) | 1.05 | 0.22 |
| | MA957 ^b | 0.10 | 14.2 | 0.07 | 0.31 | 0.03 | 0.02 | _ | _ | (b) | 0.004 | 1.07 | 0.25 |
| | HT9 ^c | 0.60 | 11.8 | 0.62 | 1.06 | 0.29 | 0.21 | 0.33 | 0.52 | _ | _ | _ | _ |
| | HT9 ^d | 0.51 | 11.8 | 0.50 | 1.03 | 0.21 | 0.21 | 0.33 | 0.52 | - | - | - | _ |

(a) Two distinct sets of values were present, one with a value of about 0.05 wt% and one with about 0.11 wt%.

(b) Two distinct sets of values were present, one with a value of about 0.015 wt% and one with about 0.22 wt%.

(c) Two distinct sets of values were present, one with a value of <0.005 wt% and one with about 0.02 wt%.

^a Heat DBB0111, 760 °C/30 min/air cool.

^bHeat DBB0122.

^cCDE Heat, 1100 °C/2 min/air cool + 650 °C/2 h/air cool.

^d Heat 91353, 1038 °C/5 min/air cool + 760 °C/30 min/air cool.

from sections of tubing. As a result of the reduction operations, the grains in the tubing were highly elongated in the direction of the tubing axis. The aspect ratio was about 10:1.

For each target irradiation temperature, a two sets of pressurized tubes were fabricated, one set from the gun drilled tubing and the other set from the drawn tubing. Each tube in a set was filled to a different pressure to provide a range of stresses. For the present work, a set of pressurized tubes shall be called a tubeset. The range of pressures, and thus stresses, was chosen based on the target irradiation temperature and specimen geometry. In general, a tube-set consisted of between four and six pressurized tubes with one tube having zero pressure at the irradiation temperature. The zero-stress tube was used to estimate swelling. Uncertainty in the magnitude of the applied stress is discussed in [3].

Stress is reported as the von Mises effective stress which, for the pressurized tube geometry, is given by $\bar{\sigma} = \sqrt{3}/2\sigma_{\rm h}$ where $\sigma_{\rm h}$ is the hoop stress [3]. Strain is reported as the effective plastic strain which for the pressurized tube geometry, is given by $\bar{\epsilon} = 2/\sqrt{3}\epsilon_{\rm m}$ where $\epsilon_{\rm m}$ is the mid-wall strain [3].

Irradiations were performed at the Fast Flux Test Facility located in Richland, Washington using the Materials Open Test Assembly [4,5]. A detailed description of the irradiation conditions can be found in [3]. Tubes were removed from reactor after each irradiation cycle, and diameter measurements were performed using a scanning laser profilometer [5].

3. Results

The diametral strain behavior of the MA957 and the CDE HT9 *stress-free* tubes after irradiation at 400 to 550 °C are shown in Fig. 1. The diametral strains for the HT9 are very small. For the MA957, the gun-drilled



Fig. 1. Diameter change of stress-free MA957 and HT9 tubes during irradiation at 400–550 $^{\circ}$ C.

tubing and the drawn tubing behave identically and are temperature dependent. Some contraction is evident at 400 °C in the MA957. This may be due to segregation-induced densification, changes in dislocation density, or further compaction of the ODS steel which always has a slight amount of residual porosity. The diameter increases observed at 500 and 550 °C may be due to swelling, however, microstructural observations after the first irradiation cycle showed no evidence of swelling at 550 °C [2]. The swelling behavior implied from the diametral strain measurements shows MA957 to have about the same swelling resistance as traditional ferritic/martensitic (F/M) steels.

The stress-normalized irradiation creep behavior of MA957 compared with HT9 is shown in Fig. 2 for temperatures ranging from 400 to 600 °C. The co-linear MA957 data at 400 °C in Fig. 2 indicate that irradiation creep of MA957 up to at least 121 MPa is linearly proportional to the applied stress at this temperature. This suggests that a climb-based creep mechanism is dominating the irradiation creep deformation [3]. The MA957 data at 400 °C is initially linear with dose, but then begins to show some evidence of a decrease in creep rate after 100 dpa which is something that has not been observed in any traditional F/M steels studied by the



Fig. 2. Biaxial stress-normalized creep as a function of dose observed in MA957 and HT9 during irradiation at 400–600 °C.

authors. The reason for this decrease in creep rate is not known, but one possible explanation is that the creep rate during the first two irradiation cycles represents a very long transient regime.

At 500 °C, the MA957 irradiation creep behavior is roughly similar to the HT9 creep behavior with both having a transient within the first irradiation interval. The post-transient creep rates for the two stress levels of MA957 are approximately equal, indicating that at 500 °C, post-transient irradiation creep of MA957 again is linearly dependent on the applied stress for stresses up to at least 87 MPa. From Fig. 2, it can be seen that the post-transient creep rate of MA957 is lower than the value for the CDE heat of HT9.

The irradiation creep behavior of MA957 at 550 °C shows a transient somewhere in the first irradiation interval just as what is observed in HT9. The post-transient stress-normalized creep rate of the MA957 is



Fig. 3. Temperature-dependent irradiation creep compliance of MA957 and HT9 during irradiation.

approximately equal to the post-transient creep rate of the CDE Heat of HT9.

At 600 °C in Fig. 2, HT9 has a very large creep transient relative to the amount of post-transient creep, while the MA957 shows very little creep transient at the same stresses. The magnitude of the creep transient in MA957 at 600 °C is more similar to the values observed at 500 and 550 °C. The post-transient creep rate of the MA957 at 600 °C is lower than that of HT9.

The post-transient value for the creep compliance of MA957 is shown in Fig. 3 as a function of temperature. The average value for HT9 is plotted for comparison. With the exception of the low value at 500 °C, the creep compliance of the MA957 is roughly independent of temperature from 400 to 600 °C. The values for HT9 are about the same up to 550 °C, but is a little over twice as large at 600 °C.

4. Discussion

Up to 550 °C, the magnitude of creep in MA957 is very similar to that of HT9. The oxide particles in MA957 serve to inhibit thermal creep by acting as obstacles to both dislocation climb and glide. Precipitates in traditional F/M steels serve this purpose. The advantage of using an ODS steel is that the size and number density of the oxide particles can be more easily controlled, and the microstructural stability of ODS steels is much better at temperatures of 600 °C or higher where precipitates in traditional F/M steels begin to show thermal aging effects. It appears that in an irradiation environment, MA957 also performs well. When attempting to rationalize the better creep resistance of MA957 at 600 °C in an irradiation environment, it is tempting to assume that in an irradiation environment at 600 °C, thermal creep mechanisms largely control the creep behavior. However, a previous comparison of creep of HT9 in a thermal environment and in an irradiation environment shows that at 600 °C, creep transients in HT9 are much larger in the irradiation environment than in the thermal environment which shows that irradiation still has a large effect on the creep behavior of HT9 at 600 °C [6]. Thus, it is reasonable to say that MA957 better resists irradiationinduced creep mechanisms (as well as thermal creep mechanisms) at 600 °C.

Some aspects of creep behavior relevant only to MA957 should also be mentioned. The low failure rate of the drawn MA957 tubes suggests that the presence of the alumina stringers had little effect on the integrity of the MA957 pressurized tubes, at least for irradiation creep at stresses that are typical for F/M steels. The high failure rate of the gun-drilled MA957 tubes suggests that this fabrication method results in tubes that are more prone to failure than the drawn tubes.

5. Summary and conclusions

Irradiation creep and swelling of the ODS steel MA957 was studied using pressurized tubes irradiated at temperatures from 400 to 600 °C. There may have been up to 0.5% swelling after about 100 dpa at 550 °C. The creep response as a function of dose was similar to traditional F/M steels except that some reduction in creep rate was observed at higher doses at 400 °C. The creep compliance values of MA957 were similar to those of

HT9 for temperatures ranging from 400 to 550 °C. However, at 600 °C the creep compliance and the magnitude of the creep transient for HT9 both jumped significantly, while for MA957, the creep compliance was unchanged from its value at 400–550 °C, and the creep transient did not increase nearly as much as it did for HT9. The results suggest that ODS steels have similar creep properties to traditional F/M steels at 400–550 °C in an irradiation environment, but at 600 °C in an irradiation environment, MA957 has better creep resistance than traditional F/M steels.

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