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CORRELATION OF SUBSTRUCTURE WITH
MECHANICAL PROPERTIES OF PLASTICALLY
DEFORMED AISI 304 AND 316 STAINLESS STEEL

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PROGRESS REPORT

For Period January 1, 1972 to March 31, 1972

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The transmission electron micrograph on the cover of this report is from a Type 316 stainless steel tubular specimen internally pressurized with argon to an initial hoop stress of 13.5 kg/mm^2 and tested in argon at 650°C . The dislocations and their interactions with the precipitates are clearly visible. An apparent "helical" array of dislocations is visible between two of the precipitates as a result of these interactions.

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SUMMARY

A program to evaluate the substructure of austenitic stainless steels (Types 304 and 316) deformed at elevated temperatures in the creep, tensile, and fatigue modes is being conducted. Transmission electron microscopy is the primary tool employed in this research. Complimentary work using optical microscopy and immersion density techniques is presently underway to characterize the micro-crack behavior and bulk density changes observed subsequent to deformation in these materials.

Initial substructure results are discussed for 316 stainless steel creep-rupture specimens from a second heat of material. The results are consistent with the previously reported substructure results for 316 material in this program. Below $\sim 0.5 T_m$, dislocation cells are the predominant feature while subgrains are evident at higher deformation temperatures.

Substructure results are reported for 304 stainless steel tested in the creep mode where testing was terminated at pre-selected levels of deformation and for tests involving an abrupt decrease in load at a pre-selected strain level.

Results are reported from a study of intergranular

crack formation in 304 stainless steel tested at 650°C in the creep-rupture and tensile modes. Optical microscopy indicates that the maximum number of cracks occurs at angles near $\sim 80^\circ$ to the direction of the applied stress for the creep specimens. A majority of the cracks in the creep specimens are one grain boundary facet in length, in agreement with the previously reported results for tensile specimens from the same heat of material. Bulk density changes due to the crack formation are discussed for both the tensile and creep specimens.

Preliminary results are reported from a study of the substructure of 316 stainless steel thin-wall tubing biaxially crept at 650°C by internal pressurization.

OBJECTIVE

The objective of this program is to evaluate (a) the time, temperature, and stress dependent properties of austenitic stainless steels and (b) the relationship of these properties to the substructure as observed principally with a 200 kV transmission electron microscope. Special emphasis has been given to operating conditions typical of LMFBR applications, including the knowledge that the radiation environment can influence the substructure of these metals leading to significant changes in the mechanical property behavior. Related substructure studies on other alloy systems will also be considered.

INTRODUCTION

Substructure results for 316 stainless steel creep specimens from a second reference heat, tested at temperatures from 538°C to 760°C, are discussed. The results are in reasonable agreement with those previously reported for tensile specimens from the same heat.^[1]

Microscopy results from 304 stainless steel creep tested at 593°C are presented. The influence of stress

history and deformation level on the substructure are being investigated.

The study of intergranular crack formation was continued for tensile and creep-rupture tested 304 stainless steel. Optical microscopy results for the creep specimens and bulk density results for both deformation modes are presented.

Initial results from a study of the biaxial creep of internally pressurized 316 stainless steel tubing are discussed.

EXPERIMENTAL PROGRAM - PHASE II [D. J. Michel and J. Moteff]

Creep Properties (316 SS)

Substructure results for 316 stainless steel tensile specimens (Reference Heat No. 332990) were discussed in a previous report.^[1] Creep-rupture specimens, fabricated and tested by WADCO from this same heat, constitute a study complimentary to the Phase I portion of the research program.^[2,3,4] Within the period covered by this report, preliminary transmission electron microscopy evaluations of the creep-rupture specimens were made.

The material characterization and creep test results have been presented in a previous report.^[5] Specimens

representing, in general, the maximum and minimum stress levels at each test temperature were selected for microscopy study. The procedures used to prepare and examine thin foils from the gage sections of these specimens were similar to those employed for the tensile specimens.^[5]

The substructure of the creep specimens tested at 538°C (1000°F) and 593°C (1100°F) exhibited an immeasurably high dislocation density and dislocation cell formation. Evidence of planar slip was frequently observed in these specimens, especially at 538°C. At 593°C, the specimen deformed at $\dot{\epsilon}_m = 2.22 \times 10^{-6} \text{sec}^{-1}$ exhibited dislocation cell formation, but no cellular substructure was observed for the specimen deformed at $\dot{\epsilon}_m = 4.16 \times 10^{-8} \text{sec}^{-1}$, where $\dot{\epsilon}_m$ is the minimum creep rate. Instead, profuse carbide precipitation on the individual dislocations was observed.

The specimens tested at 650°C (1200°F) and 760°C (1400°F) formed subgrains with well-developed regular low-angle sub-boundaries. A measurable mobile dislocation density was present within the subgrains.

Both the dislocation cell and subgrain average intercepts, d , generally increase in a regular manner with decreasing applied creep stress, σ , according to $d \propto \sigma^{-1}$. The range of average intercept values is

between $\sim 0.5 \times 10^{-3}$ mm at 538°C and $\sim 3.5 \times 10^{-3}$ mm at 760°C.

A detailed substructural analysis of these specimens will be included in future reports.

Creep Properties (304 SS)

The program to investigate the creep behavior of Type 304 stainless steel in the temperature range from 593°C to 704°C (1100°F to 1300°F) has been extended to include experiments where testing is terminated at pre-selected strain levels prior to specimen rupture. Additional tests also have been performed where the load is abruptly decreased at pre-selected strain levels. The substructures resulting from these tests are being evaluated by transmission electron microscopy techniques, and the current results are presented here. The creep test procedures and initial results for this program have been reported previously.^[5] The TEM procedures used in this work were given in an earlier report.^[4]

A summary of the creep test results and substructure data for the 304 stainless steel specimens crept at 593°C (1100°F) is given in Table I. Specimens R-10 and R-13 were crept to rupture. The tests for specimens R-15, R-12, and R-11 were terminated immediately after loading, at the onset of secondary creep, and prior to

the onset of tertiary creep, respectively. Specimen R-14 was crept to a strain of 16% at 40,000 psi before the stress was abruptly decreased to 30,000 psi and testing continued until termination prior to rupture.

A combined strain-time curve showing the location of the termination points for specimens R-15, R-12, R-11, and R-10 is shown in Figure 1. The substructure characteristic of these specimens is presented in Figure 2. The predominant substructural features of specimens R-15, R-12, and R-11 are dislocation tangles characteristic of $\{111\} \langle 110 \rangle$ slip (Figures 2a, b, and c). Limited evidence for dislocation cell formation could be seen in specimen R-15. Indications of sub-boundary formation parallel to $\{111\}$ traces were observed in specimens R-12 and R-11.

Specimen R-10 exhibited clear evidence of subgrain formation with sub-boundaries parallel to $\{111\}$ traces (Figure 2d). The average subgrain intercept for this specimen (Table I) is in good agreement with previous results obtained for 316 stainless steel (Figure 7, reference 1).

Estimates of the density of the dislocations not associated with the sub-boundaries or the tangles (mobile dislocation density) are given in Table I. The results

show that the deformation produced during initial specimen loading is substantially recovered during primary creep and that further recovery takes place until rupture occurs. This result and the observations concerning sub-boundary formation in these specimens suggest that the critical conditions required for sub-boundary formation in the regions near active $\{111\}$ slip planes in these specimens is achieved when sufficient recovery has occurred, i.e. during tertiary creep.

A separate strain-time curve showing the results for specimens R-14, R-13, and R-10 is given in Figure 3. Figure 4 illustrates the substructure of specimens R-14 and R-13 by way of comparison with specimen R-10. The substructure of the unstressed shoulder of specimen R-10 is shown in Figure 4a. Figure 4b is another view of the substructure of specimen R-10 which is similar to Figure 2d. The substructure of specimen R-13 is shown in Figure 4c where sub-boundaries are parallel to $\{111\}$ traces. No clear evidence of sub-boundary formation was observed in specimen R-14 (Figure 4d). Instead, the familiar dislocation tangles and numerous stacking faults parallel to $\{111\}$ traces were commonly seen.

The average subgrain intercepts determined for specimens R-13 and R-14 are also consistent with the

previous 316 stainless steel results.^[1] The average intercept result for specimen R-14 is seen to be closer to the value for specimen R-10 than the value for specimen R-13. This is consistent with the strain-time results for specimens R-10 and R-14 in Figure 3 which suggests that the substructure of these specimens may bear a closer resemblance to that of specimen R-10 rather than specimen R-13.

The mobile dislocation density estimated for specimen R-14 is slightly higher than the dislocation density for specimen R-10. This difference could be attributable to the higher initial stress level imposed on specimen R-14 prior to the abrupt decrease in stress. The mobile dislocation density for specimen R-14 is also quite close to the value determined for specimen R-11 suggesting that the test of specimen R-14 may have been terminated near the onset of tertiary creep.

Cavitation (304 SS)

The quantitative study of intergranular crack formation during the elevated temperature deformation of 304 stainless steel has been continued. Initial results of this study were presented in previous reports.^[1,5] Additional results from the optical microscopy portion

of this work are presented for the tensile and creep-rupture specimens (from ORNL Reference Heat No. 9T2796). Results from immersion density experiments to determine the density change due to crack formation in these specimens are also discussed.

Optical microscopy was used to determine the crack length distribution for the tensile and creep-rupture specimens. This distribution as a function of the square root of crack length (number of grain boundary facets) is shown in Figure 5. The predominant crack length for both types of specimens is one grain boundary facet, in agreement with previously published results for another austenitic steel of approximately the same grain size.^[6]

The optically measured crack percentage distribution as a function of angle from the specimen stress axis is shown in Figure 6 for the creep-rupture specimens. In all cases, the maximum fraction occurs at approximately 80°. Thus, no shift of the maximum fraction to a higher angle with a decrease in creep rate, similar to that observed for the tensile specimens (Figure 14 of reference 1), is seen. The results in Figure 6 are consistent with the general observation that the majority of cracks occur at angles near 90°.

to the stress axis during creep deformation.

Figure 7 is a plot of crack areal density (number of cracks which occupy one or more grain boundary facets/ mm^2) as a function of distance toward the fracture zone for the creep-rupture specimens. Straight lines have been used to illustrate the trend for each specimen even though some scatter is evident in the data. The results indicate that the crack densities increase with decreasing creep rate at all distances from the fracture zone. The rates at which the crack densities increase as a function of distance are also inversely related to decreasing creep rate until approximately 5 mm from the fracture zone. Beyond this point, the data suggest that the crack density first decreases, then increases, on a point-to-point basis, until the fracture surface is reached. These results are considerably different than the tensile results presented in a previous report (see Figure 10 of reference 5), and strongly suggest that the mode of testing (tensile or creep) and the deformation rate may play an important role in the crack formation process in 304 stainless steel at 650°C.

The importance of deformation mode and deformation rate on the crack formation may be conveniently distinguished from the bulk density changes in the specimens. Immersion density techniques were used to determine the

density changes due to the crack formation. Specimen sections diametrically opposite to those used for the optical microscopy study were used for the density measurements.^[5] The experimental techniques employed were similar to those discussed by Ratcliffe.^[7]

The crack volume per unit weight (specific volume) of the specimens is shown as a function of crack areal density (number of cracks/mm²) in Figure 8. The crack volumes plotted in this figure were determined from the expression

$$v = \frac{1}{\rho} - \frac{1}{\rho_0} = \frac{\Delta\rho}{\rho\rho_0} \quad (1)$$

where ρ_0 is the unstressed density (determined for the present specimens from a portion of each specimen shoulder) and ρ is the density of each specimen after deformation. Figure 8 indicates that the crack specific volumes and crack areal densities correlate depending on the mode of deformation, but that no correlation is evident between the modes of deformation.

Figure 9 is a plot of the crack areal densities, shown in Figure 8, as a function of deformation rate. This figure shows that the relationship between crack areal density and deformation rate is dependent on the

mode of deformation. However, a plot of the crack specific volume as a function of deformation rate, Figure 10, shows a correlation of the form $V \propto -\log \dot{\epsilon}$, regardless of the mode of deformation. Thus, Figures 9 and 10 clearly illustrate that the crack formation in these specimens is dependent on the deformation rate independent of the deformation mode.

The foregoing discussion has shown that the specific volume of cracks resulting from both slow tensile and creep deformation is best described by a relationship with deformation rate. However, because deformation rate is a function of strain (ϵ), time (t), applied stress (σ), and temperature (T), it is expected that the crack volume will have a similar relationship with these variables of the form

$$V = f(\epsilon, t, \sigma, T). \quad (2)$$

For tests at a constant temperature, this relationship may be written as an equation^[8]

$$V = A \epsilon t \sigma^n, \quad (3)$$

where the constants A and n can be determined from a logarithmic plot of $V/\epsilon t$ versus σ .

Figure 11 shows the relationship between $V/\epsilon t$ and σ

for the 304 stainless steel specimens. A straight line may be drawn through all points. The values of the constants A and n determined from this line are $n = 6.3$ and $A = 5 \times 10^{-12}$. Equation (3) may then be written as

$$V = 5 \times 10^{-12} \epsilon t \sigma^{6.3}, \quad (4)$$

where V is in cm^3/gm , t is in hours, σ is in kg/mm^2 , and ϵ is the fractional true strain. This equation may now be used to gain a further understanding of the crack formation behavior of these specimens in terms of the test variables. These results will be discussed in future reports.

Biaxial Creep (316 SS)

A subprogram to investigate the substructure resulting from the plastic deformation of internally pressurized thin wall austenitic stainless steel tubing has been initiated. Type 316 stainless steel specimens fabricated and tested by GE-NSP from PNL reference heat tubular material (Heat No. 20276) were chosen for evaluation in the initial stage of this work.

Material Characterization - The 316 stainless steel tubing used had the chemical composition shown in Table II (vendor analysis). The tubing dimensions were 0.635 cm O.D.

by 0.0406 cm in wall thickness.

The room temperature tensile properties of the material as supplied are (annealed condition):

Ultimate Tensile Strength	63.60 kg/mm ²
Yield Strength	29.60 kg/mm ²
Elongation	51%

The microstructure of the as-received material indicates significant grain elongation along the longitudinal direction.

The test specimens were tube sections approximately 10 cm long to which appropriate fittings were attached. A small (~ 0.3 cm O.D.) thick wall tube at one end of each specimen provided access for pressurization. The specimens were pressurized with argon to achieve the desired initial hoop stress (biaxial stress ratio 1:2) and sealed by welding the smaller access tube prior to testing. After proper leak checking, the specimens, usually in groups of three, were tested at 650°C in an argon atmosphere furnace. The temperature was monitored by thermocouples located at the ends and at the center of the specimens. At various time intervals during each test, the specimens were removed from the furnace, and room temperature diametral measurements to determine the

strain were made using a micrometer capable of reading to 2.5×10^{-4} cm. Three circumferential and five longitudinal measurement locations (15 separate measurements) were employed to determine the creep-strain-profile for each specimen.

The circumferential strain results are presented in Figure 12. These results are for three specimens tested in the annealed condition and for three specimens tested in the annealed plus 20 percent cold-worked condition. The strain values plotted in Figure 12 represent the average of 15 measurements. The results show that after about 500 hours at 650°C, the annealed and annealed plus 20% cold-worked specimens (specimens 3 and 4) tested at the same initial hoop stress exhibit strains of about 10 and 0.2 percent, respectively.

Figure 13 shows specimens 1, 2, and 3 in the as-tested condition and prior to the removal of longitudinal wall sections from the central portion of each specimen for transmission electron microscopy. Specimen 3 ruptured during testing which produced a small longitudinal fissure in the tube wall. A section was also removed from the vicinity of the closed end fitting of specimen 1 for microscopy purposes. Procedures, similar to those used for the uniaxial creep-rupture specimens, were

employed to electrochemically thin and examine the tubular material.

The preliminary results indicate that the substructure of specimens 1, 2, and 3 may be generally characterized as $M_{23}C_6$ carbide precipitation associated with dislocations. The precipitates were identified by selected-area diffraction and dark-field techniques. Considerable dislocation interaction with the precipitates is evidenced by the bowing of dislocations whose ends are pinned by adjacent precipitates and by loops generated by the interactions. No subgrains were observed in specimen 1. Specimen 2 exhibited areas where limited low-angle boundary formation had occurred. The subgrains present in specimen 3 were usually in the vicinity of grain and annealing twin boundaries. Additional results of this work will be presented in future reports.

PLANS FOR FUTURE WORK

The substructure study will be completed for the creep specimens from the second heat of 316 stainless steel.

The substructure of additional 304 and 316 stainless steel creep specimens tested to pre-selected strain levels and after abrupt stress changes will be investigated.

Optical microscopy and bulk density studies of intergranular crack formation resulting from elevated temperature creep and tensile deformation will continue for the 304 stainless steel. Specimens tested at both higher and lower temperatures will be examined.

The investigation of substructure and deformation resulting from hot microhardness testing and the correlation with hot tensile and creep data is planned.

The program to study the substructure of biaxially crept 316 stainless steel tubing will be further developed.

A study of the substructure of creep and tensile tested nickel-base alloys will be developed.

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TABLE I

CREEP AND SUBSTRUCTURE PROPERTIES OF 304 STAINLESS STEEL
HEAT 55697 AT 593°C (1100°F)

Specimen Number	Stress (kg/mm ²)	Creep Rate, ^a $\dot{\epsilon}_m$ (sec ⁻¹)	Total Strain at Rupture (%)	Initial Strain on Loading (%)	Rupture Life (hrs)	Average Subgrain Intercept (10 ⁻³ mm)	Mobile Dislocation Density (cm ⁻²)
R-10	21.0	1.69×10^{-7}	25.5	5.84	230.4	1.2	5.9×10^9
R-11	21.0	1.40×10^{-7}	(14.9)	5.03	(150.0)	-	6.4×10^9
R-12	21.0	-	(10.2)	6.25	(41.3)	-	7.1×10^9
∞ R-13	28.0	3.84×10^{-6}	35.9	11.77	14.0	0.92	n.m. ^c
R-14	28.0 → 21.0	3.12×10^{-6} → 1.42×10^{-8}	(17.9)	12.90	(164.0)	1.1	6.7×10^9
R-15	21.0	-	(4.7)	4.70	(0)	-	1.0×10^{10}

a. $\dot{\epsilon}_m$ is the minimum creep rate.

b. Values in parenthesis are at test termination prior to rupture.

c. n.m. - not measured.

TABLE II

CHEMICAL COMPOSITION OF TYPE 316
STAINLESS STEEL TUBING
(Heat No. 20276)

<u>ELEMENT</u>	<u>WEIGHT PERCENT</u>
C	0.06
Mn	1.51
P	0.018
S	0.011
Si	0.49
Ni	13.40
Cr	17.60
Mo	2.30
Cu	0.12
Co	0.09
N ₂	0.043
B	0.0006

FIGURE 1

COMBINED STRAIN-TIME RESULTS FOR 304 SS CREEP SPECIMENS R-15, R-12, R-11, AND R-10; 593°C (1100°F).

FIGURE 2

A. SPECIMEN R-15

B. SPECIMEN R-12

C. SPECIMEN R-11

D. SPECIMEN R-10

SUBSTRUCTURE OF 304 SS CREEP SPECIMENS R-15,
R-12, R-11, AND R-10; 593°C (1100°F).

FIGURE 3

STRAIN-TIME RESULTS FOR 304 SS CREEP SPECIMENS R-13, R-14, AND R-10; 593°C (1100°F).

FIGURE 4

A. SPECIMEN R-10
UNSTRESSED SHOULDER

B. SPECIMEN R-10
CREPT TO RUPTURE (30,000 PSI)

C. SPECIMEN R-13
CREPT TO RUPTURE (40,000 PSI)

D. SPECIMEN R-14
STRESS ABRUPTLY DECREASED FROM
40,000 PSI TO 30,000 PSI

SUBSTRUCTURE OF 304 SS CREEP SPECIMENS R-13,
R-14, AND R-10, 593°C (1100°F).

FIGURE 5

MICROCRACK LENGTH DISTRIBUTION AS A FUNCTION OF
SQUARE ROOT OF CRACK LENGTH FOR 304 SS TENSILE AND
CREEP-RUPTURE TESTED AT 650°C.

FIGURE 6

MEASURED MICROCRACK DISTRIBUTION AS A FUNCTION OF ANGLE FROM THE SPECIMEN AXIS FOR 304 SS CREEP-RUPTURE TESTED AT 650°C.

FIGURE 7

MICROCRACK AREAL DENSITY AS A FUNCTION OF DISTANCE
TOWARD FRACTURE ZONE FOR 304 SS; SPECIMENS CREEP-
RUPTURE TESTED AT 650°C.

FIGURE 8

FIGURE 9

FIGURE 11

ULTIMATE TENSILE STRESS OR INITIAL APPLIED
CREEP STRESS (KG/MM^2)

RELATIONSHIP BETWEEN $V/\epsilon T$ AND STRESS FOR
304 SS AT 650°C .

FIGURE 12

CIRCUMFERENTIAL STRAIN VERSUS TIME FOR 316 SS TUBULAR SPECIMENS AT 650°C.

FIGURE 13

TYPE 316 SS TUBULAR SPECIMENS (AS-RECEIVED),
ANNEALED; BIAXIAL CREEP TESTED AT 650°C IN
ARGON FOR ~ 1500 HOURS.

SPECIMENS (TOP TO BOTTOM):

1. HOOP STRESS = 13.5 KG/MM²
2. HOOP STRESS = 15.6 KG/MM²
3. HOOP STRESS = 18.0 KG/MM²

(SPECIMENS SHOWN 0.8 ACTUAL SIZE)

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