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METALLOGRAPHIC AND FRACTOGRAPHIC OBSERVATIONS
OF POSTTEST CREEP-FATIGUE SPECIMENS
OF WELD-DEPOSITED TYPE 308 CRE STAINLESS STEEL

by

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ABSTRACT

Type 308 CRE stainless steel weld specimens were subjected to metallographic and fractographic analysis after failure in elevated-temperature (593°C) creep-fatigue tests. The failure mode for specimens tested under continuous-cycle fatigue conditions was predominantly transgranular. When the test cycle was modified to include a hold time at the maximum tensile strain, the failure mode became predominantly interphase. Sigma phase was observed within the delta-ferrite regions in the weld. However, the presence of sigma phase did not appear to affect the failure mode.

I. INTRODUCTION

Type 308 stainless steel weld metal with controlled residual elements (CRE's)¹ is used to join Type 304 austenitic stainless steels. These materials are under investigation to determine their suitability for use in the main steam piping and reactor vessels in power-generation plants associated with the Liquid Metal Fast Breeder Reactor (LMFBR) program. In the present investigation, specimens of different orientations and locations within a single weld were subjected to elevated-temperature (593°C) low-cycle fatigue testing with various tension hold times and two different fully reversed total strain ranges. The intent was to characterize the failure mode and to correlate that mode with the test conditions and with microstructural changes. The results indicate that transgranular failure occurs under low-cycle fatigue conditions. However, if the test cycle is modified to include a hold time at the maximum tensile strain, the failure results from interphase separation.

II. EXPERIMENTAL PROCEDURE

Specimens for fatigue testing are from a single shielded-metal-arc (SMA) weld produced by Combustion Engineering. The double U-groove weld joined two Type 304 stainless steel base-metal plates that were 60 mm thick. The weld required approximately 40 passes to complete. Chemical

analysis of the weld metal is given in Table I. The weld was sectioned and machined into specimens with 5.08-mm-dia gauge sections. Specimens both longitudinal and transverse with respect to the weld were produced from the center, surface, and an intermediate location within the weld (see Fig. 1). The surface of the gauge section was polished to reduce crack initiation at surface scratches. All specimens were aged in argon for 3.6×10^6 s at 593°C before fatigue testing.

TABLE I. Chemical Analysis of Type 308 CRE Weld Metal

Element	wt %	Element	wt %
C	0.051	Nb + Ta	<0.01
Mn	1.87	Ti	0.06
P	0.042	Co	0.07
S	0.009	Cu	0.17
Si	0.60	B	0.007
Cr	20.10	V	0.10
Ni	10.14	N	0.042
Mo	0.24

(a) LONGITUDINAL SPECIMENS

(b) TRANSVERSE SPECIMENS

(c) SPECIMEN DETAILS

Fig. 1

SMA Type 308 CRE Stainless Steel Weld Metal
Test Specimens. All dimensions are in mm.
Neg. No. MSD-64600.

The apparatus for fatigue testing is described in detail in Ref. 2. Specimens were tested in air at 593°C. The temperature, maintained by induction heating, was uniform to $\pm 4^\circ\text{C}$ over the entire gauge section. Strain rates were constant at $4 \times 10^{-3} \text{ s}^{-1}$. Tests were strain-controlled with maximum strains of 0.25 or 0.50% both in tension and compression ($\Delta\epsilon_t = 0.50$ or 1.00%). Hold times of 0, 600, or $1.08 \times 10^4 \text{ s}$ at the maximum tensile strain were used. Because previous investigations of wrought Type 304 stainless steel³ have demonstrated the greater influence of tensile hold time on failure mode, no compressive hold times were used. Table II lists the test conditions and failure times for the specimens examined in the present investigation.

TABLE II. Specimens, Test Conditions, and Failure Times

Specimen Number	Location and Orientation ^a	Strain Range $\Delta\epsilon_t$, %	Tension Hold Time, s	Cycles to Failure, N_f	Time to Failure, s
CE7L1	LS	1.00	0	9102	4.55×10^4
CE36L1	LS	1.00	1.08×10^4	1403	1.51×10^7
CE36L3	LC	1.00	6.0×10^2	1944	1.18×10^6
CE7T22	TI	1.00	6.0×10^2	1288	7.78×10^5
CE89TS1	TS	0.50	0	11,996	3.00×10^4
CE89LI2	LI	0.50	0	35,716	8.92×10^4
CE89TS2	TS	1.00	0	2441	1.22×10^4
CE87TC	TC	1.00	0	1709	8.53×10^3

^aSee Fig. 1 for nomenclature.

After failure of the weld specimens, the fracture surfaces were examined both optically and by scanning-electron microscopy. In addition, the specimens were examined metallographically to reveal microstructural features and any secondary cracking that may have occurred on the surface of the gauge section or on the fracture surface.

III. RESULTS

The microstructure of the weld area is shown in Fig. 2. The primary austenite phase is separated into long columnar regions by the secondary delta-ferrite phase. Higher magnification (see Fig. 3) shows the secondary ferrite region with carbide precipitates at the interphase boundaries as identified by previous investigators.⁴ In another specimen (Fig. 4), the formation of sigma phase within the ferrite region is apparent.⁵ The transformation of delta ferrite to sigma phase was most complete in the longest-lived test specimen. However, the extent of the transformation was not uniform in any of the specimens, and islands of sigma phase were evident, even in the specimens with the shortest test periods.

Fig. 2

Microstructure of SMA Type 308 CRE Stainless Steel Weld Metal. Electrolytic etch, 10% oxalic acid. (a) Coordinate axes; (b) parallel to 2-3 plane; (c) parallel to 3-1 plane; (d) parallel to 1-2 plane. Neg. No. MSD-64599.

Fig. 3

Microstructure of Specimen CE89L12. Murakami's reagent. Secondary ferrite phase (δ) decorated with carbides (c) is visible. Neg. No. MSD-65444.

Fig. 4

Microstructure of Specimen CE36L1. Murakami's reagent. Secondary ferrite phase (δ) has undergone partial transformation to sigma phase (σ). Some phase-boundary carbides (c) are still visible. Neg. No. MSD-65435.

Further substantiation of the presence of sigma phase was sought through magnetic etching.⁵ All specimens contained a ferromagnetic phase (delta ferrite). The ferrite phase appeared discontinuous when observed by the magnetic-etching technique. Presumably, the ferrite regions are separated by the growing sigma phase. These microstructural changes are consistent with those observed in earlier studies of Type 308 CRE stainless steel weld metal.^{4 5}

Metallographic investigation of secondary cracking of the test specimens showed some correlation with the amount of tension hold time in the fatigue test. Specimens tested at 0- or 600-s hold times had secondary cracks both at the fracture surface and at the surface of the gauge section which propagated predominantly in a transgranular fashion (as shown in Figs. 5 and 6). Cracks appeared to initiate at interphase boundaries. One transverse specimen (CE7T22), which contained some base metal within the gauge section, clearly showed cracking at the grain boundaries in the base metal, but transgranular cracking in the weld area (see Figs. 7 and 8).

Fig. 5
Secondary Crack at Fracture Surface of
Specimen CE34B3. Murakami's reagent.
Neg. No. MSD-65434.

Fig. 6
Secondary Cracks at Fracture Surface
of Specimen CE89TS1. Murakami's
reagent. Neg. No. MSD-65433.

Fig. 7

Intergranular Cracks in Base-metal Region of Specimen CE7T22. Electrolytic etch, oxalic acid. Neg. No. MSD-65432.

Fig. 8

Transgranular Secondary Cracks at Fracture Surface of Specimen CE7T22. Electrolytic etch, oxalic acid. Neg. No. MSD-65445.

The 1.08×10^4 -s hold-time specimen showed interphase cracking, even in the weld area. This was also the only specimen in which internal damage was visible in addition to cracking initiated at the surface (see Fig. 9). The internal cracks were at interphase boundaries.

Another striking difference among the failed specimens was in the appearance of the fracture surface. All the surfaces were somewhat irregular, and in most cases the columnar structure of the weld was evident on visual examination at low magnification ($\sim 10\times$). Those specimens tested with no tension hold time had fracture surfaces that were smoothly sloped at angles of $45\text{--}65^\circ$ with the tensile axis. Those specimens tested at 600-s hold times had irregular surfaces with no definable slope. The 1.08×10^4 -s hold-time test produced a fracture surface almost perpendicular to the tensile axis (see Fig. 10).

Fig. 9. Crack at Surface of Gauge Section and Internal Cracking in Specimen CE36L1. Murakami's reagent. Neg. No. MSD-65446.

Fig. 10

SEM Macrographs of Fracture Surfaces of Test Specimens. W indicates weld-solidification direction; I indicates initiation site. (a) CE87TC (no tension hold time); (b) CE7T22 (600-s tension hold time); (c) CE36L1 (1.08×10^4 -s tension hold time). Neg. No. MSD-65450.

The difference among failure modes for different tension hold times was further exhibited by the appearance of the fracture surface at higher magnifications. Striations typical of ductile fatigue failure were visible on the fracture surface of the zero-hold-time specimens. Ductile tearing was also evident in the fast-fracture region of these surfaces. In one specimen (CE87TC), a continuous series of about 600 striations with spacings of 0.8-30 μm was visible over ~ 2 mm. Figure 11 shows the dependence of crack length on the number of test cycles. The orientation of the propagating crack front bore no discernible relationship to the solidification direction of the weld (see Figs. 12 and 13). Less extensive series of striations could be seen in all the other zero-hold-time specimens (see Fig. 14). Striations were also visible in the 600-s hold-time specimens. However, their appearance was less regular and they were frequently separated by surface cracks (as shown in Figs. 15 and 16).

Fig. 11

Crack Length vs Number of Cycles for Specimen CE87TC, Neg. No. MSD-65479.

Fig. 12

SEM Fractograph of Specimen CE87TC (No Tension Hold Time) Showing a Portion of the Fatigue Striations. Crack-propagation direction is toward the lower right. Weld-solidification direction is approximately vertical. Neg. No. MSD-65436.

Fig. 13

SEM Fractograph Specimen CE87TC (No Tension Hold Time) Showing Ductile Tearing in Fast-fracture Region. Neg. No. MSD-65437.

Fig. 14

SEM Fractograph of Specimen CE89L12 (No Tension Hold Time) Showing Fatigue Striations (Upper Left) and Ductile Tearing in Fast-fracture Region (Lower Right). Neg. No. MSD-65438.

Fig. 15

SEM Fractograph of Specimen CE36L3 (600-s Tension Hold Time). Irregular striations and secondary cracks are visible. Neg. No. MSD-65439.

Fig. 16

SEM Fractograph of Specimen CE7T22 (600-s Tension Hold Time). Irregular striations and secondary cracks are visible. Neg. No. MSD-65440.

In contrast to the above behavior, the 1.08×10^4 -s hold-time specimen (CE36L1) exhibited few striations. Considerable secondary cracking was noted on the fracture surface, and the few striations visible were very irregular and were frequently separated by secondary cracks (shown in Fig. 17a). Portions of the fracture surface clearly showed interphase separation (see Fig. 17b). Much of the fracture surface was featureless (see Fig. 17c).

(a)

(b)

(c)

Fig. 17

SEM Fractographs of Specimen CE36L1 (1.08×10^4 -s Tension Hold Time). (a) Irregular striations and secondary cracks; (b) interphase separation; (c) featureless region near center of fracture surface. Neg. Nos. MSD-65441, -65442, and -65443.

An energy-dispersive X-ray spectrum from regions on opposite sides of a secondary crack on the fracture surface of the same specimen showed different relative amounts of iron and chromium, indicating different phases in the two regions. An X-ray spectrum from two sides of a secondary crack on the polished surface of the same specimen mounted for metallography yielded similar results.

IV. DISCUSSION

The results of this investigation show that the failure mode of Type 308 CRE weld specimens is predominantly transgranular in specimens subjected to low-cycle fatigue testing with no tensile hold time. When the test conditions are altered to include a hold time at the maximum tensile strain, the crack propagation becomes predominantly interphase.

These results should be compared with two previous investigations. A study, similar to the present one, of 16-8-2 stainless steel weld metal also showed a change in failure mode from transgranular to interphase separation when a tensile hold time was added to the fatigue test cycle.⁶ However, elevated-temperature creep testing of Type 308 weld metal both with and without CRE resulted in interphase separation in the standard weld metal, but predominantly transgranular shear failure in the CRE weld metal.⁷ Where interphase separation was observed, it occurred along sigma-phase/austenite boundaries. The latter investigators also observed crack propagation through sigma particles.⁴ In the present investigation, crack propagation through sigma particles was not seen. Nor was it possible, because of the fineness of the microstructure, to be certain whether intergranular cracking occurred through the secondary phase (sigma or delta ferrite) or between the austenite and the secondary phase.

In the present investigation, interphase separation occurred in the specimen with the longest test time (measured in seconds, not cycles). That specimen also had the most extensive sigma-phase formation. The correlation of sigma-phase formation with the change in failure mode suggests that the microstructural changes might be the cause of the transition from transgranular to interphase cracking. That is unlikely to be the case, however, because in the shorter-lived test specimens, even in regions with extensive sigma-phase formation, the propagating crack showed essentially no interaction with the sigma phase. Thus, the increased development of sigma phase in the longest hold-time specimen apparently cannot be the principal reason for the change in failure mode.

Another possibility is that the age of the specimens, rather than the nature of the strain cycle, is the determinant of the failure mode. Some changes, possibly atmospheric effects or segregation at the interphase boundaries, may be occurring undetected by the techniques used in the present

investigation. The specimens tested at the 0.50% strain range had longer lifetimes than those tested at the 1.00% strain range. The failure modes under the two strain ranges were not observed to be different. However, the times to failure were not extended as much by reduction of the strain from 1.00 to 0.50% as by the addition of the tension hold time. Studies by Cheng and Diercks³ and by Majumdar and Maiya⁸ on wrought Type 304 stainless steel showed correlation of the failure mode with the nature of the test cycle, rather than with the duration of test. That evidence, together with the microstructural observations in the present investigation, indicates that it is indeed the addition of the tension hold time, rather than changes caused by aging, that results in the alteration in fracture characteristics.

V. CONCLUSIONS

The failure mode of Type 308 CRE weldments under elevated-temperature creep-fatigue conditions depends on the duration of the tension hold time. Under low-cycle fatigue testing with no tension hold time, the failure mode is predominantly transgranular. As the tension hold time is increased, interphase crack propagation predominates. This change in failure mode occurs coincidentally with the formation of sigma phase within the delta-ferrite regions. However, the absence of interaction between the propagating crack and the sigma phase indicates that the presence of the sigma phase cannot be the principal reason for the change in failure mode with increasing tension hold time.

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