## 2 Experimental

Fatigue tests have been conducted on two heats of Type 304 SS in the mill–annealed (MA) as well as MA plus additional heat treatment conditions. The chemical compositions of the heats are given in Table 1. Heat 10285 was heat treated at 600°C for 24 h whereas two heat treatments were used for Heat 30956, 0.67 h at 700°C and 24 h at 700°C. These heat treatments correspond to EPR (electrochemical potentiodynamic reactivation) values of ≈16 C/cm$^2$ for Heat 10285,[12] and ≈8 and 30 C/cm$^2$, respectively, for Heat 30956.[13]

Table 1. Composition (wt.%) of austenitic stainless steels for fatigue tests

| Material | Source | C | P | S | Si | Cr | Ni | Mn | Mo |
|---|---|---|---|---|---|---|---|---|---|
| Type 304[a] | Supplier | 0.060 | 0.019 | 0.007 | 0.48 | 18.99 | 8.00 | 1.54 | 0.44 |
| Type 304[b] | Supplier | 0.060 | 0.025 | 0.011 | 0.59 | 18.31 | 8.51 | 1.58 | 0.38 |

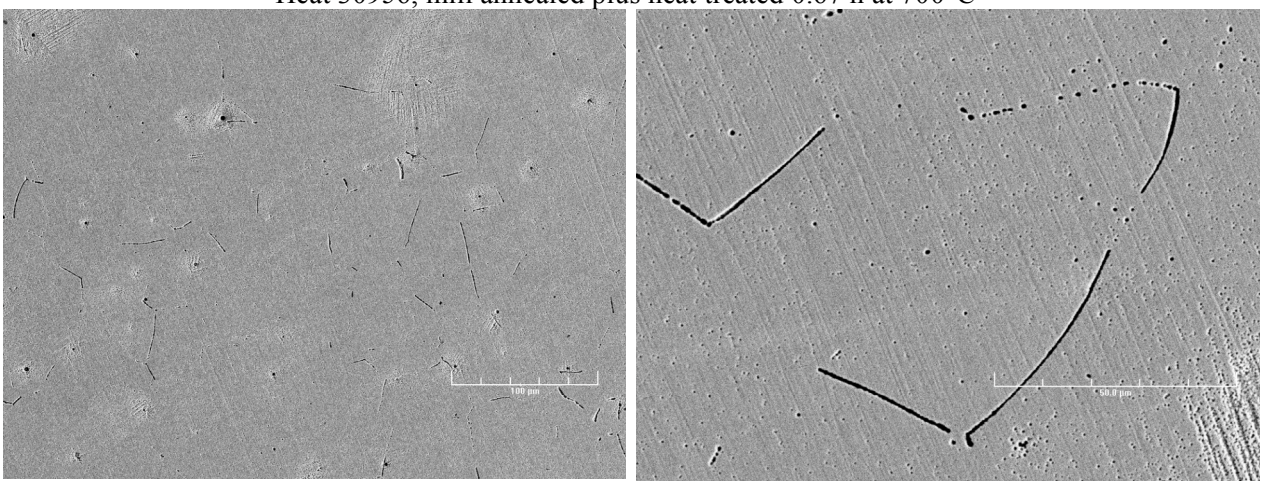
[a]76 x 25 mm bar stock, Heat 30956. Solution annealed at 1050°C for 0.5 h.
[b]25–mm–thick plate, Heat 10285. Solution annealed at 1050°C for 0.5 h.

The metallographic examination of the sensitized alloys was carried out on 10 x 10 x 10–mm specimens that were ground and polished with SiC paper by successively increasing the grade of the paper up to #4000, and subsequently finished with 1–μm diamond paste. Next, the samples were electrochemically etched in a solution of $HNO_3$ (10%) and distilled water at 8 V for ≈15 s. The examination of the microstructure was performed by scanning electron microscopy (SEM) in a JEOL JSM–6400 microscope.

Typical photomicrographs obtained from the sensitized alloys are shown in Fig. 1. Etching revealed a partially sensitized microstructure for the MA Heat 30956 that was heat treated for 0.67 h at 700°C. This is most evident in the higher–magnification photomicrograph (Fig. 1b) showing that sensitization occurred selectively, most likely at curved, high–energy boundaries. A somewhat more uniform degree of sensitization was observed in MA Heat 10285 (heat–treated for 24 h at 600°C), where almost all nontwin boundaries were sensitized. Stringers, also observed in this heat, most likely were parts of the microstructure before the sensitization treatment. Sensitization of Heat 30956 for 24 h at 700°C affected all of the boundaries, especially the curved, high–energy ones; also, it appears that some incoherent twin boundaries were also affected (Fig. 1f).

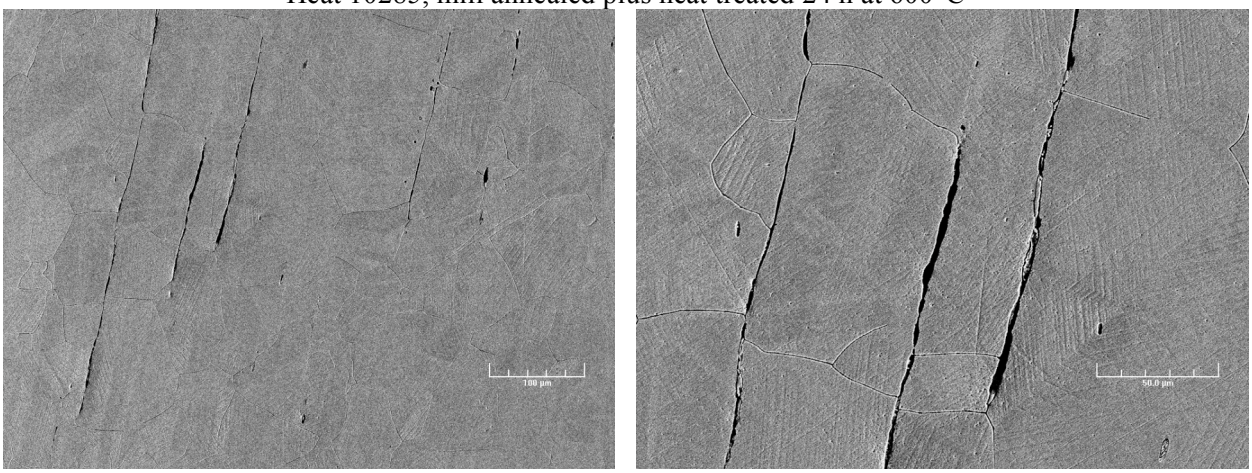
Smooth cylindrical specimens, with a 9.5–mm diameter and a 19–mm gauge length, were used for the fatigue tests (Fig. 2). The test specimens were machined from a composite bar fabricated by electron–beam welding of two 19.8–mm–diameter, 137–mm–long pieces of Type 304 SS bar stock on to each side of an 18.8–mm–diameter, 56–mm–long section of the test material, Fig. 3. The gauge section of the specimens was oriented along the rolling direction for the bar and plate stock. The gauge length of all specimens was given a 1–μm surface finish in the axial direction to prevent circumferential scratches that might act as sites for crack initiation.

Heat 30956, mill annealed plus heat treated 0.67 h at 700°C



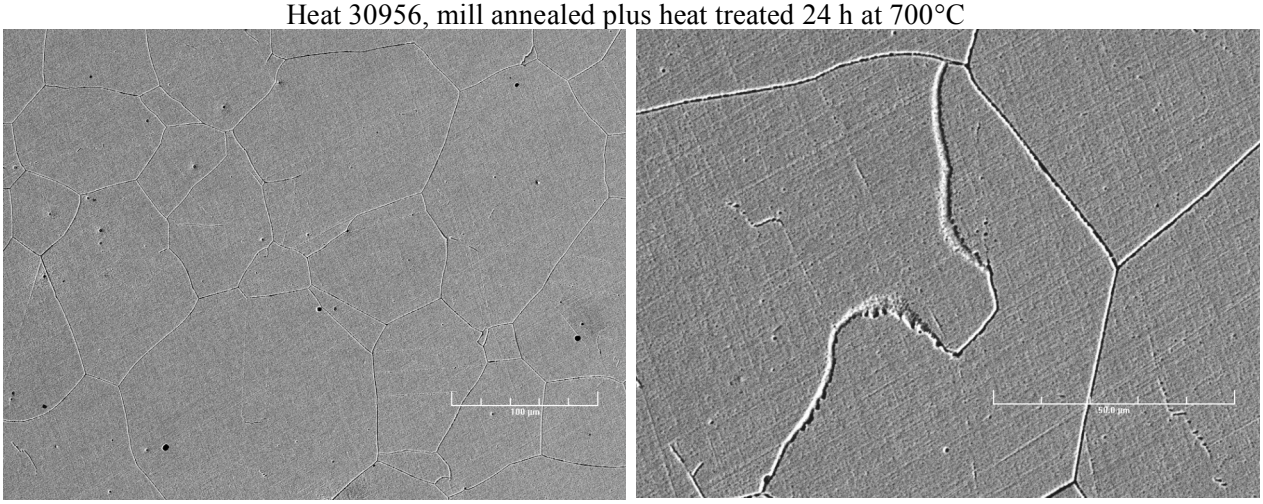
(a) (b)

Heat 10285, mill annealed plus heat treated 24 h at 600°C



(c) (d)

Heat 30956, mill annealed plus heat treated 24 h at 700°C



(e) (f)

Figure 1. Typical microstructures observed by SEM, showing degree of sensitization for alloys used in this study: (a), (c), (e), low magnification; (b), (d), (f), high magnification.

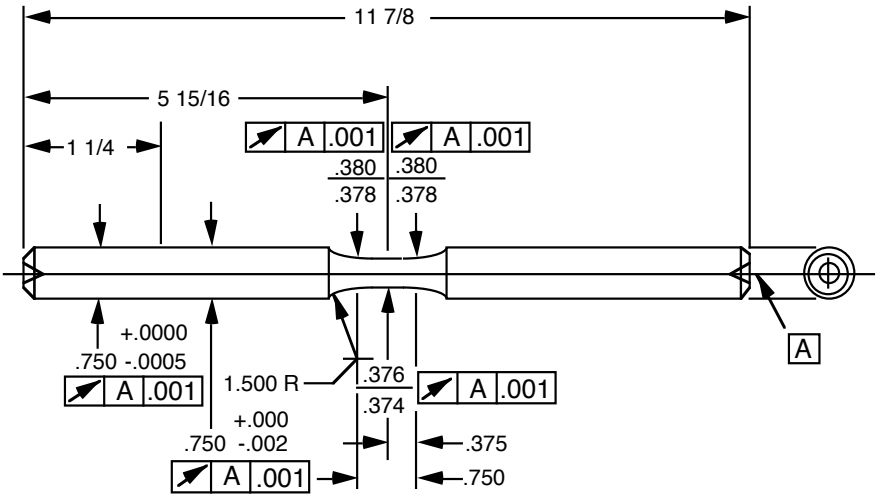



Figure 2. Configuration of fatigue test specimen (all dimensions in inches).

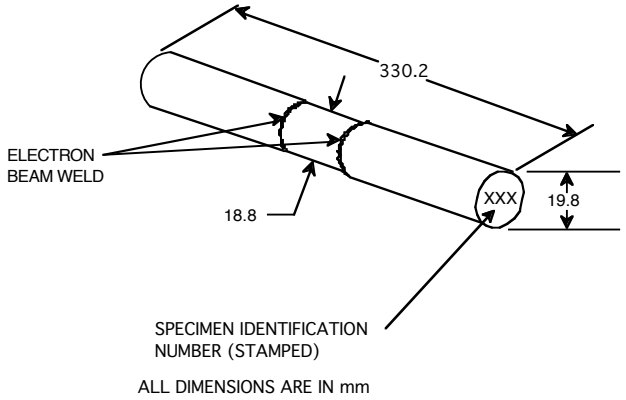



Figure 3. Schematic diagram of electron–beam–welded bar for machining A302–Gr B fatigue test specimens.

Tests in water were conducted in a 12–mL autoclave (Fig. 4) equipped with a recirculating water system that consisted of a 132–L closed feedwater storage tank, Pulsafeeder™ high–pressure pump, regenerative heat exchanger, autoclave preheater, test autoclave, electrochemical potential (ECP) cell, back-pressure regulator, ion exchange bed, 0.2–micron filter, and return line to the tank. Water was circulated at a rate of 10–15 mL/min. Water quality was maintained by circulating water in the feedwater tank through an ion exchange cleanup system. An Orbisphere meter and CHEMetrics™ ampules were used to measure the DO concentrations in the supply and effluent water. The redox and open–circuit corrosion potentials, respectively, were monitored at the autoclave outlet by measuring the ECPs of platinum and an electrode of the test material, against a 0.1–M KCl/AgCl/Ag external (cold) reference electrode. The measured ECPs, $E_{(meas)}$ (mV), were converted to the standard hydrogen electrode (SHE) scale, $E_{(SHE)}$ (mV), by solving the polynomial expression[14]

$$E_{(SHE)} = E_{(meas)} + 286.637 - 1.0032(\Delta T) + 1.7447 \times 10^{-4}(\Delta T)^2 - 3.03004 \times 10^{-6}(\Delta T)^3, \quad (2)$$

where $\Delta T$(°C) is the temperature difference of the salt bridge in a 0.1–M KCl/AgCl/Ag external reference electrode (i.e., the test temperature minus ambient temperature). A description of the test facility has been presented earlier.[6,15]

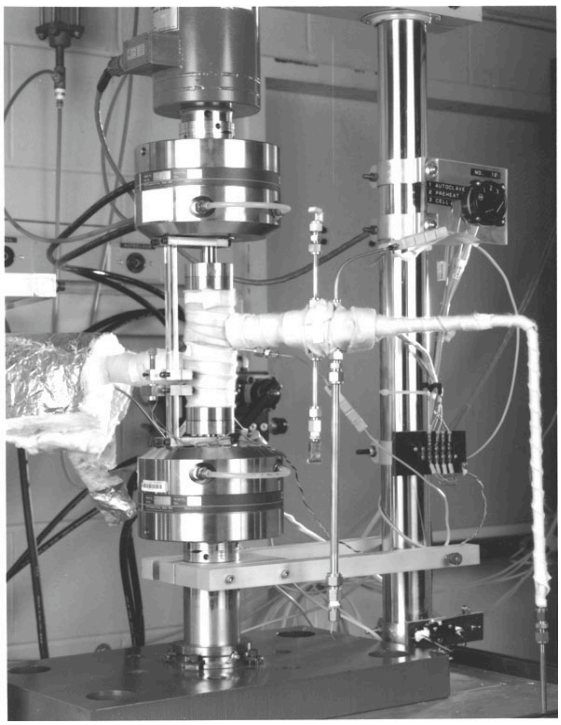


Figure 4.
Autoclave system for fatigue tests in water.

Boiling water reactor conditions were established by bubbling $N_2$ that contained 1–2% $O_2$ through deionized water in the supply tank. The deionized water was prepared by passing purified water through a set of filters that comprise a carbon filter, an Organex–Q filter, two ion exchangers, and a 0.2–mm capsule filter. Water samples were taken periodically to measure pH, resistivity, and DO concentration. When the desired concentration of DO was attained, the $N_2/O_2$ gas mixture in the supply tank was maintained at a 20–kPa overpressure. After an initial transition period, during which an oxide film developed on the fatigue specimen, the DO level and the ECP in the effluent water remained constant. Test conditions are described in terms of the DO in effluent water.

Simulated PWR water was obtained by dissolving boric acid and lithium hydroxide in 20 L of deionized water before adding the solution to the supply tank. The DO in the deionized water was reduced to <10 ppb by sparging it with either pure $N_2$ or a mixture of $N_2$ plus 5% $H_2$. A vacuum was drawn on the tank cover gas to speed deoxygenation. After the DO was reduced to the desired level, a 34–kPa overpressure of $H_2$ was maintained to provide ≈2 ppm dissolved $H_2$ (or ≈23 $cc^3$/kg) in the feedwater.

All tests were conducted at 289°C, with fully reversed axial loading (i.e., R = –1) and a sawtooth waveform. During the tests in water, performed under stroke control, the specimen strain was controlled between two locations outside the autoclave. Companion tests in air were performed under strain control with an axial extensometer. During the test, the stroke at the location used to control the water tests was recorded. Information from the air tests was used to determine the stroke required to maintain constant strain in the specimen gauge. To account for cyclic hardening of the material, the stroke that was needed to maintain constant strain was gradually increased during the test, based on the stroke measurements from the companion strain–controlled tests. The fatigue life $N_{25}$ is defined as the number of cycles for tensile stress to decrease 25% from its peak or steady–state value.

Following testing, ≈10–mm–long sections that contained the fracture surface were cut from the gauge length. These were further stripped of oxides by boiling in a 20 wt.% NaOH and 3 wt.% $KMnO_4$ solution, followed by boiling in a 20 wt.% $(NH_4)_2C_6H_6O_7$ solution. The samples were examined by SEM. Special attention has been paid to crack morphology at the sites of initiation on the fracture surface, and the occurrence of striations. Also, lateral surfaces were inspected to determine the morphology of lateral cracks.