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doi:10.1017/S1431927611005976

Microsc. Microanal. 17 (Suppl 2), 2011

Precipitation and "Sensitization" of Type 304L Stainless Steel: Correlation of the ASTM A262 Practice A Test with Analytical Electron Microscopy

B. D. Miller and M. G. Burke

Bechtel Marine Propulsion Corporation, Bettis Laboratory, PO Box 79, West Mifflin, PA 15122

Cr-rich intergranular (IG) precipitates, generally $M_{23}C_6$ carbides, form in Type 304/304L austenitic stainless steels during thermal treatment in the temperature range from ~500 to 900°C. During precipitation, the level of Cr at the grain boundaries and adjacent to the carbides may decrease, depending on the aging temperature. If the Cr content decreases to <~12 wt.%, the grain boundaries are considered "sensitized" and susceptible to intergranular attack. Frequently, the ASTM A262 Practice A test method, which involves an electrolytic etch using dilute oxalic acid for 90 seconds with a current of 1 A/cm^2 [1], is used for determining susceptibility to IG attack. These etch conditions are known to preferentially attack Cr carbides [2] and leave a "dual" or "ditched" grain boundary structure that is visible using optical microscopy; in the absence of Cr carbides, the grain boundaries are "stepped". The Practice A standard requires that materials exhibiting the "ditched" structure undergo the ASTM A262 Practice E test; however, grain boundary Cr depletion has been inferred based solely on the Practice A results (for example, [3]).

In this study, as-received Type 304L stainless steel containing 18 ppm boron exhibited a "dual" grain boundary structure following the Practice A test even though the alloy had a low carbon content (~0.02 wt.%). Detailed microstructural characterization utilizing analytical electron microscopy (AEM) coupled with laboratory solution-anneal (~1080°C/30 min + water-quench(WQ)), sensitization (700°C/2hrs + WQ) and stabilization (900°C/2hrs + WQ) heat treatments were performed to assess the precipitation behavior in this steel. The sensitization and stabilization heat treatments were chosen to nucleate IG precipitates with concomitant Cr-depleted regions and nucleate IG precipitates with reduced grain boundary Cr content, respectively. Following the Practice A etch, the laboratory solution-annealed material exhibited a "stepped" boundary structure while both the sensitized and stabilized materials, presented in Figure 1, were strongly "ditched".

Intergranular, Cr-rich M_2B-type borides, identified by electron diffraction, were detected in the as-received, laboratory solution-annealed, and aged conditions. Semi-quantitative measurements of grain boundary Cr content using energy dispersive x-ray spectroscopy (EDXS), examples are presented in Figure 2, revealed Cr levels from 15 - 17 wt.% in the mill solution-annealed material, with Cr-depleted zones ranging from 10 - 40 nm in extent. Isolated IG borides were observed in the laboratory solution-annealed material and the grain boundary Cr content was comparable to that in the surrounding grains (18 - 19 wt.%). In contrast, samples aged at 700°C ("sensitized") exhibited Cr levels in excess of 14 wt.% and concomitant Cr-depleted zones less than 50 nm in extent. Interestingly, despite exhibiting fully "ditched" grain boundaries, Cr levels of ~18 - 20 wt.% Cr with no Cr-depleted zones were detected in specimens aged at 900°C (i.e.,"stabilized"). These results establish that the ASTM A262 Practice A test preferentially attacks Cr-rich IG borides in the absence of a Cr-depleted zone. As such, the Practice A test method can yield a "false-positive" indication of IG susceptibility and should not be solely used to infer grain boundary Cr-depletion.

References
[1] 2007 Annual Book of ASTM standards, Vol. 1.03
[2] M.A. Streicher, *J. Electrochemical Soc.* **106** (1959) 161-180.
[3] S.C. Bali, et al. *Corrosion*. **65** (2009) 726-740.

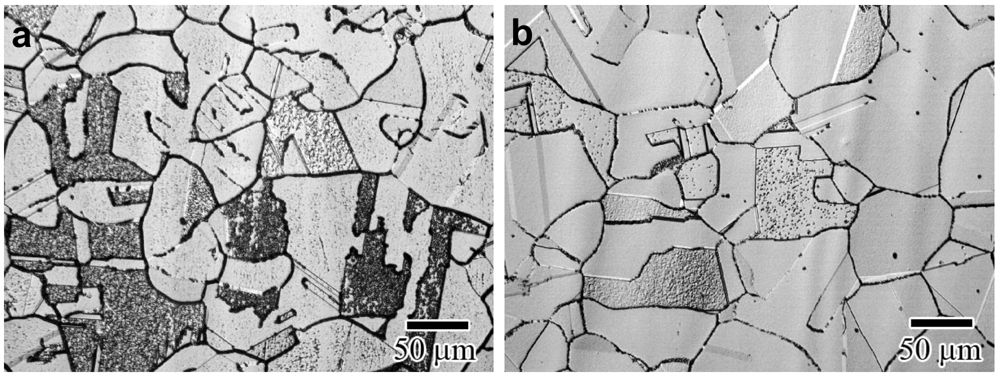

Figure 1. Optical micrographs following the ASTM A262 Practice A test showing "ditched" grain boundaries in both (a) "sensitized" and (b) "stabilized" Type 304L stainless steel.

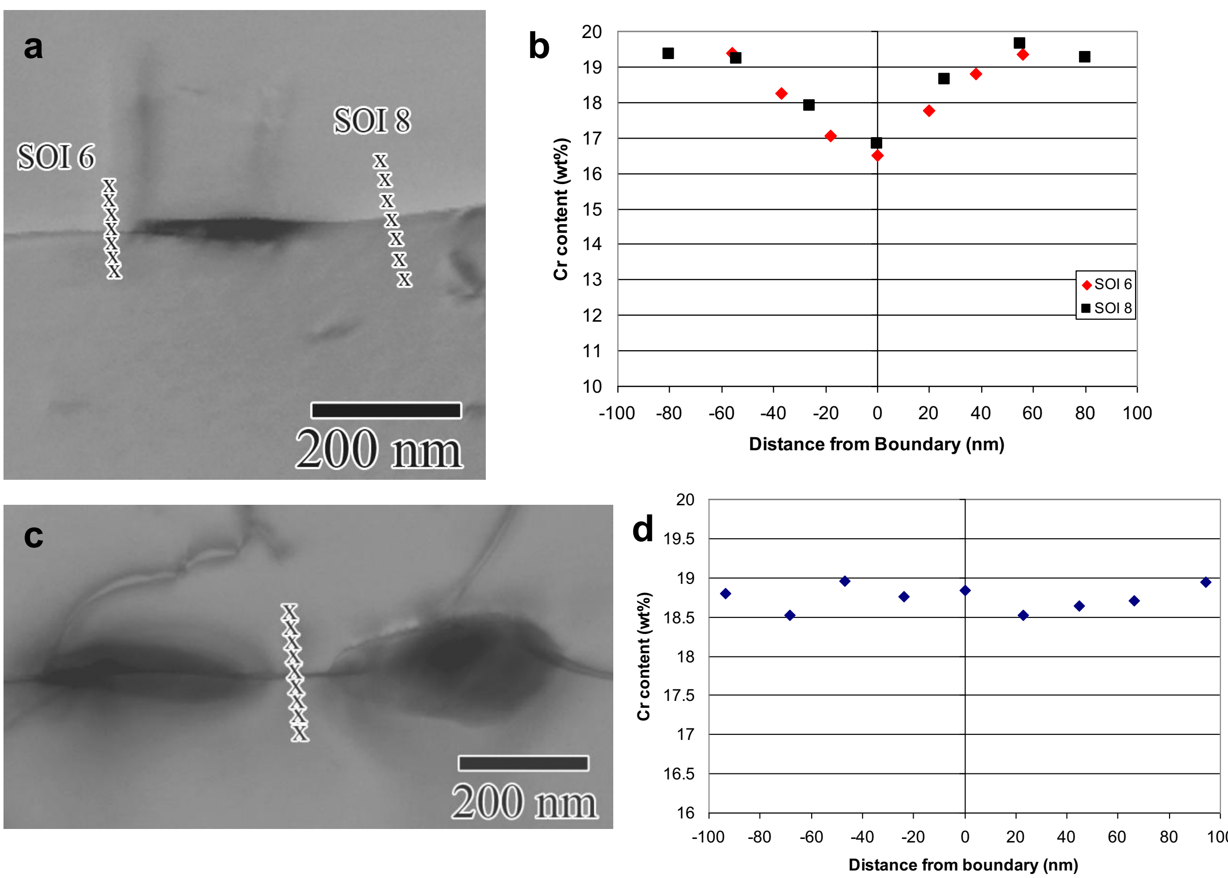

Figure 2. Bright Field-Scanning Transmission Electron Microscopy (STEM) images and associated STEM-EDXS grain boundary Cr concentration profiles for (a), (b) "sensitized" and (c), (d) "stabilized" Type 304L stainless steel.

https://doi.org/10.1017/S1431927611005976 Published online by Cambridge University Press