Title: INTERFACE CHARACTERIZATION TECHNIQUES FOR 304L STAINLESS STEEL RESISTANCE UPSET WELDS

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Interface Characterization Techniques
for 304L Stainless Steel Resistance
Upset Welds

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Abstract

In an effort to better characterize and classify austenitic stainless steel resistance upset welds, standard methods have been examined and alternative methods investigated. Optical microscopy yields subjective classification due to deformation obscured bond lines and individual perception. The use of specimen preparations that better reveal grain boundaries aids in substantiating optical information. Electron microscopy techniques produce quantitative information in relation to microstructural constituents. Orientation Imaging Microscopy (OIM) is a relatively new technique for obtaining objective, quantitative information pertaining to weld integrity, i.e., percent grain boundary growth across the interface.

Introduction

The type of weld investigated is pictured in Figure 1. Here, a 304L stainless steel tube stem is pressed into a fixed 304L SS pressure vessel cap creating an interference fit while a current is applied thus welding the side wall regions.

Characterization of a side bond, resistance upset weld interface presents unique problems related to material deformation in the bond area. Features of interest in weld line classification may be obscured when using optical microscopy with standard specimen preparation techniques. Alternative specimen preparation methods may provide a clearer representation of the microstructure. Other methods, such as electron and orientation imaging microscopies can potentially yield more objective and quantitative information, as well as reveal finer features in the microstructure.

This paper describes several approaches for better characterization of resistance upset welds, thereby aiding in bond line classification.

![Figure 1 - Diagram of the stem in relation to the cap area.](image1)

![Figure 2 - Photos showing the specimen after transverse sectioning.](image2)
Experimental

Specimen Preparations

- Weld samples are sectioned transversely as pictured in Figure 2.
- Specimens are mounted and ground through 800 grit silicon carbide paper.
- For unetched macro photos, specimens are polished with Struers MD MOL cloth using 3 μm aqueous diamond slurry at 40N force for ~4 minutes.
- Prior to etching, specimens are final polished with Struers MD NAP cloth at 25N force for ~1 minute.
- Etchant #1 - specimens are submerged in 100ml of 10% oxalic acid solution and etched electrolytically for 30-45 seconds at 6 volts. Cathode material is 300 series stainless steel mesh. Surface connection is made via a tantalum wire.
- Etchant #2 - specimens are submerged in 100 ml of 60% nitric acid and 40% water solution and etched electrolytically for ~3 minutes at 1.1 volts. Cathode material is 300 series stainless steel mesh. Surface connection is made via a tantalum wire.
- TEM specimens are prepared by sectioning with a diamond saw, grinding to 250 μm thickness, and punching 3 mm discs centering the bond region. Discs are thinned to perforation in a Fischione instrument using a 10% perchloric acid / 90% acetic acid solution at 35 volts, room temperature.
- After etching, no additional preparation is needed for OIM analysis.

Optical Microscopy

- Specimens etched with the oxalic solution are photomicrographed under differential interference contrast (DIC) conditions.
- Specimens etched with the nitric solution are photomicrographed under bright field conditions.
- Unetched, polished (3 μm aqueous diamond slurry) specimens are photomacrographed under dark field conditions.

Electron Microscopy

- TEM imaging, electron diffraction and microchemical analysis are performed on a Phillips CM30 microscope equipped with a Kevex EDS detector.
- OIM is performed on a Phillips XL30 FEG with a CCD camera coupled to a silicon graphics work station that employs TSL software.

Results and Discussion

Due to the very short amount of time that the current is applied, material deformation is still evident in cross-sectional optical micrographs as shown in Figures 3 and 4. These figures also illustrate two optical preparation methods. In Figures 3A and 4A, the oxalic etch (commonly used on many austenitic stainless steels) is applied, while in Figures 3B and 4B the specimen has been polished to 3 μm and left unetched. While both methods reveal bond line deformation and the forging characteristics of both stem and cap, the unetched, polished technique more clearly defines the bond line and material processing information.

When these transverse specimens are etched with the oxalic solution, a relief-type surface is produced, i.e., grain interiors are attacked according to their crystallographic orientations. Grains can be optically differentiated employing DIC conditions. One disadvantage of using this etchant is the lack of grain boundary definition, especially along the bond line. A more useful etchant for grain boundary definition is the 60% nitric solution as shown in Figure 5. The nitric etchant primarily attacks the grain boundaries in a uniform fashion, producing a surface with less relief. Grain boundaries are revealed all along the bond line, even the tiny recrystallized grains in the more deformed bottom region of the weld.

Figure 6 and 7 illustrate a sample weld region after the oxalic etch. One notable characteristic is the numerous flow lines in the vicinity of the weld, actually obscuring detection of the bond line. These lines primarily result from deformed grain boundaries decorated with fine precipitates or areas where precipitates were attacked. Figure 8 shows this decoration in the base metal structure. Scanning electron microscopy (SEM) yielded no useful information regarding the nature of the precipitates, so TEM was performed. Energy dispersive spectroscopy (EDS) analysis revealed the particles to be chromium rich, indicating chromium carbides. Electron diffraction patterns were consistent with the crystallographic structure of the M23C6 phase. Figure 9 contains TEM photos of the carbides in both the weld interface region and in the base metal. Tiny, recrystallized grains, evident in these photos of the weld region, are most likely formed by dynamic recrystallization, resulting from the high degree of deformation and a combination of adiabatic and resistance heating. The oxalic etchant is a more useful
choice in this instance due to its ability to reveal location and degree of carbides.

One of the primary criteria in metallographic analysis for weld classification is percent grain growth across the bond line. The nitric etchant does an excellent job of revealing the grain boundaries along the bond line, but grain growth is still a subjective determination relying on the visual perception of the person performing the analysis. A somewhat newer, more quantitative method called orientation imaging microscopy (OIM) can be used to determine grain growth across the weld interface. This method employs electron backscatter diffraction patterns (EBSPs) for automated determination of crystallographic orientations. A grain map is generated by automated single orientation measurements using a predetermined step size. Orientations of neighboring points are compared, producing delineation of grains. By comparing misorientations along the bond line, a grain growth percentage can be calculated. In Figure 10, an OIM map is shown along with a comparison optical photo. Orientation measurements were taken on both sides and along a specified length of the bond line. Via computer software, these measurements are compared by the computer to generate an orientation map in which the presence or absence of grain growth across the interface can be determined directly from crystallographic orientation, rather than inferred by the appearance of grain boundary lines in the image. Figure 11 contains an OIM map with a bond line appearance indicative of a substandard weld.

Conclusions

Side bond, resistance upset weld classification using optical microscopy characterization techniques is facilitated by use of the nitric etchant. Well defined grain boundaries are essential in determining growth across the bond line. If carbides are of interest, the electrolytic oxalic acid is a better choice.

Preparing the specimen without the use of an etchant, leaving it in 3 μm polished condition and photographing under dark field conditions, can reveal a defined bond line and material processing information.

TEM analysis revealed a dynamically recrystallized structure due to both adiabatic and resistance heating in the weld region. Chromium carbide (M₇C₃) precipitates decorate grain boundaries that form flow lines, obscuring determination of the actual bond line after an oxalic etch.

OIM analysis proved to be a better technique in determining grain growth across the bond line. The objectiveness of comparing orientation points along the bond line is far superior to visual interpretation. Unfortunately, percent grain growth is still a manual calculation.

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References

Figure 3 - Transverse sections illustrating material deformation in the weld region and process history. (3a) electrolytic 10% oxalic etch (3b) unetched, 3 μm aqueous diamond slurry polish.

Figure 4 - Views of one side of the weld. (a) electrolytic 10% oxalic etch, (b) unetched, 3 μm aqueous diamond slurry polish.
Figure 5 - Two separate bond line areas are shown for etchant comparisons. (a) and (c) etched with electrolytic 10% oxalic etchant, (b) and (d) etched with electrolytic 60% nitric etchant.
Figure 6 - Montage of one side of the weld. (electrolytic 10% oxalic etch)

Figure 7 - Increased magnification of the area in figure 6. Note the deformation lines from the faying.
Figure 8 - Cap base metal microstructures. Note decoration of the grain boundaries. (10% electrolytic oxalic etch)

Figure 9 - TEM photos of (a,b) recrystallized grains and carbides (insets) in the weld interface region and (c) carbides in base metal.
Figure 10 - Bond line area depicted photographically and with OIM mapping. Bond line is barely visible in photograph and virtually undetectable in the OIM map.
Figure 11 - OIM map of the weld area with the bond line clearly visible.