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ELEVATED TEMPERATURE EXPOSURE HAS SIGNIFICANT EFFECTS ON THE STRUCTURE OF AUSTENITIC STAINLESS STEEL WELDS

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ABSTRACT

In order to minimize the tendency for hot-cracking in austenitic stainless steel weld metals, the chemical compositions are generally adjusted to produce small amounts of  $\delta$ -ferrite in the microstructure. Generally, levels of ferrite in the range of 2 to 15 FN are acceptable for welds in these materials. This paper describes an investigation of effects of aging and testing at  $593^{\circ}\text{C}$  on the structures of type 308 stainless steel weld metals.

Shielded metal-arc (SMA) welds were made with electrodes formulated by a commercial manufacturer to produce four different concentration levels of  $\delta$ -ferrite. By varying the chromium-to-nickel concentration ratios (within the type 308 limits) in the deposits, weld metals with nominally 2, 5, 10, and 15 FN were produced. Specimens from these materials were aged and creep tested in air at  $593^{\circ}\text{C}$  for times up to 36 Ms. Aged specimens were Charpy impact tested at room temperature. Both creep and impact-tested specimens were then examined metallographically.

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Metallographic procedures included color staining and magnetic etching techniques. Using these techniques, we can identify sigma (common transformation product from ferrite), martensite, and ferrite phases in the microstructure. After about 14 Ms at 593°C most of the original δ-ferrite in the structures had transformed. In the lower ferrite welds (2-5 FN as welded), δ-ferrite transformed to austenite with carbide precipitation, while in the higher ferrite welds (10-15 FN as welded), most of the ferrite transformed to sigma phase. Also, some coarsening of carbides could be observed after longer times at temperature. The impact tested specimens, in most cases, fractured along inter-substructural boundaries (austenite to ferrite, austenite to carbide, or austenite to sigma). In some cases the fractures propagated through individual islands of sigma phase. Creep specimens generally ruptured along austenite-to-sigma phase boundaries for the higher ferrite welds and were associated with carbides in the lower ferrite welds.

#### INTRODUCTION

A significant problem in the production of fully austenitic stainless steel welds is their tendency for hot-cracking and microfissuring. To minimize this tendency the compositions of welding materials are generally modified to produce small amounts of δ-ferrite (usually 2-15 vol %) in the as-welded structure.<sup>1</sup> However, when these materials are exposed to elevated temperatures (500-900°C) for extended periods of time, the ferrite can transform to a hard, brittle phase known as sigma phase.<sup>2</sup> This transformation has been shown to lead to low-ductility creep rupture (and to low strength in some cases) when sufficiently high stresses are applied at elevated temperatures. When this happens rupture occurs along inter-substructural boundaries between the austenite and sigma phases.<sup>3</sup> Our concerns for the effects of varying levels of ferrite on properties and structures of stainless steel

welds exposed to elevated temperatures led us to pursue the program described in this paper.

#### EXPERIMENTAL APPROACH

Type 308 stainless steel SMA welds with varying ferrite levels were produced by a commercial manufacturer. The four different ferrite levels, nominally 2, 5, 10, and 15 FN (Ferrite Number -Welding Research Council standardized measure of magnetic phases), will be referred to as extra-low-, low-, medium-, and high-ferrite welds, respectively. Specimens were machined from welds from each of the four ferrite levels. Part of the specimens were creep tested in air at 593°C for times up to 36 Ms. The other specimens were aged at 593°C in air for varying times up to 36 Ms, and room temperature Charpy impact testing was performed on the aged and some as-received specimens. The measured FN of these specimens decreased very rapidly with aging and testing. Most of the original δ-ferrite (ferromagnetic) in the structures had transformed to nonmagnetic phases after about 14 Ms at 593°C.

Selected creep and impact specimens from the four ferrite levels were examined metallographically. Broken Charpy impact specimens were sectioned in the manner shown in Fig. 1. Ruptured creep specimens were sectioned along their axes. Specimens were ground through 600-grit paper, then polished on Syntron vibratory polishing units<sup>5-7</sup> in two steps. Preliminary polishing was done in a Syntron bowl covered with Pellon cloth PAN-W\* with an abrasive slurry of 0.3-μm alumina in water. Final polishing was done in another Syntron unit with a nylon cloth and an abrasive aqueous slurry of 0.5-μm diamond paste.

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\*Product of Pellon Corp., 1120 Avenue of the Americas, N.Y., N.Y. 10036.

Optical metallographic examination of the specimens used color and magnetic colloid etching techniques. The color etching technique can distinguish austenitic, ferritic, and sigma phases optically.

¶ 1 Each specimen is preheated in water at 95°C and immersed in an alkaline potassium ferricyanide solution etchant (30 g KOH, 30 g  $K_3Fe[CN]_6$ , 100 ml  $H_2O$ ) at 95°C for 5 s. This treatment turns sigma phase brownish red, turns ferrite dark gray, leaves austenite light gray, and turns carbides black. The magnetic colloid etchant<sup>8</sup> is used to determine microscopic patterns of ferromagnetic phases in a structure. Before application of the colloid (and before color etching) the specimens were electropolished to remove any surface effects from the mechanical polishing. With the magnetic technique, Ferrofluid\*--a colloidal dispersion of fine iron oxide in an organic or aqueous fluid--is placed between the polished-and-etched specimen surface and a thin cover glass.

The assembly is then placed on a metallograph and is surrounded by a dc electromagnet (coil). When the coil is energized, the iron particles are attracted to ferromagnetic constituents--ferrite and martensite--in the microstructure. Because the specimens have already been etched by conventional techniques, the ferromagnetic characteristics of microscopic constituents can be defined. With this technique the ferrite and strain-induced martensite have been detected and identified in austenitic stainless steel<sup>8 9</sup>.

#### RESULTS AND DISCUSSION

General microstructures of the low-, medium-, and high-ferrite welds are shown in Figs. 2, 3, and 4, respectively. Microstructures after aging

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\*Product of Ferrofluid Ferrofluidics Corp., 144 Middlesex Turnpike, Burlington, Mass. 01803.

for 18 and 36 Ms at 593°C are also shown in these figures. Because these photomicrographs are black and white, austenite appears very light gray, ferrite is slightly darker gray, and sigma and carbides are black. The unaged microstructures consisted of ferrite and carbides in austenitic matrices. Strain-induced martensite was not found in any of these specimens. However, martensite in austenitic stainless steels is sometimes very difficult to detect and may be masked by other constituents. Finally, in the unaged materials the ferrite islands become continuous at about 10 FN (Fig. 3).

Aging at 593°C had different effects on the microstructure of the extra-low- and low-ferrite welds than on the medium- and high-ferrite welds. For the medium- and high-ferrite welds (Figs. 3 and 4), the measured decrease in FN upon aging resulted primarily from the transformation of ferrite to sigma phase. However, in the low- (Fig. 2) and extra-low-ferrite welds, little of the ferrite phase transformed to sigma. Most of the ferrite formed carbides--probably chromium-rich carbides--which nucleated and grew at the ferrite-austenite boundary, leaving the phase deficient in chromium. This is believed to allow some of the ferrite phase to transform to austenite (Fig. 3c), thus decreasing the measured FN. These transformed areas were not the characteristic brownish-red color of sigma phase, nor was there a visible boundary between these areas and the austenite matrix.

An extra-low-ferrite weld was examined with the magnetic colloid technique to confirm that the above transformation was indeed to austenite (Fig. 5). Several of the ferrite islands that typically show a ferromagnetic response (black hazy areas) were no longer ferromagnetic after aging (Fig. 5b) for 18

Ms. Note that the number of areas that attract the Ferrofluid is larger in Fig. 5a than in Fig. 5b. However, the amount of Ferrofluid attracted to each area is greater in Fig. 5b. This does not mean, however, that the ferromagnetic response or the size of these areas is greater for the aged materials (Fig. 5b). This effect results from the fact that for a given amount of Ferrofluid on each specimen surface, more Ferrofluid is available for each ferromagnetic area when fewer such areas are present. This technique detected no martensite in the structure. Transformation to austenite and carbide precipitation also occurred in the medium- and high-ferrite welds, but not as much as in the lower ferrite welds.

In all specimens some ferrite was still present after aging for 36 Ms. The transformation to sigma phase in the high- and medium-ferrite weld began very quickly and was observed after only 8 h in one of the creep specimens (Fig. 6). Small stringers of ferrite transformed to carbides, austenite, or sigma phase more readily than did the larger islands of ferrite. When the ferrite islands transformed to sigma phase, they most often became more agglomerated than the original ferrite (Fig. 4c).

Micrographs transverse to the fracture surface of the low- and high-ferrite impact specimens are shown in Figs. 7 and 8. Fracture profiles for the extra-low-ferrite welds resembled those shown in Fig. 7, and the medium-ferrite welds resembled those in Fig. 8. For all unaged specimens the fractures did not appear to relate directly to the substructure. Also, no secondary cracks were found in any of the unaged specimens. However, the substructure significantly affected the fractures in the aged specimens. In the aged low- and extra-low-ferrite welds, fractures appeared to propagate more readily

along carbide-austenite boundaries (Fig. 7). In the aged medium- and high-ferrite welds, fractures propagated more readily along the austenite-sigma phase boundaries (Fig. 8). Secondary cracks were found in all aged specimens.

Similar results were found for the ruptured creep specimens (Figs. 6 and 9) tested at 593°C. Ruptures in the lower ferrite welds were associated with austenite and carbide precipitation, while ruptures in higher ferrite welds were associated with transformation to sigma phase. A classic creep void forming along austenite-sigma phase boundaries in a medium-ferrite weld (tested for 28 Ms at 593°C) is shown in Fig. 10.

Therefore, two mechanisms appear to operate in these welds upon exposure to elevated temperatures. For the lower ferrite welds, the transformation of ferrite to carbides and austenite predominates, while for the higher ferrite welds, transformation of ferrite to sigma phase predominates. We should again point out that both transformations occur to some extent in all exposed specimens, but that the above transformation predominated for the materials in this study.

## CONCLUSIONS

The following conclusions were drawn from this evaluation:

1. In the as-welded structures, ferrite morphology became continuous at a level of approximately 10 FN.
2. Decreases in measured FN with aging at 593°C resulted predominately from transformation of ferrite to sigma phase for the higher ferrite welds and of ferrite to austenite and carbides for the lower ferrite welds.
3. Morphology changes occurred with the transformation of ferrite to sigma phase. The sigma islands were more agglomerated than the original ferrite.
4. For all specimens in the as-welded condition, Charpy impact fractures did not appear to be directly related to the substructure, and no secondary fractures were found.
5. For specimens aged at 593°C, Charpy impact fractures propagated along austenite-to-sigma phase boundaries for welds in the 9-15 FN range, and fractures were associated with carbides for welds in the 2-5 FN range.
6. Secondary fractures were found in all aged Charpy impact specimens investigated.
7. Similar results were found from the investigation of ruptured creep specimens. That is, the ruptures propagated along austenite-to-sigma phase boundaries for the higher ferrite welds and were associated with carbides for the lower ferrite welds.

Fig. 1. Locations for Photomicrographs of General Microstructures and Fracture Profiles of Tested Charpy Impact Specimens.

Fig. 2. Micrographs Showing the Predominance of Ferrite-to-Austenite Phase Transformation in Low-Ferrite Type 308 Stainless Steel Welds Exposed at 593°C in Air. Phases: Austenite,  $\gamma$ ; Ferrite,  $\delta$ ; Transformed Austenite,  $\gamma_t$ ; Carbide, C; Sigma,  $\sigma$ . (a) As welded: 4.0 FN. (b) Exposed 18 Ms 1.9 FN. (c) Exposed 36 Ms 1.4 FN.

Fig. 3. Micrographs Showing the Predominance of Ferrite-to-Sigma Phase Transformation in Medium-Ferrite Type 308 Stainless Steel Welds at 593°C in Air. Phases: Austenite,  $\gamma$ ; Ferrite,  $\delta$ ; Carbide, C; Sigma,  $\sigma$ . (a) As welded: 15.8 FN. (b) Exposed 18 Ms 3.4 FN. (c) Exposed 36 Ms 2.8 FN.

Fig. 4. Micrographs Showing the Ferrite-to-Sigma Phase Transformation Predominating in High-Ferrite Type 308 Stainless Steel Welds Exposed at 593°C (1100°F) in Air. Phases: Austenite,  $\gamma$ ; Ferrite,  $\delta$ ; Carbide, C; Sigma,  $\sigma$ . (a) As welded: 15.8 FN. (b) Exposed 5000 h 3.4 FN. (c) Exposed 10,000 h 2.8 FN.

Fig. 5. Ferrite-to-Austenite Phase Transformation as Detected by Magnetic Colloid Etching in Extra-Low-Ferrite Type 308 Stainless Steel Welds. Key: Austenite,  $\gamma$ ; Ferrite,  $\delta$ ; Carbide, C; Sigma,  $\sigma$ ; Magnetic Colloid, m; Transformed Austenite,  $\gamma_t$ . (a) As welded: 1.8 FN. (b) Exposed 18 Ms 0.3 FN.

Fig. 6. Creep Ruptures Associated with Transformed Sigma Phase in High-Ferrite Type 308 Stainless Steel Welds Tested at 593°C in Air. Key: Austenite,  $\gamma$ ; Ferrite,  $\delta$ ; Interphase Separation, i; Carbide, C; Sigma,  $\sigma$ . (a) Weld Exposed to 165 MPa Stress Until Rupture at 21 Ms; Total Elongation, 3.4%. (b) Weld Exposed to 310 MPa Stress Until Rupture at 7.6 h; Total Elongation, 30.2%.

Fig. 7. Charpy Fracture Profiles Associated with Precipitated Carbides for the Low-Ferrite Type 308 Stainless Steel Weld Exposed at 593°C in Air. Key: Austenite,  $\gamma$ ; Ferrite,  $\delta$ ; Interphase Separation, i; Carbide, C; Sigma,  $\sigma$ . (a) As welded: 4.0 FN. (b) Exposed 18 Ms 1.9 FN. (c) Exposed 36 Ms 1.4 FN.

Fig. 8. Charpy Fracture Profiles Associated with Transformed Sigma Phase in High-Ferrite Type 308 Stainless Steel Welds Exposed at 593°C in Air. Key: Austenite,  $\gamma$ ; Ferrite,  $\delta$ ; Interphase Separation, i; Carbide, C; Sigma,  $\sigma$ . (a) As welded: 15.8 FN. (b) Exposed 18 Ms 3.4 FN. (c) Exposed 36 Ms 2.8 FN.

Fig. 9. Creep Ruptures Associated with Precipitated Carbides in the Extra-Low-Ferrite Type 308 Stainless Steel Welds Tested at 593°C in Air. Key: Austenite,  $\gamma$ ; Ferrite,  $\delta$ ; Carbide, C; Sigma,  $\sigma$ . (a) Weld Placed under 210 MPa Stress until Rupture at 14 Ms; Total Elongation, 3.4%. (b) Weld Placed under 310 MPa Stress until Rupture at 10.7 h; Total Elongation, 28.5%.

Fig. 10. Color Etching Reveals Interphase Separation between Austenite and Sigma Phases in Medium-Ferrite Type 308 Stainless Steel Weld. Rupture life, 7797 h total elongation, 4.1% test temperature, 593°C (1100°F); stress, 176 MPa. NOTE: The transformed, brittle sigma phase served as a fracture route in an area composed predominately of untransformed ductile ferrite in austenite.

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#### INTRODUCTION

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