The Influence of Composition and Microstructure on the HAZ Toughness of Duplex Stainless Steels at −20°C

HAZ toughness decreases as a function of both ferrite content and grain size

BY J. C. LIPPOLD, I. VAROL AND W. A. BAESLACK III

ABSTRACT. The toughness of two commercial duplex stainless steels, Ferralium Alloy 255 and Alloy 2205, was evaluated over a range of cooling rates representative of conditions in the weld heat-affected zone (HAZ). Both alloys exhibited a loss in toughness at the cooling rate extremes, 90° and 2°C/s, resulting from high ferrite content and large prior ferrite grain size, respectively. Alloy 255 also showed a drop in toughness at an intermediate cooling rate of 50°C/s. This intermediate loss in toughness, not observed in Alloy 2205, results from the interrelationship between austenite and Cr-rich precipitate formation along ferrite grain boundaries. The precipitation mechanisms and their subsequent effect on toughness are described. The practical implications of HAZ microstructure control are also discussed.

Introduction

Duplex stainless steels offer a number of advantages over conventional austenitic stainless steels in mildly aggressive environments due to the combination of strength and corrosion resistance (particularly pitting and stress corrosion cracking resistance) that these alloys possess. In contrast to the austenitic grades, the duplex alloys are formulated and thermomechanically processed to produce a two-phase microstructure containing nearly equal proportions of ferrite and austenite.

The weld fusion zone and regions of the heat-affected zone (HAZ) experiencing temperatures above the ferrite solvus (~1275°C) followed by rapid cooling to room temperature have been shown to exhibit an increased level of ferrite relative to the base metal. An increase in the ferrite/austenite balance has been shown to promote deterioration of both the corrosion resistance and toughness of the weld region (Refs. 1–6). This microstructural balance is primarily a function of composition, peak temperature (for the HAZ) and cooling rate through the ferrite plus austenite phase field. In the fusion zone the phase balance can often be controlled by appropriate selection of filler metals, while in the HAZ the only alternative for microstructural control is through adjustment of the weld thermal cycle.

The relationship between composition, thermal history and microstructure has not been clearly defined for the duplex stainless steels. The normal guideline for increasing the proportion of HAZ austenite is to increase heat input, preheat and/or interpass temperature (for multipass welds) in order to reduce the weld cooling rate, thereby promoting a more complete ferrite-to-austenite transformation on-cooling. It is unclear what effect this practice may have on other metallurgical reactions, such as grain growth and precipitation. In addition, the relationship between the resultant microstructure and mechanical properties has not been clearly established. Thus, the purpose of this investigation was to develop a better understanding of the effect of thermal variables on microstructure and associated toughness. In order to accomplish this in a controlled, systematic manner, a range of microstructures representative of those achievable in the HAZ of fusion welds in commercial duplex stainless steels was simulated using the Gleeble™ 1500 system.

Experimental Procedure

The chemical compositions of the two commercial duplex stainless steels studied are presented in Table 1. Both steels were in the form of 0.5-in. (12.7-mm) thick plate, which had been solution annealed in the temperature range from 1020° to 1100°C (1870°–2010°F) followed by quenching in water. Both Ferralium Alloy 255 (UNS S32550) and SAF Alloy 2205 (UNS S32205) were wrought alloys and contained essentially equivalent proportions of ferrite and austenite (75–80 FN), as measured by the Magne gauge) aligned along the rolling direction of the plate.

The Gleeble 1500 was employed to produce a range of representative HAZ microstructures in Charpy blanks of dimension 12 x 12 x 55 mm (0.473 x 0.473 x 2.165 in.) of each alloy. Samples were heated above the ferrite solvus to 1300°C (2372°F) at a rate of 130°C/s (240°F/s), held for either 1 or 10 s and then cooled at rates ranging from 90° to 2°C/s (162°–3.6°F/s).
TEM samples were cut from the thermal ly-
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sisting of 275 mL methyl alcohol, 1 75 mL
cron, followed by jet polishing at -10°C
and mechanically thinned to 100 mi-
cycled regions of the Gleeble samples
microstructure and identify precipitates.
200CX at 200 kV to further evaluate the
(TEM) was performed using a JEOL-
fected electrolytically in 10% oxalic acid at 6 V
allographic features. Samples for metal-
sections were taken normal to the frac-
ification tests for duplex stainless steels
alloy and cooled at 75°, 50° and 20°C/s
shown in Figs. 1 and 2 are representative
microstructures. The microstructures
varied directly with cooling rate (from
2205. Hold time (10 s) at peak tempera-
ture had little effect on the ferrite content,
since more austenite was available to dis-
solve the nitrogen. Ferrite content ranged
from 110 to 90 FN and
down from approximately 110 to 90 FN and
varied directly with cooling rate (from
75° to 2°C/s) in both Alloy 255 and Alloy
2205. Hold time (10 s) at peak tempera-
ture had little effect on the ferrite content,
indicating that the transformation to ferrite
on-heating occurs readily and that prior ferrite grain size has little influence on austenite precipitation during the on-
cooling portion of the thermal cycle. The
distribution of the precipitates was more
uniform within the ferrite grains at almost
cooling rates with higher holding time
at peak temperature, indicating that ad-
ditional homogenization of the ferrite
and/or dissolution of preexisting precipi-
tates occurs upon holding for longer
times above the ferrite solvus. Grain size
varied inversely with cooling rate and
ranged from 180 to 550 µm and 195 to

Following thermal treatment, the
blanks were machined into standard
Charpy V-notch (CVN) specimens and
tested per ASTM E-23 at -20°C (-4°F).
This test temperature was selected based on the requirements of many weld qual-
ification tests for duplex stainless steels and the preponderance of toughness data at -20°C in the literature (Refs. 7-9). Per
ASTM E-23, at least three samples were
tested per condition. Following testing,
sections were taken normal to the frac-
ture plane to allow examination of met-
allographic features. Samples for metal-
lographic evaluation were etched

dustry of precipitates was more
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Transmission electron microscopy
(TEM) was performed using a JEOL-
200CX at 200 kV to further evaluate the
microstructure and identify precipitates.

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Microchemical analysis was performed using an energy dispersive x-ray spectrometer attached to the TEM. The frac-
ture behavior of the CVN samples was evaluated using the scanning electron microscope (SEM) at magnifications up
to 5000X.

Results and Discussion

Microstructure

The thermal cycles used during this investigation produced a range of mi-

Table 1—Chemical Composition (wt-%)
<table>
<thead>
<tr>
<th>Material</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Mn</th>
<th>Si</th>
<th>Cu</th>
<th>C</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>FN</th>
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<tr>
<td>Alloy 255</td>
<td>24.90</td>
<td>5.39</td>
<td>3.13</td>
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<td>1.72</td>
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<td>0.17</td>
<td>0.001</td>
<td>0.023</td>
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<tr>
<td>Alloy 2205</td>
<td>21.75</td>
<td>5.82</td>
<td>2.73</td>
<td>1.74</td>
<td>0.48</td>
<td>—</td>
<td>0.019</td>
<td>0.13</td>
<td>0.002</td>
<td>0.021</td>
<td>76</td>
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</table>

Notes: All samples cooled from a peak temperature of 1300 °C.
FN = Ferrite Number, determined by Magne Gage according to AWS A4.2-91.
ACD = average grain diameter (in microns).
γM = austenite morphology, GB = grain boundary, i = intragranular, W = Widmanstätten plates.
PPT = precipitation behavior; E = extensive, M = moderate, L = low, VL = very low.

Table 2—Summary of Microstructural Observations

<table>
<thead>
<tr>
<th>Cooling Rate, °C/s</th>
<th>Hold Time = 1 s</th>
<th>Hold Time = 10 s</th>
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<td></td>
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<td>240</td>
</tr>
<tr>
<td>2</td>
<td>88</td>
<td>650</td>
</tr>
</tbody>
</table>

Fig. 1 — Simulated HAZ microstructures in Alloy 255, cooled from 1300°C at: A — 75°C/s; B — 50°C/s; C — 20°C/s. Time at peak temperature: 1 s.
the simulated HAZ samples at -20°C are Alloys 255 and 2205, respectively. At the higher cooling rates (75°C and 50°C/s), time at peak temperature had a significant effect on grain size.

Impact Toughness

The results of Charpy impact testing of the simulated HAZ samples at -20°C are shown in Fig. 3 for both Alloys 255 and 2205. The base metal toughness of both alloys was approximately 200 J at -20°C. In general, the spread of CVN toughness for a given microstructural condition was small, typically less than 10%. Each data point in Fig. 3 represents the average of three tests.

For the simulated HAZ samples, impact toughness was found to decrease with increasing cooling rate for both alloys in the range from 20°C to 90°C/s, for holding times of both 1 and 10 s at the peak temperature of 1300°C. In contrast, impact toughness increased with an increase in cooling rate in the range from 2°C to 20°C/s. Longer holding times (10 s) at peak temperature resulted in a drop in impact toughness for both alloys at all cooling rates. The effect of holding time was particularly pronounced in Alloy 255, approaching a reduction of nearly 100 J in the cooling rate range from 20°C to 75°C/s.

An unexpected drop in toughness was observed in Alloy 255 samples cooled at 50°C/s, particularly for the 1-s hold time. A similar drop in toughness was not observed at an equivalent cooling rate in Alloy 2205. The predominantly ferritic microstructure at the highest cooling rate and the large prior ferrite grain size at the lowest cooling rate were primarily responsible for the toughness drops at the cooling rate extremes. At intermediate cooling rates, the balance between grain size and ferrite/austenite content results in good toughness relative to that of the base metal (~200 J).

Toughness Anomaly

The toughness trough exhibited by Alloy 255 at a cooling rate of 50°C/s was unexpected and has been the subject of considerable microscopic and fractographic evaluation in an effort to determine the metallurgical basis for its existence. This toughness trough was verified by two separate sets of simulation experiments — a total of 6 CVN samples were tested for a 1-s hold time. Based on optical metallography, Alloy 255 showed little difference in either grain size or ferrite content (in terms of FN) over the cooling rate range from 75°C to 20°C/s (109 to 93 FN, 180 to 200 microns grain diameter for 1-s hold at 1300°C). Alloy 2205 exhibited a similar microstructural trend but was not susceptible to a drop in toughness at an intermediate cooling rate.

A bright-field TEM image of the microstructure of Alloy 255 cooled at 50°C/s is shown in Fig. 4. Note that the ferrite grain boundaries are decorated with relatively large (0.2-0.5 micron) Cr- and Mo-rich precipitates (presumably nitrides or carbonitrides). In addition, a fine intragranular dispersion of precipitates was also observed — Fig. 4C. These particles were also enriched in Cr and Mo and are presumed to be of similar nature to the intergranular precipitates.

TEM examination of Alloy 255 samples cooled at 20°C/s revealed that the degree of intergranular precipitate formation was much less than that at 50°C/s. This is due in part to the larger amount of intergranular austenite that forms at the lower cooling rate. The austenite acts as a "sink" for carbon and nitrogen, due to the large difference in solubility relative to the ferrite (Ref. 1), and it effectively retards the formation of nitrides and carbonitrides at the grain boundary. Significant intragranular intermetallic precipitation was still observed in this microstructure despite the presence of increased intergranular austenite and grain boundary sideplates.

Based on these observations, it appears that the presence of large, blocky precipitates along ferrite grain boundaries may result in a reduction of toughness, perhaps by promoting crack initiation at these locations. The absence of intergranular precipitates at higher cooling rates (75°C/s) where precipitation is predominantly intragranular, and the presence of austenite at lower cooling rates both help to restore the toughness of the microstructure. Since these grain boundary precipitates (GBPs in Fig. 5) are Cr-rich, alloys higher in Cr, N and C would have a higher propensity for forming both inter- and intragranular intermetallic precipitates and may explain why the toughness trough is absent in the lower-Cr Alloy 2205 samples thermally cycled under the same conditions.
Representative fracture surfaces of Alloy 255 CVN specimens cooled at 75°, 50° and 20°C/s (after 1 s hold at 1300°C) are shown in Fig. 6. Fracture behavior in the Alloy 2205 CVN samples was similar. At 75°C/s, fracture was almost entirely by cleavage with little or no evidence of ductile rupture. The percentage of ductile rupture observed at 50°C/s (Fig. 6B) increased only slightly, while at 20°C/s ductile rupture was prevalent and bimodal, exhibiting regions of both coarse and fine dimples (Fig. 6C and 6D, respectively). The coarse dimples corresponded to ductile tearing through grain boundary austenite, while the fine dimples were superimposed on macroscopic cleavage facets and appear to represent microductility resulting from the presence of intragranular austenite and/or precipitates in the ferrite matrix. Based on the appearance of the fracture surfaces, it is difficult to rationalize the toughness variation between the Alloy 255 samples cooled at 75° and 50°C/s. The similarity in fracture appearance suggests that fracture initiation rather than propagation may influence the impact toughness at -20°C. This observation reinforces the TEM results, suggesting that the nature of nitride and/or carbonitride precipitation as a function of composition and cooling significantly influence toughness over a relatively narrow range of cooling rates.

Practical Implications

The results of this investigation have shown that extremes in cooling rate in the HAZ of Alloys 2205 and 255 may

Fig. 4 — Bright-field TEM image of Alloy 255 cooled at 50°C/s from 1300°C (1 s hold). A — General view; B — intergranular precipitate; C — intragranular precipitates. Composition (in wt-%) is for the metallic species detected in the intergranular precipitates in B.

It is also significant that the toughness trough in Alloy 255 is more pronounced at shorter hold times. This is undoubtedly due to the difference in grain size and the fact that grain boundary precipitation occurs more readily as grain boundary area increases (where the area is inversely proportional to the square of the grain diameter). At longer peak temperature hold times for Alloy 255, the grain size effect on toughness becomes more dominant and only a small trough is observed.

Fig. 5 — Bright-field TEM image of Alloy 255 cooled at 20°C/s. Open arrows indicate austenite precipitation at ferrite grain boundaries. Closed arrows designate an array of grain boundary precipitates.

Fig. 6 — CVN fracture appearance of Alloy 255 samples (1300°C, 1 s) cooled at: A — 75°C/s; B — 50°C/s; C — 20°C/s, (all 1000X); D — 20°C/s (5000X).
promote a significant drop in impact toughness relative to the base metal. As has been reported previously (Refs. 2, 6, 10), highly ferritic HAZ microstructures reduce toughness, as was shown in samples cooled at 75° and 90°C/s in this investigation. Increasing the austenite content of the HAZ is not singularly sufficient to increase toughness, however, since increasing ferrite grain size can significantly reduce toughness. This latter observation is important, since most current guidelines on controlling HAZ microstructure in duplex stainless steels refer only to the ferrite/austenite balance. The data reported here suggest that low to medium heat input welding processes that promote HAZ cooling rates in the range from 20° to 50°C/s should be most effective in ensuring HAZ toughness down to -20°C. This cooling rate range produces a good balance between grain size and ferrite/austenite balance. In addition, the level of Cr-nitride precipitation in this range is relatively low, thereby promoting improved corrosion resistance. By proper control of welding conditions, and associated HAZ cooling rate, it should be possible to achieve toughness levels of 150 to 200 J in the HAZ at -20°C without compromising the mechanical or environmental integrity of the weldment.

Conclusions

1) Simulated HAZ microstructures in Ferralium Alloy 255 and Alloy 2205 with cooling rates ranging from 75° to 2°C/s from a peak temperature of 1300°C exhibited a significant variation in ferrite content (approximately 110 to 90 FN) and a wide range in grain size (180 to 650 microns, average grain diameter).

2) CVN toughness at -20°C in samples cooled at rates of 75° and 90°C/s was significantly below that of the base metal. This decrease was primarily due to the high proportion of ferrite in the microstructure, which promoted a predominantly cleavage-type failure.

3) CVN toughness also decreased at a 2°C/s cooling rate due to the large prior ferrite grain size and despite a higher austenite content than observed at higher cooling rates.

4) Peak impact toughness levels of approximately 200 J at -20°C were observed at a cooling rate of 20°C/s for both alloys. This level was essentially equivalent to that of the base material.

5) Increasing the hold time at 1300°C from 1 to 10 s resulted in a decrease in toughness, particularly in Ferralium Alloy 255. This decrease is associated with the increase in ferrite grain size.

6) A toughness trough was observed at a cooling rate of 50°C/s in Ferralium Alloy 255. This phenomenon was attributed to the intergranular precipitation behavior of this alloy as influenced by alloy composition. A similar drop in toughness was not observed in Alloy 2205.

Acknowledgments

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CHARACTERIZATION OF PWHT BEHAVIOR OF 500 N/mm² CLASS TMCP STEELS

The objective of this research project was to clarify the effects of PWHT conditions on the properties of TMCP steel in comparison with conventional heat-treated steel. A study on the possibility of eliminating PWHT with TMCP steels was the main subject of this cooperative research.

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