Title
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INTRODUCTION

Previous research in this laboratory led to the development of thermal cycling techniques which establish an extremely fine grain effective size in 12Ni and 9Ni cryogenic steels. In the grain-refined condition these alloys offer a good combination of strength and toughness at 4K and have possible applications in the structures of cryogenic devices such as high field superconducting magnets. But the structures of these devices are usually welded, and candidate structural alloys must hence retain good properties in the welded condition. To weld the grain-refined steels one must overcome the dual problems of brittleness in the weld deposit and brittleness due to grain coarsening in the heat affected zone (HAZ).

The problem of welding grain-refined Fe-12Ni-0.25Ti for 4K service was first approached in this laboratory by using high nickel filler metals such as are often specified for ferritic steel weldments at 77K. This approach led to an undesirable brittleness in the fusion zone and a low yield strength in the weld metal. Similar problems were encountered by Ishikawa and Maruyama in the welding of 13Ni cryogenic steel.
A more promising approach was developed in joint research between the Japanese steel companies, Nippon Kokan, K.K. and Kobe Steel, who showed that quench-and-tempered 9Ni steel may be welded for 77K service with a matching ferritic filler if a multi-pass GTAW technique is employed. The GTAW process provides a relatively clean weld deposit and permits a largely independent control of the heat input and metal deposition rate. In controlled GTAW welding subsequent weld passes may be made to impose thermal cycles on the solidified material, hence refining the structures of the weld deposit and preserving a refined microstructure in the heat-affected zone. This process is now used commercially in Japan in the manufacture of vessels for inert cryogens and has been successfully employed in a prototype spherical LNG tank. Exploratory work at the NASA Lewis Research Center showed that GTAW could also be used for autogenous welding of Fe-12Ni-0.5Ti, and yields weldments with good impact properties at 77K. Continuing this line of research, we have recently developed a 14Ni filler metal and multi-pass GTAW welding process for grain-refined Fe-12Ni-0.25Ti. The resulting welded plates show high strength and good impact toughness in liquid helium in both the weld metal and heat affected zone and show promising properties in fracture toughness tests.

The present paper reports the initial results of similar studies on ferritic GTA weldments in grain-refined 9Ni steel. The microstructural problem in welded 9Ni steel is superficially more formidable than that in the 12Ni alloy since the 9Ni
steel base plate only has good 4K properties when it is given a final intercritical temper to introduce a distribution of stable high-temperature austenite phase into the grain-refined structure. The austenite provides an additional grain refinement and getters carbon and carbides from the matrix. Its retention near the fusion line and in the heat-affected zone may be important to 4K toughness of welded plates.

**ALLOY PREPARATION AND EXPERIMENTAL PROCEDURE**

The 9Ni steel used in this work was a commercial grade provided by Nippon Kokan, K.K. Its composition is given in Table I. The material was received in the form of 15mm thick plates in the quench-and-temper (QT) condition. The plates were solution-annealed at 900°C for 2 hours to remove the effects of prior deformation and thermal treatment. They were then given the 2BT heat treatment diagrammed in Fig. 1. This treatment consists of a four-step alternating thermal cycle to refine the effective grain size followed by an intercritical temper to introduce an appropriate distribution of precipitated austenite. A transmission electron micrograph of the 2BT microstructure is shown in Fig. 2.

The weld filler metal was cast in this laboratory in 4.5kg ingots of nominal composition Fe-14Ni-0.2Ti-0.003B. The ingot composition is given in Table I. After homogenizing at 1200°C for 24 hours the ingots were hot-rolled and swaged into wire of 1.6 diameter. This wire was used as the filler for manual GTA welding.

The plates to be welded were machined into one of two joint configurations, a 60° single V or a 45°single bevel. The plates
were welded manually using one of the two sets of welding conditions shown in Table 2. No significant defects were found in the completed weldments in non-destructive x-ray examination.

Specimens for mechanical testing were cut from the welded plates, as described below. Specimens for x-ray diffraction analysis of the residual austenite content were sliced parallel to the fusion boundary (as shown in the insert to Fig. 4), ground, and chemically polished. The distribution of austenite was found by sequential grinding and surface preparation. The retained austenite volume fraction was calculated using the method proposed by Miller.

Charpy impact and fracture toughness tests at 77K were conducted according to ASTM procedures. The 4K Charpy impact tests employed a modification of the "lucite-box" technique proposed by Jin, et al.

RESULTS AND DISCUSSIONS

Microstructure

Grain Refinement. As in the case of grain-refined 12Ni steel welded with the same filler metal, the multi-pass GTA weld of 9Ni steel were efficiently grain refined in both the weld metal and heat-affected zone. While there were some islands of larger grain size which apparently represent a poor overlap of subsequent weld passes, a reasonably uniform grain size approximately 5µm was obtained through the weld region. A full thickness microstructure is shown in Fig. 3.

Retained Austenite. The volume fraction of retained austenite in the welded 9Ni steel is plotted as a function of distance.
from the weld center line in Fig. 4. In agreement with results obtained earlier from the welding of 9Ni\textsuperscript{16} and 12Ni\textsuperscript{13} steel there is no retained austenite within the weld metal. The volume fraction of austenite decreases gradually through the heat-affected zone from a value of approximately 15% in the base metal. These results are in some disagreement with the earlier observations of Tamura et al.\textsuperscript{17} on the welding of quench-and-tempered 9Ni steel with ferritic filler metal. They reported an increase in retained austenite in the heat-affected zone to approximately 10% from a lower value of 4% within the base metal. They also found a significant austenite retention within the weld metal, and argued that the austenite fraction is well correlated to cryogenic impact toughness\textsuperscript{17,18}. In the present work the austenite volume fraction increases monotonically through the heat-affected zone and has no obvious correlation with the variation of impact toughness through the weld region.

**Cryogenic Toughness**

**Impact Toughness.** Charpy impact tests were conducted at 77K and 4.2K to give a general indication of the toughness of the welded plate. The results are listed in Table 3, and are compared with those obtained for 12Ni steel welded with the same filler metal and similar procedure. The variation of impact toughness through the heat-affected zone at 4K is plotted in Fig. 5. The Charpy impact energy of the welded 9Ni steel is relatively flat through the heat-affected zone and reaches a maximum within the weld metal. The sharp maximum in impact toughness within the
heat-affected zone which was found in 12Ni steel is not present. There is, moreover, no obvious correlation between the impact toughness of the welded plate and the retained austenite level.

All of the broken specimens in both 9Ni and 12Ni fractured in a completely ductile mode. The relevant 9Ni scanning electron fractographs are presented in Fig. 6. The impact ductile-brittle transition temperature of the ferritic weldment is, therefore, below liquid helium temperature. There was no evidence of significant fusion zonebrittleness in these tests. While there is a slight relative drop in Charpy impact energy near the fusion line, the fusion zone fracture was ductile, and cracks initiated from the heat-affected zone did not follow the fusion boundary.

**Cryogenic Fracture Toughness.** The cryogenic fracture toughness tests conducted to date on ferritic welded 9Ni plate have been at 77K. Tests at 4K are now in progress. The completed test series employed three-point bend test specimens of 1cm thickness prepared according to ASTM specifications and tested on a MTS machine equipped with a liquid nitrogen cryostat. The load-crack opening displacement (COD) curves are plotted in Fig. 7. The specimens from the base plate and weldment showed no evidence of unstable crack propagation at 77K. The HAZ specimen showed only a slight indication after the peak load had been passed. Fractographic examination of the surfaces showed that the fracture mode was completely ductile rupture in all cases.

Since the specimens tested were well away from plane strain conditions, valid $K_{lc}$ values were not obtained. The fracture
toughness was estimated using the equivalent energy method\textsuperscript{20}, the result of the calculation is shown in Table 4. Fracture toughness of the weld metal is near 300 ksi$\sqrt{\text{in}}$ (330 MPa$\sqrt{\text{m}}$) at 77K. High fracture toughness was preserved even in the heat-affected zone.

Fracture toughness tests of the ferritic welded 9Ni plates are now in progress. The 4K toughness of the weld metal itself should, however, be nearly the same as that obtained in tests on 12Ni steel welded with the same filler metal and welding procedures\textsuperscript{13}. Tests of the fracture toughness of the ferritic weldment in 12Ni have been complicated by the difficulty of obtaining good grain refinement in the final weld passes. The tests nonetheless demonstrate that the grain-refined weld material remains ductile and tough near 4K.

CONCLUSIONS

The results presented above show that it is possible to weld grain-refined 9Ni steel with ferritic weld filler metal so as to retain good toughness at cryogenic temperatures. The results of this work may permit the utilization of retreated commercial grade 9Ni steel in structural applications within helium-cooled cryogenic devices where high strength and good toughness are required.

ACKNOWLEDGMENTS

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REFERENCES

11. I. Watanabe, Central Research Laboratory, Nippon Kokan, K.K., private communication.


Table I. Chemical Compositions of Base and Filler Metal (wt.%).

<table>
<thead>
<tr>
<th></th>
<th>Ni</th>
<th>Ti</th>
<th>Mn</th>
<th>Si</th>
<th>C</th>
<th>P</th>
<th>S</th>
<th>B</th>
<th>Fe</th>
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</thead>
<tbody>
<tr>
<td>9Ni Steel</td>
<td>9.18</td>
<td>0.06</td>
<td>0.60</td>
<td>0.21</td>
<td>0.06</td>
<td>0.01</td>
<td>0.002</td>
<td>-</td>
<td>Bal.</td>
</tr>
<tr>
<td>Filler</td>
<td>13.87</td>
<td>0.16</td>
<td>*</td>
<td>*</td>
<td>0.003</td>
<td>0.001</td>
<td>0.002</td>
<td>0.003</td>
<td>Bal.</td>
</tr>
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</table>

* Negligible

Table II. Welding Conditions.

<table>
<thead>
<tr>
<th>Heat Input (KJ/cm)</th>
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<tr>
<td>Arc Voltage (V)</td>
<td>14 - 18</td>
</tr>
<tr>
<td>Welding Current (A)</td>
<td>150 - 180</td>
</tr>
<tr>
<td>Welding Speed (cm/sec)</td>
<td>0.4</td>
</tr>
<tr>
<td>Shielding Gas Flow Rate</td>
<td>Pure argon, 25 ft³/hr.</td>
</tr>
<tr>
<td>Root Gap</td>
<td>1.6 mm</td>
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<tr>
<td>Interpass Temperature</td>
<td>50°C - 150°C</td>
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</table>

Table III. Charpy V-notch impact values of 9Ni and 12Ni welded joint.

<table>
<thead>
<tr>
<th>Impact Energy, Joule (ft-lb)</th>
<th>Weld Metal</th>
<th>HAZ</th>
<th>Base Metal</th>
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</thead>
<tbody>
<tr>
<td>9Ni joint at 77°K</td>
<td>184(136)</td>
<td>169(125)</td>
<td>156(115)</td>
</tr>
<tr>
<td>at 4.2°K</td>
<td>153(113)</td>
<td>149(110)</td>
<td>146(108)</td>
</tr>
<tr>
<td>12Ni joint at 77°K</td>
<td>182(135)</td>
<td>217(160)</td>
<td>156(115)</td>
</tr>
<tr>
<td>at 4.2°K</td>
<td>173(128)</td>
<td>179(132)</td>
<td>136(100)</td>
</tr>
</tbody>
</table>
Table IV. Fracture toughness of 9Ni welded joint at 77°K

<table>
<thead>
<tr>
<th></th>
<th>Fracture Toughness*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MPa $\sqrt{m}$</td>
</tr>
<tr>
<td>9Ni Base Metal</td>
<td>220</td>
</tr>
<tr>
<td>HAZ (0.5mm)</td>
<td>247</td>
</tr>
<tr>
<td>HAZ (2.0mm)</td>
<td>212</td>
</tr>
<tr>
<td>Weld Metal</td>
<td>328</td>
</tr>
</tbody>
</table>

* All values are calculated using Equivalent Energy method.\(^{20}\)
Figure 1

The 2BT heat treatment of 9Ni steel plotted alongside the Ni-rich segment of the Fe-Ni phase diagram
TEM micrographs of retained austenite in 2BT 9Ni steel.
(a) bright field, (b) dark field taken from (002)$\gamma$ diffraction spot in (c).
Macro- and Microstructures of 9Ni welded joint: (a) macrostructure of welded joint, (b) microstructure of well refined region marked B in (a), (c) grain coarsened last pass region marked C in (a).
Figure 4

Variation of the retained austenite volume fraction through the heat-affected zone in welded 9Ni steel.
The variation of Charpy impact energy with notch location for samples impacted in liquid helium.
Figure 6

Scanning electron fractographs of 9Ni Charpy specimens broken at 4.2K (a) weld metal (b) HAZ (c) base metal.
Figure 7

Load-crack opening displacement curves of three-point bend specimens at 77°C.
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