Weldability of High-Strength, Low-Expansion Superalloys

Weld solidification and HAZ liquation cracking in 900-series superalloys are related to Nb and Si levels

BY S. C. ERNST, W. A. BAESLACK III AND J. C. LIPPOLD

ABSTRACT. Heat-affected zone (HAZ) liquation and weld metal solidification cracking in high-strength, low-expansion superalloys have been investigated using the spot- and mini-Varestraint weldability tests, respectively. Specific alloys evaluated included Incolloys 903, 907 and 909. Varestraint test results revealed that the relative susceptibility to both HAZ liquation and weld solidification cracking in these alloys was 903 < 907 < 909. In general, the cracking susceptibility of these alloys was much higher than that of 300-series austenitic stainless steels tested in a similar manner. Metallurgical evaluation of spot-Varestraint samples via optical and electron microscopy revealed that HAZ liquation cracking resulted from the presence of a low-melting, Laves phase/gamma eutectic liquid that was enriched in Nb, Si and Ti. This liquid originated in part from the constitutional liquation of Nb-rich carbides in the 903 and 907 alloys and G-phase and/or Laves phase in the 909 alloy. Weld solidification cracking was associated with the partitioning of Nb, Si and Ti to solidification boundaries and the subsequent depression of the solidification temperature locally along those boundaries. For both fusion zone and HAZ cracking, an increase in the Nb + Si content was found to promote increases in amounts of grain boundary liquid, which correlated well with the relative cracking susceptibility of these alloys. The mechanisms of both solidification and HAZ liquation cracking in these alloys are discussed in detail.

Introduction

Metallurgy of the 900-Series Low-Expansion Superalloys

The 900-series superalloys represent a relatively new class of aerospace materials designed to provide high room- and elevated-temperature strength and a low coefficient of thermal expansion (CTE) (Refs. 1, 2). This unique property combination has promoted their increased use in gas turbine engine applications requiring exacting tolerance control over a wide range of temperatures, including compressor seals, shafts and support rings. The 900-series, low-expansion superalloys are also highly resistant to high-pressure hydrogen embrittlement, which has led to their use in the space shuttle main engine.

The 900-series, low-CTE superalloys are iron-based and contain appreciable amounts of nickel and cobalt. In contrast to alternate high-temperature alloys, the chromium content is limited to optimize their low-expansion characteristics. The alloys are fully austenitic, yet ferromagnetic at room temperature. The coefficient of thermal expansion of 900-series alloys is typically low and relatively constant up to a characteristic inflection temperature, at which point the CTE rises more rapidly with increasing temperature. Values of CTE for the 900-series, low-expansion superalloys are well below those of alternate commercial superalloys, being approximately one-half that of Inconel 718 at the inflection temperature of 427°C (800°F).

In order to exploit the Fe-Ni-Co system for elevated-temperature structural applications, Al, Nb and/or Ti were added to the Fe-Ni-Co solid solution to promote precipitation hardening. With proper solutionizing and aging heat treatments, the 900-series superalloys can develop excellent short-term tensile properties comparable to those of Inconel 718. Unfortunately, the absence of chromium reduces oxidation resistance and promotes susceptibility to stress-accelerated grain boundary oxygen (SAGBO) embrittlement. Sensitivity to the SAGBO phenomena, in turn, significantly reduces elevated-temperature notch-rupture strength. During the past decade, development efforts at Huntington Alloys (now Inco Alloys International) have resulted in a series of age-hardenable Fe-Ni-Co low-CTE alloys (designated Incoloy 903, 907 and 909) that offer improvements in elevated-temperature notch-rupture properties via both compositional tailoring and heat treatment (Refs. 3, 4).

Incoloy 903 was introduced as the first commercial low-CTE superalloy in the mid-1970's, and today remains the most widely used alloy in this family. The nominal compositions of Incoloy 903 and subsequent generation alloys 907 and 909 are provided in Table 1. As indicated above, high tensile strengths can be obtained in Incoloy 903 using a conventional solution heat treatment [982°C (1800°F)/1 h, air cool] followed by a standard Inconel 718 aging treatment [718°C (1325°F)/8 h, furnace cool at 50°C/h to 621°C (1150°F)/8 h, air cool]. This heat treatment cycle promotes a fine, recrystallized gamma (austenite) grain structure and the uniform precipitation of Nb(Al,Ti)gamma prime. Obtaining adequate notch-rupture strength in Incoloy 903, however, requires the use of special thermomechanical processing and heat treatment which involves warm working followed by a moderate anneal [843°C (1550°F)/1 h, below the
recrystallization temperature and gamma-prime solvs.] and the standard aging treatment. This processing sequence results in a highly textured microstructure and corresponding anisotropic mechanical properties, as exemplified by a transverse notch-rupture strength less than half that exhibited in the longitudinal direction. Time-temperature transformation (TTT) studies on Incoloy 903 (Ref. 5) have determined that strengthening is achieved primarily by the precipitation of Ni$_3$(Al,Ti,Nb) gamma prime, and that long exposure at elevated temperatures may promote formation of a needle-like Ni$_3$(Al,Ti,Nb) eta phase.

Evidence that a decreased aluminum content promotes significantly improved SAGBO resistance and a corresponding improvement in notch-rupture strength led to the development of a second generation low-CTE alloy, Incoloy 907. As shown in Table 1, the aluminum content was reduced from 0.9 wt-% in Incoloy 903 to 0.03 wt-% in Incoloy 907. Correspondingly, the Nb and Ti contents were increased to maintain the desired strength levels. As in Incoloy 903, satisfactory notch-rupture strength cannot be achieved using a standard solutionize and age heat treatment. Rather, an overaging treatment [774°C (1425°F)/12 h + 621°C (1150°F)/8 h, air cool] is required to provide acceptable notch-rupture strength at the sacrifice of reduced yield and tensile strengths. Phase transformation studies on Incoloy 907 (Ref. 6) have shown that overaging promotes a more complex microstructure (relative to Incoloy 903) that is comprised of inter- and intragranular Ni$_3$(Al,Nb,Ti) epsilon phase or epsilon double-prime and gamma prime.

Incoloy 909 represents the most recent low-CTE superalloy. This alloy differs compositionally from Incoloy 907 primarily in its four-fold increase in silicon content, a modification which markedly improves notch-rupture properties. Significantly, these improvements can be achieved using a higher temperature, solution heat treatment [774°C (1425°F)/12 h + 621°C (1150°F)/8 h, air cool] is required to provide acceptable notch-rupture strength at the sacrifice of reduced yield and tensile strengths. Phase transformation studies on Incoloy 907 (Ref. 6) have shown that overaging promotes a more complex microstructure (relative to Incoloy 903) that is comprised of inter- and intragranular Ni$_3$(Al,Nb,Ti) epsilon phase or epsilon double-prime and gamma prime.

Table 1—Nominal Compositions of 900-Series Superalloys (wt-%)

<table>
<thead>
<tr>
<th>Element</th>
<th>Alloy 903</th>
<th>Alloy 907</th>
<th>Alloy 909</th>
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<tr>
<td>Ni</td>
<td>38</td>
<td>38</td>
<td>38</td>
</tr>
<tr>
<td>Co</td>
<td>15</td>
<td>13</td>
<td>13</td>
</tr>
<tr>
<td>Nb</td>
<td>3.0</td>
<td>4.7</td>
<td>4.7</td>
</tr>
<tr>
<td>Ti</td>
<td>1.4</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>Al</td>
<td>0.9</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>Si</td>
<td>0.1</td>
<td>0.1</td>
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</tr>
<tr>
<td>C</td>
<td>0.02</td>
<td>0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Fe</td>
<td>bal.</td>
<td>bal.</td>
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Weldability of the 900-Series Low-Expansion Superalloys

The weldability of Nb-containing superalloys is defined primarily in terms of susceptibility to heat-affected zone (HAZ) liquation cracking, fusion zone liquation cracking (also termed microfissuring) and fusion zone solidification cracking. The sensitivity of a particular alloy to each of these cracking forms is dependent primarily on alloy composition, although HAZ cracking may also be influenced by the base metal microstructure.

HAZ Liquidation Cracking

Heat-affected zone liquation cracking occurs from the formation of a liquid film at grain boundaries during the weld thermal cycle and the inability of this film to accommodate thermally induced stresses during weld cooling. Fully austenitic alloys that contain Nb and/or Ti can be highly susceptible to this form of weld cracking due to the formation of Nb- and/or Ti-rich low-melting-point liquids at HAZ grain boundaries. Numerous mechanisms have been proposed to explain the origin of this liquid, including:

1) the constitutional liquation of Nb-and/or Ti-rich second-phase particles, including carbides, Laves phase and silicides, (Refs. 8–12).
2) the accumulation of solute at gamma-grain boundaries during their migration (i.e., grain boundary sweeping) (Ref. 13).
3) "pipeline": diffusion of Nb from Nb-enriched solidification boundaries in the fusion zone that are continuous across the fusion line into the HAZ (Ref. 14).
4) the penetration of Nb-enriched liquid along gamma-grain boundaries from the fusion zone into the HAZ.

Two additional metallurgical factors influencing the susceptibility of these alloys to liquation cracking are the effects of minor and tramp elements (e.g., B, P, S) on grain boundary wettability and the influence of gamma-grain size (Refs. 15, 16).

A weldability study of Incoloy 903 performed by the authors (Refs. 17, 18) using the spot-Varestraint test found a HAZ liquation cracking susceptibility greater than the Fe-base alloys Incoloy 800 and A-286, and comparable to that of Inconel 718. The HAZ grain boundary liquidation and subsequent cracking was attributed primarily to the constitutional liquation of Nb-rich carbides, although the high degree of liquation in the HAZ also suggested the contribution of additional liquation mechanisms.

Weld Metal Liquidation Cracking

Weld metal liquidation cracking occurs in the underlying pass of a multipass weldment. The susceptibility to this type of cracking generally parallels that of the HAZ. However, the inherent microsegregation remnant from weld solidification, the presence of terminal second phases that exhibit lower liquidation temperature and a coarse fusion zone grain size promote a greater susceptibility to this form of cracking relative to HAZ liquidation cracking. Increased cracking susceptibility was demonstrated by the authors (Ref. 18) for Incoloy 903 using a double-weld, spot-Varestraint technique. The greater susceptibility of Alloy 903 to weld metal liquidation cracking was attributed to the lower constitutional liquidation temperature of Nb-rich Laves phase present at grain and dendrite boundaries in the weld metal versus Nb-rich carbides in the base metal, and an appreciably larger grain size in the weld fusion zone. It is of interest to note that recent weld overlay trials of Incoloy 903 on Inconel 718 have indicated significant difficulties with underbead cracking in multipass overlays (Ref. 19). The intergranular cracking morphology and evidence of incipient melting at grain boundaries in the overlay microstructures strongly suggested a weld metal liquidation cracking phenomenon.

Weld Solidification Cracking

Weld metal solidification cracking occurs during the final stages of solidification due to an inability of the nearly solidified weld metal to accommodate thermal and/or mechanical shrinkage stresses. In austenitic high alloys that contain Nb and Ti, non-equilibrium solidification in the weld
Table 2—Chemical Compositions and ASTM Grain Sizes of Incoloy 903, 907 and 909 heats (wt-%)

<table>
<thead>
<tr>
<th>Element</th>
<th>903/A</th>
<th>903/B</th>
<th>907/A</th>
<th>907/B</th>
<th>907/C</th>
<th>909/A</th>
<th>909/B</th>
<th>909/A1,A2(a)</th>
<th>909/B</th>
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<td>Fe</td>
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<td>bal.</td>
<td>bal.</td>
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<tr>
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</tr>
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<td>&lt;0.01</td>
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<tr>
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<td>0.004</td>
<td>0.002</td>
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</table>

ASTM Grain Size

(a) Incoloy 909/Heat A was rolled from two different ingot product forms and are designated Heats A1 and A2.

Fig. 1—Light and SEM micrographs of solution annealed base metal microstructures in Incolloys 903, 907 and 909. A,B—Incoloy 903/Heat A, note coarse, Nb-rich MC carbides and solute banding parallel to rolling direction; C,D—Incoloy 907/Heat B, note coarse, Nb-rich MC carbides and stringers of fine, Nb-rich carbides; E,F—Incoloy 909/Heat B, note fine, Si-rich particles at prior-gamma grain boundaries.

**Objectives**

Improvements in the high-temperature mechanical behavior of the 900-series superalloys through compositional modifications make these alloys more amenable to widespread commercial usage, particularly by the aerospace industry. However, the nature of the compositional changes required to achieve these property improvements, in particular, increases in Nb and Si, might also be expected to impact the susceptibility of these alloys to cracking during welding. The purpose of this study was to quantify HAZ liquation and weld solidification cracking using weldability test techniques and investigate the mechanics of cracking in Incolloys 903, 907 and 909. Quantitative HAZ liquation and fusion zone solidification cracking susceptibility data were determined for multiple heats of each alloy using the spot-Varestraint and mini-Varestraint weldability tests, respectively. In addition, in-depth metallurgical analyses of weldability test specimens were performed to characterize cracking morphologies and identify cracking mechanisms.

**Experimental procedures**

**Materials**

Multiple heats of Incolloys 903, 907 and 909 were received in the form of hot-rolled strips 6.4 mm (0.25 in.) in thickness. Chemical compositions for each heat are provided in Table 2. Following thermomechanical processing, the Incoloy 903 plate was mill annealed at 940°C (1725°F)/1 h, water quenched, and the Incoloy 907 and 909 plates were solution annealed at 982°C (1800°F)/1 h, water quenched. Since the various heats were rolled from different initial product forms, a range of mechanical processing, the Incoloy 903 plate was mill annealed at 940°C (1725°F)/1 h, water quenched, and the Incoloy 907 and 909 plates were solution annealed at 982°C (1800°F)/1 h, water quenched. Since the various heats were rolled from different initial product forms, a range of as-received microstructures was obtained. As these variations were expected to influence HAZ cracking characteristics, the alloy microstructures were subjected to an additional solution heat treatment. The purpose of this final heat treatment was to both solutionize all precipitates with the exception of high-temperature carbides and carbonitrides and fully recrystallize the gamma grains while limiting grain growth. The heat treatment of Incolloys 903 and 907 at 982°C (1800°F)/1 h, water quenched, and Incoloy 909 at 1038°C
Spot weld

Top View

15.25 cm

2.5 cm

Force

Die block

GTAW torch

Side View

Cracks

Fig. 2 — Illustration of spot-Varestraint weldability test apparatus

Fig. 3 — Schematic illustration of mini-Varestraint weldability test apparatus

(1900°F)/1 h, water quenched, produced nearly equivalent microstructures in all the materials tested. As shown in Fig. 1, the alloys exhibited comparable recrystallized gamma microstructures with grain sizes ranging from ASTM 4 to 6 (Table 1). Incoloy 903 and 907 both contained randomly dispersed coarse carbides, with Incoloy 907 exhibiting stringers of fine carbides running parallel to the rolling direction. Scanning electron microscopy (SEM)/energy-dispersive spectroscopy (EDS) revealed that the coarse and fine carbides were enriched in Nb and Ti. Incoloy 909 (with its reduced C content) showed a distribution of fine particles along prior-gamma grain boundaries and only a few large carbide particles. Microstructural analysis at increased magnification and EDS analysis indicated the fine particles to be enriched in Nb and Si, suggesting Laves or G-phase.

Weldability Testing

Weldability testing was performed on small, laboratory-scale samples 15.2 X 2.5 X 0.65 cm (6.0 X 1.0 X 0.25 in.) machined such that the 15.2 cm (6.0 in.) dimension was oriented parallel to the rolling direction of the original plate. The HAZ liquation cracking susceptibility was evaluated using the spot-Varestraint weldability test, which is schematically illustrated in Fig. 2 (Ref. 22). During this test, a gas tungsten arc (GTA) spot weld is initiated and, after a predetermined time, a pneumatically actuated ram is triggered, forcing the sample to conform to the radius of a pre-selected die block. By maintaining a short delay time between arc extinction and the application of strain, cracking is restricted to the weld HAZ.

Weld fusion zone solidification cracking susceptibility was evaluated using the mini-Varestraint weldability test, which is illustrated in Fig. 3 (Ref. 23). In this test, straining occurs as a moving GTA weld is made with cracks forming in the nearly solidified weld fusion zone at the trailing edge of the molten weld pool.

The tangential (augmented) strain imparted to the outer fibers of the test specimen during the Varestraint tests is approximated by the relationship:

\[ \varepsilon = \frac{t}{2R} \]

where \( t \) is the thickness of the sample and \( R \) is the radius of the die block. In this study, augmented strains of 1, 2 and 3% were applied, with three specimens from each alloy heat tested at the individual strain levels. Test conditions for spot-Varestraint and mini-Varestraint testing are listed in Table 3.

Quantitative cracking data for both tests were obtained by measuring the length of each crack on the as-tested surface using a binocular microscope equipped with a filar eyepiece. Total crack length, maximum crack length and number of cracks were recorded for each sample. All crack measurements were performed at 70X magnification.

Metallurgical Characterization

Representative Varestraint test specimens were metallographically prepared by sectioning, mounting in epoxy, grinding and polishing through 0.06 micron colloidal silica. Microstructural details were revealed by etching with a mixed acid solution comprised of equal parts of concentrated nitric, hydrochloric and lactic acids. Characterization methods included light and scanning electron microscopy and energy-dispersive x-ray analysis.

In order to confirm the liquation nature of the cracking processes, regions of the Varestraint test samples containing cracks were carefully sectioned and then fractured to reveal the crack surfaces for fractographic analysis using SEM/EDS.

Results

HAZ Liquation Cracking

Macroscopic Cracking Characteristics

Figure 4A shows HAZ liquation cracks in an Incoloy 903 spot-Varestraint specimen tested at 3% augmented strain. Cracks were observed to propagate intergranularly and perpendicular to the fusion line and extend from the location of the solid-liquid interface at the instant the sample was strained several grain diameters into the HAZ. Although the epitaxial nature of fusion zone solidification resulted in grain boundary continuity across the fusion line, cracks did not extend into the fusion zone.
Table 3—Spot-Varestraint and Mini-Varestraint Weldability Test Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Spot-Varestraint Test</th>
<th>Mini-Varestraint Test</th>
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</thead>
<tbody>
<tr>
<td>Current</td>
<td>110 A</td>
<td>180 A</td>
</tr>
<tr>
<td>Voltage</td>
<td>12-13 V, DCEN</td>
<td>11 V, DCEN</td>
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<tr>
<td>Travel Rate</td>
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<td>2.5 mm/s (6 in./min)</td>
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<tr>
<td>Electrode</td>
<td>2.4 mm (0.094 in.) dia. thoriated tungsten</td>
<td>2.4 mm (0.094 in.) dia. thoriated tungsten</td>
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<tr>
<td>Electrode-to-work distance</td>
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<td>0.060 in.</td>
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<tr>
<td>Arc Time</td>
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<tr>
<td>Delay Time</td>
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<td>NA</td>
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<tr>
<td>Shielding</td>
<td>Argon, 11.5 L/s (13 cfh)</td>
<td>Argon, 23.0 L/s (30 cfh)</td>
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</table>

Fig. 4 — Macrographs of spot-Varestraint tested Incolloys 903 and 907. A—Top Surface of Incoloy 903 specimen tested at 3% augmented strain, large arrow indicates fusion boundary; B—Top surface of Incoloy 907 specimen tested at 3% augmented strain, large arrow indicates outer boundary of presolidified region.

Occasionally, analysis of the weld fusion boundary region revealed the presence of a presolidified region located between the HAZ and the normal spot weld fusion zone. Microstructural analysis of this region (Fig. 4B) revealed a dendritic solidification substructure appreciably coarser than that of the "normal" weld fusion zone, indicating solidification during the arc-on time at slower cooling rates. The formation of this region was attributed to vigorous, nonuniform fluid flow in the weld pool during the later stages of the test when heat flow had reached nearly steady-state conditions. The observation of presolidification, primarily in Incoloy 909, suggested an effect of silicon on fluid flow, although evidence of the phenomena in Incoloy 907 and heat-to-heat variations also indicated an influence of other minor alloying elements.

The formation of liquidation cracks in these weld metal regions during straining preceded the accurate analysis of HAZ liquidation cracking susceptibility, and consequently data from specimens exhibiting significant presolidification were ignored.

Quantitative Cracking Analysis

The Varestraint weldability test is unique in offering several indices of cracking susceptibility for quantitative analysis and comparison, including total crack length (TCL), total number of cracks (TNC), maximum crack length (MCL), average crack length (ACL) and the threshold strain for cracking. During the past decade, the Varestraint testing of a wide variety of high alloys has shown TCL to generally provide the most representative cracking index.

Total crack lengths for each Incoloy 900-series alloy heat are presented in the form of a bar graph in Fig. 5. Although Incoloy 907/Heat A showed the highest threshold strain required to cause cracking (over 1%), the Incoloy 903 heats exhibited the lowest TCL values at 2 and 3% augmented strain and, thus, the lowest overall susceptibility to HAZ liquidation cracking. With the exception of the relatively high cracking susceptibility of Incoloy 909/Heat A1 at 2% strain, heat-to-heat variations in TCL were within the normal variation observed in spot-Varestraint testing. Figure 6 provides a clearer alloy-to-alloy comparison of relative cracking susceptibility by plotting the averaged TCL for each alloy (average of all heats per alloy) versus augmented strain level. Based on this analysis, Incoloy 903 exhibited the lowest cracking susceptibility, particularly at higher augmented strain levels, followed in order of increasing susceptibility by Incoloy 907 and 909.

The total number of cracks generally correlated with the TCL. For example, the average total number of cracks at 3% augmented strain for Incoloy 903, 907 and 909 were 19, 23 and 28, respectively. In contrast, MCL values were relatively constant and did not show a clear trend. Average MCL values for Incoloy 903, 907 and 909 tested at 3% augmented strain were 0.50, 0.56 and 0.50 mm, respectively.

Microstructure Characterization

Metallographic sections parallel to the specimen's top surface (plan view) were examined at higher magnification to determine the metallurgical changes that occurred in the HAZ as a result of the spot-
Varestraint test and to relate the onset of hot cracking to microstructural features in this region.

Microstructural changes in the Incoloy 903 weld HAZ were first evidenced as a coarsening of the gamma-grain structure and, closer to the fusion line, the onset of a constitutional liquation reaction at the interface between the gamma matrix and coarse, Nb-rich carbides. As shown in Fig. 7 by a widened grain-boundary appearance, the coincidence of these carbides with gamma-grain boundaries due to grain boundary migration allowed the penetration of Nb-rich liquid along gamma-grain boundaries. The extent of carbide dissolution and liquation increased progressively nearer to the fusion zone, with complete dissolution occurring immediately adjacent to the fusion boundary. Liquated grain boundaries that were relatively narrow (on the order of a few microns) were observed to solidify entirely to Nb-enriched gamma. In contrast, liquated regions surrounding carbides and the widened, more extensively liquated grain boundaries nearer to the fusion boundary solidified to a eutectic constituent comprised of gamma and a fine second phase. The EDS analysis (Fig. 7) of the second phase showed considerable enrichment in Nb, Ti and Si to levels that indicated an (Fe,Ni,Co)(Nb,Ti) Laves phase. Although definitive phase analysis via electron diffraction or x-ray diffractometry was not performed in this study, the observation of morphologically and compositionally similar Laves phase in alternate Fe- and Ni-base superalloys further suggested the presence of a Laves phase (Refs. 9, 21). In addition to extensive grain-boundary liquation, intragranular melting was also observed within gamma grains directly adjacent to the fusion boundary (lower right-hand portion of Fig. 7). Such melting may have resulted from the presence of localized Nb-rich regions residual from the dissolution of fine carbides during solution annealing.

As shown in Fig. 1, the Incoloy 907 base metal microstructure differed from Incoloy 903 by the presence of fine, intragranular carbide particles. In the far-HAZ of the 907 spot-Varestraint samples, these fine carbides experienced complete solid-state dissolution in the gamma matrix. Nearer to the fusion boundary, the limited growth of gamma grains (due to liquid film pinning) and constitutional liquation of Nb-rich carbides appeared comparable to those described previously for Incoloy 903. Figure 8 shows evidence of extensive liquation around decomposing carbides and at gamma-grain boundaries in the Incoloy 907 HAZ, and the resolidification of this liquid to a gamma/Laves phase eutectic (location C in Fig. 8). Considering the spatial resolution limitations, compositions of the Laves phase and eutectic gamma in Incoloy 903 and 907 alloys were quite comparable.

Microstructural transitions across the weld HAZ in Incoloy 909 are shown in Fig. 9. Remote from the fusion boundary, only solid-state microstructural changes were observed, including gamma-grain growth and a coarsening of intragranular Laves or C-phase particles. Nearer to the fusion boundary (Fig. 9B), grain growth was more substantial and the second-phase particles experienced a constitutional liquation reaction with the gamma matrix, as suggested by the observation at high magnification of a fine, lamellar eutectic structure where discrete particles had originally existed—Fig. 10. Although these reaction zones were located principally within the grains, grain boundary migration and impingement of the liquated regions promoted continuous grain boundary liquation, as shown in Fig. 9C. Directly adjacent to the fusion line, liquation within the grains and along grain boundaries became extensive—Fig. 9D. As in Incoloy 903 and 907, liquated regions were observed to solidify to a fine gamma/Laves phase eutectic constituent.

Figures 11 and 12 show HAZ liquation cracks and backfilled crack regions in Incoloy 903 and 909, respectively. Cracks in both alloys propagated several grain diameters into the HAZ and, at their farthest extent, were observed along grain boundaries that showed no apparent evidence of liquation. Backfilling of liquid from the fusion zone was observed near the crack mouth, and in some instances strong capillary effects promoted the flow of eutectic back to the crack tip, thus providing complete crack healing. In Incoloy 903 and 907, the liquid solidified to fine, Nb-rich carbides (via a gamma/carbide eutectic reaction) and Laves phase (via the gamma/Laves phase eutectic reaction). As expected, the low-carbon content of Incoloy 909 resulted in solidification en-

![Fig. 7—SEM micrographs of the HAZ in Incoloy 903 and corresponding EDS analysis. Inset shows liquated Nb-rich MC carbide along gamma grain boundary at high magnification](image-url)
Fractographic Analysis

HAZ liquation crack surfaces appeared very similar for each of the alloys tested. As shown in Fig. 13A, the crack surface farthest from the fusion line exhibited a distinct intergranular appearance. Careful examination of these fracture surfaces at higher magnification (Fig. 13B) showed a fine, wavy pattern on the grain faces, and evidence of melting along grain boundaries and at triple points, indicating the presence of a thin liquid film prior to cracking. These observations clearly suggested that fracture occurred by a liquation mechanism.

Nearer the fusion line, surfaces appeared increasingly dendritic due to increased grain boundary liquid resulting from both constitutional liquation and the backfilling of liquid into cracks from the fusion zone and near-HAZ regions. This dendritic resolidification of backfilled liquid on the crack surface is apparent in Fig. 13A. The SEM/EDS examination of the smooth and dendritic-appearing surfaces showed appreciable enrichment in Nb and Si. Nb-rich particles were also observed on the fracture surfaces, with the density of these particles and compositional enrichment greater on the dendritic surface relative to the smooth-appearing surfaces. It is important to recognize that the backfilling of liquid from the fusion zone alters the topography of the crack surfaces as it existed during initial separation. Consequently, the actual mechanism of HAZ cracking may be masked by the presence of this backfilled liquid.

Fusion Zone Solidification Cracking

Macroscopic Cracking Characteristics

Figure 14 shows fusion zone solidification cracks in an Incoloy 903 mini-Varestraint specimen tested at 2% augmented strain. Intergranular cracks were observed to emanate radially outward from the location of the trailing edge of the weld pool at the instant of straining. At the highest strain levels, crack propagation was primarily normal to the trailing edge of the weld pool. The backfilling of liquid from the fusion zone into the cracks was also evident, with a greater degree of backfilling noted in Incoloy 907 and 909 versus Incoloy 903. Completely backfilled solidification cracks and occasional HAZ liquation cracks were not considered in the quantitative cracking analysis of mini-Varestraint specimens.

Quantitative Cracking Analysis

Total crack lengths for each Incoloy
900-series alloy heat are presented in Fig. 15. As indicated, Incoloy 903/Heat B exhibited both the lowest TCL and threshold strain for cracking. Cracking susceptibilities of Incoloy's 907 and 909 were progressively higher and noticeably greater than that of Incoloy 903, particularly at the higher strain levels. Figure 16 provides a clearer alloy-to-alloy comparison of relative cracking susceptibility by plotting TCL averaged for each alloy versus augmented strain level. Interestingly, Incoloy 907 showed a lower cracking susceptibility versus Incoloy 909 at augmented strains of 1 and 3% but a greater cracking susceptibility at 2% augmented strain. Total number of cracks and MCL data generally corresponded with TCL data, although alloy-to-alloy differences in these indexes were less significant. For example, at 3% augmented strain, average TNC's of 7.7, 7.5 and 7.8, and average MCL's of 1.7, 1.8 and 2.0 mm, were determined for Incoloy's 903, 907 and 909, respectively.

Microstructure Characterization

Solidification of the weld fusion zone for each alloy occurred to gamma (austenite) in a cellular-dendritic mode (Figs. 17A, 18A). The partitioning of Nb, Ti and Si to dendrite interstices during solidification (i.e., partition coefficient, \( k < 1 \)) promoted appreciable compositional variations at solidification substructure boundaries and the formation of a terminal gamma/Laves-phase constituent in the last-to-solidify regions. A comparison of solidification structures among alloys qualitatively showed an increase in the quantity of the interdendritic terminal Laves/gamma eutectic constituent with an increase in Nb and Si level (i.e., 909 > 907 > 903). An additional distinction between Incoloy's 903 and 907, and Incoloy 909, was the presence of fine, Nb-rich carbides in interdendritic regions of the 903 and 907 weld metals. The proximity of these carbides nearer to the gamma dendrite cores versus the Laves phase indicated their formation via a eutectic reaction at temperatures above the gamma/Laves-phase eutectic. The apparent absence of carbides in Incoloy 909 was consistent with the low carbon content of this alloy.

Fusion zone solidification cracking in each of the alloys occurred intergranularly with no evidence of crack propagation along solidification substructure (i.e., dendrite) boundaries. Analysis of the cracking morphologies at increased magnification showed a microscopically irregular crack path and evidence of a greater quantity of gamma/Laves-phase eutectic constituent along the grain boundary crack path ver-
sus the interdendritic regions (Fig. 17B). This increase was attributed to greater segregation along grain versus subgrain boundaries and the backfilling of solute-enriched liquid from the fusion zone into the crack. Structure and compositional analysis of the backfilled crack region in Incoloy 903 (Figs. 17B and 17C) show a gamma/Laves-phase eutectic constituent essentially identical morphologically and compositionally to that observed in the backfilled HAZ liquation cracks in this alloy.

Figure 18 shows the solidification substructure and backfilled crack region in an Incoloy 909 mini-Varestraint test specimen. The greater quantity of eutectic constituent in this alloy relative to Incolloys 903 and 907 was qualitatively apparent in both these regions. Compositions of the Laves phase and eutectic gamma, however, were found to be quite similar for all three alloys. The bright-field transmission electron micrograph and associated diffraction pattern in Fig. 19 of the Incoloy 909 fusion zone interdendritic region confirms final solidification to a hexagonal Laves phase.

Fractographic Analysis

SEM fractographs in Fig. 20 show fusion zone solidification crack surfaces in Incoloy 903. As was previously reported (Refs. 24, 25) that for high alloys which experience single-phase solidification, the solidification crack surfaces exhibited a gradual transition from flat fracture near the rear of the crack (farthest from the solid-liquid interface at the time of straining) to an increasingly dendritic fracture near the front of the crack. These fractographic features are a function of the temperature at which cracking occurs, the quantity of liquid present along the boundary at separation, and the nature of the boundary at the instant of separation. As discussed earlier, backfilling of the cracks by liquid from the fusion zone may also influence fracture surface topographies, particularly near the crack mouth. Despite apparent increases in the quantity of interdendritic eutectic liquid in Incolloys 909 and 907 versus 903, fractographic fea-

![Fig. 13 - SEM fractographs of a HAZ liquation crack surface in Incoloy 903. Note backfilling of liquid into the crack as evidenced by dendritic-appearing fracture surface of upper right portion of A](image)

![Fig. 14 - Light macrograph showing the top surface of an Incoloy 903 mini-Varestraint specimen tested at 2% augmented strain. The large arrow indicates the instantaneous solid-liquid interface at the instant of straining, the small arrow indicates the fusion boundary](image)

![Fig. 15 - Bar graph showing total crack length versus alloy heat for Incolloys 903, 907 and 909 mini-Varestraint tested at 1, 2 and 3% augmented strain. Note that Incoloy 903/Heat B did not crack at 1% augmented strain](image)

![Fig. 16 - Total crack length versus percent augmented strain for Incolloys 903, 907 and 909 mini-Varestraint tested at 1, 2 and 3% augmented strain. Note that data obtained for different heats of each alloy have been averaged to provide an alloy-to-alloy comparison](image)
Discussion

During the past decade, the evolution of 900-series, low-expansion superalloys via compositional modifications has been driven principally by requirements for improved elevated-temperature properties. Unfortunately, compositional changes which impart acceptable notch-rupture strength, specifically increases in Nb and Si, are generally detrimental with respect to HAZ liquation (Refs. 9, 15) and fusion zone solidification cracking susceptibility (Refs. 20, 21, 26). Quantitative cracking results obtained in the present study via spot-Varestraint and mini-Varestraint testing confirmed this compositional effect by demonstrating that the relative HAZ liquation cracking susceptibilities of the 900-series, low-CTE superalloys increased with increasing Nb and Si content (i.e. Incoloy 909 was more susceptible than 903 and 907).

Relationships between alloy composition, liquation cracking susceptibility, and cracking mechanisms are complex and multiaxied in these alloys. The metallurgical analysis performed in the present study, however, was useful in providing important insights into the rationale for cracking differences observed among the 900-series, low-expansion superalloys.

HAZ Liquation Cracking

Effect of Liquation Mechanism(s)

An important prerequisite for HAZ liquation cracking is the formation of a continuous or semicontinuous liquid along gamma-grain boundaries in the weld HAZ. If such continuity is not achieved due to lack of sufficient liquid or the poor wetting of grain boundaries by the liquid present, sufficient solid-solid contact along HAZ boundaries may allow welding stresses to be accommodated without fracture. Of the mechanisms that have been proposed to explain the origin of liquation in Fe- and Nb-based superalloys, the constitutional liquation of Nb- and/or Ti-rich second phases remains the most widely reported (Refs. 8-12). In the present study, a constitutional-liquation reaction between coarse, Nb-rich carbides and the gamma matrix was observed in both Incolloys 903 and 907. In these alloys, liquation initiated on heating at the interface between these phases upon reaching the gamma/carbide eutectic temperature. In regions of the HAZ heated above this eutectic temperature, the liquation reaction continued until the carbide was completely dissolved or until the temperature decreased below that of the eutectic reaction.

As shown in Figs. 7 and 8, the resolidification of liquated regions occurred to a gamma/Laves eutectic constituent versus a gamma/carbide eutectic. This difference in solidification product was attributed to the rapid diffusion of carbon and nitrogen from the liquid into the surrounding gamma matrix, which depleted the liquid in carbon and effectively prevented the reformation of Nb-rich carbides. Since the diffusion rates of Nb, Ti and Si in gamma are too sluggish to allow appreciable diffusion into the surrounding matrix, the liquid rapidly reaches a composition that is enriched in these elements relative to the surrounding matrix. On cooling, resolidification of grain boundaries occurs by epitaxial growth from the gamma matrix. In narrow liquated regions, solidification occurs completely to Nb- and Ti- and Si-enriched gamma. However, in widened liquated regions, the segregation of Nb, Ti and Si to the last-to-solidify regions promotes formation of the gamma/Laves eutectic. Although the gamma/carbide and gamma/Laves phase eutectic temperatures have not been precisely determined for the 900-series superalloys, differential thermal analysis (DTA) studies by Knorovsky, et al. (Ref. 27) on Inconel 718 have shown the gamma/Laves eutectic temperature to be appreciably lower than that of the gamma/carbide eutectic temperature (1198°C versus 1298°C). Although gamma matrix compositions differ between the Incoloy 900-series alloys and Inconel 718, a similar difference in the eutectic temperatures may be expected.

In the Incoloy 909 weld HAZ, constitutional liquation was observed between the gamma matrix and fine Laves- or G-phase particles. A lower eutectic-liquation temperature of the Laves- or G-phase versus the Nb-rich carbides in Incolloys 903 and 907 would be expected to promote the onset of HAZ liquation at an even lower temperature, and, consequently, a larger region of partial melting would be expected in Incoloy 909. Although microstructural examination did, indeed, reveal a wider region of liquation in the Incoloy 909 HAZ, a noticeably greater MCL was not observed. Previous investigators have correlated MCL values obtained at high, saturated augmented strain levels to the locus of the eutectic liquation temperature isotherm in the HAZ (i.e. the effective solidus), assuming crack propagation occurs exclusively along liquated grain boundaries. The observation of similar MCL values for the 900-series superalloys, despite differences in the constitutional liquation temperatures of second phases in the HAZ, may be attributed to the grain boundary penetration of the gamma/Laves eutectic liquid formed in Incoloy 903 and 907. This explanation assumes similar
compositions and eutectic liquation temperatures for Laves or G-phase in the Incoloy 909 and Laves phase in Incoloy 903 and 907, which was suggested by EDS analysis. Alternatively, the lack of a clear correlation between MCL and HAZ eutectic liquation temperatures may be related to the incomplete saturation of strain at the 3% augmented strain level.

In addition to a lower constitutional liquation temperature, an appreciably greater number of second phase particles in Incoloy 909 versus carbides in Incoloy 903 and 907 promoted more extensive intragranular and intergranular liquation. The greater availability of liquid at grain boundaries may have further contributed to an increased cracking susceptibility of Incoloy 909.

The high degree of HAZ grain boundary liquation in the Incoloy 900-series alloys suggested that the constitutional liquation of Nb-rich second phases was not the only operative liquation mechanism. Studies by Lippold (Ref. 13) on the Fe-base superalloy Incoloy 800 suggested that titanium present in the gamma matrix due to the solid-state dissolution of fine carbides on heating may be "swept up" by migrating gamma grain boundaries. The gradual enrichment in titanium content reduces the effective solidus temperature of this region, ultimately promoting melting. A subsequent microanalytical study of simulated HAZ microstructures in the same alloy system, however, failed to detect significant titanium at these boundaries (Ref. 28). This suggests that even if some niobium is swept into HAZ grain boundaries in 900-series alloys, the amount of niobium assimilated into the moving boundary could not explain the substantial liquation along these boundaries.

In the present work, however, EDS analysis could not detect Nb enrichment along grain boundaries that had not experienced liquation or liquid penetration. If a grain boundary sweeping mechanism was operative, then the enriched grain boundary layer was too narrow to allow detection by SEM/EDS. In this context, it is important to recall that crack propagation identified by fractographic analysis to be liquation-related (Fig. 13) occurred principally along HAZ grain boundaries devoid of observable liquid, thereby suggesting the presence of such an extremely thin liquid film.

Since gamma grain boundaries are continuous across the fusion boundary, an alternate mechanism for Nb-enrichment at HAZ grain boundaries may be the diffusion of Nb concentrated along fusion zone grain boundaries due to partitioning during solidification across the fusion boundary into the HAZ. Termed pipeline diffusion, this solid-state, diffusion-controlled mechanism could significantly increase the HAZ grain boundary concentration in Nb and also interstitial elements such as Si, P, and B. The continuity of gamma-grain boundaries across the fusion boundary may also promote the penetration of Nb-rich liquid from the fusion zone into the HAZ, which may be enhanced by the presence of a stress field across the boundaries (particularly during severe deformation imposed during the Varestraint test). Unfortunately, as with other mechanisms, it is both analytically and experimentally difficult to determine the exact contribution of the pipeline diffusion and/or liquid-penetration mechanisms to HAZ liquation and cracking.

Effect of Minor Alloying Elements

Minor alloying elements in Fe- and Ni-base superalloys, particularly Si and B, have been shown to have detrimental effects on HAZ liquation cracking susceptibility. Brooks (Ref. 9) has shown that increased Si in A-286 promotes the formation of a gamma/Laves eutectic and suppresses the eutectic temperature, thereby increasing cracking susceptibility. Studies in A-286 (Ref. 9) and Inconel 718 (Ref. 15) both indicate detrimental effects of boron due to the constitutional liquation of borides and the effectiveness of boron in increasing the wettability of liquid films. This latter effect is of particular significance, since the extent of grain boundary liquation is determined both by the volume of liquid available and the effectiveness with which this liquid wets and penetrates grain boundaries. Based on these observations, it is suggested that an increased Si content may have contributed to the increased susceptibility of Incoloy 909 versus Incoloy 903 and 907 to HAZ liquation cracking.

Conversely, Incoloy 903 and 907 exhibited appreciably greater boron levels than Incoloy 909, indicating that, at the levels present, boron was ineffective in influencing liquation cracking, or that its influence was overshadowed by competing effects.

Effect of Grain Size

The detrimental effect of large grain
size on HAZ liquation cracking has long been recognized in Ni-base superalloys. This effect is generally explained by considering the distribution of a constant volume of liquid on the grain faces, where large-grained materials provide less surface area for liquid coverage than fine-grained microstructures, thus reducing the solid-solid contact area. In addition, it has been suggested that cracks initiate by a grain boundary sliding mechanism at triple points (Ref. 16). A large grain size increases stresses at these points, and thereby provides an increased opportunity for crack initiation.

Since gamma grain sizes in the present study varied from approximately ASTM 4 to 6, a systematic analysis of grain size effects was not possible. Comparing base metal grain sizes (Table 2) with heat-to-heat variations in TCL for a specific alloy, it is apparent that the coarser ASTM 4 grain sizes exhibited by Incoloy 903/Heat B and Incoloy 907/Heat A may contribute to their greater cracking susceptibilities relative to other heats of the same alloys. A similar relationship was not apparent for Incoloy 909.

It is also important to note that it is not the base metal grain size, per se, that is significant, but rather the grain size in the HAZ over the liquation temperature range. Since grain growth may be rapid at temperatures approaching the alloy solidus, base metal grain size should only be used as a rough guideline for determining the influence of grain size on cracking susceptibility.

**Fusion Zone Solidification Cracking**

Solidification of the weld fusion zone in the 900-series superalloys was associated with the significant partitioning of Nb, Ti and Si to the last-to-solidify interdendritic regions. As shown by EDS data presented in Figs. 17 and 18, this partitioning promoted appreciable differences in the concentration of these elements between the dendrite cores and the interdendritic regions (where the concentration reached the maximum solid solubility and promoted the formation of a gamma/Laves terminal eutectic).

Solidification cracking in the Incoloy 900-series alloys was observed to occur exclusively along fusion zone solidification grain boundaries and to be associated with the segregation of Nb, Ti and Si to these boundaries via both microsegregation effects, operative on a solidification substructure scale, and macrosegregation effects. Enrichment due to microsegregation results from the impingement of solute-enriched liquid boundary layers that form ahead of the advancing solid-liquid interface. This macrosegregation is best manifested by the distinct solidification grain boundaries along which cracking is most prevalent. In addition to enhanced segregation effects, cracking is predominant along the solidification grain boundaries due to the macroscopic straightness of these boundaries and their orientation relative to the principal shrinkage stresses. It is also likely that these boundaries are more easily wet than subgrain boundaries due to the higher concentration of impurity elements.

Alloy susceptibility to solidification cracking is also influenced by the alloy freezing range, or more precisely the brittleness temperature range (BTR). Solidification over a large temperature range permits shrinkage stresses to concentrate along liquid or semi-liquid boundaries during the final stages of freezing. Above a threshold level of stress, these boundaries will separate and form a crack if sufficient liquid is not present to "heal" the separation. In this respect, the quantity of eutectic liquid is often critical in determining whether crack healing occurs. If the amount of liquid present is just sufficient to completely wet the solidification grain boundaries, cracking susceptibility will be maximum. A decrease in this amount allows solid-solid contact along the boundary and increases the load-bearing capability of the boundary, thus reducing its susceptibility to cracking. If the quantity of liquid increases above the level to just promote complete wetting, additional liquid will then be available for healing.

In the 900-series alloys, as in other alloy systems, it is often difficult to determine the critical level of eutectic liquid that produces maximum susceptibility. It is evident from Figs. 16 and 17 that eutectic liquid was present along solidification grain and subgrain boundaries in the alloys evaluated. Assuming a binary eutectic system between the gamma solid solution and Laves phase, and Scheil-type solute partitioning in the weld fusion zone (Ref. 29), increases in the alloy Nb content would be expected to reduce the liquidus temperature, decrease the freezing range (since the eutectic temperature is fixed) and increase the quantity of eutectic constituent formed at solidification boundaries. The influence of increased Si is not so apparent, although it might be expected that segregation of Si could further lower the eutectic temperature and thereby increase the solidification range and BTR.

As indicated above, MCL data obtained in Varestraint testing under saturated strain conditions are frequently correlated with the BTR and the effective solidus temper-
ature. Although strain saturation at 3% augmented strain was not confirmed in this study, higher average MCL values observed in Incoloy 909 (1.95 mm) versus those in Incolys 903 and 907 (1.7 and 1.6 mm, respectively) did suggest a larger BTR and lower effective solidus (i.e. gamma/Laves eutectic temperature) in the higher-Si Incoloy 909.

Microstructural comparisons between the Incoloy 903, 907 and 909 fusion zone microstructures revealed greater quantities of the gamma/Laves eutectic constituent with increased Nb and Si content (i.e., 909 > 907 > 903). For correlation purposes, a Scheil calculation (Ref. 29) was performed to predict the approximate volume fraction of gamma/Laves constituent in the weld fusion zone. Based on EDS data for Nb concentrations at the dendrite cores and in the interlamellar gamma (this concentration approximates the maximum solid solubility) eutectic liquid volume percentages of 2.9, 4.6 and 8.5% were calculated for Incolys 903, 907 and 909, respectively. This progressive increase in eutectic liquid correlated well with microstructural observations. It is important to note that higher carbon content levels in Incolyos 903 and 907, and the formation of Nb-rich carbides during solidification, would actually result in the formation of less terminal gamma/Laves eutectic constituent in these alloys than the Scheil calculation predicts.

As in other alloy systems, relating the quantity of eutectic liquid to solidification cracking susceptibility in the 900-series alloys is not straightforward. As noted previously, a critical level of liquid exists at which boundary wetting is essentially complete. This value is of course dependent on the wetting characteristics of the liquid. At low levels of eutectic liquid, increased liquid may promote greater grain boundary wetting and increased cracking. However, above the critical level, additional liquid becomes beneficial due to crack healing effects. Quantitative cracking results obtained from the mini-Varestraint test indicating a greater susceptibility of the higher Nb + Si Incoloy 909 versus Incolyos 903 and 907 suggest that eutectic liquid levels in these alloys were below this critical value for crack healing.

Conclusions

1) Spot-Varestraint weldability testing determined the relative HAZ liquidation cracking susceptibilities of the 900-series, low-expansion superalloys to be 903 < 907 < 909.

2) Heat-affected zone liquidation cracking was attributed to the presence of a low-melting, Laves phase/gamma eutectic liquid at gamma-grain boundaries that was compositionally enriched in Nb, Ti and Si. Origins of this liquid included the constitutional liquation of Nb-rich carbides in Incolys 903 and 907 and Laves or Gphase in Incoloy 909.

3) Heat-affected zone liquidation cracking susceptibilities were correlated to the quantity of liquid present at HAZ grain boundaries, with volume increasing with increased Nb + Si content.

4) Mini-Varestraint weldability testing determined the relative fusion zone solidification susceptibilities of 900-series, low-expansion superalloys to be 903 < 907 < 909.

5) Weld solidification cracking susceptibility originated from the partitioning of Nb, Ti and Si to grain and solidification substructure boundaries during solidification and the formation of low melting-point terminal Laves/gamma eutectic.

6) The relative susceptibility to fusion zone cracking among alloys was related to the effects of increased Nb, Ti and Si levels on the quantity and freezing temperature of the terminal eutectic liquid.

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