Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

Materials & Molecular Research Division

MASTER

THE WELDABILITY OF THERMALLY GRAIN-REFINED Fe-12Ni-0.25Ti
FOR CRYOGENIC STRUCTURAL APPLICATIONS

Douglas Edwards Williams
(M.S. thesis)

February 1980

Prepared for the U.S. Department of Energy under Contract W-7405-ENG-48

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

THE WELDABILITY OF THERMALLY GRAIN-REFINED Fe-12Ni-0.25Ti FOR CRYOGENIC STRUCTURAL APPLICATIONS

By

Douglas Edwards Williams
Lawrence Berkeley Laboratory
Master of Science in Engineering University of California, Berkeley Physic Metallurgy
Berkeley, California 94720

ABSTRACT

The weldability of a research alloy designed for structural use in liquid helium temperature, cryogenic environments was investigated. Plates of iron-12 weight percent nickel-0.25 weight percent titanium were grain refined by the four-step, grain-refining thermal treatment developed for this alloy and welded with Inconel Number 92 weld wire using the Gas Metal Arc (GMA) welding process with argon-15% helium gas shielding. Both a single pass and a double-sided, 2 pass electron beam (EB) weld were also made without filler metal addition. Weldments were radiographed and sectioned and the charpy V-notch specimens removed were tested at liquid nitrogen and helium temperatures.

Although radiographically acceptable weld deposits were somewhat difficult to achieve (only 4 out of 7 GMA welds and 2 out of 4 EB welds were acceptable), the problems encountered during EB welding were largely due to alignment and weld metal fluidity while GMA welds were confounded by the sensitivity of the weld metal to oxidization and the production of inclusions rich in titanium. The minimum charpy V-notch impact values obtained for the heat affected zones of an acceptable GMA weld were 50 and 62 foot-pounds at liquid helium ($\sim 4^\circ\text{K}$) and liquid nitrogen (-196°C) testing temperatures, respectively. Weld metal toughness for Electron Beam

welds were 15 and 23 foot-pounds at these test temperatures, but HAZ values rose from 18 and 29 foot-pounds, respectively, for the single pass to 33 and 96 foot-pounds, respectively, for the two pass welds. Typical values for the base metal are 95 and 120 foot-pounds at these two temperatures.

Full sized charpy specimens were subjected to simulated HAZ thermal treatments, although heating rates were one-third slower than those measured. Testing at liquid helium and nitrogen testing temperatures revealed an increase in impact toughness for any treatment in which the temperature exceeded the transformation temperature, regardless of the duration.

I. INTRODUCTION

Requirements for structural materials in the expanding cryogenic technology demand combinations of high strength coupled with high toughness at temperatures to that of liquid helium ($\sim 4^\circ\text{K}$). For example, it would be desirable for superconducting magnet cases under the projected twelve tesla magnetic field strength, to have in excess of 180 ksi yield coupled with fracture toughness above about $150 \text{ ksi}\sqrt{\text{in}}^1$. The scale of this application will most assuredly preclude any form of post weld heat treatment, thereby eliminating materials such as precipitation hardening steels from the already short list of potential materials. Since, in all but possibly the nitrogen strengthened grades (e.g. Nitronics), austenitic stainless steels do not have a sufficiently high yield strength at cryogenic temperatures, ferritic steels are being studied even though these materials are ferromagnetic and undergo a brittle transition. One such steel, which was designed² and developed^{3,4} at UCB/LBL, meets these requirements with less than 13% alloying elements.

Although in the as-normalized state ($\sim 15\mu$ grain size: ASTM #9) the fracture toughness of Fe-12Ni-0.25Ti is only $75 \text{ ksi}\sqrt{\text{in}}$ at 6°K , over 200 $\text{ksi}\sqrt{\text{in}}$ are obtained when the grain size is reduced to approximately 1μ by a thermal cycling procedure developed for this alloy³. Since its yield strength at this temperature is 195 ksi, this alloy seems to be well suited to the need. There are, however, two potential problems to be addressed before this alloy could appear in such a design specification. The commercial feasibility of the four-step heat treatment and the weldability of the grain-refined alloy must be proven. The research project described herein attempts to determine the weldability of this alloy in view of the above requirements while utilizing commercial welding techniques suitable

for construction on the appropriate scale. A fusion welding process suitable for both initial research studies and construction on the required scale is the gas metal arc (GMA) welding technique. GMA welding is characterized by the lack of any fluxing additions since the shielding medium is a gas and by the lack of heating other than through the filler metal itself. An electric arc is maintained between the work and a spool-fed wire of filler metal, thereby allowing a high deposition rate to be achieved. From a researcher's standpoint, the analysis of deposited metal and inclusions is simplified by the lack of fluxing compounds since virtually all additions can be accounted for in the weld deposit as none are lost in slag.

The heat input inherent in any fusion welding process can be anticipated to significantly alter the base plate microstructure within a heat affected zone (HAZ), that region within which temperature excursions above the transformation range were attained. The heat input is defined as the rate of arc power consumption:

$$\text{Heat Input} = \frac{VI}{S} \text{ (j/mm)}$$

where V = the arc voltage, I is the effective current through the arc and S is the travel speed in mm/sec. This arc energy is sufficient to melt both the filler wire, the end of which acts as the anode, and the base plate which is the grounded cathode. During welding, therefore, a temperature gradient exists which extends from above the liquidus temperature within the weldpool, through the two phase, liquid-plus-solid region found at the fusion zone, to the adjacent metal which has been heated above the transformation temperature and is thus called the heat affected zone (HAZ), to, finally, the unaffected parent metal beyond.

At the fusion zone the cellular solidification structure of the weld metal can be seen to grow from base metal grains even if the weld metal is

austenitic and the base metal ferritic. This interaction can be seen in Figure 1 which shows the root pass area from a double bevel weld of Fe-12 Ni-0.25 Ti with Inconel #92 GMA weld wire. The prior austenitic grain size of the base plate in this region has grown from approximately $1-2\mu$, as shown in Figure 2, to $15-20\mu$ by the heat treatment caused by the passage of two adjacent weld passes. The combination of mechanical and cathodic vacuum etching clearly shows the austenite grains are contiguous across the 1μ fusion zone even though the base metal is no longer austenitic. Farther away from the fusion zone in the HAZ, a region within which the peak temperature only just exceeded the AC_3 temperature will generally be finer grained. Since the time at temperature is short and the kinetics of grain growth are far slower than those of austenite nucleation, this grain refining process will occur in a ferritic weldment regardless of whether the martensite (α') to austenite (γ) transformation is shear or diffusional. A simple shear reversion is not expected due to the presence of the complicated stress and temperature gradients associated with the fusion welding process. Since the ductile-to-brittle transition temperature of this alloy is strongly dependent on grain size, significant problems were anticipated in full penetration fusion welds in which a grain coarsened region could extend continuously through the weldment.

Although weldability studies have been performed on this and two similar alloys⁵, the present study was based upon the desire to extend the usefulness of this alloy to larger structures, higher deposition rates and lower temperatures. Where the Develietian, et al.,⁵ study demonstrated successfully the weldability of this alloy using autogeneous gas tungsten arc (GTA) welding for thin-wall, liquified natural gas containers, the present investigation sought to determine the effects of using the GMA process in thicker (1/2 inch) sections.

The approach used in the present investigation sought to determine the extent of the degradation of properties within the heat affected zone through the use of dissimilar weld metal, GMA weldments, autogeneous electron beam (EB) weldments and HAZ simulation specimens. These techniques complemented each other since a weakness detected in the HAZ near the fusion line of the GMA welds could be seen in contrast to comparable specimens from EB welds and HAZ simulations. The effects of weld metal chemistry and weld heating on the fusion zone could thereby be sorted. Thus, following the current practice of welding ferritic materials for cryogenic service⁶, GMA weldments were made using Inconel #92 filler wire and were impact tested at liquid nitrogen and liquid helium testing temperatures. In addition; single and two pass autogeneous EB weldments were similarly evaluated. In both processes, welding was accomplished on fully automatic equipment. To isolate fusion zone effects from HAZ properties, specimens were heat treated to simulate HAZ conditions and similarly impact tested at the two cryogenic temperatures.

II. EQUIPMENT

An automated GMA welding process using Inconel #92 weld wire with non-reactive shielding gases was chosen in an attempt to minimize the number of variables which had to be dealt with during the course of the investigation. Since the primary question appeared to be the effect of the welding heat input on the base plate microstructure and, thus, properties, the selection of the GMAW process using Inconel filler metal was based on both current industrial practice in similar cryogenic alloys⁶ and the ability to separate weld metal effects from base metal characterization. Thus, a ferritic filler metal would have been undesirable since its ductile-to-brittle transition temperature (DBTT) would be confused with that of the HAZ, assuming that the weakest part of the weldment lay within the fusion zone. Inconel filler metal was also chosen for its constancy in absorbing impact energy down to liquid nitrogen temperature (119.5 ft. lb. at room temperature compared to 118.0 ft. lb. at -296°C)⁷. A chemical analysis of the Inconel alloy used is given in Table I. The non-reactive gases were used for shielding to avoid introducing any interstitial impurities into the weld as these are known to decrease the toughness of ferritic alloys at cryogenic temperatures. It was also of concern to minimize the variance of weld travel speed and direction during each pass so that the HAZ would be both straight and uniform. Accomplishing this objective by using a fully automated arrangement allowed easier and more accurate notch placement for HAZ specimens. It also afforded the maximum confidence in the mechanical test data and HAZ characterization taken from individual specimens. Virtually completely reproduceable weld passes were obtainable on the automated welding table designed and constructed as a part of this investigation (Figure 10).

The design of the weld table to automate a Miller Spoolmatic welding gun was based on the ease of use, repeatability of parameters and adaptability to other welding research. A stationary gun with a moving work piece was deemed desirable to facilitate the attachment of leading and trailing gas nozzles, cameras, arc spectographs or other equipment for analyzing the arc and weld pool during welding. The problem of vibration and constant travel speed were minimized in the threaded drive shaft design by employing a free floating split nut to transmit only a linear driving force. Adjustable spring pressure was utilized to clamp the split nut to the driveshaft, thus providing a safety release, while the nut halves were pinned like reverse pliers for manual table positioning. A tilting capability was incorporated into the gun mount to allow forehand, backhand or vertical gun position techniques to be used. Vertical height was made adjustable over a two foot range with a four inch fine adjustment available at each coarse height setting. This range proved useful when it became apparent that a square bevel on one plate (i.e. single and double bevel, or K, edge preparations) was necessary to realistically evaluate the mechanical properties of the HAZ. A hinged plate was used to incline the centerline bisecting the 45° and the square (90°) plate bevel angles to the welding gun axis. The additional six inches that this arrangement added to the weldment height was easily accommodated by the vertical height adjustments. Both gun trigger controls and weld table movement controls were re-routed to a central control box. Oscilloscope pictures sampling (chopped) simultaneous voltage and current waveforms enabled an independent check on arc transfer characteristics.

III. MATERIAL PREPARATION WELDING AND SPECIMEN PREPARATION

Alloys of nominal composition Fe-12Ni-0.25Ti with low carbon were induction melted in an argon atmosphere then cast into 20 pound ingots in a copper chill mold. A typical chemical analysis is shown in Table II. Homogenization was performed under vacuum at 1200°C for 24 hours. The ingots were then upset cross forged at 1100°C into 1/2 inch thick by 7-9 inch wide plates. Each plate was then annealed at 900°C for two hours to remove prior deformation strain and to provide a constant starting point for subsequent grain refinement.

Grain refinement was accomplished through the four step (2B) heat treatment process previously developed for this alloy³ with the exception that heating rates were generally slower (12-14°C/min vs 15°C/min) and water, rather than air, quenching was necessary to compensate for the larger specimen mass. Grain refinement through this four step heat treatment is accomplished by repeatedly heating first to a temperature just above the austenite transformation temperature, or A_{c3} , holding for two hours and quenching, and, secondly, heating to a temperature just below the A_{c3} , holding for two hours and quenching. The practical limit to grain refinement of this material with this process (approximately one-half micron average grain size) will usually be reached in a few repetitions for small samples. The process of grain refinement is based on providing the conditions preferential to nucleation while carefully avoiding those which promote growth. During the first part of the cycle, many austenite grains are nucleated but few grow since there is insufficient thermal energy for the necessary diffusion. The resulting martensite laths are subsequently altered by the two-phase decomposition step since the stable austenite nucleating is chemically different from that previously precipitated. It is

important to note, however, that it is not possible to avoid conditions favorable to grain growth during welding.

The plates were then cut and the edge preparations machined. Single and double Vee and single and double bevel end preparations were used. In all cases a 1/32 to 1/16 inch land and a 1/32 to 3/32 inch root gap were employed to attempt full penetration on the root pass.

The parameters used during welding were derived from optimizing the weld contour and penetration by running beads-on-plate with varying open circuit voltages, currents, weld stickouts, travel speeds and shielding gas mixtures. Spray transfer, which was necessary to minimize the introduction of defects, was detected by recording sample oscillographs of simultaneous voltage and current wave forms. The welding parameters determined by these tests remained approximately constant throughout the course of the research and were an open circuit voltage of 41-43 volts, a stickout of 3/4 inch, and a shielding gas mixture of Ar + 15% He except as noted in Table III. The variable which was manipulated in order to change the heat input to accommodate differences in weld joint configurations was primarily the travel speed (from 10 to 30 inches per minute) although weld numbers 11 and 12 had arc currents between 270 and 310 amperes while all other GMA welds were made between 240 and 280 amperes. Except for the root pass which was ground out in all but weld 11, welding parameters were kept constant to the extent possible between weld passes of the same weld. No pre-heat was used and interpass temperatures were kept below 60°C with water-cooled, copper coils to increase cooling rates. Out of the seven GMA welds attempted, one was rejected for an excessive amount of distortion and two more were found to have lack of fusion by radiography.

Electron beam welds were made at 60 kV, 50 ma and 10 inches per minute under high vacuum. Four welds were made since the first two contained

continuous lack of penetration defects found by radiography. These defects were assumed to be at the plate mid-thickness since these were both two pass welds made from opposite sides. Weld 7 was successfully made from one side with the use of a back-up strip, while weld 8 was again a two pass weld although radiographically sound. Again, no preheat was used and interpass temperatures were kept below 100°C.

Radiography and relevant surface examinations were performed on all welds. When necessary, liquid penetrant examination was performed between weld passes and ultrasonic examination was used to determine the depth of indications observed on radiographs. Using the radiographs to circumvent any weld defects, the welds were sectioned with all but the first and last 3/4 of an inch capable of providing specimens.

Since no post weld heat treatment was performed, a procedure had to be developed to accommodate the distortion of specimens due to residual stress relaxation. The convex side of the weld plate (if the plates had become bowed transverse to the weldment) was first milled flat, to a distance of 1-1/4 inch from the weld centerline on the beveled side and an equal distance from the fusion line on the square bevel side. This section of the plate was then cut from the remaining plate and the cut edges milled parallel to the weld centerline before the backside was milled (to 0.430 inch). A photographic print of the radiograph was then superimposed, the specimens laid out and subsequently cut on a water-cooled abrasive wheel. One side of each specimen was then milled to 0.415 inch leaving the smooth, cut side for macroetching and specimen centerline location. The specimen was then turned 90° and milled to 0.415 inch, thus insuring squareness. The notch centerline was then scribed. With calipers set at 1.088 inch for a reasonable narrow scribe, the ends were then scribed, saw cut, and one side milled before milling the other end to size. Metal stamp numbers were

oriented such that the notch would be on top with the specimen number placed above the weld number.

The specimens removed included charpy V-notch, bend and optical. Optical metallography specimens taken from welds made with the Inconel weld wire were polished and either differentially etched chemically or slightly mechanically etched followed by a cathodic vacuum etch⁸. Due to the drastically different etching responses between the weld and the base metal, only cathodic vacuum etching was successful in allowing high magnifications of the fusion zone. Only high energy boundaries such as prior austenite grains are expected to be etched since the process, which is similar to the etching of gold during sputtering, depends on the ejection of metal atoms by ion bombardment.

Mechanical testing was performed at both liquid nitrogen (LN) and liquid helium (He) temperatures. Standard charpy V-notch specimens were impacted within 5 seconds of LN immersion in accordance with the ASTM specification⁹. Impact specimens of 51mm length were broken at liquid helium temperature in accordance with a procedure described elsewhere¹⁰.

Temperature profiles occurring during welding were established by recording temperatures from different weld passes with a stationary thermocouple located on the plate surface located 3/16 inch away from the square bevel. The thermocouple was thus approximately 3/8 inch away from the root pass and within 1/32 inch from the final pass. Specimens 1/2 by 1/2 by 2-1/2 inches were then subjected to similar heat treatments although heating rates were slower (by 1/3) and cooling rates were somewhat faster. Since the complete cycle in both cases took less than thirty seconds for heating from room temperature to $\sim 1000^{\circ}\text{C}$ and quenching to below the M_f temperature, a rough comparison could be made between the effect of the

actual weld HAZ with its partially melted (fusion) zone and residual stress field and that of the heat treatment alone. The simulation study was conducted on a 25 kW induction furnace with a water quenching medium. Temperatures were determined by an optical pyrometer for temperatures above 750°C and a K-type thermocouple for temperatures below.

IV. RESULTS AND DISCUSSION

GAS METAL ARC WELDS

The results of mechanical testing on welds done in this study are shown in Table III. Except in the case of simulation studies, base metal impact test specimens were taken transverse to the major rolling axis and only one specimen was tested per plate because they were used primarily to determine the effectiveness of the grain refining (2B) heat treatment. Owing to limitations in base plate material and, thus, weld length, the majority of mechanical test specimens taken from the Inconel/GMA welds were extracted so as to test heat affected zone properties rather than those of the weld metal.

The initial welding procedure was based on the expectation that degradation of the grain refined structure through both grain growth and tempering would be a principal source of the loss of cryogenic toughness. As the welding of a metal which has been thermally grain refined to an ASTM grain size of 18 or smaller is a new form of welding problem, the procedures used in welding quench and tempered materials were taken as a guide. Thus stringer passes, low interpass temperatures and low heat input were considered desirable although these considerations were modified by the necessity to minimize the defect level.

Although defect types such as lack of complete penetration or fusion are notorious in GMA welding and would be expected to be considerably worse in a fully automated GMA welding situation, the automated system was chosen out of consideration for improving the confidence that each specimen was representative of the weld since many more variables could be controlled and repeated. The most significant of these variables include travel speed, stickout and oscillation. With this apparatus, however, the

optimum transfer mode was necessarily spray. To minimize the heat input to the base plate during welding, a high travel speed (often above 25 inches/minute) and long stick out (over 3/4 inch) were necessary to compensate for the required increase in electrical power through the arc.

An optimum shielding gas, argon with approximately 15% helium, was determined by running beads-on-plate with widely varying gas ratio mixtures. The Inconel 92 alloy was found to be extremely sensitive to oxidation. At the welding speeds used, unless a four-inch long trailing gas nozzle (pure argon) immediately followed the gun nozzle, a wrinkled oxide skin often obscured even the weld ripples. Oxide inclusions were common on fracture surfaces even in weld beads with bright, unoxidized surfaces. Brittle inclusions or the remains of inclusions, such as in Figure 3, which shows an inclusion from the fracture surface of a HAZ specimen from weld 3, always registered high in titanium in EDAX analysis, usually in proportion to their volume, but were never sufficiently wide and thick to obtain a background-free count. As thin and broken-up as the remains of the inclusion in Figure 3 obviously are, an EDAX computer estimate of the weight percent composition (assuming $\text{Fe}+\text{Ni}+\text{Cr}+\text{Mn}+\text{Ti} = 99\%$) determined titanium at above 83%. Since the beryllium window of the EDAX counter did not allow detection of elements lighter than fluorine, any titanium oxide, nitride and carbide, etc., which was large enough would then show high titanium percentages. Fracture surfaces of Inconel weld metal specimens sometimes revealed large areas of titanium-rich surfaces such as the 150 by 50 μm film evidenced in Figures 4 and 5.

As mentioned earlier, titanium-rich inclusions were by no means restricted to the Inconel 92 weld deposit but were visible on virtually all base plate fracture surfaces as the small, numerous particles at the base of ductile "cones" (Figure 6). Although the fracture surfaces of the simulation

study specimens were very similar to those of the as-grain-refined (2B) base metal, very little ductility was evident in the electron beam weldments, especially in fracture surfaces representing the weld metal.

As the degree of gas shielding used was far more elaborate than that expected in the field, it was concluded that there was an excessive amount of titanium in Inconel Alloy 92 for successful shop welding with gas shielding unless close attention was paid to the effectiveness of the gas coverage. Using semiautomatic techniques, short circuit transfer could probably reduce the problem with large oxide inclusions. Fewer inclusions would be created during deposition since the exposed surface area is drastically reduced in this transfer mode. Short circuiting welds, however, are more susceptible to lack of fusion defects.

Titanium-based, non-metallic inclusions attract the arc since the thermionic work function of titanium is more than 0.5 eV below that of either iron or nickel. Under such a concentrated arc titanium carbide or nitride would melt even though their liquidus temperatures, at 3140°C and 2950°C¹³ respectively, are more than twice that of the iron-12 nickel base metal (~1500°C)¹⁴ and over 1500°C above the 1430°C liquidus of the Inconel. Solidification of those three phases could be assumed to follow this order on cooling since the solidus of the base plate lies above the liquidus of weld metal. The formation of titanium films could be explained by the physical pressure of arc force and weight of molten weld metal. The presence of these films at the fusion zone, as seen in Figure 3, is also aided by the numerous small inclusions present in the base plate as shown in Figure 6.

The combination of weld metal fluidity and melting range difference between the weld and base metals also required a further change in welding procedure. A forehand gun tilt of 15° was necessary to control the size of the weld pool. At smaller angles the weld pool front was almost vertical

so that when the arc wandered or became attracted to a titanium inclusion, the associated lack of arc cleaning action on the adjacent weld groove would produce lack of fusion regions where the pool collapsed. Figures 7 and 8 depict such a region where the resulting void produced clusters of cactus-like metallic growths precipitated as deposition from the vapor onto the still-visible, machined bevel. Based on $\text{Fe}+\text{Ni}+\text{Cr}+\text{Mn}+\text{Ti} = 99\%$, an EDAX computer estimate of weight composition gave approximately 35% Ni, 27-31% Cr, 11-16% Fe and 1-1/2-2% Ti. Transfer was spray/globular with periodic short circuiting during the deposition of the double bevel groove weld.

Problems with lack of fusion and especially porosity were further compounded by the fact that when the base metal was molten, the weld metal was superheated and when the base plate was solidified, the weld metal was within the two-phase liquid plus solid region.

In spite of the problems in obtaining weld deposits with acceptable defect levels, impact toughness was usually good. Most toughness values were greater than 50 ft-lbs in LHe and 60 ft-lbs in LN for HAZ specimens near the fusion zone, which was generally the weakest region of the weldment. The only exceptions to these minima were for specimens with defects observable in non-destructive examinations.

The observation that the fusion zone was the region of minimum toughness in the weldment in all Inconel/GMA welds, except those deposited in the 'jet-rotation' transfer regime (welds 11 and 12) where HAZ values were unchanged while weld metal properties deteriorated, suggests three likely mechanisms. The first, a brittle layer formed at the fusion zone by segregation, was not in evidence consistently enough in SEM examination of fracture surfaces and therefore was considered only a contributing factor. Specimens with large Ti films or inclusions evident on fracture surfaces usually absorbed less than average impact energy. A second possibility lay in a

weakening mechanism caused by the heat treatment to the grain-refined base metal such as is known to occur to the 18% Ni maraging steels¹¹. The third possibility extended the known correlation of DBTT to grain size, as discussed earlier, to observed grain growth near the fusion zone in multipass welds, as seen in Figure 1, or even to the partially-melted region of the fusion zone itself. To distinguish these required two additional experiments with similar heat treatments given to the metal: one without added elements in the vicinity of the fusion zone was represented by the EB welds and one without a fusion (partially melted) zone was represented by the HAZ simulation studies.

ELECTRON BEAM WELDS

Single and double pass, full penetration electron beam (EB) welds were made in grain-refined (2B) Fe-12Ni-0.25Ti plates. Square bevels with neither preheat nor oscillation were used to fuse the two plates without filler metal or flux. The fluidity of the weld metal, presumably attributable to the lack of delta ferrite formation in this material, precluded full penetration, single pass welds being made without a backing strip. Two-pass welds were made from opposite sides and factors such as the lack of critical alignment left long areas of incomplete penetration in two of the three two-pass welds. The fourth EB weld (number 7) incorporated a back-up strip to circumvent this problem.

Although impact toughness was virtually negligible for EB weldments, low values were expected based on the extremely coarse solidification structure of the weld metal. Optical cross sections revealed a cellular structure with cell diameters ranging from 5-10 microns while weld metal fracture surfaces were deeply ribbed with virtually through-thickness ridges (Figure 9). What was significant, however, was the impact toughness of the HAZ which rose from that of the weld metal (14 ft-lbs) in the single pass weld where the fusion zone was adjacent to the through-thickness solidification structure, to 33 ft-lbs in the HAZ of the two-pass weldment where it was further removed from the fusion zone of the final pass.

HAZ SIMULATION

The HAZ simulation studies were initiated to attempt to isolate the source of the degradation of properties observed at least to some extent in the HAZ of all welds tested. Using available induction heating equipment, the 1/2 inch square specimens were suspended within the r-f coils and released into water when the required temperature had been reached. Heating rates of 50°C/sec to 1350°C and 1050°C were achieved in this manner although heating rates of 135°C/sec to 1050°C were recorded during welding. However, since the total time above the transformation temperature was approximately the same for both the actual and the simulated heat-affected specimen, the use of this technique for the determination of the trend of the effect of rapid heating and cooling upon the grain-refined microstructure should be justified.

The results of mechanical and Mossbauer tests are given in Table IV.

Although the accuracy of the temperatures estimated with an optical pyrometer on a constantly heating induction furnace was not closer than $\pm 30^\circ\text{C}$, HAZ simulated studies provided a surprising result: every specimen subjected to this fast heating rate/immediate quench treatment showed enhanced impact toughness. A specimen heated to 620°C (thermocouple pyrometry), 60°C below the A_{c3} temperature, was 15 ft-lbs above the base metal while those heated above 750°C all had values greater than 160 ft-lbs, regardless of whether the heat treatment included single or multiple heatings. Multiple heatings included permutations of 1350°C, 1050°C, and 750°C. Retained austenite was measured by Mossbauer spectroscopy in three samples and was never significant past the error inherent in the technique ($0.5\% \pm 0.5\%$). This also is a surprising result since, although similar increases in impact toughness were found in tempering studies with this alloy¹², the optimum conditions at 525°C for eight hours were attributed, at least partially, to an

introduction of two volume percent retained austenite. The coincident loss of stable crack growth and peak K_{IC} fracture toughness values observed in the previous study could be used to determine whether the same mechanism was at work; however, this exercise was not performed.

V. CONCLUSIONS

The research undertaken to determine the weldability of grain-refined (2B) Fe-12Ni-0.25Ti steel plate in the as-welded state for cryogenic, structural applications revealed that reasonable impact toughness could be retained in spite of the grain coarsening and tempering effects of fusion welding.

Gas metal arc welding, using Inconel Number 92 welding wire with argon-15% helium shielding gas, produced welds with 50 and 62 ft-lbs minimum HAZ toughness at liquid helium and nitrogen temperatures, respectively, for welds which would be radiographically acceptable under AWS D1.1 or ASME Section VIII, Division 1, Article UW-51 codes. A weld which contained a clearly excessive amount of porosity (weld 11) yielded a minimum HAZ specimen of 32 ft-lbs in the HAZ, a value which is still acceptable in many designs.

Inconel 92 filler metal, however, contains an excessive amount of titanium and is too sensitive to atmospheric contamination to be an optimum filler metal.

Electron Beam Welds without any oscillation or filler metal failed at low impact energies (<15 ft-lbs) both in the weld metal and HAZ when the as-solidified structure was within the fracture path. When the as-solidified structure was tempered by a second pass, the HAZ specimen tested in liquid helium was comparable to the GMA/HAZ specimens tested in L.N which had rejectable porosity.

Samples of the plate material which were subjected to thermal treatments comparable to those of a GMA welded HAZ, but while in an essentially stress-free state, showed uniform improvement of impact properties over the base metal, increasing to over 160 ft-lbs for any specimen whose simulation

temperature traversed the A_{C3} temperature regardless of the duration at elevated temperatures.

Based on the results of this study, it can be concluded that the weakest link of a fusion weldment with this material is at the fusion zone. Fractures of HAZ specimens usually tended to pass through this region and impact values decreased as the notch was placed closer to the fusion line. Since simulation studies imply that these values might be increased if the heat treatment alone were active, it seems probable that the fusion zone itself contains a region of either poor cohesion, segregated embrittling species, or effectively large prior austenite grains. The mechanical and cathodic vacuum etching technique revealed the fusion zone along with other austenite and prior austenite grains as seen in Figure 1. This region could then be expected to lower the DBTT by effectively raising the grain size. This theory would also explain the higher toughness values in the Devletian, et al., study⁴ than would be expected here since the joint preparation in that study was milled in a single plate which was never separated and "full penetration" was defined as a simple melting of the base metal at the bottom of the grooves. Thus, where the weldment in the present study had a through thickness fusion zone adjacent to deposited weld metal, the weldment studied in the previous investigation was modified by the prior microstructure.

Although beyond the scope of this work, fracture toughness studies would be necessary to determine the actual usefulness of this material in a cryogenic structure. The earlier work of S. K. Hwang¹² demonstrated that unstable crack growth accompanied gains in impact toughness from tempering. The increases in impact strength seen in the simulated specimens correlate well with those of the earlier work where a simultaneous decrease in K_{IC} along with a change from stable to unstable crack growth was observed.

ACKNOWLEDGMENTS

The author wishes to express his appreciation to Professor J. W. Morris, Jr. and Ms. Kim W. Mahin for their encouragement and help during the course of this investigation.

The assistance and cooperation of the support staff of the Inorganic Material Research Division was also greatly appreciated. The author particularly wishes to thank Duane Newhart for his contributions in the design and construction of the automated welding table, Fred Lockwood, Contra Costa Community College, for his generous help in the non-destructive evaluation of welded plates, and my wife, Louise, for contributions in the preparation of this manuscript.

This research was performed under the auspices of the United States Department of Energy through the Inorganic Materials Research Division of the Lawrence Berkeley Laboratory.

REFERENCES

1. Daldor, E.: Private communication.
2. Horwood, Wade Alan: Factors Controlling the Fracture Toughness of Cryogenic Alloys, M.S. Thesis, December, 1972.
3. Jin, S., Morris, J. W. Jr., and V. F. Zackay: Grain Refinement through Thermal Cycling in an Fe-Ni-Ti Cryogenic Alloy, Met Trans A, vol 6A, p. 141, 1975.
4. Jin, S., Hwang, S. K., and Morris, J. W. Jr.: Comparative Fracture Toughness of an Ultrafine Grained Fe-Ni Alloy at Liquid Helium Temperature, Met Trans A, August, 1975.
5. Devletian, J. H., Stephens, J. R., and Witzke, W. R.: Weldability of High Toughness Fe-12% Ni Alloys Containing Ti, Al or Nb, Welding Journal, April, 1977.
6. Pense, A. W. and Stout, R. D.: Fracture Toughness and Related Characteristics of the Cryogenic Nickel Steels, WRC Bull, 205, May, 1975.
7. Shoemaker, L. E., Huntington Alloys, Technical Service and Specifications Department: Private communication.
8. Padden, T. R. and Cain, F. M. Jr.: Cathodic Vacuum Etching, Metal Progress, p. 108, July 1, 1954.
9. ASTM E 23 and ASTM A 370.
10. Jin, S., Horwood, W. A., Morris, J. W. Jr., and Zackay, V. F.: A Simple Method for Charpy Impact Testing Below 6°K, Advances Cryog. Eng., vol. 19, p. 373, 1974.
11. Prager, M. and Sines, G.: A Mechanism for Cracking during Post Welding Heat Treatment of Nickel Base Alloys, WRC Bull. 150.
12. Hwang, S. K., Jin, S., and Morris, J. W. Jr.: A Study of Retained Austenite in a Fine Grained Fe-12Ni-0.25Ti Alloy, Met Trans A, vol. 6A, p. 2015, 1975.

13. Handbook of Physics and Chemistry, 37th Edition, Chemical Rubber Co., pp. 614-615.
14. Metallography, Structures, and Phase Diagrams, Metals Handbook, Volume 8, eighth edition, 1973, American Society for Metals.

Table I. Chemical Analysis of Inconel No. 92 Weldwire (Heat No. HT1961MD)

C	MN	FE	S	SI	CU	NI	CR	TI	OTHERS
0.03	2.27	8.28	0.008	0.05	0.11	69.99	16.04	3.22	<0.50

Table II. Typical Composition of Fe-12Ni-0.25Ti Ingots.

NI	TI	C	N	S	P	MN	O	FE
11.5	0.23	0.009	0.003	<0.005	<0.005	<0.005	0.02	Balance

Table III. Weld Results.

Weld	Type	Bevel	No. of Passes (Front + Rev. Sides)	Heat Input (kJ/mm)	Shielding	Charpy V-notch Impact Energies (Ft-lbs)				
						Given as: Average value (No. of specimens) Minimum value				
						Weld Metal		HAZ		Base Metal
						L.N	L.H	L.N	L.He	L.N
2	GMA	2-V	4 + 4	0.32	Ar-20%He	88(2) 87		88(4) 83	96(1)	96(1)
3	GMA	K	5 + 4	0.66	Ar-40%He	72(1)		76(3) 52		106(1)
11	GMA	sgl. bvl.	5	0.98	Ar-17%He	53(1)	88(1)	75(4) 60	80(1)	100(1)
12	GMA	sgl. bvl.	6 + 1	0.95	Ar-14%He	66(2) 65		43(4) 32	50(2) 50	106
7	EB	sqr.	1	0.71	vac.	23(1)	15(1)	29(4) 26	18(2) 13	106
8	EB	sqr.	2	0.71	vac.	23(2) 21	13(1)	81(3) 50	33(1)	100

Table IV. Simulation Results.

Specimen	Heat Treatment	Retained Austenite (Mossbauer)	Charpy V-notch Imp. Energies (ft-lbs)	
			L.N	L.He
14,15	2B	0-1/2% $\pm$ 1/2%	108	
1,2,3	+1050°C WQ+1350°C WQ+750°C WQ		161	132
4,6,7	+1350°C WQ+1050°C WQ+750°C WQ		163	135
8	+1350°C WQ+750°C WQ		167	
9	+1350°C WQ	1/2% $\pm$ 1/2%	163	
10	+750°C WQ	0-12% $\pm$ 1/2%	166	
11	+700°C WQ		131	
12	+650°C WQ		148	
13	+620°C WQ		123	

FIGURE CAPTIONS

- Fig. 1 Root area of double vee weld #2: Inconel/GMAW.
- Fig. 2 Base plate microstructure: Grain refined 2B. Plate used in welds 1-4 and all simulated specimens.
- Fig. 3 Heat affected zone specimen (3W9) fracture surface: Inconel/GMAW.
- Fig. 4 Weld metal specimen (3W4) fracture surface: Inconel/GMAW
- Fig. 5 Detail of Figure 4.
- Fig. 6 Base metal region (3W2) fracture surface.
- Fig. 7 Heat affected zone specimen (3W6) fracture surface: Inconel/GMAW
- Fig. 8 Detail of Figure 7.
- Fig. 9 Weld metal specimen (7W1) fracture surface: EBW.
- Fig. 10 Detail of welding table.

Figure 1

NBB 799-12313

Figure 2

XBB 799-12814

Figure 3

XBB 799-12815

Figure 4

XBB 799-12820

Figure 5

XBB 799-12816

Figure 6

XBB 799-12817

Figure 7

XBB 799-12812

Figure 3

NBB 799-12812

Figure 9

XBB 799-12819

Figure 10

NBB 799-12811