Mechanical Properties of Nb-1Zr Weldments

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Abstract. The objective of this work was to measure the mechanical properties of Nb-1Zr weldments, and, more specifically, to evaluate whether Charpy V-notch impact testing may be a more meaningful indication of weld deposit ductility than the slow bend testing. Manual gas-tungsten-arc welds were made in Nb-1Zr plates using 4 beads of filler wire from the same heat. The cold-rolled plates were recrystallized by heat treatment at 1773 K for 1 hour prior to welding. Welded plates were made in a stainless steel welding glove box backfilled with either argon or helium . Two plates were made with each gas, with one plate post-weld heat treated at 1373 K. Weld metal from each of the four welded plates was subjected to chemical analysis, tensile testing at room temperature, and Charpy V-notch testing at 93-473 K. Also, microhardness testing was used to evaluate property gradients in the weldments. Impurity concentrations in the weld deposits compared well with the base metal concentrations and were within the limits of the relevant ASTM specification, B393 Type 3. All specimens with one minor exception had yield strengths, tensile strengths, and ductilities that exceeded the ASTM specified minimums. The Charpy V-notch specimens from the as-welded He weld deposit, and from both post-weld-heat-treated weld deposits showed similar behavior with upper shelf energies of 9.7-10 J and ductile-brittle transition temperatures of 85-100 K. These Charpy V-notch properties were comparable to those of the unwelded plate. The as-welded Ar weld deposit had a slightly higher ductile-brittle transition temperature near 150 K. Microhardness testing indicated that in the as-welded conditions the average hardness of the weld deposit made in Ar was higher than that made in He. Post-weld heat treatment reduced the average hardnesses of base metal and weld metal. The range of hardnesses in the weld deposits was increased by the post weld heat treatment.

INTRODUCTION

A primary objective of this project was to determine whether Charpy V-notch impact testing may be a more meaningful indication of weld deposit ductility than the slow bend and tensile testing that has traditionally been used (Franco-Ferreira and Slaughter, 1963; Lessmann, 1964; Lessmann, 1966; Stephens, 1977; Harwig, Bryhan, and Ring, 1989; Lessmann, 1984). This initial experiment consisted of making manual gas tungsten arc (GTA) welds in 3-mm-thick plates of Nb-1Zr using Nb-1Zr filler metal. The welds were made in a glove box backfilled with either Ar or He. Both gases were used because both have been used in previous studies (Kearns, Young, and Redden, 1966; Franco-Ferreira and Slaughter, 1966), but there was no clear indication of whether one might be preferred. It is well known that arc characteristics and weld shape are influenced by shielding gas composition (Helmbrecht and Oyle, 1957; Key, 1980; Katsaounis, 1993).

EXPERIMENTAL

Both the 3-mm-thick plate material and the 1.6-mm-diameter filler metal wire used to make the welds were produced from Nb-1Zr heat no. 531029 (Wah Chang). The initial condition of the plate was cold rolled. Eight weld plates with dimensions of 125 mm x 38 mm were sheared from the source plate for welding. A 45° bevel was machined on one 125-mm edge of each weld plate. Specimens about 12 mm x 12 mm were also saw cut from the plate for an annealing study.

The machined welding plates, welding filler metal wire, and annealing specimens were degreased and then chemically cleaned using a fresh solution of 3 parts distilled water + 1 part HF (48%) + 1 part HNO_3 (70%).

Specimens were lowered into the solution, slowly agitated until chemical reaction started, and held in the solution until they were bright and shiny. After acid cleaning the various parts were rinsed in running distilled water and then sprayed with ethanol from a squirt bottle. They were then air dried in a dust-free environment, handled with cotton gloves, and stored in plastic bags.

Based on the results of an annealing study, it was decided to heat treat the weld plates for 1 h at 1773 K as this condition produced a recrystallized microstructure with relatively large grain size. The weld plates were heat treated in lots of 4 in a furnace with refractory metal heating elements that was evacuated to below about 1.3 x 10^{-4} Pa. Each weld plate was wrapped in Nb foil prior to annealing. The annealed plates were then loaded into the welding glove box, also in lots of 4.

The beveled weld plates were held for welding in a fixture constructed of tungsten plates that were backed with stainless steel plates. In this arrangement, the Nb-1Zr weld plates were only in contact with the W plates. The beveled edges of the weld plates were set with a gap between them of slightly less that 1.6 mm. After fixturing was completed, the atmosphere in the glove box was stabilized to the lowest oxygen and water vapor concentrations that could be achieved. Typical impurity levels at the commencement of welding were O < 2 ppm, and H_2O < 5 ppm. Oxygen and H_2O concentrations were continuously monitored and welding was stopped if either O exceeded 5 ppm, or H_2O exceeded 20 ppm. Continuous purging with high purity Ar or He was then used to reestablish acceptable impurity levels.

A total of 4 welded plates was made, two using an Ar backfill in the glove box, and two using a He backfill. Each plate was completed with 4 manually deposited weld beads. The arrangement of the beads was essentially identical in each plate. First was a root pass that completely sealed the two individual coupons together. Second was a "fill" pass that nearly welded the plates to the full 3-mm thickness. Third and fourth were "cover" passes deposited side-by-side to complete the welds and produce a thickness at the joints of greater than 3 mm. The welded plates were each radiographed to confirm quality.

One welded plate with each backfill gas was reserved in the as-welded condition. The second plate was given a post-weld heat treatment (PWHT) of 1 hour at 1373 K (Franco-Ferreira and Slaughter, 1963) with the same vacuum furnace used for annealing of the unwelded plates.

All 4 of the welded plates were then sectioned for machining of miniature tensile specimens and Charpy V-notch impact specimens. The tensile specimens were completely prepared by electrical discharge machining (EDM). The gage dimensions of the specimens were nominally 5.08 mm long x 1.22 mm wide x 0.762 mm thick. The tensile specimens were tested at room temperature using an MTS HYD-02 machine. The nominal strain rate used for the tests was 0.001 s^{-1}. Blanks with dimensions of 3 mm x 4 mm x 27 mm were cut for Charpy specimens by EDM. The 1-mm-deep notches with 60° angle were produced with a cutting tool, and they were oriented in the weld metal as shown in ASME Section II, Part C, Specification SFA 5.1. The Charpy tests were done on an instrumented apparatus of ORNL design following procedures outlined in ASME Section II, Part A, Specification SA 370. A 20 mm span was used to break specimens at temperatures of 93-473 K. A slice about 6-mm thick was also cut from each end of each weld and used for microscopic examination and hardness testing.

RESULTS AND DISCUSSION

Specimens for chemical analysis of the weld deposits were cut from the remnant pieces that were used for making the all-weld-metal tensile specimens. The analyses were done at Wah Chang, Albany, Oregon. A standard analysis procedure for Nb-1Zr was requested. The elements Al, B, Cr, Fe, Hf, Mo, Ni, Si, Sn, Ta, Ti, V, W, Zn, and Zr were determined by inductively coupled plasma optical emission spectroscopy (ICP-OES). The element C was determined by LECO combustion analysis. The element Cu was determined by atomic absorption. The elements H, O, and N were determined by the appropriate LECO analyzer based on inert gas fusion techniques. The elements Cu, Sn, V, and Zn were included in the analyses even though their concentrations are not specified in ASTM B393. The elements Be and Co were not included in the analyses even though their concentration limits are specified in ASTM B393. The reasons for these discrepancies are not presently known.

The results from the chemical analyses of the welds and the base metal are shown in Table 1 where the concentration limits (in wt%) from ASTM B393 are included for reference. The concentrations of Al, B, Cr, Fe, H, Ni, Si, Sn, V, and Zn were below their respective detection limits. The C concentrations in the welds were in the 23-35 wppm range and slightly higher than the base metal which was below its detection limit of 20 wppm. Elevated C concentrations in the welds could originate from a number of sources including decomposition of organic materials like gloves or lubricants, or from back streaming of vacuum oils inside the glove box chamber. The Cu concentrations of the welds were elevated with one specimen containing 230 wppm. In comparison, the base metal Cu concentration was below its detection limit of 10 wppm. It is most likely that the source of the Cu contamination was from brass wires used to machine the tensile specimens by EDM. The Hf, Mo, Ta, Ti, and Zr concentrations of the welds were in the ranges of the base metal. The O and N concentrations of the welds were also within the range of the base metal. One weld had an elevated W concentration, but the others were within the range of the base metal. The source of the elevated W concentration most likely was the welding electrode used for the GTA welding process. All of the analyzed elements in the welds and the base metal were within the ASTM B393 Type 3 specified limits. Post-weld heat treatment of the welds produced no systematic variations in their chemistries.

TABLE 1. Chemical Analysis Results for Nb-1Zr Base Metal and Manual GTA Weld Deposits, Both As-Welded and After PWHT. Results Are Reported as WPPM Except as Noted.

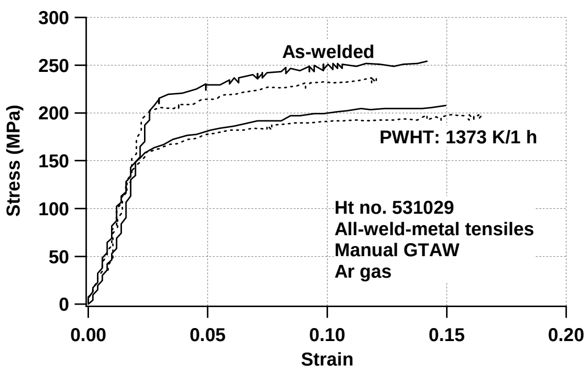
Element	Max. wt% ASTM B393	Heat 531029	Ar As-Welded	Ar PWHT	He As-Welded	He PWHT
Al	0.002	<20	<20	<20	<20	<20
B	2 ppm	<5	<5	<5	<5	<5
Be	0.005	---	---	---	---	---
C	0.01	<20	35	24	24	23
Co	0.002	---	---	---	---	---
Cr	0.002	<20	<20	<20	<20	<20
Cu	---	<10	230	63	54	53
Fe	0.005	<35	<35	<35	<35	<35
H	0.0015	<3	<3	<3	<3	<3
Hf	0.02	140	150	150	150	160
Mo	0.010	<30	40	<30	<30	<30
N	0.01	30	27	39	<20	25
Ni	0.005	<20	<20	<20	<20	<20
O	0.015	70	70	90	70	60
Si	0.005	<50	<50	<50	<50	<50
Sn	---	<40	<40	<40	<40	<40
Ta	0.1	870	840	840	840	820
Ti	0.02	54	54	50	<40	46
V	---	<20	<20	<20	<20	<20
W	0.03	63	71	67	130	76
Zn	---	<40	<40	<40	<40	<40
Zr(wt%)	0.8-1.2	1.06	1.08	1.08	1.07	1.04

All-Weld-Metal Tensile Tests

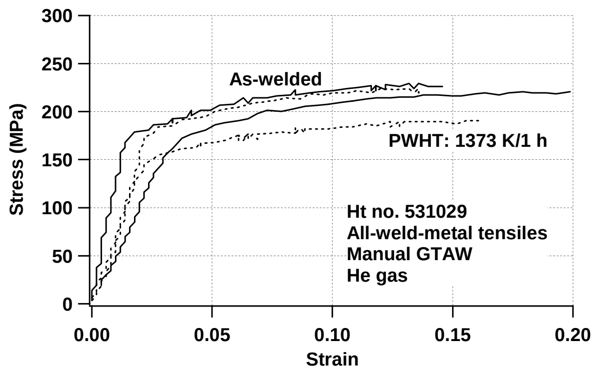
The results from the all-weld-metal tensile tests done at room temperature are shown in Figure 1(a) for the welds made with Ar, and in Figure 1(b) for the welds made with He. These plots show the behavior up to the point of maximum load, i.e., they represent only the uniform elongation portion of the tensile curves. Strains were estimated from crosshead displacements rather than extensometers. Consequently, the elastic portions of the curves do not accurately represent the true elastic behavior. The jagged appearance of the curves is largely the result of translation of the load-displacement data from the data acquisition software to plotting software. The tensile data are also summarized in Table 2 where minimum values specified in ASTM B393 Type 3 are included for reference.

Figure 1(a) shows that Ar-shielded, as-welded specimens had very high yield strengths and excellent tensile ductilities. Post-weld heat treatment reduced flow stresses by about 25% from as-welded values, but increased uniform elongations by about 20%. Figure 1(b) shows that the He-shielded, as-welded specimens also had high yield strengths both before and after PWHT. For He, the PWHT decreased the flow stresses by about 15%. Overall elongation values were slightly higher than for the welds made in Ar.

With one exception, all of the all-weld-metal tensile specimens resulted in properties that exceed the minimums specified by ASTM B393. One of the PWHT specimens made in He had a tensile strength slightly lower than the specification minimum, 189 MPa compared to 195 MPa. It is possible that this tensile strength reduction was associated with a defect in the weld deposit, but the exact cause was not determined.



(a) Ar-Shielded Welds.



(b) He-Shielded Welds.

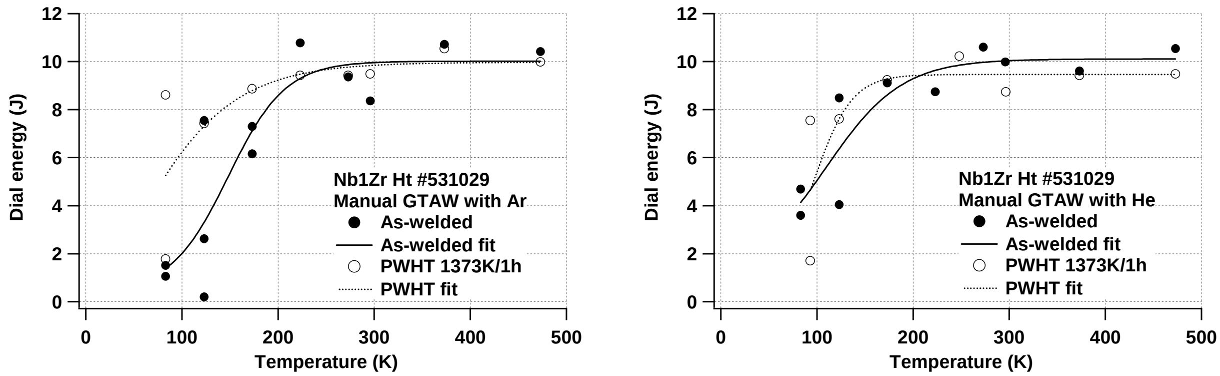
FIGURE 1. Results from All-Weld-Metal Tensile Tests at Room Temperature.

TABLE 2. Tensile Test Results.

ID	0.2% Y.S. MPa	U.T.S. MPa	Uniform Elong., %	Total Elong., %
Ar As-Welded 1	213	251	9.9	30.9
Ar As-Welded 2	204	235	10.3	29.1
Ar PWHT 1	149	196	12.5	30.6
Ar PWHT 2	149	206	12.6	37.6
He As-Welded 1	176	227	11.8	28.1
He As-Welded 2	178	223	11.4	28.8
He PWHT 1	143	189	13.2	29.4
He PWHT 2	160	220	15.4	44.3
ASTM B393	125	195	---	>20

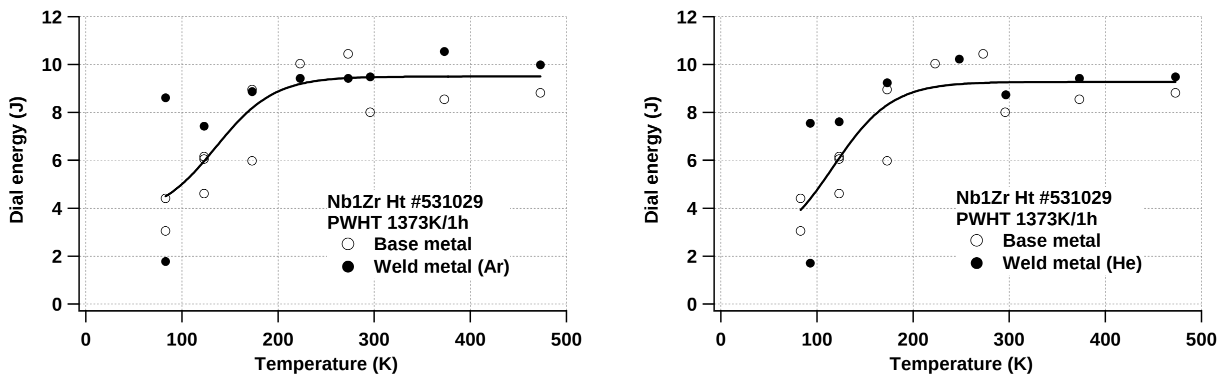
All-Weld-Metal Charpy Impact Tests

The results from all-weld-metal Charpy impact testing are shown in Figure 2(a) for welds made in Ar, and in Figure 2(b) for welds made in He. For the PWHT conditions, comparisons of the all-weld-metal data with base metal data are shown in Figure 3(a) for Ar shielding and in Figure 3(b) for He shielding. In Figure 2, each data set was fit to a sigmoid function by a regression technique. The mid-transition ductile-brittle temperatures (DBTT) and upper shelf energies were estimated from the fit curves and they are shown in Table 3. For the DBTT estimate the lower shelf energy was assumed to be zero. The as-welded He specimens and the PWHT specimens made with either Ar or He showed similar behavior with upper shelf energies of 9.7-10 J and DBTTs of 85-100 K. The as-welded Ar specimens also had an upper shelf of about 10 J, but their DBTT appeared to be near 150 K. In Figure 3, all-weld-metal and base metal data from PWHT material are plotted together and fit to a single function. The DBTT and upper shelf energy for only the PWHT base metal were 105 K and 9.1 J, respectively, and these values are also included for reference in Table 3.



(a) Ar-Shielded Welds. **(b)** He-Shielded Welds.

FIGURE 2. Plots of Dial Impact Energy Against Test Temperature From All-Weld-Metal Charpy V-Notch Impact Tests.



(a) Ar-Shielded Weld. **(b)** He-Shielded Weld.

FIGURE 3. Plots of Dial Energy Against Test Temperature Comparing All-Weld-Metal and Base Metal Data.

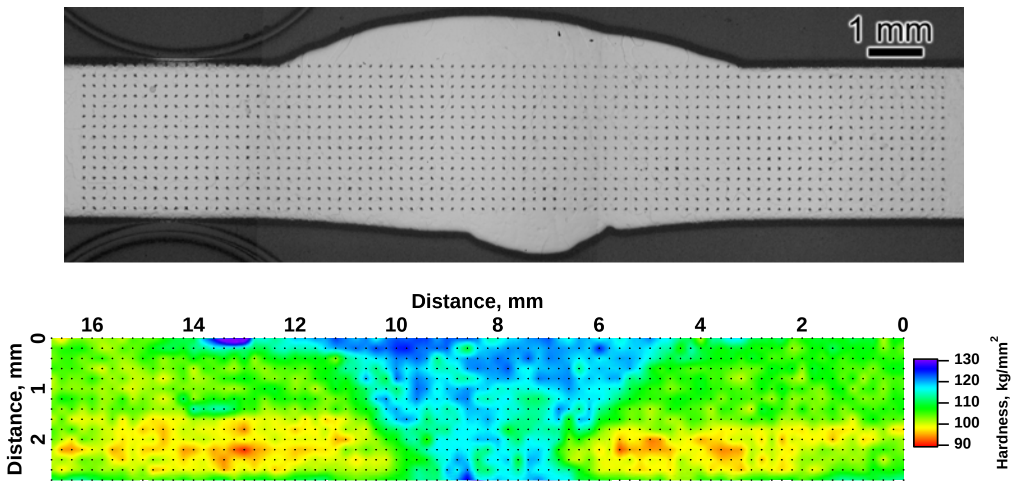
The data from the all-weld metal specimens indicate that the PWHT of 1373 K for 1 h lowers the DBTT of the Ar weld deposit substantially but it had little effect on the He weld. The upper shelf energies were not influenced by the PWHT. The data further indicate that in the PWHT condition the weld deposits have slightly lower DBTTs than the base metal. However, both the weld and base metal data are represented reasonably well by single behavior pattern. These results confirm that the DBTTs of manual GTA weld deposits are well below room temperature. There are no Charpy impact requirements in ASTM B393.

Weldment Hardness Tests

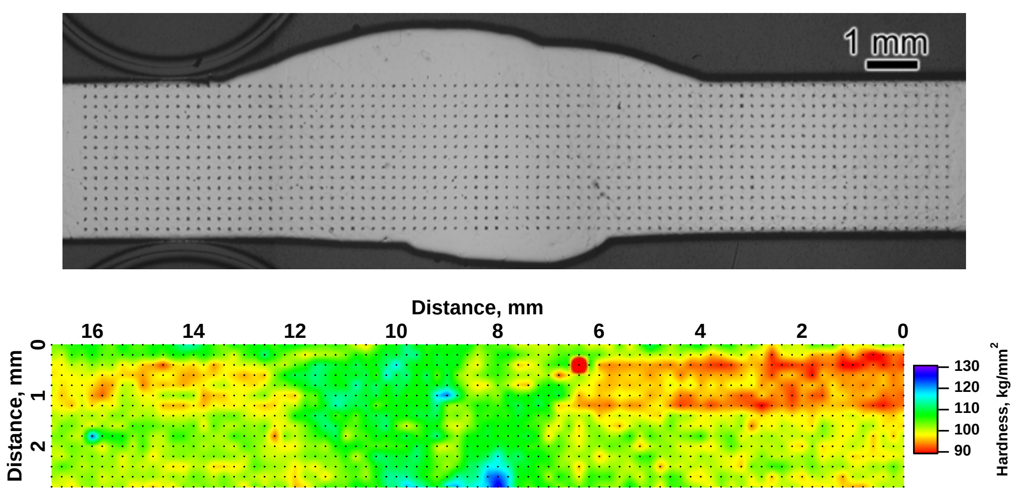
The hardness characteristics of the welds were measured with indentation grids that extended well into the base metal on either side of the weld deposits. The overall grid dimensions were 2.8 mm x 16.8 mm, and the indentations were made at 200 μm intervals using a Vickers indenter under a 100 g load. The results for the Ar-shielded welds are presented in Figure 4 where the actual grid on a weld cross section is shown with a hardness contour plot. The results for the He-shielded welds are shown in Figure 5.

TABLE 3. Charpy V-Notch Impact Test Results.

ID	Estimated mid-transition DBTT, K	Estimated upper shelf energy, J
Ar As-Welded	150	10
Ar PWHT	85	10
He As-Welded	100	10
He PWHT	97	9.7
Base PWHT	105	9.1
ASTM B393	Not specified	Not specified

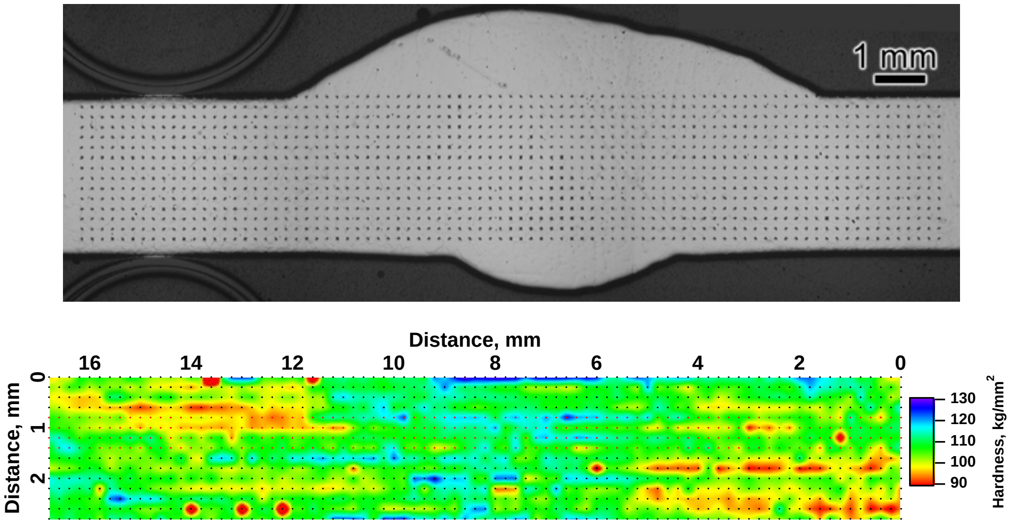


(a) As-Welded Condition.

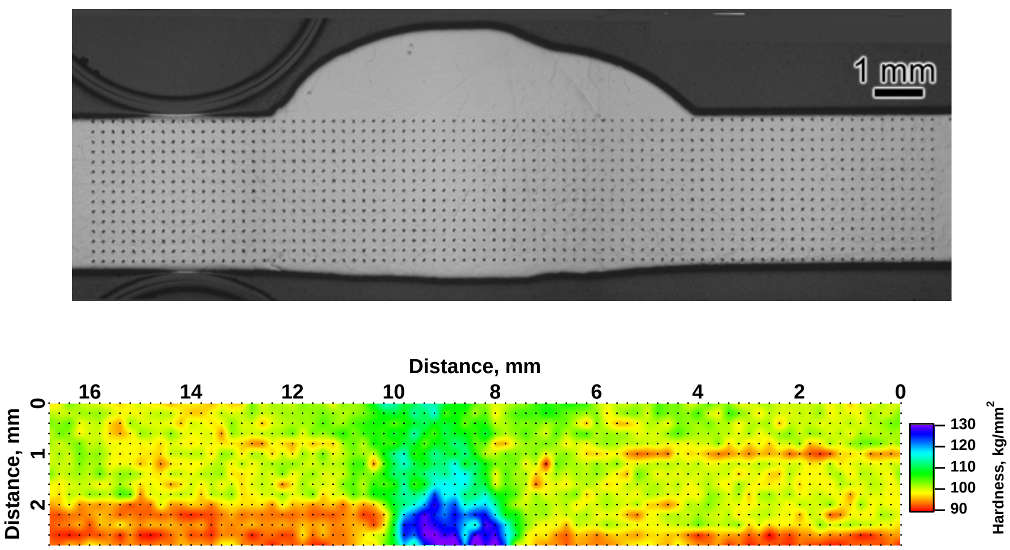


(b) PWHT Condition.

FIGURE 4. Contour Plots of Vickers Hardness Measurements from Ar-Shielded Welds.



(**a**) As-Welded Condition.



(b) PWHT Condition.

FIGURE 5. Contour Plots of Vickers Hardness Measurements from He-Shielded Welds.

Several clear features of the weldments are illustrated by the contour plots. The weld deposits are harder than the base metal for both shielding gases in both the as-welded and the PWHT conditions. Overall, the PWHT decreased the average hardness in both the weld and the base metal. In the as-welded condition, the weld made in Ar was slightly harder than the one made in He, and this is consistent with their tensile behavior. Both Figures indicate that weld deposit hardness may be fairly nonuniform even after PWHT, and they further suggest that some regions of the weld deposits may experience hardness increases following PWHT. Banding of base metal hardness is also clearly illustrated, and it persisted after the PWHT. These bands were associated with bands of nonuniform grain sizes in the base metal plate.

CONCLUSION

Manual gas tungsten arc welds of Nb-1Zr were made in both Ar and He atmospheres and tested in both the as-welded and the post-weld-heat-treated condition (1373 K/1 h). With the exception of the tensile strength of one PWHT He specimen, all of the weld metal chemistries and room temperature tensile properties met or exceeded the requirements of ASTM B393 Type 3. The highest tensile strengths were achieved for the Ar weld in the as-welded condition. These specimens also had the lowest tensile ductilities, although they still exceeded the ASTM B393 minimum value. Charpy V-notch testing showed that this weld deposit also had a higher DBTT than the other three welded plates. A direct comparison of Charpy impact energies of weld metal and base metal was only made after PWHT. In this condition, both weld and base metal had DBTTs near 100 K and upper shelf energies of 9-10 J. Microhardness testing showed that weld deposits had higher average hardnesses than base metal in both the as-welded and PWHT conditions.

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