CANADIAN WESTINGHOUSE COMPANY LIMITED,
ATOMIC ENERGY DIVISION,
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WELDING OF ZIRCONIUM-2.5 w/o NIOBIUM AND ZIRCONIUM-3.0 w/o ALUMINUM 0.5 w/o MOLYBDENUM ALLOYS

M. J. LAVIGNE

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WELDING OF ZIRCONIUM-2.5 w/o NIOBIUM AND ZIRCONIUM-3.0 w/o ALUMINUM-0.5 w/o MOLYBDENUM ALLOYS

by

M.J. Lavigne, Manager,

Metallurgical-Mechanical Section,
Research and Development Laboratories

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SUMMARY

The high strength zirconium alloys, Zr-2.5 w/o Nb and Zr-3.0 w/o Al-.5 w/o Mo were welded under various atmospheres and the welds were evaluated by nondestructive testing, chemical analysis, metallographic examination and by tensile testing at room temperature and 315° C (600°F). The Zr-Nb welds were also evaluated by corrosion testing in 360°C (680°F) water after various heat treatments and surface treatments before and after welding. In addition the welding of Zr-2.5 Nb to Zircaloy-2 was investigated.

The alloys are readily weldable by the inert tungsten-arc welding process and although the weld-shielding atmosphere has no apparent effect on the mechanical properties of the welds, the corrosion resistance of the Zr-Nb alloy is affected. The best corrosion resistance is obtained with the highest purity, gas-shielding conditions. Vapour blasting and pickling after welding resulted in some improvement in corrosion resistance of welds made under the various atmospheres. Heat treating after welding followed by vapour blasting and pickling definitely improves the corrosion resistance of the Zr-Nb alloys. The corrosion resistance of the heat-affected zone is definitely impaired in the Zr-Nb material, whether welded as-received or heat-treated, and a complete solution and aging treatment would probably be required to restore the corrosion resistance. Welding in a pre-evacuated box (0.01μ), back-filled with purified argon, is considered to be more suitable for the welding of thick sections or complicated shapes of Zr-Nb and Zr-Al-Mo alloys.

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WELDING OF ZIRCONIUM - 2.5 w/o NIOBIUM AND ZIRCONIUM - 3.0 w/o ALUMINUM - 0.5 w/o MOLYBDENUM ALLOYS

INTRODUCTION

The Zirconium - 2.5 w/o Niobium and Zirconium - 3.0 w/o Aluminum - 0.5 w/o Molybdenum alloys exhibit much greater strengths (1,2,3,4) and lower capture cross sections than the Zircaloy-2 alloy (3). The corrosion resistance of the Zr-2.5 Nb alloy in high temperatue water (360°C) and steam (400°C) is not as good as that for Zircaloy-2 (1,2,5), but is considered acceptable (6). The Zr-3.0 Al-0.5 Mo alloy which shows better strength than the Zr-2.5 Nb alloy at high temperatures has poor corrosion resistance in high temperature water but may be clad with a more corrosion resistant material. For the above reasons both alloys have been proposed as replacements for Zircaloy - 2 in Canadian water-cooled power reactors, particularly for use in pressure tubes (6).

As the reactor components produced from these alloys should be weldable and little information was available in the unclassified literature on the welding of these alloys an investigation was carried out to determine their welding characteristics. The investigation included the welding of the alloys under various atmospheres and the evaluation of the welds by nondestructive testing, chemical analyses, metallographic examination and by tensile testing at room temperature and 350°C (600°F). In the case of the Zr-Nb alloy the welds were also evaluated by corrosion testing in water at 360°C (680°F) after receiving various surface treatments. The effects of pre-and post-weld heat treatments on the mechanical properties and the corrosion resistance of the Zr-Nb alloy were determined.

The welding of Zr-2.5 Nb alloy to Zircaloy-2 was also investigated in order to obtain information on the joining of Zr-Nb pressure tubes to Zr-2 connecting tubes for a water-cooled power reactor system. The Zr-Nb/Zr-2 welds were evaluated by the same procedures used for the Zr-Nb/Zr-Nb welds. For the convenience of the reader the investigation on the welding characteristics of the alloys is presented in two sections:-

1 - Welding of Zr-2.5 w/o Nb alloy

2 - Welding of Zr-3 w/o Al-.5 w/o Mo.

SECTION I

WELDING OF Zr-2.5 w/o Nb ALLOY

1 - Materials

A 235 in. long x 11 in. wide x 0.030 in. thick sheet of Zr-Nb alloy fabricated by Carborundum Metals Company, Akron, N Y., from Ingot No. AE-7, was supplied by Atomic Energy of Canada Limited (AECL) for the original investigation. When additional material was required to study other welding variables AECL supplied approximately 5 feet of 4 in. diameter tubing with 0.030" wall which had been formed at NTH by bending and welding a sample-sheet bearing the same heat number as the sheet supplied for the earlier welding tests. No information was made available on the condition of the sheet and the tubing materials as-received, but the structure of the materials and their surface condition indicated that they were supplied in the hot rolled and pickled condition.

The chemical composition of the Zr-Nb sheet, as determined by R&D Laboratories of Canadian Westinghouse Company Limited, is shown in Table 1. This chemical analysis is in close agreement with that of the manufacturer. The certified analysis of the Zr-2 used in the welding of Zr-Nb to Zr-2 is also shown in Table 1. This Zr-2 was received from Atlas Titanium Ltd., Welland, in the form of a .250 in. thick plate in the annealed condition. It was cut into bars 3" wide and hot rolled down to 0.125 in. strips. After annealing at 750°C for 1/2 hour in a vacuum furnace (0.02μ Hg) the strips were cold rolled down to 0.030 in. and vacuum annealed at 750°C for 1/2 hour. The gas analysis of one of the finished strips is included in Table 1.

2 - Experimental Procedures

2.1 Preparation of Samples

Samples 2-3/4 in. wide x 12 in. long were sheared from the 235 in. long sheet with their long axes parallel to the rolling direction of the material. Similar samples were also removed from the tubing material after discarding the weld and 1/2 in. of the adjacent material and straightening the remaining tube in a press. The samples were deburred and cleaned with acetone and those which had been heat treated before welding were also vapour blasted with 220 grit silicon carbide abrasive to remove the oxide film before washing in acetone. Attempts made to remove the oxide film with an aluminum oxide abrasive were unsuccessful and for this reason silicon carbide was used. In the preparation for welding the samples were handled only by personnel wearing lint-free gloves.

2.2 Equipment and Welding Conditions

All the welds for this investigation were made semi-automatically by the inert-gas-shielded tungsten-arc process, using straight-polarity and direct current. The welds consisted of fusion butt welds made along the long axis of the samples.

The welding was done in a welding box equipped with a vacuum system capable of evacuating the entire welding chamber to less than 0.03 microns Hg, a gas purification train to permit back-filling of the welding chamber with purified argon, a translation fixture with motor drive for positioning the work, and an automatically positioned welding torch (Figures 1 and 2).

The power supply was a Westinghouse 300 amps. RST welder equipped with an amperage remote control foot switch. The electrode used was a centerless ground 1/16" diameter, 2% thoriated tungsten electrode ground on a 15° taper and finished with a flat tip having a diameter one-quarter of that of the electrode. A chrome-plated copper back-up plate with a central groove 1/4" wide and 3/16" deep was used to control weld penetration and to ensure alignment between the abutted sheets (Figure 3).

Preliminary tests were performed to determine the optimum welding current necessary to produce fully penetrated joints and then five series of welding tests were made to establish the effect of welding atmosphere. Several feet of the alloy were welded in the welding box under the following atmospheres:

A) Box outgassed to 0.01μ and back-filled with purified argon to atmospheric pressure - using a heat-sink type welding torch.
B) Box outgassed to 0.01μ, back-filled with air to 2.2μ and then back-filled to atmospheric pressure with purified argon - using a heat-sink type welding torch.
C) Box opened - using a standard Line HW-13 torch equipped with a #8 nozzle plus an argon back-up.
D) Same as in (C) plus trailing shield.
E) Same as in (C) plus leading and trailing shields.

Details of the welding conditions are summarized in Table 2. The heat-sink torch is shown in Figure 2, and the set up for welding with the Linde torch and shields is shown in Figure 4. The trailing shield consisted of a copper tube with a 3 in. long x 1 in. wide canopy and

1/16" diameter perforations facing the fusion joint. It was attached to the torch by means of a clamp. The leading shield, which was subsequently connected in series with the trailing shield, consisted of a perforated copper tube (1/16" holes) and circular canopy formed to fit the contour of the nozzle. The assembly of leading and trailing shields can be seen in Figure 5. Only high purity argon (99.99% Argon) was used as shielding gas.

Two series of welding tests were also made on heat treated samples of Zr-Nb alloy to determine the effect of welding on the corrosion resistance and the mechanical properties of the heat treated alloy. All samples were welded as per Condition A described previously and summarized in Table 2. The first welding test consisted of welding 24 specimens, 2-3/4 in. long x 1.5 in. wide, abutted in pairs on their small axes (parallel to rolling direction) to produce one 18 in. long weldment. The reason for using such small samples was the limitation imposed by the heat treating requirements. The heat treatment specified by AECL was solution annealing at 880°C for one hour followed by water quenching and then aging at 500°C for 24 hours. For solution annealing the samples were sealed in quartz tubes (12 only per capsule) and outgassed to less than 0.05µ. At the time of quenching the capsules were broken over the quenching tank and the specimens were allowed to drop into water at room temperature. Great care was taken in performing a fast quench because the tensile properties of the alloy were said to be most affected by the rate of quenching from the solution treating temperature. The samples were subsequently vapour blasted with silicon carbide abrasive, aged in a vacuum furnace, outgassed to less than 0.02µ, and cooled in the cold zone of the furnace.

The second welding test consisted of welding together two 12 in. long x 2-3/4 in. samples, previously solution treated at 890°C for 15 minutes in Houghton #1450 salt bath using a charcoal cover and a rectifier for degassing the bath, followed by quenching in water, vapour blasting with silicon carbide abrasive and aging, as previously described, at 500°C for 24 hours.

To investigate the welding of Zr-2.5 Nb alloy to Zr-2, two series of welding tests were performed in the welding box under Condition A described in Table 2. In the first test a 12 in. long x 2-3/4 in. wide sample in the as-received condition was welded to an annealed sample of Zr-2 of the same dimension (annealed at 750°C for 1/2 hr. in vacuum furnace). In the second test 4 weldments of Zr-Nb to Zr-2 were made. In this case, the Zr-Nb had been solution treated in the salt bath and then aged, as previously described, before welding, and the Zr-2 had been annealed for 1/2 hr. at 750°C in a vacuum furnace.

3 - Evaluation of Weldments

After welding, all weldments were examined by the florescent penetrant method for surface defects. None of the welds showed defects. The weldments were thereafter inspected by x-raying with a 260 KV Baltograph unit for internal defects. The settings for x-raying were 145 KV, 4 ma, 5 minutes exposure at 36 in. focal distance, using Kodak M films with no screen. The radiographs revealed that all the welds were sound.

For evaluation by destructive testing, the experimental weldments were sampled as shown in the sketch of Figure 6. Sampling in this manner took into account the position of the specimen with respect to arc start or arc stop.

3.1 Gas Analyses

Oxygen, nitrogen and hydrogen analyses were made (in duplicate) on as-received and as-heat-treated sheet samples as well as on the material in the welds. The gas determinations in the welds were made on longitudinal sections of weld metal (full thickness); in some cases the heat affected zones of the base metal may have been partially included in the sample because of the difficulties in separating the narrow welds from the base metal.

Additional hydrogen analyses were made on sheet samples and on the weld metal and the base metal of some welded specimens which had been exposed to distilled and deionized water at 360°C (680°F) for 35 or 70 days, to determine the extent of hydrogen pickup during corrosion testing. The results of gas analyses on sheet materials and welded specimens before and after corrosion testing are presented in Tables 3 and 4. Data on specific weight gains due to corrosion are also included for better evaluation of the hydrogen pickup.

A few of the samples welded under the worst shielding atmospheres (Conditions C and D) showed a nitrogen pickup in the weld metal as low as those of welds made under the better shielding atmospheres (Conditions A, B and E). These low nitrogen contents may be due to the fact that the samples included a large proportion of the base metal or because the contamination was confined mainly to the top surface of the welds.

In general heat treating in the salt bath resulted in base metal with higher nitrogen, oxygen and hydrogen contents than when heat treated in quartz capsules.

3.2 Tensile Testing

Tensile tests were conducted at room temperature and at 315°C (600°F) on welded specimens, as-welded and welded and heat treated, with the weld positioned at the centre of the reduced section (Figure 7). Samples of sheet material were also tensile tested in the as-received and heat treated conditions for comparison with the welded specimens. The heat treatment involved was the solution treatment carried out in capsules or a salt bath at approximately 880°C for 24 hours. The results of tensile testing are presented in Tables 5, 6 and 7.

3.3 Corrosion Testing

Small coupons, 1 in. x 1 in., containing a weld across the mid-point as shown in Figure 6, were exposed in stainless steel autoclaves for 35 or 70 days to 360°C (680°F) distilled and deionized water, in the as-welded, welded and pickled, welded-heat-treated and vapour blasted, and welded-heat-treated-vapour blasted and pickled conditions. Coupons of sheet materials similarly heat treated and surface treated were included in the corrosion tests for comparison.

Pickling of the samples was carried out in a solution containing 30 w/o $H_2 SO_4$, 30 w/o HNO_3, 30 v/o H_2O and 10 v/o HF at approximately 50°C. The specimens were then immersed for 5 minutes in a warm aqueous solution containing 50 v/o HNO_3, rinsed in running cold water and boiling deionized water. The heat treatments involved were the treatments described previously.

The specimens were examined and weighed every 7 - 11 days. Before weighing, the specimens were dried for 1 hour in an oven at 110°C; fresh deionized water was employed for each exposure. After 35 days of exposure some of the autoclaved specimens were sectioned for hydrogen analyses and metallographic examinations. The hydrogen content of the corroded specimens was compared with that of reference specimens which had been retained in the various metallurgical conditions and with the same surface treatments. Data pertinent to the pickup of hydrogen during the 35 or 70 day corrosion test are shown in Tables 3 and 4.

Data on specific weight gains for samples from Zr-Nb sheets, specimens of the alloy welded under various atmospheres, samples of Zr-Nb welded after heat treating and samples welded to Zr-2, are included in Tables 8, 9, 10 and 11 respectively. Figures 8 to 11 inclusive illustrate the surface condition of typical corroded specimens.

Two of three sheet samples corrosion tested in the as-received condition showed a tightly adherent dark grey film and a low weight gain;

the other samples exhibited a relatively high weight gain and the white film characteristic of accelerated corrosion. The difference in the behaviour of these samples could not be explained.

All the pickled samples exhibited a black oxide film whereas the vapour blasted and unpickled samples showed a loose white powdery film with a black film underneath; this surface was observed on all the samples, welded or unwelded, exposed to corrosion after vapour blasting with the silicon carbide abrasive.

The gas contamination which occurred during welding showed a definite effect on the samples as-welded and corrosion tested for 35 days (Figure 9). The weld metal of samples welded under Conditions A, B and E showed an adherent dark grey film, whereas that of samples welded under Conditions C and D showed a thick white flaky film on its top surface. It was interesting to note that very little white oxide film formed on the bottom surface of all the welds made with the standard torch. Very likely the argon backup adequately protected the underside of the welds from gas pickup. In all cases the base metals and heat affected zones of the coupons showed dark grey and white powdery films respectively. Pickling after welding appears to have reduced the contamination in the welds but not affected the appearance of the film at the heat affected zones (Figure 9). Vapour blasting and pickling the coupons autoclaved for 35 days seems to have eliminated the contamination in the welds and none of the coupons corrosion tested for an extra 35 days showed the white corrosion product on the welds; only the heat affected zone/base metal interface showed a thin white film (Figure 10).

Heat treating after welding followed by vapour blasting appeared to have reduced the corrosion at the weld and only the samples welded with the standard torch and no shields showed a considerable buildup of corrosion product on the welds. In the latter condition all the samples exhibited a white powdery film (Figure 9).

Coupons solution treated, tempered at 500°C and pickled, all showed a black film except the coupons from welds performed in the open with the standard torch and no shields. In the latter case a white film built up on the weld metal (Figure 9). None of the heat treated coupons vapour blasted and pickled after the first 35 day exposure to water and corrosion tested for another 35 days, showed any significant white film with the exception of the Condition B specimen which appeared to have been contaminated in the pickling operation (Figure 10).

On the corrosion coupons from Zr-Nb sheets welded after heat treating, the welds showed a black film, the heat affected zone a thick white film and the base metal a dark grey film when as-vapour blasted, and a black film when as-pickled.

The Zr-Nb portion of the Zr-Nb/Zr-2 corrosion coupons showed the same surface films as observed on the Zr-Nb corrosion coupons which had been welded in the as-received, or welded after heat treating. The weld showed a black film and the Zr-2 base and heat affected zones showed no white corrosion product.

The metallographic examination of typical corroded specimens is reported in the following section.

3.4 Metallography and Hardness Survey

Cross sections of all welds were examined metallographically, after etching in a solution of 45 v/o HNO_3, 10 v/o HF, 45 v/o H_2O. The width and soundness of the weld and heat-affected zones were noted and a study made of the structural changes which had occurred in these areas and in the base metal during welding and heat-treatment.

A coupon from each series of corrosion tests was sectioned and a metallographic examination made of the hydrogen pickup in the base metal and in the various parts of the weld.

Welds made between as-received Zr-Nb samples under the various atmospheres were all sound and no discernable differences in microstructure were evident. Figure 12 illustrates the microstructure of a sample welded in the welding box outgassed to 0.01 and back-filled with purified argon to atmospheric pressure. The photomacrograph at 10X shows three well defined zones: (1) weld zone (2) heat-affected zone, and (3) unaffected base material.

The weld zone contains large transformed beta grains* and represents the actual melting of the Zr-Nb. Adjacent to the weld zone is the heat affected zone which has smaller transformed beta grains with alpha grain boundaries. In the region near the unaffected base metal the material showed small transformed beta grains with coarse interconnected alpha regions. The unaffected base material represents a region entirely excluded from the heating affects of welding and shows a structure typical of hot rolled Zr-Nb i.e., a coarse alpha structure with retained beta zirconium or beta niobium.

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*These grain boundaries outline what was the original beta structure now transformed to a martensitic type of alpha, referred to as acicular or transformed beta.

The samples welded with the standard torch with one or two argon shields showed grains approximately 0.030 in. in diameter at the center of the weld zone whereas the samples welded in the pre-evacuated box and in the open with the standard torch and no extra shields, showed grains between 0.010 to 0.015 in. in diameter. The width of the weld zone and heat affected zones varied from one sample weldment to another for weldments made under the same welding conditions. In general the welds and the heat affected zones were 0.160 to 0.420 in. and 0.024 to 0.075 in. respectively. Typical hardness readings across a weld made under Condition A are shown in Table 12.

Metallographic examination of sections of samples from each series of welds corroded for 35 days in water at 360°C (680°F) showed that in all cases the hydrogen pickup was greater in the weld than in the unaffected as-received base metal. The same observation was made on samples vapour blasted and pickled after 35 days of a total 70 day exposure to water. Figures 13 and 14 show equal areas of weld metal and base metal of a sample welded under Condition A and tested for 70 days in water.

Heat treating after welding did not change the grain size of the weld metal and heat-affected zones but altered the structures in the various zones of the weldments. Figure 15 shows the structures developed by the heat treating at 880°C for 1 hour, water quenching and tempering at 500°C for 24 hours. The weld shows coarse transformed beta grains with a few small alpha grains within the grain and a nearly continuous precipitate of alpha at grain boundaries. In the heat affected zone the transformed beta grains are smaller than in the weld zone and the grains appear to be more defined in the transformed beta and the alpha phase less numerous at the grain boundaries. The unaffected base metal in the heat treated condition shows alpha grains in a matrix of transformed beta. The hardness readings across a typical heat treated weld is shown in Table 12.

Metallographic examination of welded samples corrosion tested for 35 days in 360°C (680°F) water in the heat-treated and vapour-blasted condition revealed that the hydrogen pickup in the base metal was as great as in the weld zone although in the latter the hydrides were much coarser. The heat treated and pickled samples picked up approximately the same amount of hydrogen as the heat treated and vapour blasted samples and its distribution in the various zones was the same.

Figure 16 shows that welding after the solution heat treatment and furnace aging produced a fine acicular alpha and retained beta zirconium or beta niobium in the weld zone and a coarse acicular alpha and retained beta zirconium or beta niobium in the heat affected zone.

The unaffected base metal showed alpha grains in a matrix of transformed beta. Hardness measurements on a sample showed that the weld zone was harder than the heat treated base metal and that welding reduced slightly the hardness of the sheet material at the base metal/heat affected zone interface. Typical readings are shown in Table 12.

Metallographic examination of samples as-welded, and welded and pickled, corroded for 35 days in 360°C water revealed that the concentration and distribution of hydrides were the same in all zones. Pickling showed no effect on the pick up of hydrogen.

The microstructures of the Zr-Nb base metal and heat affected zone of welds made between Zr-Nb and Zr-2 were the same as those observed in weldments of Zr-Nb made in as-received, and after heat treatment. The weld zones showed transformed beta grains with very few alpha grains at grain boundaries. The Zr-2 showed a Widmanstatten structure in the heat affected zone and alpha grains in the base metal. Microscopic examination of a corrosion coupon from each series of Zr-Nb/Zr-2 welds indicated that the hydrogen pickup was greater in the Zr-Nb heat affected zone than in the other sections of the welded specimens (Figures 17 and 18).

The results of Knoop hardness survey made across a weld from each series of Zr-Nb/Zr-2 weldments are shown in Table 13.

4 - Discussion of Results

The following discussion of the test results indicates that Zr-Nb is readily joinable to itself or Zircaloy 2 by the inert-gas-shielded tungsten arc process and that butt-welded joints made under controlled fabricating conditions exhibit attractive elevated temperature tensile properties and relatively good corrosion resistance in water at 360°C (680°F).

4.1 Weldability

The welding experiments carried out on as-received or hot rolled Zr-Nb under various atmospheres and on Zr-Nb heat treated before welding indicated that the material is readily weldable. As anticipated, the alloy as-received or fully heat-treated was readily joinable to Zr-2. None of the welds showed cracks, porosity, blow holes, or voids and weld penetration could be accurately controlled.

The variations in the width of the welds were probably due to differences in cooling rates caused by the lack of good contact of the samples with the backing plates and the distortion of the specimens during welding.

The larger grain size in the weld zones of samples welded with a standard torch and extra shields is believed to be due to the lower welding speed employed. Shielding the weld zone with a leading and trailing shield is probably adequate for thin sheets (up to 0.050 in. thick) but when thick sections are considered it is preferable to perform the welding in a welding box pre-evacuated and back-filled with purified argon.

4.2 Mechanical Properties

Tensile measurements made at room temperature and 600°F showed that welded samples of Zr-Nb, as-received or heat treated after welding, had mechanical properties equal or slightly better than that of the sheet samples in the same metallurgical conditions and that welding under various atmospheres did not affect significantly the mechanical properties of the welds. However, the weld metal and the heat affected zones being hard, all the tensile specimens broke in the base metal. Samples heat treated after welding showed a tendency to fail in the weld, especially when tested at 315°C (600°F). The failure in the weld may be attributed to the presence of alpha at the transformed beta grain boundaries. Very likely the time at the solution heat treating temperature was not long enough to homogenize the structure and on quenching some alpha precipitated preferentially at the transformed beta grain boundaries. Welding after heat treating did not affect the properties of the alloy seriously and the Zr-Nb still showed about the same ductility. Only one of the samples broke in the weld. The weld zone showed the same structure as that of as-received and welded Zr-Nb but the heat affected zone, perhaps as a result of heating in the alpha and beta region, showed a coarse acicular alpha with retained beta zirconium or beta niobium.

The tensile samples of Zr-Nb as-received and heat treated welded to annealed Zr-2, showed approximately the same mechanical properties. When heat treated after welding the specimens showed an increase in ultimate and yield strengths by about 10,000 psi. In all cases the welded specimens broke in the Zircaloy-2. Hardness measurements across the welds indicated that the fusion of Zr-2 with the Zr-Nb produced a weld zone softer than the Zr-Nb heat affected zone on the Zr-Nb heat-treated base metal.

4.3 Corrosion and Hydrogen Pickup

In general, as-received Zr-Nb sheet material showed better corrosion resistance and lower hydrogen pickup than the fully heat treated Zr-Nb (6 to 16 ppm H_2 for as-received and 26 to 30 ppm H_2 for as-heat treated). Pickling resulted in the formation of a black adherent corrosion film but a higher weight gain. Vapour blasting resulted in the formation of a loose white powdery corrosion film which probably fell off the specimens in the autoclave and caused the lower weight gain of the specimens.

The base metal of the samples welded under the various atmospheres behaved like the sheet samples submitted to corrosion in the various metallurgical and surface treated conditions. Welding seriously affected the corrosion resistance of the as-received Zr-Nb, the effect being less for the samples welded under the best shielding atmospheres, i.e. in the box evacuated and back-filled with argon or in the open atmosphere using a standard torch with leading and trailing shields. The effect does not appear to be due only to the structure of the weld metal and heat affected zone because pickling or vapour blasting followed by pickling practically eliminated the formation of the white film on the welds; only the metal at the interface between the heat affected zones and the base metal showed the white corrosion product when corrosion tested after these surface treatments. In this case it is believed that the transformed beta grains with a fine precipitate of alpha is responsible for the greater corrosion in this area.

In all cases the weld metal picked up more hydrogen than the as-received base metal, (approximately 6 to 16 ppm for base metal and 25 to 40 ppm for weld metal). The pickup of hydrogen by as-welded and as-welded and pickled samples corroded for 35 days in water was similar and it is just about double in samples corrosion tested for 70 days.

Heat treatment, followed by two vapour blasting and pickling treatments, definitely improved the corrosion resistance of all the welded coupons and showed that most of the gas contamination which occurred during welding under the poor shielding atmospheres (Condition C and D) must have been on the surfaces of the welds. The heat treated samples showed no preferential distribution of hydrides.

The corrosion behaviour of samples welded after heat treatment was identical for salt bath heat treated and furnace heat treated samples. The white film which formed in the heat affected zones is believed to be due to the presence of fine alpha grains at the boundaries of the transformed beta grains. Very likely a complete beta solution treatment followed by aging at 500°C would be required to restore the corrosion resistance of the material in the heat affected zone. The heat treated and welded samples shows the same hydrogen pickup as the welded and heat treated samples.

The corrosion behaviour of the heat affected zone and base metal on the Zr-Nb side of the Zr-Nb/Zr-2 welded coupons was similar to that observed on Zr-Nb/Zr-Nb welds prepared and tested under the same conditions. Although the data on hydrogen pickup in Zr-Nb/Zr-2 welds is not conclusive, in general it appears to be approximately the same as that for Zr-Nb/Zr-Nb welds prepared under similar conditions.

As most of the welded samples showed a loosely adherent white corrosion product on or near the welds the weight change measurements cannot be considered an absolute measure of corrosion.

5 - Conclusions

From the information presented the following conclusions may be drawn:

1. Zr-Nb hot rolled or heat treated may be successfully inert tungstenarc butt-welded to itself or to Zircaloy-2.

2. The weld-shielding atmosphere has no effect on the mechanical properties of the welds but shows a definite effect on the corrosion resistance of welded Zr-Nb.

   a) With the best shielding atmospheres obtained by welding in a box evacuated and back-filled with argon or using a standard torch with leading and trailing shields, welds with relatively good corrosion resistance may be produced.
   b) Welds made with the standard torch with and without trailing shields corrode rapidly in the autoclave.

3. Pickling after welding improves the corrosion resistance of welds made under good or poor atmospheres.

4. Solution heat treating at 880-890°C followed by water quenching and aging at 500°C for 24 hours and vapour blasting improves the corrosion resistance of all welds, particularly when the welds have been pickled before testing.

5. Heavy vapour blasting followed by pickling improves the corrosion resistance of heat treated or un-heat treated welds.

6. Welding under any shielding atmosphere impairs the corrosion resistance of the Zr-Nb base metal (as-received or heat treated) at the heat affected zones and complete solution and aging heat treatment is probably required to restore the corrosion resistance of the Zr-Nb.

7. The atmosphere obtained when welding with the standard torch equipped with leading and trailing shields produced welds as good as those performed in a pre-evacuated box (0.01μ) and back-filled with purified argon; however, the latter is considered more suitable for the welding of thick sections and complicated shapes of Zr-Nb alloy.

8. Variations in mechanical properties and corrosion resistance of welds with previous history are discernible from the few specimens available, but a more detailed study of these effects would require a larger number of specimens and a statistical evaluation of the results.

SECTION 2

WELDING OF Zr-3 w/o Al - 0.5 w/o Mo ALLOY

1 - Materials

A 22.5 in. long x 12 in. wide x 0.100 in. thick strip of Zr-Al alloy produced by Harvey Aluminum Co. Inc., Torrance, California was supplied by Atomic Energy of Canada Limited for an investigation of the weldability of the alloy. As the strip was too thick for the welding experiments proposed, the Zr-Al was fabricated into strips 0.030 in. thick and 2-3/4 in. wide. Attempts made to reduce the material by cold rolling were unsuccessful, even after intermediate anneals at 980°C. The Zr-Al alloy was finally reduced to thickness of 0.038 in. to 0.040 in. by hot rolling at 980°C, using reductions of 0.010 in. per pass.

Satisfactory cleaning of the hot rolled strips was obtained by vapour blasting, using 220 grit silicon carbide powder, and by pickling in a bath composed of 45 v/o nitric acid and 5 v/o hydrofluoric acid. About 0.002 in. of metal was removed by pickling for approximately 2 minutes at room temperature. As the strips required straightening and a better surface finish they were annealed in vacuum (less than 0.02μ) at 980°C, cold rolled a few per cent and annealed again at 980°C. This treatment yielded strips with clean uniform surfaces.

The composition of the 0.100 thick strip of Zr-Al and the nitrogen and oxygen contents of six 0.030 in. thick hot rolled strips used in the welding experiment are shown in Table 13.

2 - Experimental Procedure and Results

2.1 Preparation of Samples

Six hot rolled strips which had been analysed for oxygen and nitrogen were sheared into samples 2-3/4 in. wide and 12 in. long, deburred and cleaned with acetone. In the preparation for welding they were handled only by personnel wearing lint-free gloves.

2.2 Equipment and Welding Conditions

The welds were all made semi-automatically by the inert-gas-shielded tungsten-arc process, using straight-polarity and direct current, using the equipment described in the investigation on the weldability of the Zr-Nb alloy. The welds consisted of fusion butt welds made along the long axis of the samples and parallel to the rolling direction.

Once the optimum welding current necessary to produce full-penetration joints was established, three series of welding tests were made to determine the effect of welding atmosphere on the mechanical properties of the alloy. Two strips of Zr-Al alloy were welded together in the welding box under each of the following atmospheres:

1. Box outgassed the 0.01μ and back-filled with purified argon to atmospheric pressure-using a heat-sink type welding torch (Strips 1 and 2).

2. Box opened - using a standard Line HW-13 Torch equipped with a #8 nozzle plus an argon back-up (Strips 3 and 4).

3. Same as in (2) plus leading and trailing shields (Strips 5 and 6 - Shields were the same as used in the Zr-Nb investigation).

Details of the welding conditions are summarized in Table 14. The Zr-Al alloy expanded much more than the Zr-Nb during welding, and to avoid the distortion of the weldments the current had to be decreased to 33 amperes.

3 - Evaluation of Weldments

Examination of the weldments by the fluorescent penetrant method and by radiography revealed that all the welds were free from defects and the weldments were cut into 1 in. wide specimens as shown in Figure 6. Samples A and K were chosen for gas analysis, Sample G for metallography and Samples B, C, E, F, I and J for tensile testing.

3.1 Gas Analyses

Oxygen and nitrogen analyses were made on the material in the welds to determine the extent of gas pickup during welding under the various atmospheres. The welds were not analysed for hydrogen because the welded samples were not to be corrosion tested. The results of the gas analyses are presented in Table 15.

3.2 Tensile Testing

Tensile tests were conducted at room temperature and at 315°C (600°F) on welded specimens similar to those used in the testing of Zr-Nb welds, (Figure 7,). The strips being too narrow to machine tensile specimens having the same dimensions, the tensile tests on the sheet material were carried out on specimens prepared by welding the remaining portions of strips 1, 2 and 3 and machining samples with a 2 in. gauge length free of weld metal from the welded assembly. The tensile results are shown in Table 15.

3.3 Metallography and Hardness Survey

A cross section of each of the welds made under the three shielding atmospheres was examined under the microscope after mechanical polishing and anodizing for 10 seconds in a solution containing 60 ml ethyl alcohol, 35 ml distilled water, 5 ml phosphoric acid, 10 ml lacticacid, 20 ml glycerine and 2 grams citric acid using a stainless steel cathode and 60 - 80 volts. All the welds appeared to be sound and showed no structural differences. Figure 19 shows photographs of the butt welded joint made in the pre-evacuated welding box at 10 X and 250 X. The weld zone contains large transformed beta grains. The heat affected zones shows small transformed beta grains with fine alpha regions. The unaffected base material shows a coarse acicular alpha structure with probably some scattered Zr_3Al crystals.

The welds made under Conditions 1 and 3 showed a weld zone with grains approximately 0.030 in. in diameter. The weld zone of the sample performed under Condition 2 was 2 grains thick. For the three weldments the weld plus heat-affected zones was approximately 1/4 inch wide.

The results of a Knoop hardness survey (500 kg load) made on the cross section each weld are presented in Table 16.

4 - Discussion of Results

4.1 Weldability

The welding experiments carried out on the Zr-Al under the various shielding atmospheres indicated that the material is readily joinable to itself by the inert-gas-shielded tungsten arc process. All the welds were free from defects. The material showed a greater tendency to distort during welding than the Zr-Nb alloy and welding jigs should be designed accordingly.

The smaller grain size (0.015 in. diameter) observed in the weld performed with the standard torch and no shields is probably due to the combination of low amperage (33 amps) and high welding speed (10 in./min.). Very likely the same factors were also responsible for the low gas pickup in the weld.

4.2 Mechanical Properties

The samples welded under the various atmospheres showed no difference in tensile properties and exhibited strength values as good as the sheet material. At 315°C (600°F) the samples showed an ultimate tensile strength of approximately 110,000 psi, nearly four times as great as that of Zircaloy-2. At room temperature the strength of the welded material was approximately 150,000 psi.

The weld zones of the sample welded at high speed (10 in./min.) in the box or in the open with the standard torch and no shields showed a slightly higher hardness than the base metal.

5 - Conclusions

The work reported above has shown that Zr-3 w/o Al-0.5 w/o Mo may be successfully inert tungsten-arc butt-welded to itself and that slight gas contamination has no effect on the mechanical properties of the welds. A relatively good atmosphere has been obtained when welding in the open with the standard torch with or without shields; however, welding in a box pre-evacuated (to 0.01μ) and back-filled with purified argon is considered more suitable for the forming of thick sections and complicated shapes of Zr-3 Al-5 Mo alloy.

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Table 1

CHEMICAL COMPOSITION* OF Zr-Nb & Zr-2

Elements	Zr-Nb [1] Sheet	Zr-2 Plate	Zr-2 [1] Sheet
Niobium	2.7%		
Carbon	-	192	
Aluminum	30.	23	
Oxygen	0.122%		0.095%
Nitrogen	22.	45	42
Hydrogen	7	19	12
Iron	499	1285	
Tungsten		< 25	
Nickel	15	597	
Chromium	99	850	
Manganese	10	< 25	
Silicon	≃ 130	< 50	
Cobalt	< 10	< 5	
Molybdenum	≃ 20	< 5	
Tin	30	1.7%	
Titanium	10	< 25	
Copper	≃ 20	32	
Magnesium	10	< 15	
Sodium		< 20	
Calcium		< 15	
Vanadium	Not detected	< 5	
Lead	< 20	< 10	
Boron		0.5	
Hafnium		56	
Zinc		< 50	
Cadmium		< 0.5	

*Analyses in ppm, except for alloying elements which are given in weight percent.

[1] Analyzed by Canadian Westinghouse Co. Ltd.

Table 2

WELDING CONDITIONS FOR Zr-Nb

Welding Atmospheres	Current (Amperes)	Voltage Volts	Work Speed (in./min.)	Shielding Gas Flow Rate, cfh. Through Torch	Through Shields
A	50±5	8	10	-	-
B	"	8	10	-	-
C	"	8	10	30	
D	45±5	9	5	30	35
E	"	9	5	30	35

A - Welding box evacuated to 0.01μ and back filled with argon to atmospheric pressure.
B - Welding box evacuated to 0.01μ, back filled with air to 2.2μ, then with argon as in A.
C - Welding box opened - using standard torch with argon backup.*
D - Same as in C plus trailing shield.
E - Same as in C plus leading and trailing shields.

* Using argon back up at 5 cfh - weld area was purged for 5 minutes before and after welding.

Table 3

GAS ANALYSES OF SHEET MATERIALS BEFORE AND AFTER CORROSION

Material	Condition	Nitrogen ppm	Oxygen%	Wt. gain (mg/dm^2) After 35 days	Hydrogen ppm Original	Hydrogen ppm After 35 days
Zr-Nb	As received	22	0.132	20.7	7	13
	As rec'd & Pickled			68.6	7	23
	Furnace Heat Treated (1) and Vapour Blasted	23	0.136	45.4	16	42
	Furnace Heat Treated Vapour Blasted & Pickled			68.5	13	43
	Salt bath heat treated (2) and Vapour Blasted					
	Plate #1	40	0.149		64	
	2	44	0.134		30	
	3	62	0.142		35	
	4	61	0.144		25	58
	5	47	0.145		30	
	6	41	0.125		32	
	Salt bath heat treated (2) Vapour Blasted & Pickled Plate #5				30	49
Zr-2	Annealed (3)				9	32
	Annealed and Pickled				8	42
	Annealed, Heat treated (1) in furnace and vapour blasted	36	0.140		25	38

(1) Solution treated at 880°C for 1 hour in quartz capsule evacuated to 0.05μ, quenched in water, vapour blasted, then aged for 24 hours in vacuum furnace (0.02μ) at 500°C.
(2) Solution treated at 890°C for 15 minutes in Houghton #1450 salt bath using charcoal cover and rectifier for degassing, quenched in water, vapour blasted and aged as in (1).
(3) Annealed at 750°C for 1/2 hour in vacuum furnace.

Table 4

GAS ANALYSES OF SHEET MATERIALS BEFORE AND AFTER CORROSION

Welding Atmosphere	Weld No.	Weld Condition	Nitrogen ppm** A	Nitrogen ppm** E	Nitrogen ppm** K	Oxygen %	Wt. gain (mg/dm^2) after 35 days	Wt. gain (mg/dm^2) after 70 days	Hydrogen ppm Original	Hydrogen ppm After 35 days	Hydrogen ppm After 70 days
			Zr-Nb/Zr-Nb Welded in As-Received Condition								
A	10	As-welded	29	28	30	.138			12		
	11	"	32	27	45	.138			9		
	12	"	53	40	45	.143			11		
	13	"	51	40	35	.140			9		
	14	"	67	68	74	.144			13		
	15	"	67	74	96	.137	140.7	123.1	9	43	74
	11	Welded & pickled	32	27	45	.138	122.8		9	40	
	3	Welded & Heat Treated*	74	-	77	.146			30		
	4	" " "	71	-	75	.146			15		
	5	" " "	40	-	40	.154			39		
	6	" " "	98	-	98	.163			16		
	8	" " "	107	-	109	.143	57.7	111.7	14	45	63
	5	Welded Heat Treated & Pickled	40	-	40	.146	92.8		39	58	
B	19	As-welded	80	69	74	.141	97.3	116.3	9	38	70
	16	"	61	-	55	.139			11		
	17	Welded & Heat Treated*	-	-	115	.142			20		
	18	" " "	-	-	99	.129			21		
	25	" " "	179	-	153	.132	116.8	108.9	21	45	76
	26	" " "	91	-	81	.141			22		
C	27	As-welded	165	-	194	.172	100.3	111.3	14	47	81
	29	"	208	-	158	.137			12		
	27	As-welded & pickled	165	-	194	.172	102.4		14	52	
	39	Welded & Heat Treated*	79	-	59	.139	36.1	97.9	39	41	63
	40	" " "	77	-	100	.141			31		
	39	Welded, Heat Treated & Pickled	79	-	59	.139	78.5		39	52	
D	34	As-welded	180	-	180	.141	122.0	127.0	8	52	77
	35	"	332	-	107	.139			6		
	34	Welded & pickled	180	-	180	.141	171.1		8	52	
	41	Welded & Heat Treated*	41	-	59	.149			38		
	42	" " "	57	-	45	.133	38.8	89.8	25	46	62

Table 4 (con't)

Welding Atmosphere	Weld No.	Weld Condition	** Nitrogen ppm A	E	K	Oxygen %	Wt. gain (mg/dm^2) after 35 days	70 days	Hydrogen ppm Original	After 35 days	After 70 days
E	36	As-welded	111	-	37	.139	114.3		9	47	
	37	Welded & Heat Treated*	73	-	71	.140			9		
	38	" " "	38	-	49	.140	48.5	100.2	9	52	63
		Zr-Nb/Zr-Nb Heat Treated before Welding									
A	HT	As welded	79		73	.150	167.0		14	55	
	S-5	As welded	51		67	.171	118.0		28	71	
		Welded & pickled	51		67	.171	179.1		21	77	
		Zr-Nb Welded to Zr-2									
A	44	As welded	74	-	58	-	60.6		8	30	
	45	Welded & Heat Treated*	88	-	63	.138	35.3		22	39	
	S-1	As Welded	145	-	219	.153	105.0		29	63	
		Welded & Pickled	145	-	219	.153	98.2			69	
	S-2	As Welded	251	-	234	.125	85.2		19	46	
		Welded & Pickled	251		234	.125	94.4			60	
	S-3	As Welded	-	-	137	.142	69.2		16	35	
		Welded & Pickled					101.2		14	56	
	S-4	As Welded	59		52	.148	63.1		14	30	
		Welded & Pickled					92.1			51	

* Solution treated at 880°C for 1 hour in quartz capsule evacuated to 0.05μ, quenched in water, vapour blasted, then aged for 24 hours in vacuum furnace (0.02μ) at 500°C.

**See Figure 6 for sampling arrangement.

Table 5

TENSILE PROPERTIES OF Zr-Nb SHEET MATERIAL & Zr-Nb WELDS MADE UNDER VARIOUS ATMOSPHERES

Condition	Test Temp. (°F)	No. of Specimens	U.T.S. (1000 lb/in.^{2})	Y.S. (2% offset) (1000 lb/in.^{2})	Elgonation (% in 2")	No. of Specimens Fractured in Weld
As received	RT	32	112	94	4	-
	600	3	67	53	5	-
As rec'd, heat treated*	RT	1	123	109	7	-
	600	3	88	73	6	-
As welded (cond.A)	RT	11	111	100	4	-
	600	6	60	55	3	-
As above, heat treated*	RT	3	125	116	6.5	1
	600	5	86	76	2.5	5
As welded (cond.B)	RT	3	111	98	6	-
	600	3	61	48	4	-
As above, heat treated*	RT	6	125	117	1.5	6
	600	6	87	75	2.5	6
As welded (cond.C)	RT	3	114	100	7	-
	600	3	65	57	5	-
As above, heat treated*	RT	3	133	120	5	-
	600	3	90	77	4	3
As welded (cond.D)	RT	3	111	98	5	-
	600	3	65	57	4	-
As above, heat treated*	RT	3	131	118	6	-
	600	3	87	77	4	3
As welded (cond.E)	RT	3	111	97	5	-
As above, heat treated*	RT	3	130	116	6	-
	600	3	91	78	3	3

*Solution treated at 880°C for 1 hour after sealing in quartz capsule evacuated to 0.05μ, water quenched, vapour blasted and tempered for 24 hours in vacuum furnace 0.02μ at 500°C.

Welding Conditions

A - Welding box evacuated to 0.01μ and backfilled with purified argon to atmospheric pressure.
B - Welding box evacuated to 0.01μ backfilled with air to 2.2μ, then backfilled with argon as in A.
C - Welding box opened - using standard torch with argon back-up.
D - Same as C plus trailing shield.
E - Same as C plus leading and trailing shields.

Table 6

TENSILE PROPERTIES OF Zr-Nb WELDED* AFTER HEAT TREATING

Heat Treatment	Test Temp. (°F)	Specimens	U.T.S. (1000 lb/in.^{2})	Y.S. (.2% offset) (1000 lb/in.^{2})	Elongation (% in 2")	No. of Specimens Fractured in Weld
T.C.	RT	3	123	112	5	-
	600	3	91	79	3	-
T.S.	RT	1	123	120	1	1
	600	2	94	82	3	-

* Welded in welding box evacuated to 0.01μ & backfilled to atmospheric pressure with purified argon.

T.C. - Solution treated at 880°C for 1 hour after sealing in quartz capsule evacuated to 0.05μ, water quenched, vapour blasted and tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.

T.S. - Solution treated at 890°C for 15 minutes in Houghton #1450 salt bath using charcoal cover and rectifier for degassing, quenched in water, vapour blasted and tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.

Table 7

TENSILE PROPERTIES OF Zr-Nb/Zr-2 WELDED* SPECIMENS

Base Metal Condition	Weld No.	Weld Condition	Test Temp. (°F)	Sample No.	U.T.S. (1000 lb/in.^{2})	T.S. (.2% offset) (1000 lb/in.^{2})	Elgonation (% in 2")
A.R./An	44	As welded	RT	44-D,H	71	60	17
			600	44-C,I	31	21	25
		As welded H.T.**	RT	45-C,D	79	65	15
			600	45-I,J	42	32	20
H.T./An	2	As welded	RT	3	63	52	16
			600	1,2	30	19	21
	3	As welded	RT	3	64	50	16
			600	1,2	29	18	23
	4	As welded	RT	3	75	60	15
			600	1,2	32	21	24
	5	As welded	RT	3	74	60	14
			600	1,2	32	22	24

Note: All failures occurred in Zr-2 at least 3/4" from weld.

* Welded in welding box evacuated to 0.01μ and backfilled to atmospheric pressure with purified argon.

** Welds solution heat treated at 880°C for 1 hour after sealing in quartz capsule evacuated to 0.05μ, water quenched, vapour blasted & tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.

A.R./An - Zr-Nb as received
Zr-2 annealed at 750°C for 1/2 hour in vacuum furnace (0.02μ)

H.T./An - Zr-Nb solution treated at 890° for 15 minutes in Houghton #1450 salt bath using charcoal cover and rectifier for degassing, quenched in water, vapour blasted and tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.
Zr-2 annealed at 750° for 1/2 hour in vacuum furnace (0.02μ).

Table 8

CORROSION DATA FOR Zr-Nb SHEET SPECIMENS EXPOSED TO 360°C (680°F) WATER

Specimen Condition	Specific Wt. gain (mg/dm^2) 7 Days	14 Days	24 Days	35 Days	Remarks
As received	30.6	36.4	33.0	32.2	Dark grey film
	60.3	67.8	71.1	75.2	White film
	24.8	25.7	25.6	20.7	Dark grey film
As-reç'd & Pickled	37.2	47.1	36.4	71.1	Black adherent film
	37.2	46.3	44.6	68.6	" " "
	32.2	44.6	51.2	73.5	" " "
Heat Treated & Vapour* Blasted	31.2	37.2	33.0	28.1	White powdery film
	52.1	41.3	39.7	36.4	" " "
	56.2	52.9	49.6	45.4	" " "
Heat Treated, Vapour Blasted & Pickled	34.7	53.7	60.3	81.0	Black adherent film
	34.7	48.8	51.2	71.9	" " "
	23.1	41.3	43.8	68.6	" " "

* Solution treated at 880°C for 1 hour after sealing in quartz capsule evacuated to 0.05μ, water quenched, vapour blasted and tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.

Table 9

CORROSION DATA FOR Zr-Nb WELDED UNDER VARIOUS ATMOSPHERES AND EXPOSED TO 360°C (680°F) WATER

Welding Condition	Surface Condition	No. of Samples	Average Wt. gain (mg/dm^2) 7 Days	14 Days	24 Days	35 Days
A	a)	6	66.9	99.1	112.0	131.3
	b)	3	53.7	72.4	89.2	122.8
	c)	6	55.0	63.2	53.9	55.5
	d)	3	43.5	61.4	68.6	92.8
	e)	2	32.2	46.8	73.8	123.3
	f)	2	31.6	49.8	70.4	
B	a)	6	54.2	73.0	80.0	97.3
	c)	6	56.2	53.4	41.5	37.3
	e)	2	21.6	42.7	65.0	116.3
	f)	2	19.2	46.9	70.8	108.9
C	a)	6	64.7	89.3	101.5	103.6
	b)	2	28.7	67.5	85.7	147.3
	c)	6	45.5	43.9	44.4	36.3
	d)	3	15.1	37.5	53.4	99.2
	e)	2	18.3	43.1	62.6	111.3
	f)	2	21.2	42.7	67.1	98.1
D	a)	6	63.6	93.4	110.7	127.4
	b)	2	32.8	70.0	95.7	157.6
	c)	6	43.6	47.8	51.2	39.2
	d)	1	20.7	47.1	60.3	119.0
	e)	2	27.4	49.8	72.0	127.0
	f)	2	19.2	38.6	57.2	89.8
E	a)	3	61.2	80.4	94.0	114.3
	c)	6	50.9	50.9	47.6	45.6
	f)	2	19.2	42.3	66.3	100.2

A - welding box evacuated to 0.01μ and back-filled to atmospheric pressure with purified argon.
B - welding box evacuated to 0.01μ, back-filled with air to 2.2 , then backfilled as in A.
C - Welding box opened - using standard torch with argon back-up.
D - Same as C + trailing shield.
E - Same as C + leading & trailing shields.

a) As-welded
b) Welded & Pickled
c) Welded, solution treated at 880°C for 1 hour after sealing in quartz capsule evacuated to 0.05μ, water quenched, vapour blasted & tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.
d) Same as c) plus pickling.
e) Welded, corrosion tested for 35 days, vapour blasted & pickled.
f) Samples of condition d) corrosion tested for 35 days then vapour blasted & pickled.

Table 10

CORROSION DATA FOR Zr-Nb WELDED* AFTER HEAT TREATING

AND EXPOSED TO 360°C (680°F) WATER

Heat Treatment	Specimen Condition	Wt. gain (mg/dm^2) 7 Days	14 Days	24 Days	35 Days	Remarks
T.C.	As welded	34.7	72.7	105.0	166.9	base metal & weld dark
		35.5	83.5	108.3	174.4	grey, heat affected zones
		40.5	90.9	118.2	181.0	white
T.S.	As welded	56.3	72.3	93.6	140.0	base metal grey-weld
		53.3	57.8	76.9	118.0	black-heat affected
		62.4	79.1	105.0	150.7	zones white
T.S.	Welded & Pickled	49.5	73.0	109.6	160.6	base metal black-weld
		48.7	78.3	117.2	179.1	dark grey & heat affected
		48.7	69.2	107.3	162.9	zones white

* Welded in welding box evacuated to 0.01μ & back-filled to atmospheric pressure with purified argon.

T.C. - Solution treated at 880°C for 1 hour after sealing in quartz capsule evacuated to 0.05μ, water quenched, vapour blasted & tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.

T.S. - Solution treated at 890°C for 15 minutes in Houghton #1450 salt bath using charcoal cover and rectifier for degassing, quenched in water, vapour blasted and tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.

Table 11

CORRODION DATA FOR Zr-Nb/Zr-2 WELDED* SPECIMENS

EXPOSED TO 360°C (680°F) WATER

Base Metal Condition	Weld No.	Weld Condition	Sample No.	Wt. gain (mg/dm²) 7 Days	14 Days	24 Days	35 Days	Remarks
AR/An	44	As welded	44-B	28.1	35.5	56.2	50.4	Base metals and
			-E	40.5	49.6	66.1	67.8	weld dark grey-
			-J	37.2	50.4	62.8	62.8	Zr-Nb HAZ white
		Heat treated** & Vapour Blast.	45-F	36.4	38.0	38.0	33.0	White film all
			-B	36.4	41.3	43.0	37.2	over samples
HT/An	S-1	As welded (AW)	1	33.5	44.1	57.1	87.5	Zr-Nb base and
			2	39.6	47.9	69.2	105.0	HAZ white, weld
			3	48.7	56.3	82.9	120.2	& Zr-2 black
		Welded & Pickled (WP)	4	36.5	41.1	69.2	98.2	Base metals and
			5	35.8	43.4	65.4	102.2	weld black-Zr-Nb
			6	28.2	34.2	50.2	85.2	HAZ white
	S-2	As welded	1	35.0	38.8	57.1	77.6	Same as for 2(AW)
			2	35.0	36.5	54.0	68.5	
			3	36.5	42.6	57.8	85.2	
		Welded & Pickled	4	33.5	41.8	59.4	76.1	Same as for 2(WP)
			5	37.3	47.9	73.1	94.4	
			6	39.6	47.2	67.0	85.2	
	S-3	As welded	1	32.7	26.6	41.1	60.9	Same as for 2(AW)
			2	36.5	32.7	42.6	69.2	
			3	39.6	33.5	45.7	75.3	
		Welded & Pickled	4	34.2	44.9	65.4	102.7	Same as for 2(WP)
			5	34.2	38.9	57.8	78.4	
			6	29.7	41.1	58.6	101.2	
	S-4	As welded	1	29.7	25.9	32.7	65.4	Same as for 2(AW)
			2	27.4	28.1	35.8	63.2	
			3	32.7	28.1	41.9	73.0	
		Welded & Pickled	4	33.5	38.0	57.8	92.1	Same as for 2(WP)
			5	42.6	49.5	67.0	99.7	
			6	34.2	47.9	60.9	89.8	

* Welded in welding box evacuated to 0.01μ and back-filled to atmospheric pressure with purified argon.

** Welds solution treated at 880°C for 1 hour after sealing in quartz capsule evacuated to 0.05μ, water quenched, vapour blasted & tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.

AR/An - Zr-Nb as-received
- Zr-2 annealed at 750°C for 1/2 hour in vacuum furnace (0.02μ)

HT/An - Zr-Nb solution treated at 890°C for 15 minutes in Houghton #1450 salt bath using charcoal cover and rectifier for degassing, quenched in water, vapour blasted and tempered for 24 hours in vacuum furnace (0.02μ) at 500°C. Zr-2 annealed at 750°C for 1/2 hour in vacuum furnace (0.02μ).

HAZ - Heat affected zone.

Table 12

KNOOP HARDNESS SURVEY OF Zr-Nb/Zr-Nb and Zr-Nb/Zr-2 WELDS*

(500 g. load - 10.25 mm. obj)

Specimen Identification	Zr-Nb Base Metal		Zr-Nb Heat Affected Zone			Weld Zone								Zr-2 HAZ			Zr-2 Base	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Zr-Nb/Zr-Nb (as-received) Condition A	218	228	270	265	270	265	263	270	275	270	275	270	265					
Zr-Nb/Zr-Nb Heat treated after Welding (Condition A)	304	304	300	293	295	300	295	302	309	298	300	306	298					
Zr-Nb heat treated before welding	295	304	275	281	295	321	333	348	333	340	333	348	333					
Zr-Nb (as-received) Welded to Zr-2	232	238	275	288	304			288	261	261	255			226	226	226	141	141
Zr-Nb (as-received) Welded to Zr-2 & Heat Treated	288	288	295	281	288			281	281	281	268			206	201	188	155	145
Zr-Nb (Heat Treated)	304	295	288	268	281	261	275	275	255	250	261	238	212	131	120	127	124	124

* All readings 0.020 in. apart except in weld zone where readings were made 0.040 in. apart.

Table 13

CHEMICAL COMPOSITION* OF Zr-3 w/o Al -.5 w/o Mo ALLOY

Elements	0.100 in. thick Sheet	0.030 in. Thick Sheets					
		1	2	3	4	5	6
Aluminum	2.63%						
Molybdenum	0.52%						
Oxygen	0.138%	0.150%	0.150%	0.152%	0.148%	0.164%	0.139%
Hydrogen	5						
Nitrogen	30	43	42	69	79	41	40
Iron	1187						
Chromium	164						
Nickel	15						
Manganese	60						
Silicon	350						
Cobalt	< 5						
Tin	25						
Titanium	13						
Copper	$\lesssim$ 15						
Magnesium	$\lesssim$ 5						
Vanadium -	20						
Lead -)	not						
Calcium)	detected						

* Analyses in ppm except for alloying elements which are given in weight per cent. Analyzed by Canadian Westinghouse Ltd. The analysis of the 0.100 in. thick sheet is in close agreement with that of the ingot analysis supplied by Harvey Aluminum Co., Inc.

Table 14

WELDING CONDITIONS FOR Zr-Al-Mo

Welding Atmospheres	Current (Amperes)	Voltage Volts	Work Speed (in./min.)	Shielding Gas Flow Rate, CFH Through Torch	Through Shields
1	50	9	10		
2	33	9	10	30	
3	33	9	5	30	35

1 - Welding box evacuated to 0.01μ and back-filled with argon to atmospheric pressure.

2 - Welding box opened - using standard torch with argon backup*.

3 - Same as in 2 plus leading and trailing shields.

* Using argon back-up at 5 cfh - weld area was purged for 5 minutes before and after welding.

Table 15

GAS ANALYSES AND TENSILE PROPERTIES OF Zr-3 w/o Al-0.5 w/o Mo

SHEET MATERIAL AND WELDS MADE UNDER VARIOUS ATMOSPHERES

Specimen Identification	Welding* Atmosphere	Nitrogen ppm A	Nitrogen ppm K	Oxygen %	Test Temp. (°F)	No. of Specimens	U.T.S. 1000 lb/in.²	Y.S. (.2% offset) 1000 lb./in.²	Elgonation (% in 2")
Sheet #2	-	42		0.150	RT	2	147	135	4.5
					600	3	111	91	6.5
Al 1	1	59	64	0.154	RT	3	153	143	5
					600	3	107	93	6
Al 3	2	95	80	0.154	RT	3	152	139	6
					600	3	106	92	6
Al 5	3	114	87	0.154	RT	3	152	142	4
					600	3	111	96	6

Note: All welded specimens failed at least 3/8" away from weld.

*Welding Atmosphere: 1 - Welding box evacuated to 0.01μ and back-filled with argon to atmospheric pressure.

2 - Welding box opened - using standard torch with argon backup (argon backup to 5 cfh - weld area purged for 5 minutes before and after welding).

3 - Same as in 2 plus leading and trailing shields.

Table 16

KNOOP HARDNESS SURVEY OF Zr-Al-Mo WELDS

(500 g. load - 10.25 mm. obj.)

Weldment Identification	Base Metal		Heat Affected Zone			Weld Zone			
	1	2	3	4	5	6	7	8	9
Al-1 (Welded under Condition 1)	335	329	335	350	362	344	367	372	384
Al-3 (Welded under Condition 2)	306	304	327	367	365	365	374	369	367
Al-5 (Welded under Condition 3)	344	365	348	346	355	348	327	342	350

* All readings 0.020 in. apart except in weld zone where readings were made 0.060 in. apart.

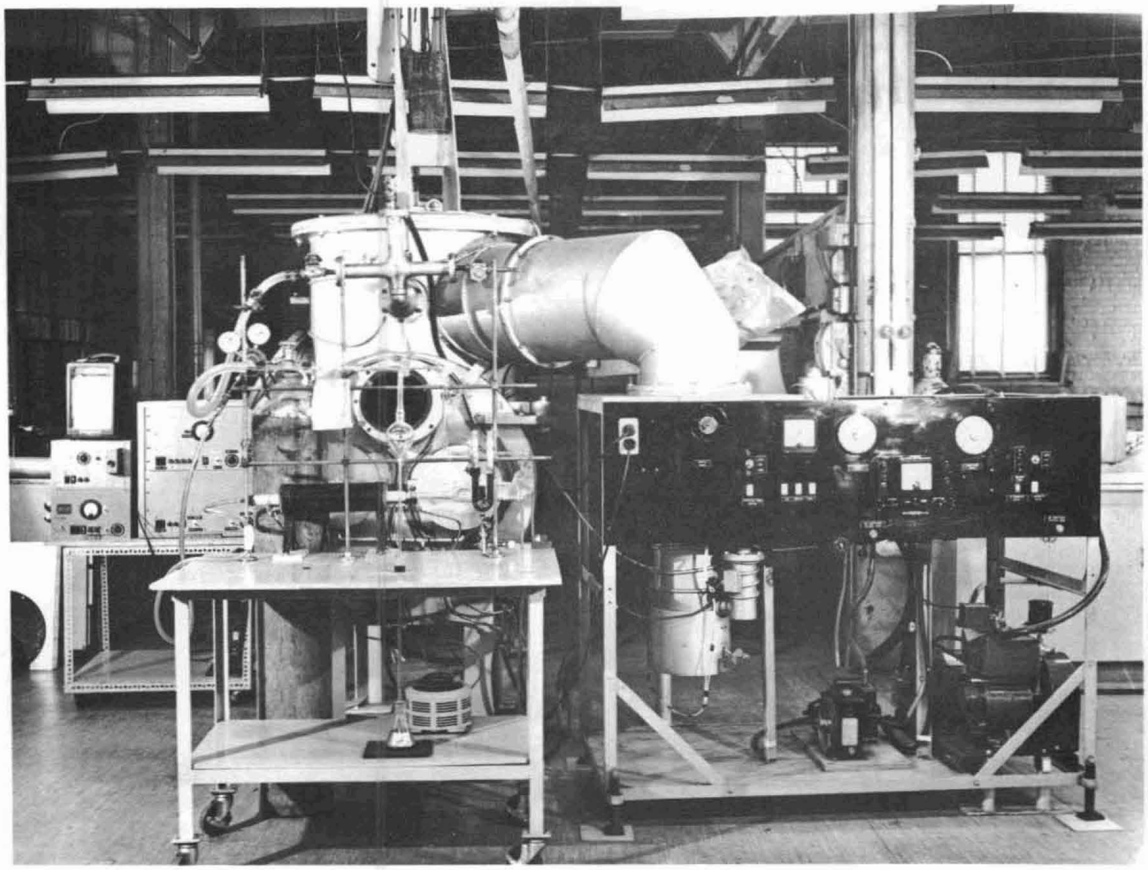


Figure 1. Welding box with vacuum system and purification train.

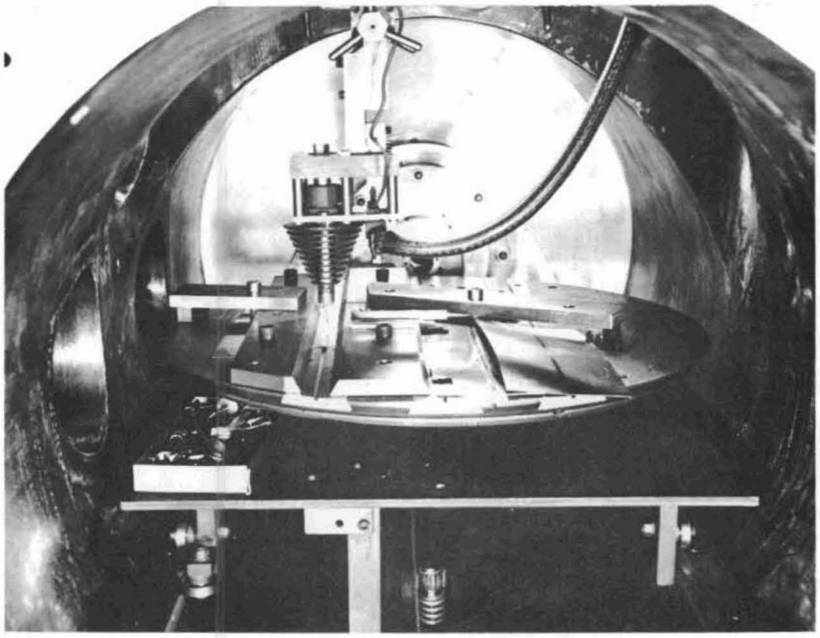


Figure 2. End view of heat-sink torch and clamping fixtures and back-up plates on welding table.

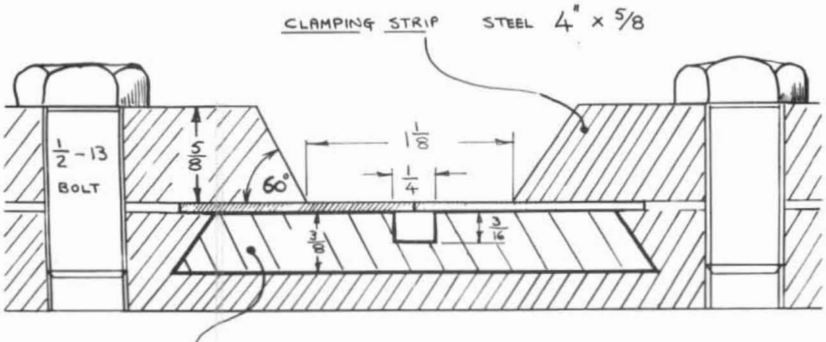


Figure 3. Cross section of chrome plated copper back-up plate and clamping device with sample in position for welding.

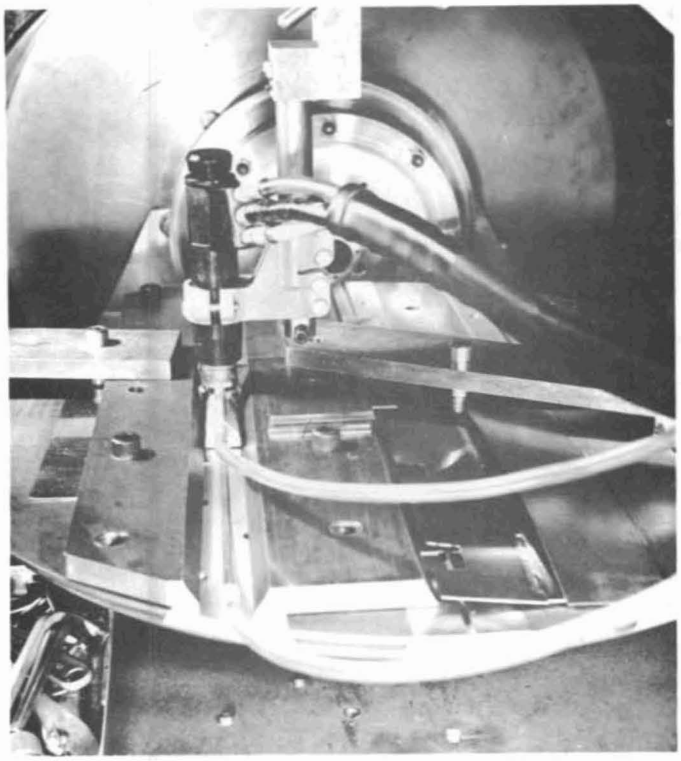


Figure 4. Linde HW-13 torch with leading and trailing shields and argon back-up tube.

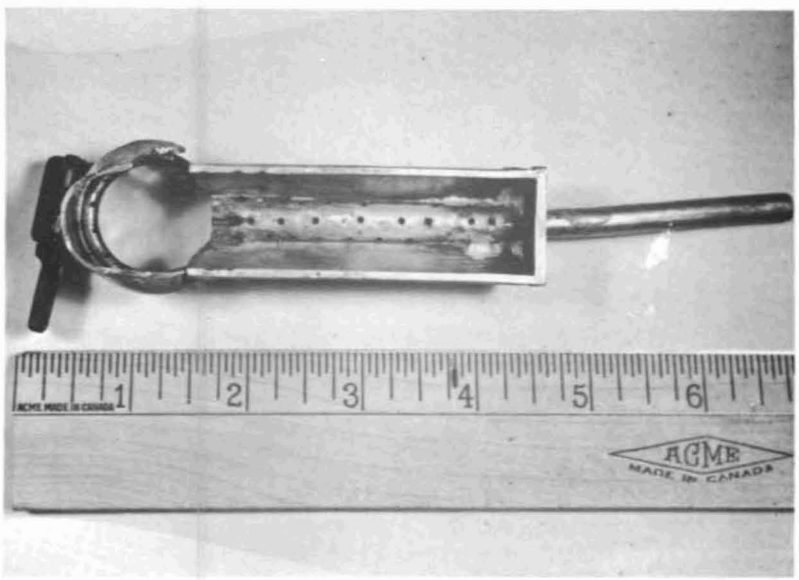
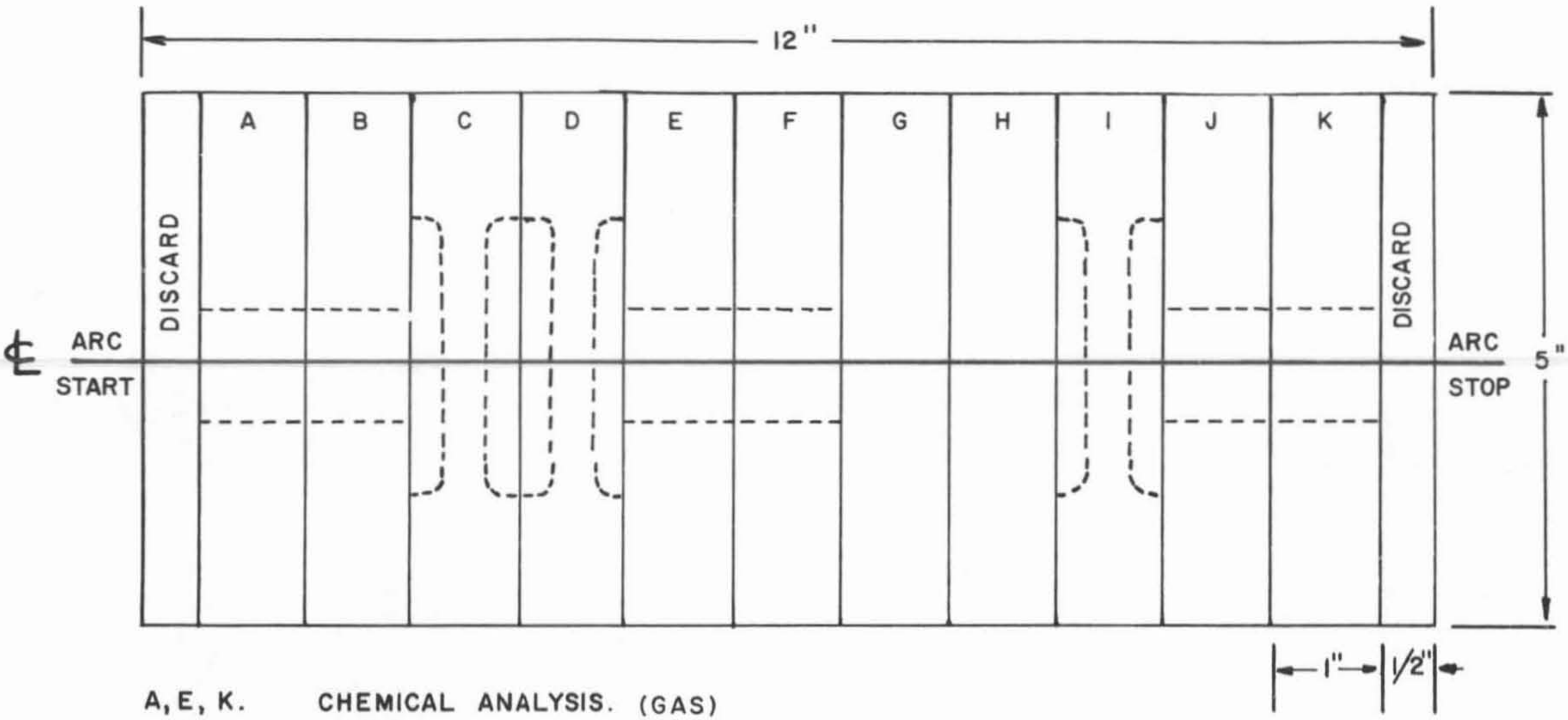


Figure 5. Inner view of leading and trailing shields.



A, E, K. CHEMICAL ANALYSIS. (GAS)
B, F, J. CORROSION.
C, D, I. TENSILE.
G. METALLOGRAPHY.
H. ARCHIVE.

FIGURE. 6. SKETCH SHOWING POSITIONS OF SPECIMENS FOR DESTRUCTIVE TESTING.

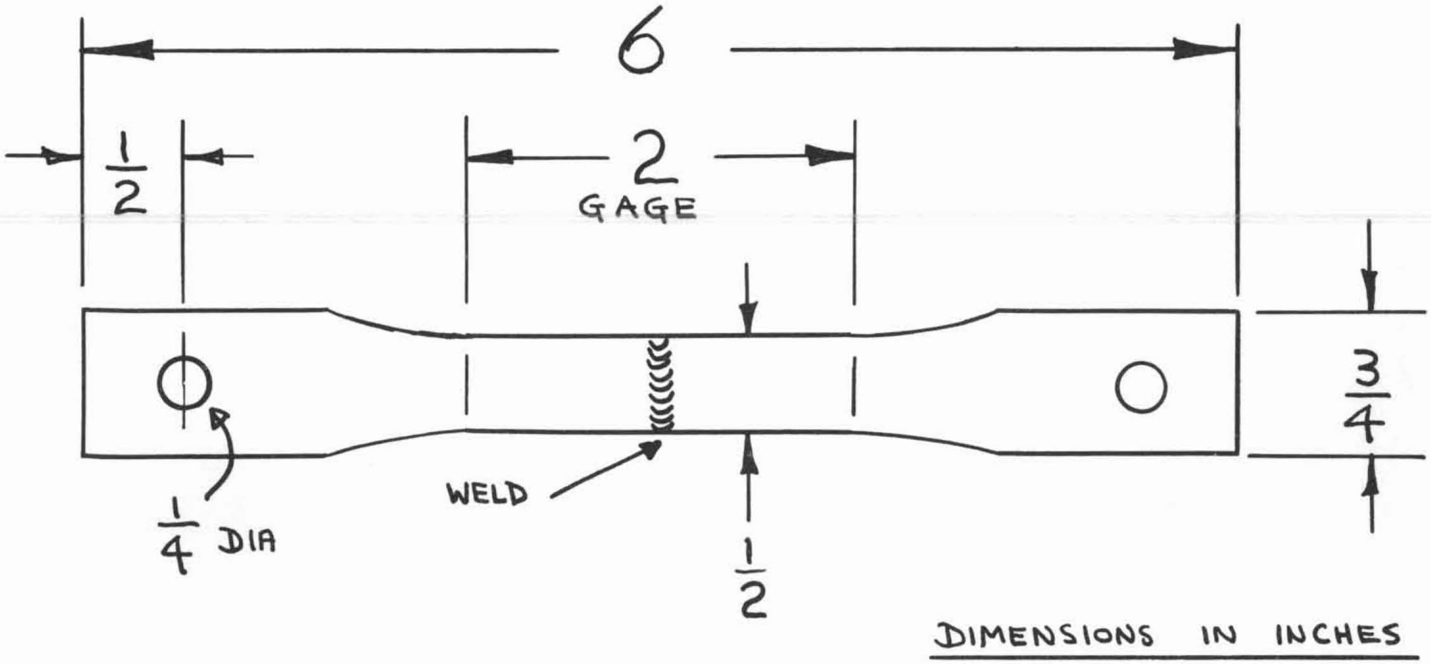


FIGURE. 7. TENSILE SPECIMEN.

Corroded Specimens of Zr-2.5% Nb Sheet Material

Surface Conditions	Remarks
As-Received	Dark grey film
As-Received & Pickled	Black film
As-Received, Heat Treated* and Vapour Blasted	White film
As-Received, Heat Treated Vapour Blasted & Pickled	Black film

* Solution treated at 880°C for 1 hour after sealing in quartz capsule evacuated to 0.05μ, water quenched, vapour blasted and tempered for 24 hours in vacuum furnace (0.02μ) at 500°C.

Corrosion Medium: Distilled and deionized water at 680°F.

Exposure Time: 35 Days

Figure 8. Corroded specimens of Zr-2.5% Nb sheet material.

Welding Atmospheres

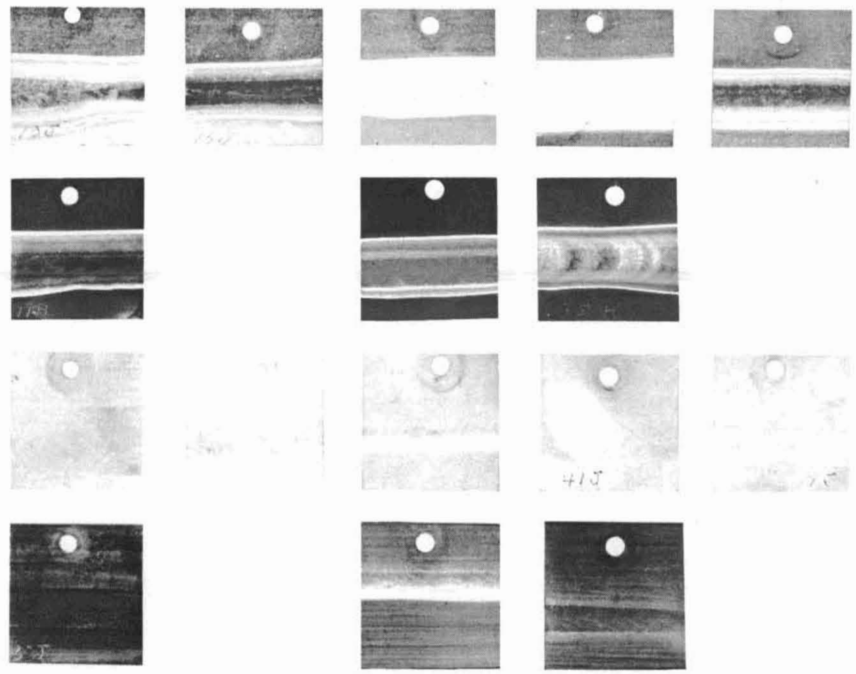
Surface Conditions — A, B, C, D, E

As-Welded

As-Welded and Pickled

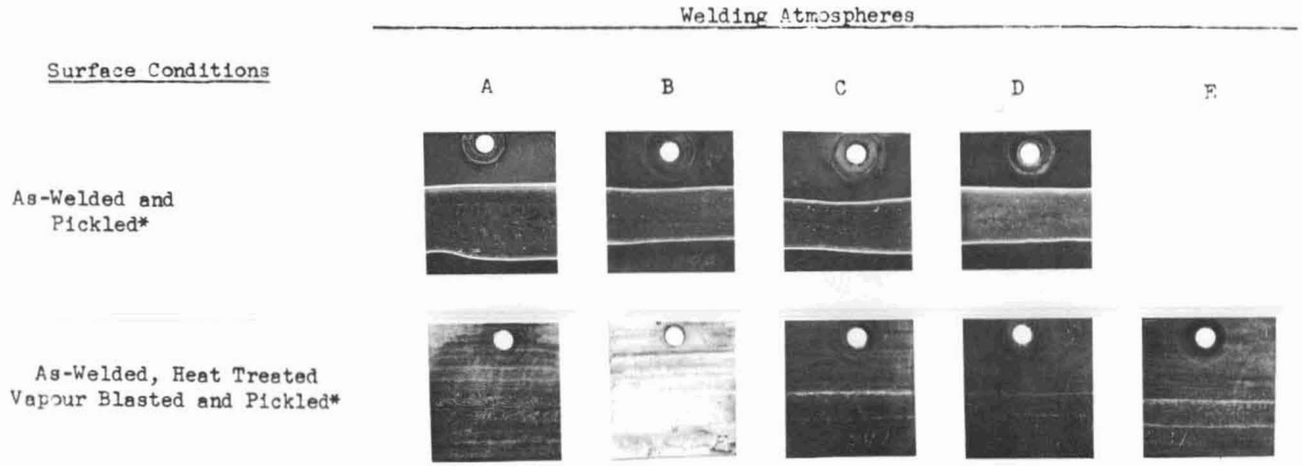
As-Welded, Heat Treated and Vapour Blasted

As-Welded, Heat Treated Vapour Blasted and Pickled



Welding Atmospheres:
A - Welding box evacuated to 0.01μ and back filled with argon to atmospheric pressure.
B - Welding box evacuated to 0.01μ, back filled with air to 2.2μ, then with argon as in A.
C - Welding box opened - using standard torch with argon backup.
D - Same as in C plus trailing shield
E - Same as in C plus leading and trailing shields.

Figure 9. **Welded specimens of Zr-2.5% Nb after 35 days exposure to distilled and deionized water at 680°F.**



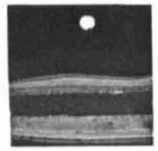
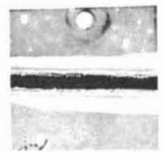
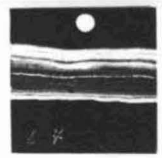
* Vapour blasted after 35 days exposure, then pickled

Welding Atmospheres: A - Welding box evacuated to 0.01μ and back filled with argon to atmospheric pressure.
B - Welding box evacuated to 0.01μ, back filled with air to 2.2μ then with argon as in A.
C - Welding box opened - using standard torch with argon backup.
D - Same as in C plus trailing shield
E - Same as in C plus leading and trailing shields.

Corrosion Medium: Distilled and deionized water at 680°F.

Figure 10. Welded specimens of Zr-2.5% Nb after 70 days exposure to 680°F water.

Heat Treating Conditions*

Surface Conditions	A	B
As-Welded		
As-Welded & Pickled		

Welding Atmosphere: Welding box evacuated to 0.01μ and back filled with argon to atmospheric pressure.

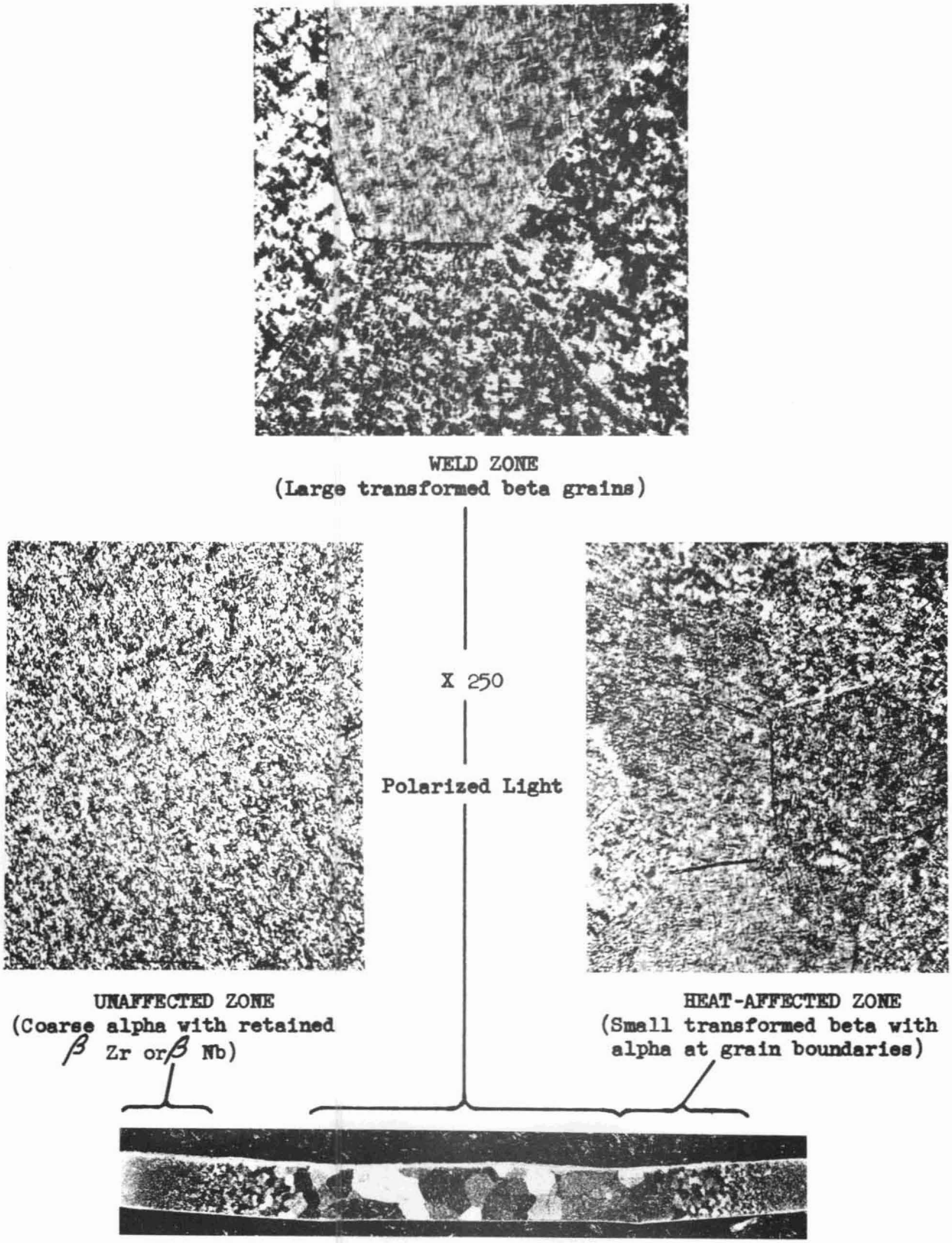
***Heat Treatments:** A - Solution treated at 880°C for 1 hour in quartz capsule evacuated to 0.05μ, quenched in water, vapour blasted and aged for 24 hours in vacuum furnace (0.02μ) at 500°C.

B - Solution treated at 890°C for 15 minutes in Houghton #1450 salt bath using charcoal cover and rectifier for degassing, quenched in water, vapour blasted and aged as in A.

Corrosion Medium: Distilled and deionized water at 680°F.

Exposure Time: 35 Days

Figure 11. Corroded specimens of Zr-2.5% Nb welded after heat treating.



<u>Figure 12</u>. Transverse Section from Welded Zr-2.5 wt % Nb.

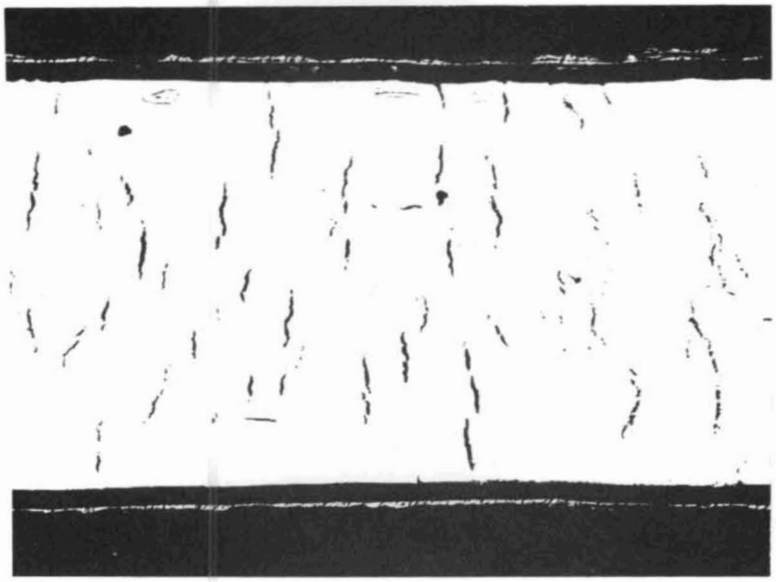


Figure 13. Distribution of hydrides in weld zone of sample (as-welded) exposed to water at 360°C (680°F) for 70 days.
Bright Field X 100

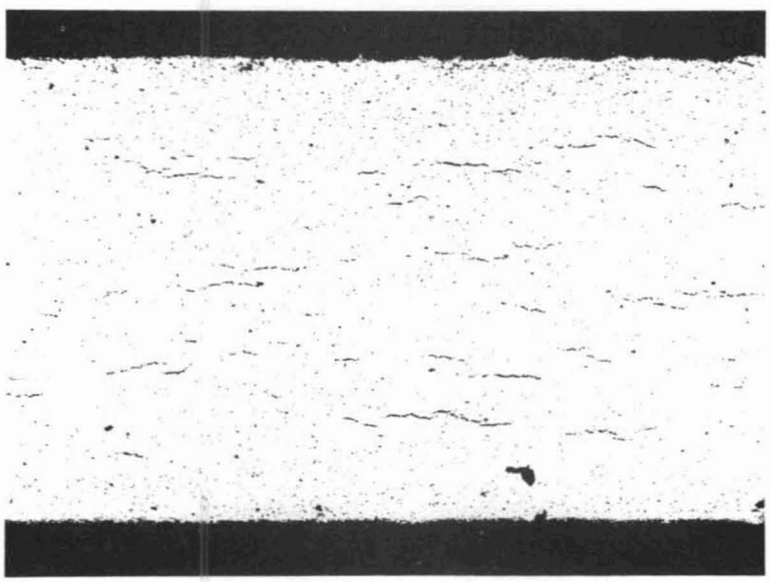
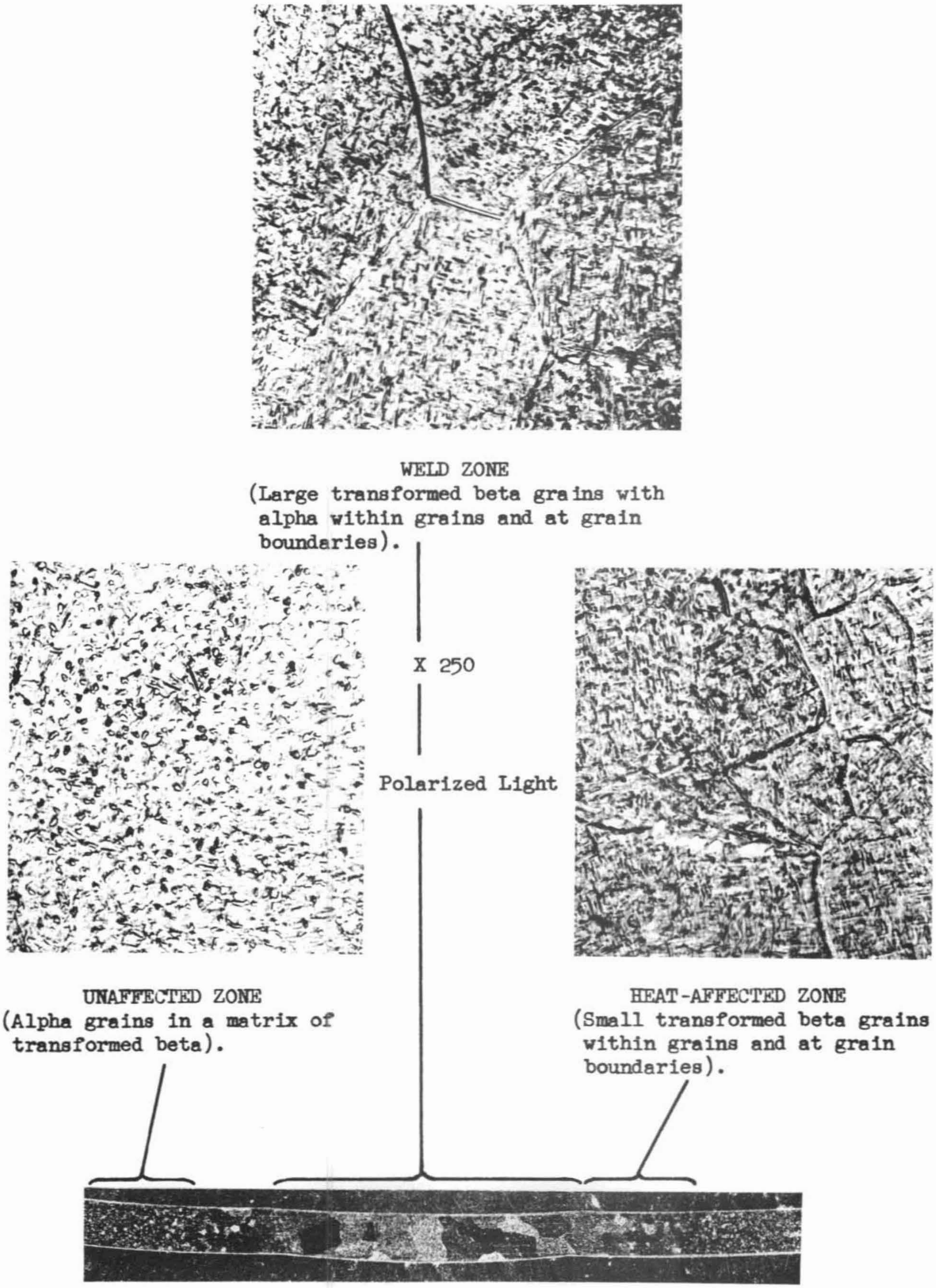


Figure 14. Distribution of hydrides in unaffected base metal as-received. (Same sample as in Fig.13)
Bright Field X 100



WELDED SHEET CROSS SECTION (X 10)

Figure 15. Transverse Section from Welded and Heat Treated Zr-2.5 wt % Nb.

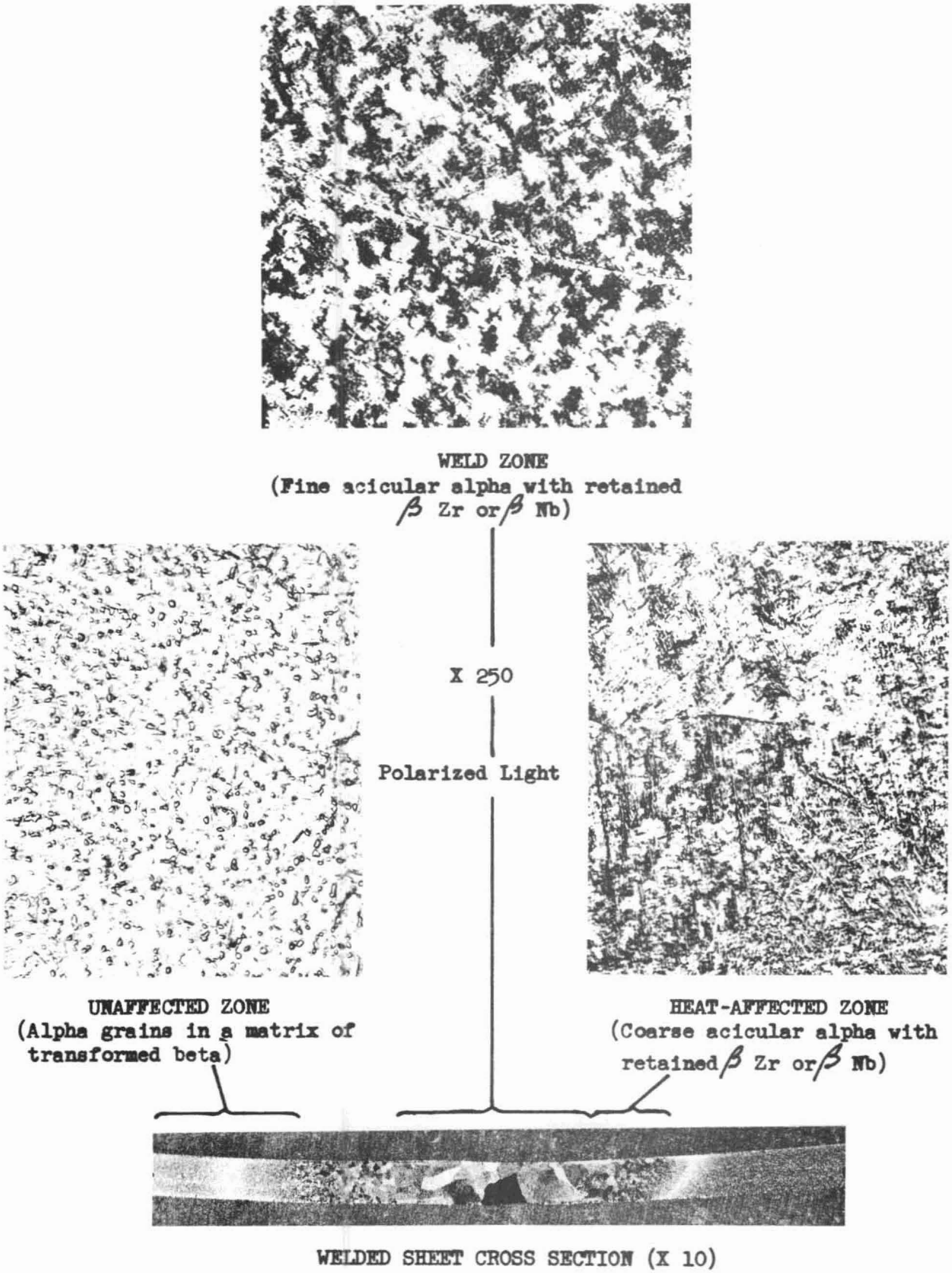


Figure 16. Transverse Section from Heat Treated and Welded Zr-2.5 wt % Nb.

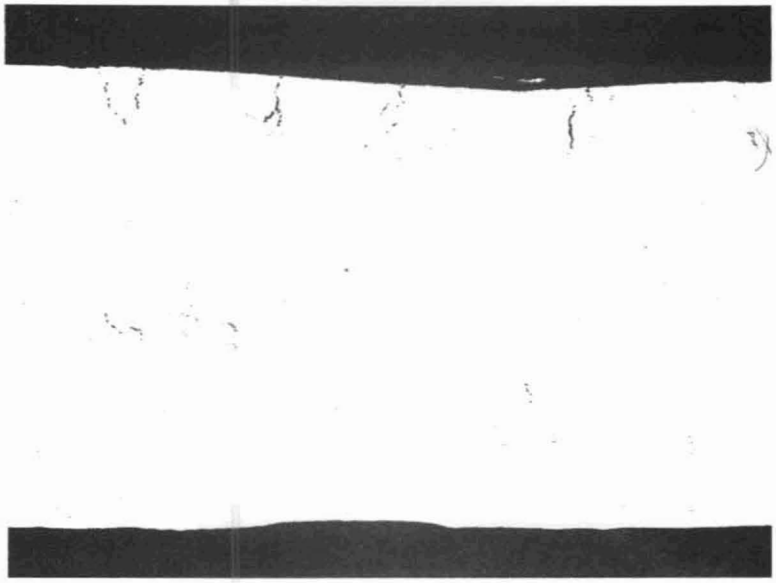


Figure 17. Distribution of hydrides in weld zone of Zr-2/Zr-Nb sample (as-welded) exposed to water at 360°C (680°F) for 35 days.
Bright Field X 100

Figure 18. Distribution of hydrides in Zr-Nb heat-affected zone. (Same sample as in Figure 17).
Bright Field X 100

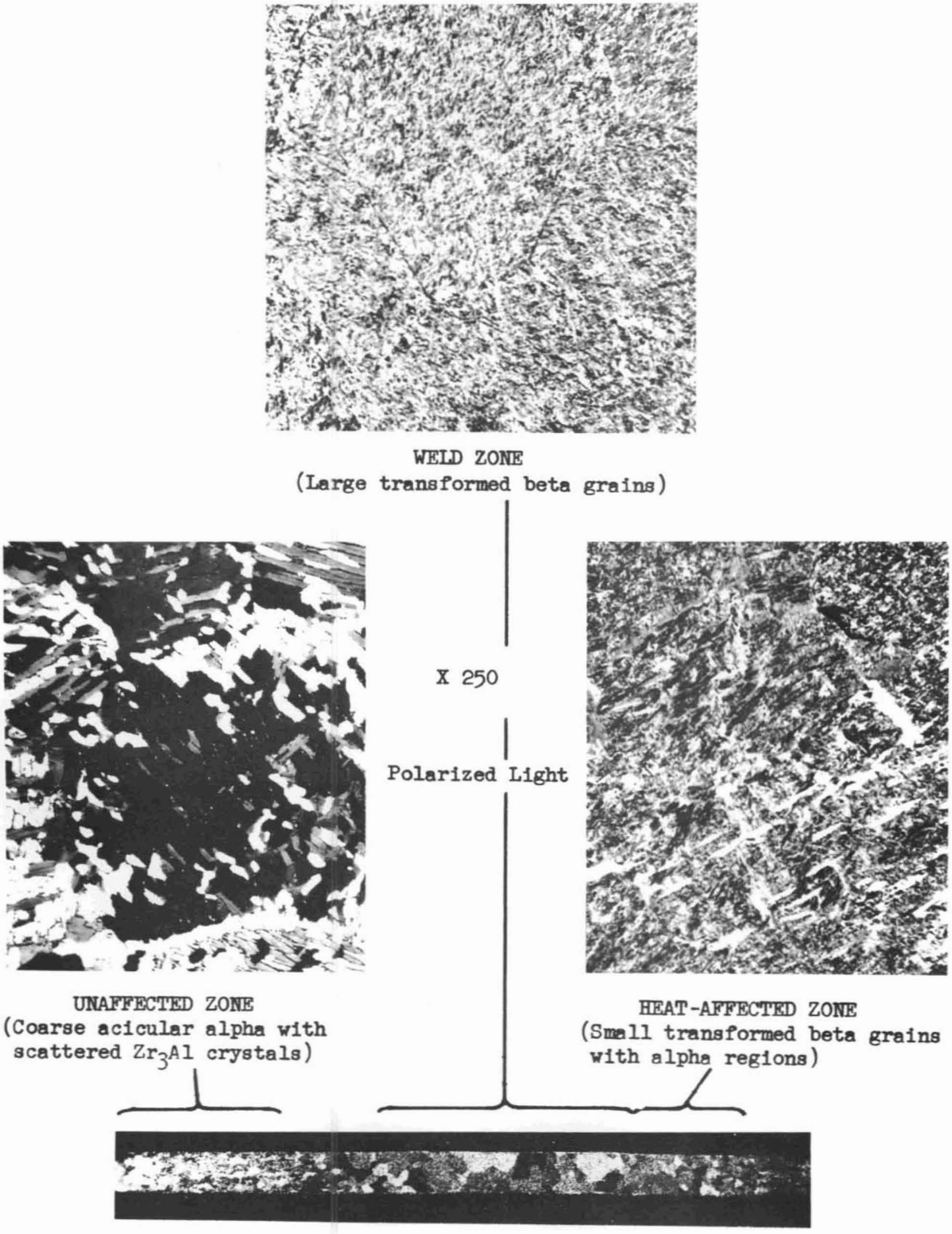


Figure 19. Transverse section from welded Zr-3 wt % Al-.5 % Mo.