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Work performed for
**U.S. DEPARTMENT OF ENERGY**
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# THE EFFECT OF ELECTRON BEAM WELDING ON THE CREEP RUPTURE PROPERTIES OF A Nb-Zr-C ALLOY

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## SUMMARY

Creep rupture tests of electron beam welded PWC-11 sheet were conducted at 1350 K. Full penetration, single pass welds were oriented transverse to the testing direction in 1 mm thick sheet. With this orientation, stress was imposed equally on the base metal, weld metal, and heat-affected zone. Tests were conducted in both the post-weld annealed and aged conditions. Unwelded specimens with similar heat treatments were tested for comparative purposes. It was found that the weld region is stronger than the base metal for both the annealed and aged conditions and that the PWC-11 material is stronger in the annealed condition than in the aged condition.

## INTRODUCTION

Advanced space power system requirements that include a service life of 7 years at 1350 K dictate the use of refractory metal systems (ref. 1). Nb-1Zr is a candidate material. However, Nb-1Zr was not developed for structural applications such as pressure vessels, heat pipes, and piping where high creep strength is required for temperatures in excess of 1100 K. In the mid 1960's Pratt and Whitney Aircraft Division of United Aircraft Corporation developed a stronger Nb-1Zr alloy by increasing the carbon content by two orders of magnitude to about 0.1 wt %. The Nb-1Zr-0.1C alloy was designated PWC-11. Long term overaging of PWC-11 in the 1175 to 1375 K temperature range has been of concern because of potential weakening effects (ref. 2).

The weldability of PWC-11 appears to be similar to that of the Nb-1Zr alloy in that it is readily weldable by fusion welding processes. Titran et al. (ref. 3) reported that sound welds were produced in PWC-11 by the electron beam welding (EBW) process and that test specimens with transverse EB welds were creep tested to 1 percent strain at 1350 K. In these tests, however, the relative strength of the base metal, heat-affected zone and fusion zone was not determined. The study to be reported on here supplements the work done by Titran et al. (ref. 3) by creep rupture testing EB welded PWC-11 specimens at 1350 K. The weak link was determined by testing specimens with a transverse EB weld. Unwelded base metal specimens were creep rupture tested to obtain base line data. The creep rupture tests were conducted in two conditions, annealed and aged. Microstructural analysis and microhardness determinations were used in evaluating the test results.

## MATERIAL AND PROCEDURE

A chemical analysis of the as received PWC-11 material was conducted at NASA Lewis with the following results:

| | |
|---|---|
| Zirconium . . . . . . . . . . . | 0.90 wt % |
| Carbon . . . . . . . . . . . . . | 0.063 wt % |
| Oxygen . . . . . . . . . . . . . | 80 ppm wt |
| Nitrogen . . . . . . . . . . . . | 53 ppm wt |
| Hydrogen . . . . . . . . . . . . | 11 ppm wt |
| Niobium . . . . . . . . . . . . | Balance |

This material was received from the Oak Ridge National Laboratory in the form of 1 mm thick tensile creep test specimens with a 6.4 mm wide by 25 mm long gauge section and with the longitudinal axis parallel to the rolling direction. All of the specimens were degreased, rinsed in distilled water and then in alcohol. Next, each specimen was wrapped in cleaned tantalum foil and then given the prescribed double anneal of 1 hr at 1775 K, furnace cooled, followed by 2 hr at 1475 K - both in a vacuum of $10^{-6}$ Pa.

Electron beam welding (EBW) of the PWC-11 creep rupture test specimens was conducted in accordance with the procedures outlined by Moore, Moorhead, and Bowles (ref. 4). Nb-1Zr starting and run-off tabs were positioned at the sides of the gauge section of the creep rupture test specimens and a single pass, full penetration transverse weld was made as shown in figure 1. Molybdenum and tungsten hold-down fixtures were used. The welding parameters were:

| | |
|---|---|
| Vacuum . . . . . . . . . . . . . . . . . | $9\times10^{-3}$ Pa |
| Voltage . . . . . . . . . . . . . . . . | 120 KV |
| Amperage . . . . . . . . . . . . . . . . . | 11 mA |
| Travel Speed . . . . . . . . . . . . | 810 mm/min |
| Gun-to-work distance . . . . . . . . . . . | 76 mm |

These welding parameters were selected in order to produce a rather wide fusion zone for an EB weld. This was done so that the heat input and weld region microstructure would be intermediate between a very narrow EB weld and a much wider gas tungsten arc weld.

Prior to creep testing or heat treating of the welded specimens, the Nb-1Zr starting and run-off tabs were removed. The face and root surfaces were ground flat and approximately 25 μm of additional material was removed from both the face and root surfaces.

Creep rupture testing was conducted in split stainless steel water cooled double walled chambers which have been described previously (ref. 5). The chambers attain a vacuum of $10^{-5}$ Pa using liquid nitrogen continuous cold trapping of an oil diffusion pump. The specimen is heated by radiation from a tubular tantalum heating element. Temperature was measured and controlled with one of three Pt-Pt13Rh thermocouples tied to the specimen surface. Specimens were wrapped with 25 μm tantalum foil. Creep stress loads were applied using a lever-arm system through a sliding o-ring seal.

The creep rupture tests were conducted at 1350 K on unwelded and welded specimens in two basic conditions:

Annealed

1. Unwelded material, double annealed 1 hr at 1755 K followed by 2 hr at 1475 K (DA)
2. Welded DA material, post weld heat treated 1 hr at 1475 K (W+HT)

Aged

1. Unwelded material DA followed by 1000 hr aging at 1350 K (DA+A)
2. Welded DA material, post weld heat treated (HT), followed by 1000 hr aging at 1350 K (W+HT+A)

The 1000 hr-1350 K aging treatment was used to simulate initial exposure of the PWC-11 alloy under no stress conditions such as might be encountered in space power component applications. This treatment was used to address the concern of overaging in this alloy. Longitudinal sections were obtained from each tested specimen for metallographic examination of the base metal, weld region, and fracture edge. Vickers microhardness determinations were made using a 50 g load with a 15 sec dwell time.

## RESULTS AND DISCUSSION

Creep rupture and microhardness test data are shown in table I. These data will be discussed following a presentation of the microstructures of the base metal, weld joint, and fracture edge. It should be noted at the outset, however, that all creep rupture specimens with transverse EB welds failed in unaffected base metal and not in the vicinity of the weld or heat-affected zone. A typical creep rupture failure with necking in the base metal on both sides of the weld is shown in figure 2.

### Microstructure

Typical microstructures of the base metal, weld, and fracture regions are shown in figure 3. The EB weld fusion zone (fig. 3(a)) was sound with an average grain size of 105 μm diameter. The coarsened heat affected zone grains averaged 40 μm in diameter (fig. 3(b)). In the unaffected base metal, the blocky grains, elongated in the rolling direction, averaged 21 μm in diameter (fig. 3(c)). In view of this difference in grain size, the fusion zone and heat-affected zone would be expected to have inherently higher creep strength than the unaffected base metal.

Weld width at the face was about 2 mm and 0.6 mm at the root of the weld (fig. 3(a)). A narrower EB weld could have been produced. But the objective in this study was to produce a relatively wide fusion zone. The coarse grain heat-affected zone varied from about 0.2 to 0.4 mm in width. The thin white diagonal band shown in figures 3(a) and (b) delineates the weld interface. This white band is an area of plane front solidification that took place prior to the cellular solidification in the fusion zone shown to the right of the white band in figure 3(b). Note that epitaxial grain growth across the weld interface into the weld fusion zone has taken place in this alloy. At the fracture tip after a 20 percent elongation, the blocky base metal grains (fig. 3(c)) were highly elongated and narrow as shown in figure 3(d). Examination of creep rupture tested base metal at magnifications to 1000X revealed the presence of some very small particles and extremely fine intragranular precipitation which was not resolvable (not shown). No significant differences could be discerned between the annealed and the aged microstructures.

## Creep Rupture Properties

Creep rupture data from table I are plotted in figure 4. Test specimens with transverse EB welds (numbers 1, 3, 6, 7, and 9) all failed away from the weld. This shows that EB welding did not degrade the creep rupture properties of PWC-11 and that the strength of the weld and heat-affected zone is greater than that of the unaffected base metal. Because of the failure location in the welded specimens, these data points represent base metal properties. This indeed proved to be the case because data points for the unwelded specimens (numbers 2, 4, 5, and 8) fall on the same curves. The upper curve in figure 4 shows that the PWC-11 base metal is stronger in the annealed condition than in the aged condition (lower curve). Aging prior to creep rupture testing produces a weakening effect.

## Microhardness

Consistent relationships were established between creep rupture strength and room temperature microhardness. Vickers hardness values for the base metal (from table I), are shown next to the creep rupture data points in figure 4. Note that hardness values are higher for the stronger, annealed material. Since grain size in annealed and aged material is the same, the intragranular carbide precipitate that produces the higher microhardness appears to be associated with superior creep strength. This condition indicates that the intragranular matrix carbide precipitate is the important factor in the creep resistance of PWC-11 material. The hardness decreases with time to rupture for both annealed and aged materials may indicate a loss of coherency of the precipitate with the matrix and/or possible agglomeration of the precipitate.

A comparison of the hardness of the weld region with the base metal and base metal fractures tip is shown in figure 5. These two creep rupture test specimens (numbers 3 and 9) had rupture lives of similar duration (table I). One specimen was tested in the annealed condition and the other in the annealed and aged condition. The following observations can be made:

(1) Base metal hardness of the stronger annealed specimen number 3 is greater than that of number 9, the aged specimen. The heat-affected zone and the fusion zone are also at higher hardness levels for the annealed specimen which also indicates higher strength in all regions of the annealed weldment compared to the aged weldment. Thus, possibly more coherency and more extensive intragranular precipitation is believed to be present for the annealed material.

(2) For both the annealed and aged weldments, the heat-affected zone is considerably harder and stronger than the base metal. This is also believed to be due to an intragranular precipitation effect.

(3) For the annealed specimen the fusion zone is even harder than the heat-affected zone and thus presumably the strongest region. The fusion zone

hardness of the aged specimen is somewhat less than that of the heat-affected zone, but still higher than that of the base metal. Precipitation effects are believed to produce these patterns.

(4) The area of highest hardness was located in the unaffected base metal within about 125 μm of the fracture tip. In this highly worked region the base metal grains were very long and narrow as shown in figure 3(d). The hardness values and the microstructure at the fracture tip were similar for specimens tested in both the annealed and the aged starting conditions.

## CONCLUSIONS

On the basis of creep rupture tests conducted at 1350 K on unwelded PWC-11 base metal specimens and on specimens with transverse electron beam welds, the following conclusions are drawn:

1. Welding does not degrade the creep rupture properties of the PWC-11 alloy.

2. The weld region is stronger than the base metal for specimens tested in both the post weld heat treated (1 hr at 1475 K) and aged (1000 hr at 1350 K) conditions.

3. Base metal creep rupture strength is significantly higher in the annealed condition than it is in the aged condition. Intragranular precipitates, which are reflected by higher room temperature microhardness in annealed material, are responsible for the improved creep strength.

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5. R.W. Hall, and R.H. Titran, "Creep Properties of Columbium Alloys in Very High Vacuum," Refractory Metals and Alloys III, ed., R.I. Jaffee, (New York, NY: Gordon and Breach, 1966), 885-900.

TABLE I. - 1350 K CREEP RUPTURE TEST RESULTS AND ROOM TEMPERATURE MICROHARDNESS OF TESTED SPECIMENS

[All failures occurred in the unaffected base metal. DA - Unwelded double annealed 1 hr at 1755 K plus 2 hr at 1475 K. W+HT - Welded plus post weld heat treated 1 hr at 1475 K. DA+A - Unwelded, double annealed plus aged 1000 hr at 1350 K. W+HT+A - Welded plus post weld heat treated 1 hr at 1475 K + aged 1000 hr at 1475 K.]

| Specimen test number | Condition prior to creep rupture test | Stress, MPa | Hours to rupture | Elong., percent | Vickers hardness,[a] 50 gram load | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | Base metal | Heat-affected zone | Fusion zone | At fracture tip |
| 1 | W+HT | 140 | 0.2 | 22 | 92(13) | 97(4) | 104(4) | 101(1) |
| 2 | DA | 110 | 80.0 | 22 | 88(7) | ----- | ------ | 93(1) |
| 3 | W+HT | 110 | 86.3 | 19 | 87(21) | 93(2) | 94(5) | 98(1) |
| 4 | DA | 100 | 625 | 17 | 85(8) | ----- | ------ | 102(1) |
| 5 | DA+A | 140 | 0.2 | 27 | 85(9) | ----- | ------ | 98(1) |
| 6 | W+HT+A | 90 | 2.6 | 38 | 83(17) | 87(2) | 86(5) | 97(1) |
| 7 | W+HT+A | 85 | 22.6 | 25 | 84(9) | 92(4) | 88(4) | 105(1) |
| 8 | DA+A | 90 | 40.1 | 45 | 80(10) | ----- | ------ | 100(1) |
| 9 | W+HT+A | 75 | 134 | 26 | 79(10) | 89(4) | 84(3) | 98(1) |

[a]Number of hardness readings in parenthesis.

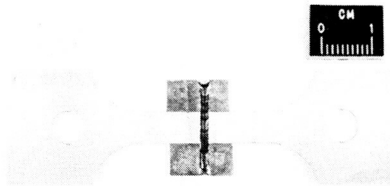



FIGURE 1. - TRANSVERSE ELECTRON BEAM WELD IN 1 MM THICK PWC-11 TEST SPECIMEN WITH NB-1ZR STARTING AND RUN OFF TABS.

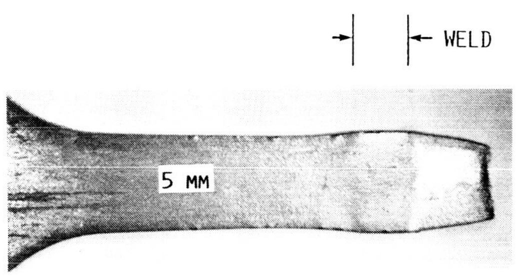
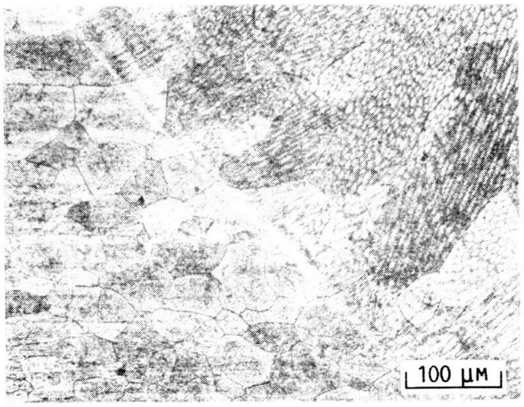


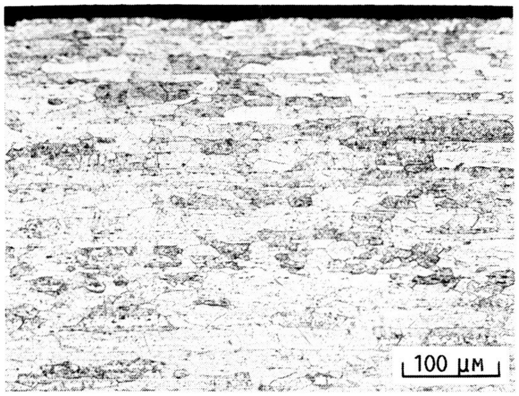

FIGURE 2. - TYPICAL BASE METAL CREEP RUPTURE FAILURE FOR PWC-11 SPECIMEN WITH TRANSVERSE ELECTRON BEAM WELD.


(A) Sound, large-grained weld joint.

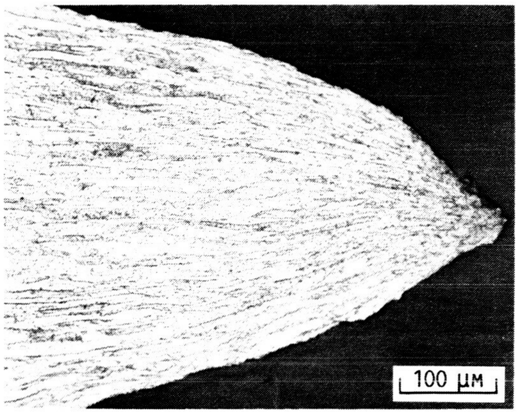



(B) Epitaxial growth of weld metal grains across the weld interface.

Figure 3. - Longitudinal sections of PWC-11 creep rupture Specimen No. 9 tested in the welded plus heat treated plus aged condition. Failure occurred in 134 hours at 1350K under 75 MPa with 26% elongation. Etchant: 10 parts $HNO_3$-15 parts HF-10 parts $H_2O$.




(C) BASE METAL AWAY FROM WELD SHOWING BLOCKY GRAINS ELONGATED IN THE ROLLING DIRECTION OF THE SHEET.




(D) HIGHLY ELONGATED GRAINS AT FRACTURE TIP IN BASE METAL.

FIGURE 3. - CONCLUDED.

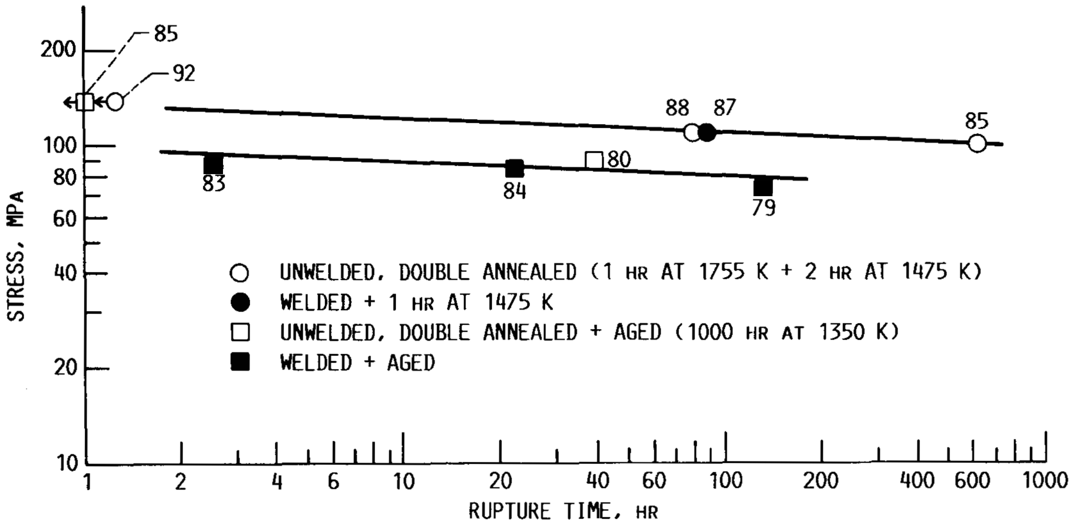



FIGURE 4. - 1350 K CREEP RUPTURE STRENGTH OF UNWELDED PWC-11 BASE METAL SPECIMENS AND SPECIMENS WITH TRANSVERSE ELECTRON BEAM WELDS. ROOM TEMPERATURE VICKERS MICROHARDNESS VALUES OF UNAFFECTED BASE METAL DETERMINED AFTER CREEP TESTING ARE SHOWN FOR EACH CREEP RUPTURE DATA POINT.

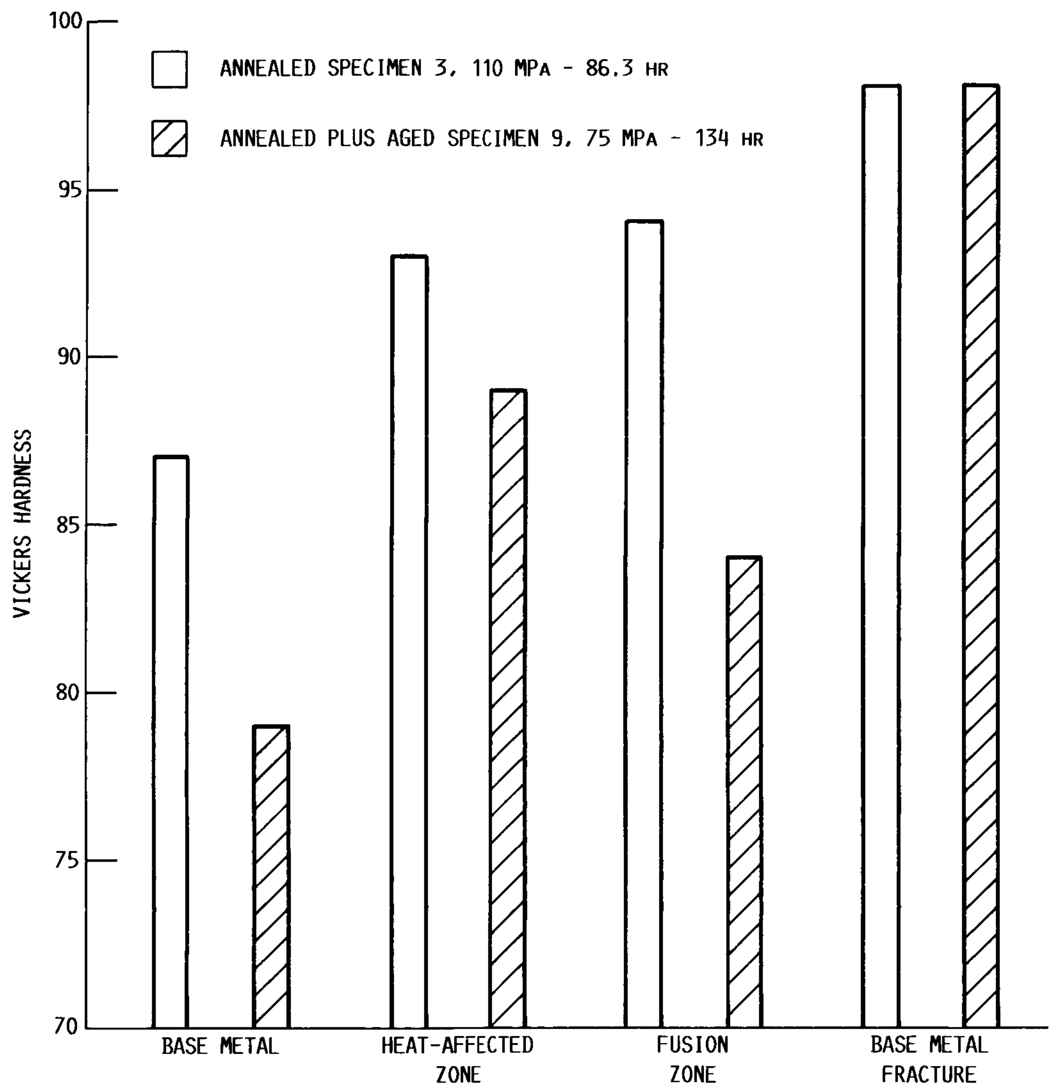



Figure 5. - Typical hardness of an annealed and an aged PWC-11 weld specimen after similar creep rupture lives at 1350 K.

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16. Abstract

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