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CREEP PROPERTIES OF PWC-11 BASE METAL AND WELDMENTS AS AFFECTED BY HEAT TREATMENT

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SUMMARY

In a preliminary study using single specimens for each condition, PWC-11 (a niobium-base alloy with a nominal composition of Nb-1%Zr-0.1%C) was creep tested at 1350 K and 40 MPa. Base metal specimens and specimens with transverse electrode beam welds were tested with and without a 1000 hr, 1350 K aging treatment prior to testing. In the annealed condition (1 hr at 1755 K + 2 hr at 1475 K), the base metal exhibited superior creep strength compared to the aged condition, reaching 1 percent strain in 3480 hr. A 1000 hr, 1350 K aging treatment prior to creep testing had a severe detrimental effect on creep strength of the base metal and transverse electron beam weldments, reducing the time to attain 1 percent strain by an order of magnitude. Extrapolated temperature compensated creep rates indicate that the present heat of PWC-11 may be four times as creep resistant as similarly tested Nb-1%Zr. The extrapolated stress to achieve 1 percent creep strain in 7 yr at 1350 K is 0.6 MPa for annealed Nb-1%Zr and 2.8 MPa for annealed and aged PWC-11 base metal with and without a transverse electron beam weld.

INTRODUCTION

Advanced space power system requirements include a nominal service life of 7 yr at 1350 K which dictates the use of refractory metal systems (ref. 1). Historically, Nb-1%Zr has been suggested for use in space power conversion applications where resistance to liquid alkali metal corrosion at temperatures near 1000 K was the primary concern (refs. 2 and 3). Nb-1%Zr was not, however, developed for applications which required high creep strength for very long times at temperatures in excess of 1100 K. Pratt and Whitney Aircraft Corp., (ref. 4) in the mid-1960's, experimented with carbon additions to Nb-1%Zr and thermomechanical processing of the new compositions in an attempt to raise the tensile creep strength and to increase the service temperature. One of the most promising alloys produced, PWC-11, has a nominal composition of Nb-1%Zr-0.1%C.

Extensive creep testing of this alloy at temperatures and conditions used for space power systems has not been done. Overaging of this alloy in the 1175 to 1375 K temperature range has been of concern because of potential weakening effects (refs. 5 to 8). Use of the alloy in the welded condition will also be required and thus warrants investigation. This preliminary study was designed to provide creep data at 1350 K on PWC-11 base material in the annealed and in the aged conditions along with a comparison test on the Nb-1%Zr alloy. In addition, specimens with transverse electron beam welds were tested in creep at 1350 K in the annealed and in the aged conditions. Because of a shortage of material at the time this study was initiated, only single tests were conducted for each type of specimen and processing condition.

MATERIAL

The material used in this study was received from the Oak Ridge National Laboratory in the only form available: 1 mm thick tensile creep test specimens. A detailed processing history was not available for this material. However, it is believed that this heat of PWC-11 was from one of the first attempts at a manufacturing scale-up from laboratory development. Initial arc melting was performed using spectrographically pure graphite sticks spot welded to the sides of a Nb-1%Zr electrode. Only one melt was performed. The single arc cast billets, 300 mm in diameter, were extruded (slowly) in one pass at temperatures in the 1350 to 1450 K range.

Chemical analysis of the as-rolled PWC-11 material and Nb-1%Zr material that was used for comparison tests was performed at Lewis with the following results:

	PWC-11	Nb-1%Zr
Zirconium	0.90 wt %	1.10 wt %
Carbon	630 ppm wt	16 ppm wt
Oxygen	80 ppm wt	170 ppm wt
Nitrogen	53 ppm wt	40 ppm wt
Hydrogen	11 ppm wt	0.5 ppm wt
Niobium	balance	balance

Although the extruded PWC-11 material met the chemistry specifications (600 to 1000 wt ppm), it was not homogeneous and did not possess the manufacturer's recommended Nb₂C and NbC precipitate composition or morphology. The desired microstructure is a very uniform distribution in the grain boundary and matrix of very small precipitates of (Nb,Zr)₂C and (Nb,Zr)C ranging in size from tens to hundreds of nanometers in diameter (ref. 9). The actual microstructure of the as-rolled material is pictured in figure 1(a), consisting of very large precipitate particles (1 to 5 μ m) dispersed throughout an elongated grain structure.

Subsequent processing of this "scale-up" material into sheet for determination of mechanical properties in support of space power systems is believed to have yielded a material which does not reflect the optimum conditioning of the Nb-Zr-C alloy. Thus, the creep properties of PWC-11 reported in this study may be less than optimum. The Nb-1%Zr alloy was processed by normal practices historically established by the refractory metal industry. The creep properties of this particular heat of Nb-1%Zr are expected to be typical given the reported heat treatment and composition.

EXPERIMENTAL PROCEDURE

The tensile creep specimen design is shown in figure 2. All of the specimens were degreased, rinsed in distilled water and then in alcohol. Next, each PWC-11 specimen was wrapped in cleaned tantalum foil and double annealed (DA) in a vacuum of 10⁻⁶ Pa (1755 K for 1 hr, furnace cooled, followed by 1475 K for 2 hr). Figure 1 compares (a) the microstructure in the as-rolled condition, and (b) the microstructure in the DA condition. The DA material had a mixture of elongated and equiaxed grains with an average grain size of approximately 25 μ m. Numerous shapes and sizes of particles were noticeable

in the microstructure. The morphology range from massive 5 μm particles delineating grain boundaries to submicron accicular particles which appeared to be oriented on as yet undetermined slip planes. The majority of particles observed are believed to be carbides resulting from the initial solidification which were not dissolved during subsequent processing to sheet or after DA.

The electron beam welding (EBW) process was used to produce weld specimens by making a weld pass on finish machined and double annealed PWC-11 creep test specimens. EBW was conducted in accordance with the procedures outlined by Moore, et al. (ref. 10). Nb-1%Zr starting and run-off tabs were positioned on the sides of the gauge section of the PWC-11 creep test specimens. A single pass, full penetration transverse EB weld was made. Molybdenum and tungsten hold-down fixtures were used to prevent any contamination of the weld. The welding parameters were:

Voltage	120 kV
Amperage	10 mA
Weld rate	810 mm/min
Gun-to-work distance	76 mm

Figure 2 shows a sketch of the weld specimen-tab arrangement and a typical weld. The macro- and microstructure of a welded specimen in the as-welded condition are shown in figure 3. The average width of the fusion zone, the area which became molten during welding, ranged from 1.6 mm at the face (top) of the weld to 1.2 mm at the root (bottom) of the weld (fig. 3(a)). The total fusion plus heat affected zone (HAZ) width was about 2 mm at the face and about 1.6 mm at the root. Thus, the weld and HAZ were a small part of the 25 mm long gauge length (fig. 2). The base metal microstructure shown in figure 3(b) is in the same DA condition represented in figure 1(b) for a different specimen. Although the large grain boundary particles are apparent in both photomicrographs, the submicron accicular precipitate is more evident in figure 1(b). The primary particles which dissolved during welding have precipitated as (1) a grain boundary network and (2) a subgrain network in the matrix consisting of "walls" of precipitate clusters surrounding areas of relatively lower precipitate density (fig. 3(c)). Much of the intragranular precipitate is in the form of short rods.

After the weld 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. One EB welded specimen was tested in each of the following conditions:

AW	As-welded
W+HT	Welded plus post-weld heat treated for 1 hr at 1475 K
W+A	Welded plus aged for 1000 hr at 1350 K
W+HT+A	Welded, post-weld heat treated, and aged

Prior to elevated temperature service, post-weld heat treatment is recommended. Thus, the W+HT condition is preferred over the AW condition. The 1000 hr, 1350 K age was chosen to partially simulate the time a fuel cladding material may be at its service temperature, free of stress, before any gas build-up or swelling (due to the fission reaction) begins. Specimen

weight changes associated with the heat treatments generally amounted to only a few milligrams, equivalent to changes in the interstitial content of the specimen of less than 10 ppm.

Figure 4 shows microstructures for the W+HT+A condition prior to creep testing. Although the samples were slightly overetched, the matrix structure retained some of the fine particulates. The unaffected base metal area (fig. 4(a)) shows little evidence of the grain boundary particles present in the DA condition (fig. 4(b)). The weld fusion zone (fig. 4(b)) has a continuous phase in the grain boundaries and a cell-like structure inside the grains. The grain size in neither the base metal nor in the weld was affected by the heat treatments.

Constant-load creep tests were conducted in the internally loaded, high vacuum creep chambers described by Hall and Titran (ref. 11). A tantalum split-sleeve resistance heater was used for heating the specimens. The pressure was generally in the 10^{-6} Pa range at the start of the test and decreased to near 5×10^{-8} Pa after several hundred hours. Strains were measured by frequent optical readings of fiducial marks in the reduced gauge section during creep. Strains on loading were of the order of 0.05 percent and are included in the creep strain data.

The creep test temperature was 1350 K, and the stress level was 40 MPa for all but one test for each alloy which was conducted at 34.5 MPa. Although tests were generally terminated after achieving 1 percent strain, a few tests were continued to strains as high as 3 percent.

RESULTS AND DISCUSSION

One specimen of Nb-1%Zr was tested in creep for comparison to the DA PWC-11 alloy. The creep curves for both, tested at 1350 K are shown in fig. 5. The time to achieve 1 percent creep strain differs by almost a factor of 50 for the tests at 34.5 MPa; Nb-1%Zr required about 75 hr while the PWC-11 required about 3200 hr. Although neither alloy was in the optimum creep-resistant condition (the Nb-1%Zr was annealed 1 hr at 1475 K with an average grain size of 20 μm , and the PWC-11 had a grain size of 25 μm with large particles present, possibly primary carbides from the melt), it is believed that the creep strength ranking would not change in samples processed at the optimum conditions. The approximately 300 hr negative difference in the time to achieve 1 percent strain (i.e., lower creep rate at a higher stress) between the two tests on PWC-11 in the DA condition (fig. 5) probably reflects the inhomogeneous microstructure resulting from the melting and fabrication processes.

The high temperature ($>1/2 T_m$) creep strength of PWC-11 (relative to the order of magnitude lower carbon Nb-1Zr alloy) has been attributed to the presence of very fine precipitates of $(\text{Nb,Zr})_2\text{C}$ and/or $(\text{Nb,Zr})\text{C}$ ranging in size from tens to hundreds of nanometers in diameter (ref. 9). As with all precipitation strengthened alloys, the long term beneficial contribution of the precipitate to high temperature strength is suspect. It has been postulated (refs. 5 to 8) that welding and/or isothermal aging of the PWC-11 alloy could result in a significant loss (greater than 50 percent) in elevated temperature creep strength.

Results of creep tests of PWC-11 specimens with transverse EB welds (fig. 2) are shown in figure 6. The as-welded specimen (AW) and the post-weld heat treated specimen (W+HT) show times to achieve 1 percent strain of 2025 and 2120 hr, respectively. Thus, the AW and W+HT specimens with a transverse EB weld in the gauge section achieved 1 percent creep in about 60 percent of the time required for the unwelded specimen, which achieved 1 percent creep strain at 1350 K and 40 MPa in 3480 hr (fig. 5).

The cause for lower creep strength in welded specimens is not believed to be related to welding effects per se. Consider that microexamination of crept specimens showed no visible reduction in thickness in the vicinity of the weld, and that the weld fusion zone plus heat affected zone comprised only about 2 mm of the 25 mm gauge length. If the majority of the 1 percent strain took place in the weld area, some reduction in thickness of the weld should have been apparent. Further, since only single tests were conducted for each condition, and base metal properties can vary (as shown in fig. 5), it is difficult to make conclusions at all.

Aging of tests specimens prior to creep testing produced severe losses in creep strength which are believed to be real. An aged AW specimen suffered a decrease of 90 percent in the time to achieve 1 percent strain for DA material; 355 versus 3500 hr (figs. 5 and 6). The post-weld heat treated plus aged specimen showed a similar but less drastic effect, achieving 1 percent strain in 700 hr. To further demonstrate that EBW of PWC-11 did not produce the decade loss in the time to achieve 1 percent creep strain, an unwelded DA plus similarly aged (A) specimen was creep tested. Figure 7 shows the resultant creep curves for an unwelded specimen in the A condition along with the curve for the specimen in the W+A condition. The times to achieve 1 percent creep strain, 320 hr for the aged-only condition and 355 hr for the transverse EB welded specimen, W+A, were quite close.

The microstructures of the unaffected base metal in the W+HT+A condition are similar prior to and after creep testing (fig. 4(a) versus fig. 8(a)), characterized by generalized precipitation in the grain boundaries and in the matrix. Thus, thermal exposure under stress (creep testing) did not produce a significant change visible at these magnifications. In the weld fusion zone, the microstructure of the W+HT+A creep tested specimen (fig. 8(b)) has a grain boundary necklace of coarse particles in place of the nearly continuous network in the grain boundaries in the fusion zone for the AW (fig. 3(c)) and W+HT+A conditions (fig. 4(b)). In addition, precipitation at the cell boundaries is heavy in the fusion zone of the AW and the W+HT+A creep tested specimens.

Both of these microstructures differ substantially from the DA microstructure (fig. 3(b)) in that:

1. The large intergranular precipitates of the DA condition are no longer evident, and
2. More of the fine matrix precipitation in the base metal is evident in the W+HT+A condition both prior to and after creep testing.

Since the PWC-11 material is measurably stronger in the DA condition (fig. 4(b)) than in the aged condition (fig. 4(a)), it appears that the presence of large intergranular precipitates and moderate matrix precipitation

are desirable, possibly acting by pinning the elongated grains to prevent grain boundary sliding. Since the creep testing was only taken to 1.2 percent strain, however, it is difficult to determine if any grain boundary sliding has taken place.

It is possible that a change in the carbide morphology is the cause of the loss of creep strength after aging. This is supported by previous work by Titran on a tantalum alloy (ref. 12), where it was found that the carbide phase transformed in the presence of a strain. In the T-222 alloy (Ta-9.6W-2.4Hf-0.01C), the HCP phase $(Ta,Hf)_2C$ was found both as primary particles from the melt and after stress-free aging. However, after aging in a high temperature regime ($7.5 T_m$) in the presence of a strain, the FCC phase $(Ta,Hf)C$ was detected. Presumably, the dicarbide phase was dissolved and the reprecipitated strain-induced monocarbide interfered with dislocation glide and climb. This dislocation motion may be the primary creep mechanism in the PWC-11 alloy, along with some grain boundary sliding.

A similar process may occur in the Nb-Zr system, although optical metallography and SEM are insufficient to identify the exact changes in the carbide microstructure which affect the creep strength. A more thorough understanding of the effect of heat treatment on the microstructure of the base metal and weld zone of PWC-11 will require the use of more sophisticated techniques such as particle extraction, microdiffraction, and/or microanalytical electron microscopy techniques on aged and on strained samples.

To aid in assessing the potential of PWC-11 as a material for long term applications in space power systems, the values of $1/t$ (where t is the time to 1 percent creep strain) for the treatments and weld conditions were analyzed and compared to base-line Nb-1%Zr (ref. 13) and PWC-11 (ref. 14) data of tests conducted in liquid lithium (ref. 4). The lithium data is used as a comparison base because the number of tests allows meaningful statistical linear analysis. Prior tests were conducted in liquid lithium because that was and currently is the intended nuclear coolant and environment that the material would most likely be exposed to. The results in both lithium and in vacuum would be influenced by the partial pressure of oxygen in the medium, since oxygen can strengthen niobium significantly in creep (ref. 15). Figure 9 shows the temperature compensated base-line 1 percent strain rates for Nb-1%Zr and PWC-11 against stress as a solid line. These lines are extended to show the relative creep lives for 1 percent strain in 7 yr at 1350 K.

The base line data which is accepted as the interim reference data base by the space power system community involves only those tests performed by PWAC (refs. 13 and 14). The time to 1 percent creep properties are presented using Orr-Sherby-Dorn (O-S-D) parameters (ref. 16). The values for these parameters were determined using least squares analysis.

The data from this study is shown as dashed lines for the various material conditions. The data points have been forced to fit parallel to the reference data by using the O-S-D values determined by least squares analysis of the PWAC reference data base. Interestingly, all PWC-11 specimens in this study appear less creep resistant than the PWAC reference base-line data shown taken from tests run in lithium, and it could be presumed that the difference reflects both nonstoichiometric carbon levels and nonoptimum thermomechanical

processing as well as variations in the creep testing technique and environment. The DA condition has a projected 7 yr, 1 percent creep strain capability of 4.0 MPa. The Nb-1%Zr tested in this study appears to have an extrapolated strength of only 0.6 MPa for a 1 percent creep stain in 7 yr, also lower than the PWAC reference base-line data tested in lithium. The vacuum data in this study and the PWAC lithium data are similar in that both show the PWC-11 as approximately 6 times as strong as Nb-1%Zr.

In the aged condition (1000 hr at 1350 K), the PWC-11 material (specimens A and W+A in fig. 9) has a projected 7 yr, 1 percent creep strain capability of approximately 2.8 MPa. Nevertheless, even in this weakened condition, the PWC-11 material is approximately 4 times as strong as the Nb-1%Zr material similarly tested. The data for the PWC-11 W+HT specimen plotted in figure 9 projects a 7 yr, 1350 K, 1 percent creep strength capability of nearly 4.0 MPa.

CONCLUDING REMARKS

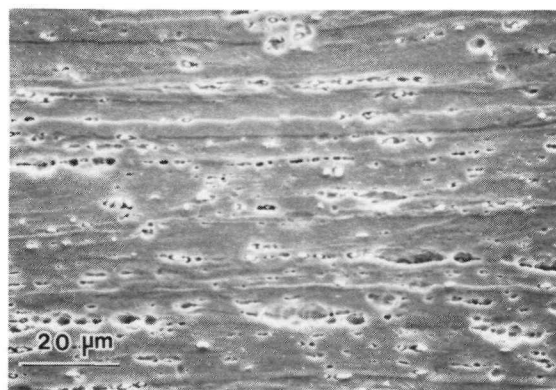
The preliminary study results obtained on the PWC-11 material evaluated in this investigation, Nb-1%Zr-0.063C, processed at less than optimum parameters has provided the following conclusions:

1. The PWC-11 base metal in the double annealed condition (1 hr at 1755 K + 2 hr at 1475 K) was the most creep resistant condition tested. The projected 7 yr, 1350 K, 1 percent creep strength of approximately 4.0 MPa exceeds the strength of the annealed (1 hr-at 1475 K) Nb-1%Zr alloy tested by greater than a factor of 6.
2. Aging for 1000 hr at 1350 K greatly reduces the creep strength of the DA base metal both with and without transverse electron beam welds. However, the projected 7 yr, 1 percent creep strength of 2.8 MPa at 1350 K appears more than adequate for service in space power systems where use of Nb-1%Zr is considered.
3. Any means possible to avoid subjecting PWC-11 to a stress-free condition at a high temperature prior to loading (simulated in this case by the 1000 hr age at 1350 K) would be highly desirable in potential space power systems.
4. The changes in the microstructure, i.e., particle specie, distribution, and morphology, which affected the creep strength was not resolvable by optical metallography. Other analytical techniques may aid in the correlation of creep behavior, second phase stability, and microstructure.

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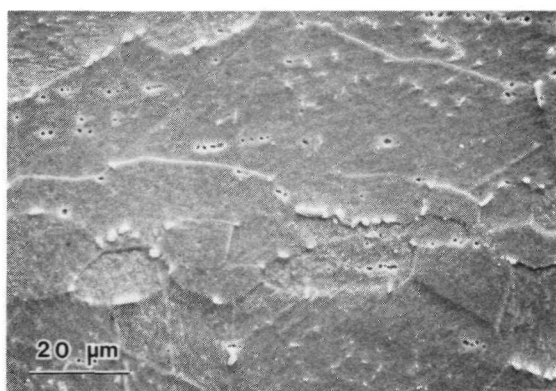
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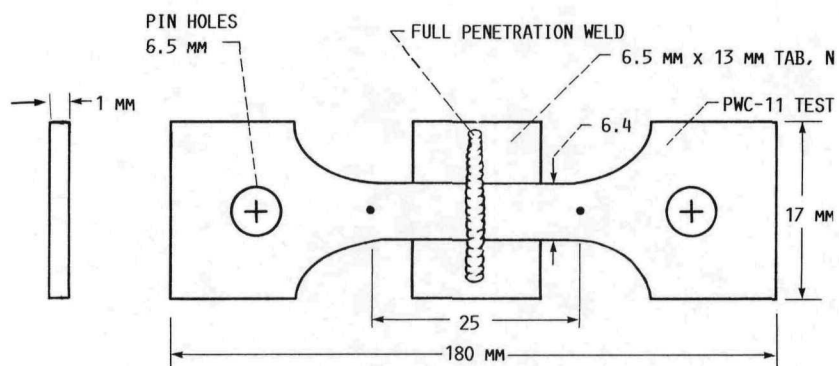
(A) AS-ROLLED SHEET.

← ROLLING DIRECTION →



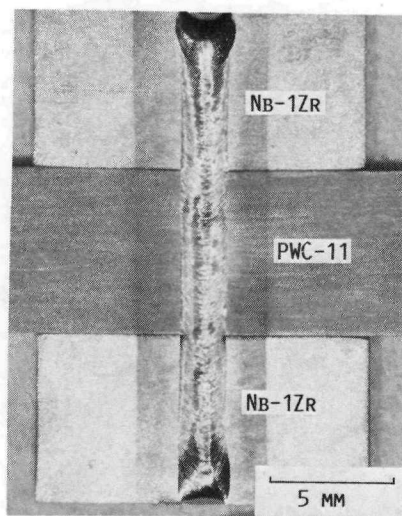
(B) DOUBLE ANNEALED CONDITION.

FIGURE 1. - MICROSTRUCTURE OF PWC-11 (A) PRIOR TO ANNEALING, SHOWING LARGE PARTICLES ALIGNED PARALLEL TO THE ROLLING DIRECTION, AND (B) THE RESULTANT ELONGATED GRAIN STRUCTURE AND PARTICLE DISTRIBUTION FOLLOWING THE 1 H-1755 K PLUS 2 H-1475 K DOUBLE ANNEAL.



(A) TENSILE CREEP TEST SPECIMEN.

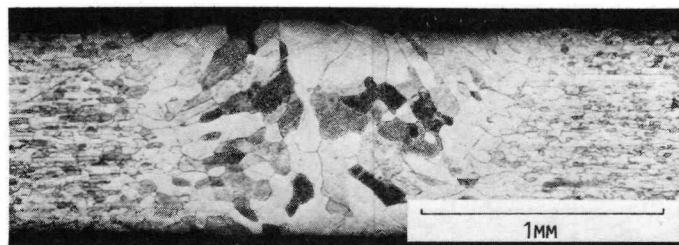
← ROLLING DIRECTION →



(B) FACE SURFACE OF TRANSVERSE ELECTRON BEAM WELD.

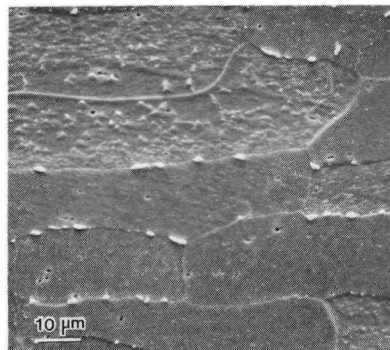
FIGURE 2. - TENSILE CREEP TEST SPECIMEN AND FACE SURFACE OF TRANSVERSE ELECTRON BEAM WELD IN PWC-11 TEST WITH NB-1Zr STARTING AND RUN-OFF TABS.

FACE

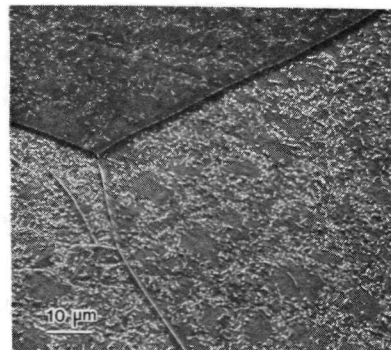


ROOT

(A) CROSS-SECTION OF SINGLE-PASS FULL PENETRATION WELD.

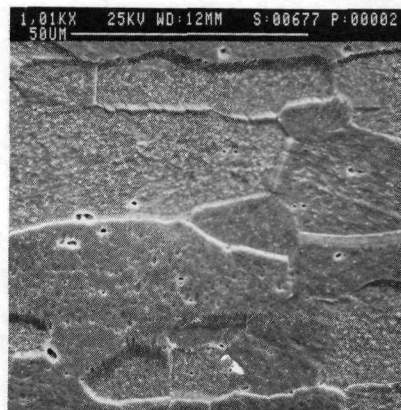


(B) UNAFFECTED BASE METAL.

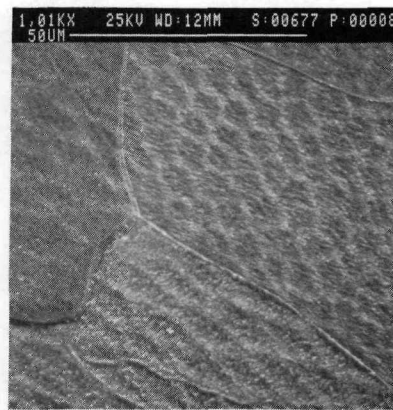


(C) WELD FUSION ZONE.

FIGURE 3. - TRANSVERSE PWC-11 ELECTRON BEAM WELDMENT IN THE AS-WELDED CONDITION PRIOR TO SURFACE GRINDING AND CREEP TESTING.



(A) UNAFFECTED BASE METAL.



(B) WELD FUSION ZONE.

FIGURE 4. - TRANSVERSE PWC-11 ELECTRON BEAM WELDMENT IN THE POST-WELD HEAT TREATED PLUS AGED CONDITION PRIOR TO CREEP TESTING.

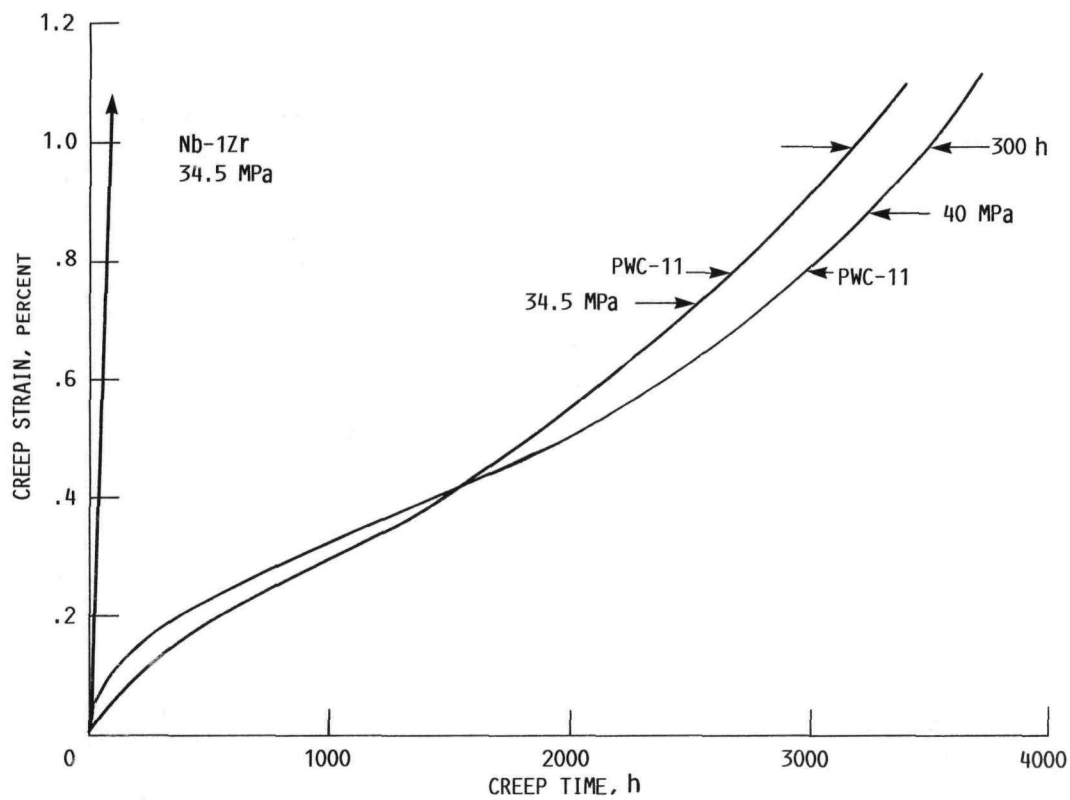


FIGURE 5.- 1350 K CREEP CURVES FOR DOUBLE ANNEALED PWC-11 AND Nb-1Zr ANNEALED FOR 1 h AT 1475 K TESTED AT INDICATED STRESS LEVELS.

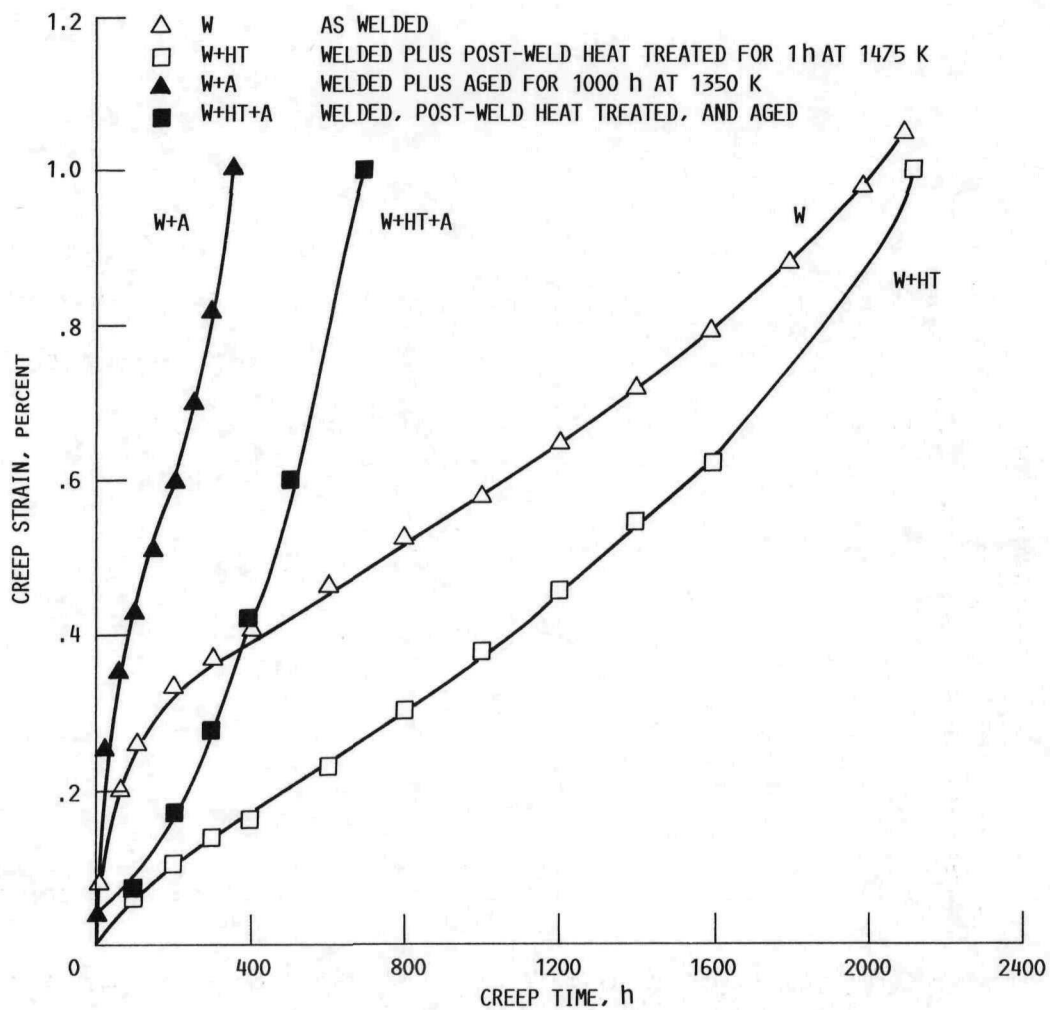


FIGURE 6.- 1350 K-40 MPa CREEP CURVES FOR PWC-11 SPECIMENS WITH TRANSVERSE ELECTRON BEAM WELDS TESTED IN VARIOUS CONDITIONS.

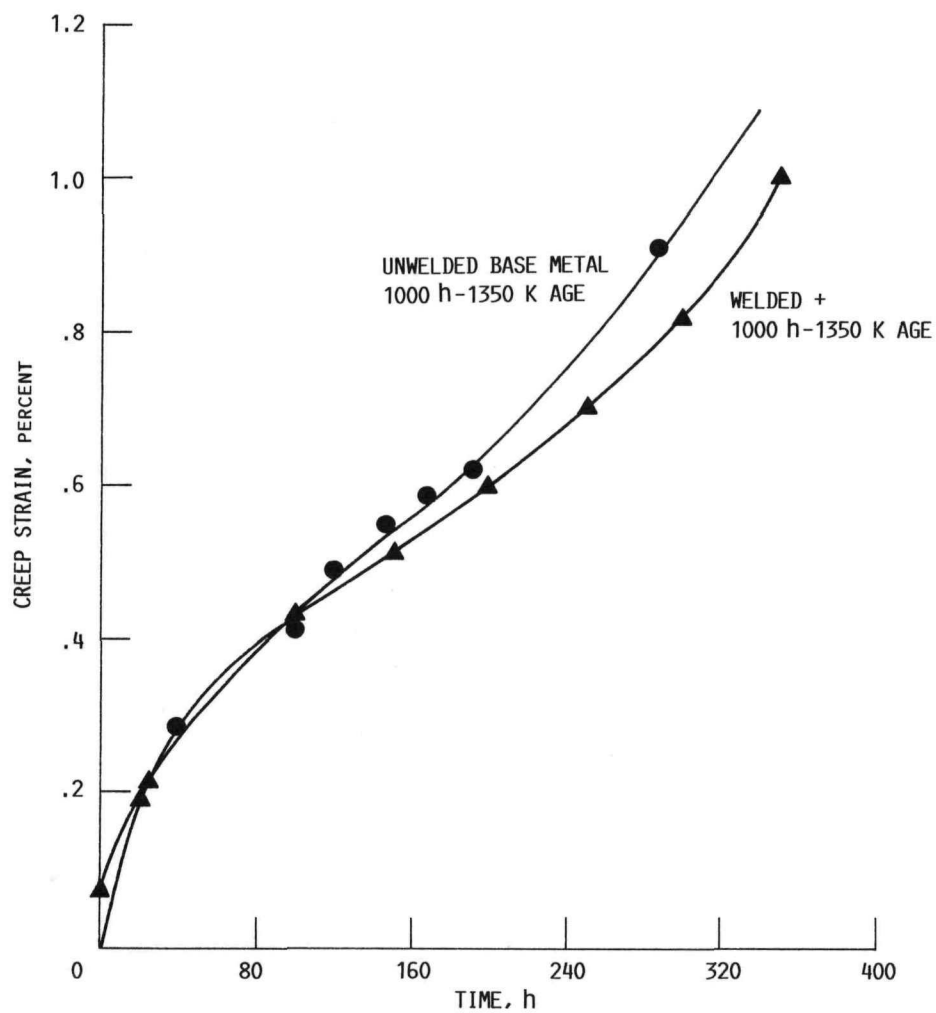
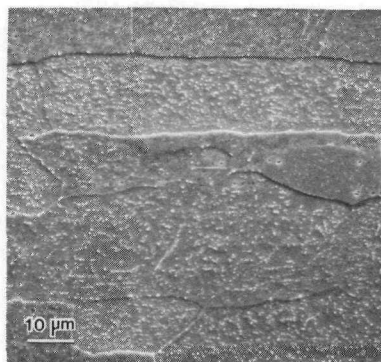
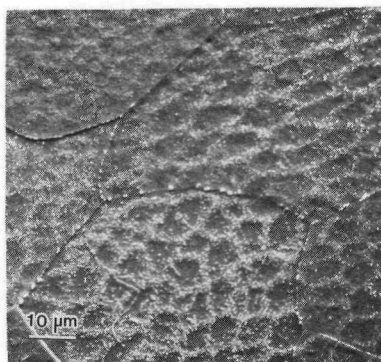


FIGURE 7.- 1350 K-40 MPA CREEP CURVES FOR A PWC-11 BASE METAL SPECIMEN AND A TRANSVERSE ELECTRON BEAM WELD SPECIMEN. BOTH SPECIMENS WERE AGED FOR 1000 h AT 1350 K PRIOR TO CREEP TESTING. THE CREEP STRENGTHS ARE SIMILAR.



(A) UNAFFECTED BASE METAL.



(B) WELD FUSION ZONE.

FIGURE 8. - TRANSVERSE ELECTRON
BEAM WELDMENT IN THE POST-WELD
HEAT TREATED PLUS AGED CONDITION;
CREEP TESTED TO 1.2 % STRAIN AT
1350 K FOR 770 H.

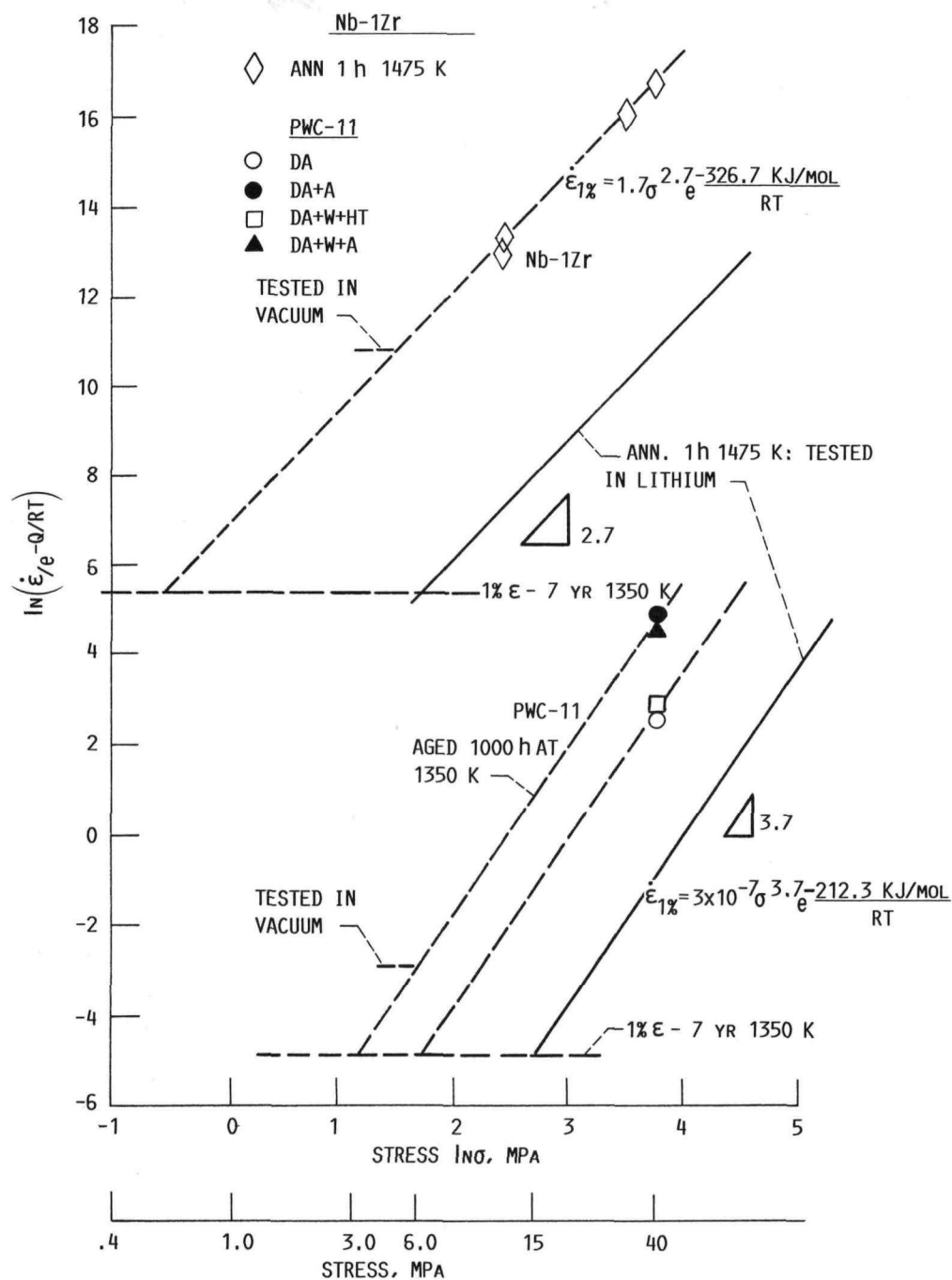


FIGURE 9.- TEMPERATURE COMPENSATED TIME TO 1% CREEP RATE VERSUS STRESS FOR Nb-1Zr AND PWC-11 MATERIALS. LITHIUM DATA FROM REFERENCES 13 AND 14.

1. Report No. NASA TM-88842		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Creep Properties of PWC-11 Base Metal and Weldments as Affected by Heat Treatment				5. Report Date	
				6. Performing Organization Code 506-41-3B	
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16. Abstract In a preliminary study using single specimens for each condition, PWC-11 (a niobium-base alloy with a nominal composition of Nb-1%Zr-0.1%C) was creep tested at 1350 K and 40 MPa. Base metal specimens and specimens with transverse electron beam welds were tested with and without a 1000 hr, 1350 K aging treatment prior to testing. In the annealed condition (1 hr at 1755 K + 2 hr at 1475 K), the base metal exhibited superior creep strength compared to the nonaged condition, reaching 1 percent strain in 3480 hr. A 1000 hr, 1350 K aging treatment prior to creep testing had a severe detrimental effect on creep strength of the base metal and transverse electron beam weldments, reducing the time to attain 1 percent strain by an order of magnitude. Extrapolated temperature compensated creep rates indicate that the present heat of PWC-11 may be four times as creep resistant as similarly tested Nb-1%Zr. The extrapolated stress to achieve 1 percent creep strain in 7 yr at 1350 K is 2.7 MPa for annealed Nb-1%Zr and 12 MPa for annealed and aged PWC-11 base metal with and without a transverse electron beam weld.					
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