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Creep Strength of Niobium Alloys, Nb-1%Zr and PWC-11

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INTRODUCTION

Space power requirements for future civilian and military missions will range from multikilowatts to megawatts of electricity. Nuclear reactor power systems have been identified as the primary power source to meet these high levels of electrical demand. Advanced materials will play a major role in meeting the stringent size and performance requirements of space nuclear power systems. The requirements for such a nuclear power system, which presently include a service life of greater than 7 years at a temperature greater than 1350 K, dictate the use of refractory metals (Cooper 1984). The alloy Nb-1%Zr has been suggested for use in space power applications where resistance to liquid alkali metal corrosion at temperatures near 1100 K was the primary concern (Lane and Ault 1965 and Buckman 1984). Although current designs of space nuclear power systems for ground demonstration specify the Nb-1%Zr alloy for reactor, heat pipe and power components (Kruger et al. 1987), future flight applications will need niobium base alloys with greater high temperature strength and increased creep resistance to provide additional design margins (Dokko et al. 1984).

A study is being conducted at NASA Lewis Research Center to determine the feasibility of using a carbide particle strengthened Nb-1%Zr base alloy, to meet the anticipated temperature and creep resistance requirements of proposed near term space power systems (Titran et al. 1987). In order to provide information to aid in determination of the suitability of the PWC-11 alloy (DelGrosso et al. 1965) as an alternative to Nb-1%Zr in space power systems this study investigated: (1) the long-time high-vacuum creep behavior of the PWC-11 material and the Nb-1%Zr alloy; (2) the effect of prior stress-free thermal aging on creep behavior; (3) the effect of electron beam (EB) welding on creep behavior, and (4) the stability of creep strengthening carbide particles.

MATERIAL AND PROCEDURE

The PWC-11 alloy used in this study was received from the Oak Ridge National Laboratory (ORNL) in the form of 1-mm thick machined tensile creep specimens. The material contained 0.90 wt % Zr and 0.063 wt % C, slightly below the PWC-11 alloy minimum specification of 0.075 wt % C. Unfortunately, a detailed processing history of this heat is not available. All material was annealed in high-vacuum for 1 h at 1755 K plus 2 h at 1475 K as per the manufacturer's recommendations (DelGrosso et al. 1967) prior to any aging, creep testing, or welding. Following the anneal, various creep and creep-rupture specimens were subjected to one or both of the following treatments: (1) single pass, full penetration EB welding across the width of the test specimen, perpendicular to the tensile axis; and (2) aging for 1000 h at 1350 K in high vacuum. The Nb-1%Zr alloy was received from ORNL in the form of cold worked 1-mm sheet. Machined tensile creep specimens were annealed in high vacuum for, (1) 1 h at 1475 K and (2) 1 h at 1755 K plus 2 h at 1475 K prior to creep testing. The

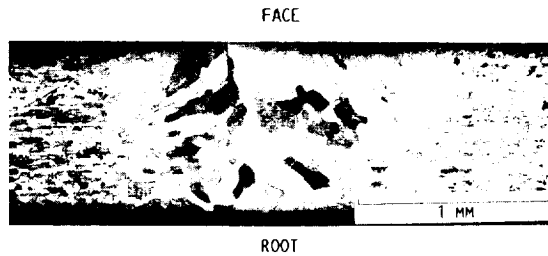
results of chemical analyses following the high vacuum annealing are given in Table 1.

RESULTS

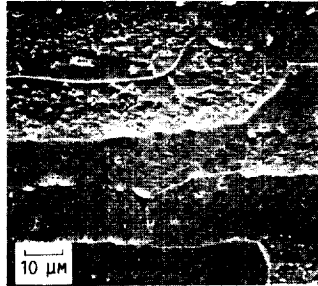
Typical microstructures of the annealed base metal and the EB weld area for the PWC-11 material are shown in Figure 1. The base metal area shows that the annealed condition has an average grain size of 25 μm measured by the circle-intercept method, with an aspect ratio of approximately 5:1 orientated in the sheet rolling direction. The EB weldment has a highly oriented grain structure with a grain size ranging from about 45 μm up to over 200 μm . The annealed condition has an extensive amount of second phase precipitated in the grain boundaries and matrix. The EB weld zone also shows extensive second phase precipitation; however, the particles appear to be finer and form cell-like domains within the grains. The Nb-1%Zr annealed for 1 h at 1475 K had an average grain size of 20 μm with a fine dispersion of ZrO_2 particles. The material annealed 1 h at 1755 K + 2 h 1475 K is expected to have an average grain size of about 80 μm based upon results of 1 h annealing studies conducted at 1600, 1700 and 1800 K. Constant load creep tests were conducted in the internally loaded, high vacuum (10^{-7} MPa) creep chambers described by Hall and Titran (1966). Creep tests were run at 1350 K to assess the effect of carbon content, grain size, thermal aging and EB welding. Figure 2 shows the creep curves for the annealed Nb-1%Zr and the PWC-11 material tested at an applied stress of 10 MPa. The fine grain (20 μm) Nb-1%Zr required about 3500 h to achieve 1 percent strain whereas the coarse grain (~80 μm) Nb-1%Zr required 11 200 h. The coarse grained Nb-1%Zr which had the identical anneal as that recommended for the PWC-11 material achieved 2 percent strain in 18 000 h and 3 percent in 24 100 h. The PWC-11 material, in both the recommended annealed condition and the annealed plus 1000 h at 1350 K thermal aged condition, it has exhibited essentially zero creep strain after 28 000 h at 1350 K and an applied stress of 10 MPa.

TABLE 1 Chemical Analyses of the Nb-1%Zr Alloy and the PWC-11 Material after High Vacuum Annealing prior to Creep Testing.

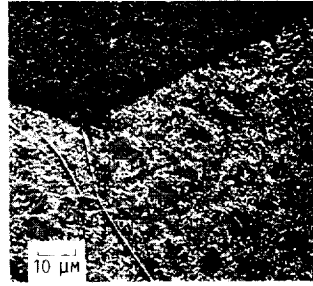
	Nb-Zr-C	Nb-1%Zr
Zirconium	0.90 wt %	1.1 wt %
Carbon	630 ppm wt	16 ppm wt
Oxygen	80 ppm wt	170 ppm wt
Nitrogen	53 ppm wt	41 ppm wt
Hydrogen	11 ppm wt	0.4 ppm wt
Niobium	Balance	Balance



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



(b) UNAFFECTED BASE METAL.



(c) WELD FUSION ZONE.

FIGURE 1. Transverse Electron Beam Weldment of the PWC-11 Material in the As-welded Condition prior to Surface Grinding and Creep Testing. Material vacuum annealed 1 h at 1755 K plus 2 h at 1475 K prior to welding.

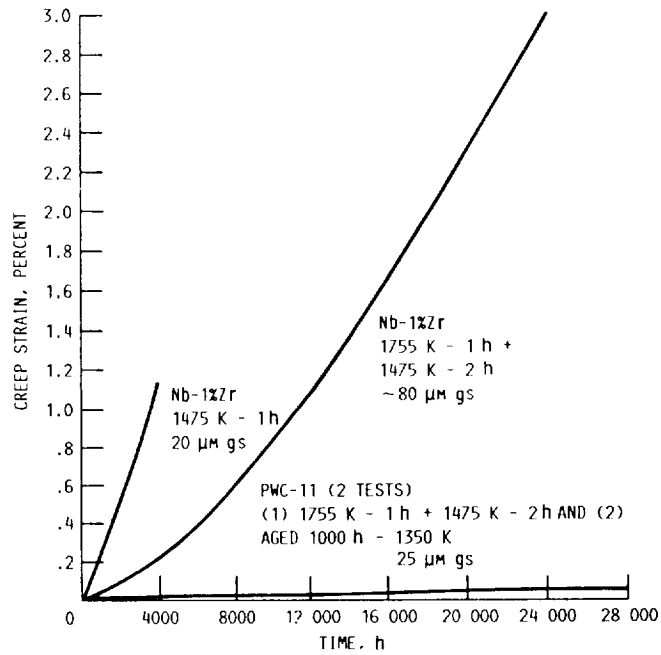


FIGURE 2. Creep Curves for Nb-1%Zr and PWC-11 Material Annealed at Indicated Conditions and tested at 1350 K and 10 MPa.

Creep tests on the EB welded PWC-11 material were conducted in high vacuum (10^{-7} Pa) at 1350 K and 40 MPa to assess the effect of welding on creep strength. The creep curves, Figure 3, show that a sample in the annealed condition required about 3200 h to achieve 1 percent strain. A similarly treated sample with an EB weldment required 2200 h to reach 1 percent strain. Moore et al. (1986) conducted short-time creep-rupture tests to further characterize the effect of EB welding on the PWC-11 material. Figure 4 shows a typical PWC-11 material creep-rupture specimen with a transverse EB weld. In all creep-rupture tests, failure occurred in the unaffected base metal, which is a clear demonstration that the weld region was stronger. The cause for the lower creep strength in welded specimens is not believed to be related to welding effects per se. Since only single test were conducted for each condition and base metal properties can vary (Titran et al. 1986), it is difficult to make conclusions at all.

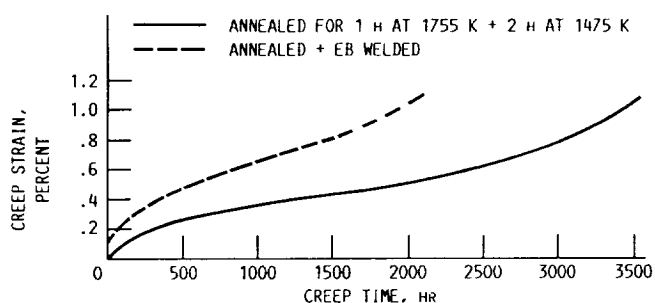


FIGURE 3. Creep Curves for PWC-11 Material with and without a Transverse EB Weld showing the Time to reach 1 Percent Creep Strain. Samples were tested at 1350 K with an applied stress of 40 MPa.

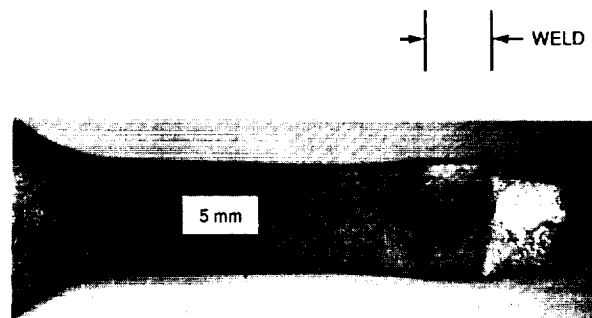


FIGURE 4. Photomicrograph of a Typical EB Welded PWC-11 Material Specimen after Creep-Rupture Testing showing that Failure occurred in the Base Metal Material.

The high temperature ($>0.5 T_m$) creep strength of PWC-11, relative to the order of magnitude lower carbon content Nb-1%Zr alloy, has been attributed to the presence of very fine precipitates of $(Nb,Zr)_2C$ and/or $(Nb,Zr)C$ ranging in size from 1 to 10 μm in diameter (PWAC 1965). Results of metallographic analysis of several samples annealed and aged with and without an applied stress confirmed the stability of the strengthening carbide particle (Grobstein et al. 1986). The concern about overaging of the precipitate particles during high-temperature exposure for long times was addressed by microstructural characterization of the precipitate (complex carbide) morphology. Several techniques were used including light microscopy, scanning and transmission electron microscopy, x-ray diffraction, and chemical analysis of extracted particles. Table 2 summarizes the results of this study. In the as-rolled condition, the precipitates were relatively coarse, 1- to 10- μm in diameter and were found to be hcp Nb_2C . After an initial heat treatment of 1 h at 1755 K and 2 h at 1475 K, a different finer precipitate formed. These particles were 0.05 to 0.1 μm in diameter and were determined to be fcc $(Zr,Nb)C$ with the Zr/Nb ratio approximately 70:30. After approximately 5000 h at 1350 K ($0.5 T_m$), these fine precipitates almost doubled in size, but did not "overage." The stability and effectiveness of this fcc $(Zr,Nb)C$ precipitate in pinning dislocations and grain boundaries, thus resisting plastic deformation is self-evident from the two 28 000-h creep tests at 1350 K and 10 MPa (Figure 2).

Multiple linear regression analysis of the time to achieve 1 percent creep strain was performed on the high vacuum (10^{-7} Pa) creep data for both Nb-1%Zr (Horak 1989) and the PWC-11 (Titran 1986) material. The Orr-Sherby-Dorh parameter (Orr et al. 1954) was determined and the results used for extrapolation of

TABLE 2 Characterization of Precipitates in the PWC-11 Material
(Nb-1%Zr-0.06%C).

	As-rolled	After initial heat treatment	After long-term high temperature exposure
Size	1 to 10 μm	0.05 to 0.1 μm	0.1 to 0.15 μm
Structure	HCP	FCC	FCC
Composition	Nb_2C	(Zr,Nb)C	(Zr,Nb)C

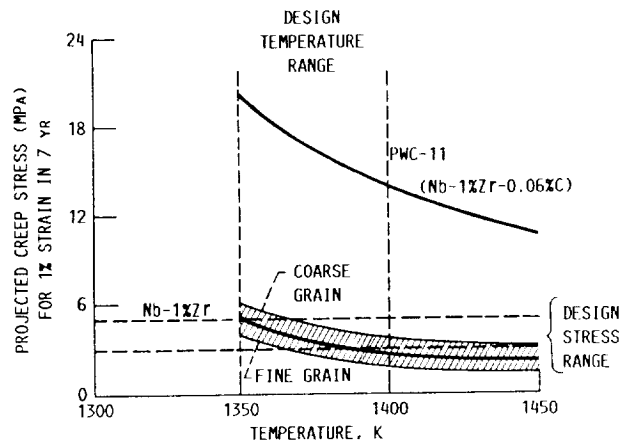


FIGURE 5. Temperature and Creep Strength Potential of the PWC-11 Material compared to Nb-1%Zr Alloy.

the vacuum creep data to 7 years (an extrapolation of approximately one order of magnitude in time). For the PWC-11 material, a projected nominal stress of 20 MPa would be required to achieve 1 percent creep strain in 7 years at 1350 K. For the Nb-1%Zr alloy, the projected 7 year 1350 K nominal creep strength is 4 MPa for the fine grain size and about 6 MPa for the coarse grain size as shown in Figure 5.

SUMMARY OF RESULTS

Based upon the preliminary studies of the high vacuum (10^{-7} Pa) creep and creep-rupture behavior of the Nb-1%Zr alloy and PWC-11 material at 1350 and 1400 K, the following observations could be drawn:

1. The PWC-11 material in the annealed state (1 h at 1755 K plus 2 h at 1475 K) is more creep resistant than the similarly annealed Nb-1Zr alloy by at least a factor of three in applied stress.
2. Aging the PWC-11 material for 1000 h at 1350 K prior to low stress (10 MPa) creep testing does not appear to affect the long term (>28 000 h) strength.
3. The electron-beam weld region was stronger than the base metal for specimens creep-rupture tested at 1350 and 1400 K.

4. The (70% ZrC - 30% NbC) fcc monocarbide was the only extracted phase identified after high temperature exposure of the PWC-11 material and does not appear to overage.

Acknowledgments

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