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Tensile and Creep Rupture Behavior of P/M Processed Nb-Base Alloy, WC-3009

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ABSTRACT

Due to its high strength at temperatures up to 1600 K, fabrication of niobium base alloy WC-3009 (Nb-30Hf-9W) by traditional methods is difficult. Powder metallurgy (P/M) processing offers an attractive fabrication alternative for this high strength alloy. Spherical powders of WC-3009 produced by electron beam atomizing (EBA) process were successfully consolidated into one inch diameter rod by vacuum hot pressing and swaging techniques.

Tensile strengths of the fully dense P/M material at 300-1590 K were similar to the arc-melted material. However, the P/M material showed somewhat less tensile elongations than the arc-melted material. Creep-rupture tests in vacuum indicated that WC-3009 exhibits a Class I solid solution (glide controlled) creep behavior in the 1480-1590 K temperature range and stress range of 14 to 70 MPa. The creep behavior was correlated with temperature and stress using a power law relationship. The calculated stress exponent n, was about 3.2 and the apparent activation energy, Q , was about 270 kJ/mol. The large creep ductility exhibited by WC-3009 was attributed to its high strain rate sensitivity.

INTRODUCTION

Niobium (Nb) base alloys are attractive for advanced aerospace propulsion applications because of their favorable combination of low density, high melting point and elevated temperature mechanical properties. However, none of the currently available commercial Nb alloys have the desired combination of strength, ductility and oxidation resistance required for these applications. High strength alloys lack ductility, oxidation resistance and are difficult to fabricate whereas ductile alloys lack sufficient strength at high temperatures. Oxidation resistant alloys such as niobium aluminides are hard and brittle. If significant improvements in strength,ductility and oxidation resistance are to be achieved for structural alloys for advanced aerospace propulsion components, it is most probable that these improvements will be provided by innovation in processing and production technology. An understanding of the structure and composition of the protective oxide scales on niobium alloys combined with unique processing techniques such as rapid solidification and mechanical alloying, the desired materials can be produced.

Thus the NASA-Lewis Research Center has undertaken a broad program to develop niobium base alloys with improved oxidation resistance, ductility and strength for advanced aerospace applications. In this overall program, a commercially available high strength Nb-alloy WC-3009 (Nb-30Hf-9W) in a powder form and the most oxidation resistant niobium aluminide intermetallic compound developed (1) will be mechanically alloyed in a high energy attritor mill. The mechanical alloying hopefully will cause the brittle intermetallics to fragment and embed in the surface of the more ductile WC-3009 alloy. It is felt that this type of microstructure of powder particle may help in selective oxidation of Al from the well dispersed intermetallic phase to form a more protective oxide scale. However, the addition of a brittle intermetallic to ductile alloy may degrade some of its mechanical properties. Therefore, the aim of the present investigation was first to identify the powder processing parameters and then to evaluate the tensile and creep-rupture behavior of P/M processed WC-3009.

EXPERIMENTAL PROCEDURE

Spherical powders of WC-3009, procured commercially from Teledyne Wah-Chang Albany (TWCA), were produced by electron beam melting followed by centrifugal atomization in a laboratory size electron beam atomizer. The chemical analysis of as-received powder is given in Table I.

Table I
Chemical Composition Of As-Received WC-3009 Powder (In Wt%)

Nb	Hf	W	Zr	Ta	O	N	C
56.0	33.2	9.6	1.02	0.5	0.020	0.0015	0.0085

The spherical powders were canned in niobium tubes, 38 mm dia and 500 mm long, followed by vacuum hot pressing at 1590 K under a pressure of 200 MPa for 8 hours. The bars were then hot and warm swaged to 12.5 mm dia rod. The fully consolidated rods were vacuum annealed at 1590 K for one hour. With an intention of investigating the influence of temperature and pressure on the consolidation of the powders, two trials of hot pressing were carried out at 1590 and 1810 K under a pressure of 150 MPa for 8 and 3 hours respectively.

ASTM standard tensile and creep specimens of 25.4 mm gage and 6 mm dia round were machined from the heat treated rod. Sheet specimens of 25.4 mm gage were also machined from recrystallized sheets of WC-3009 obtained from TWCA. These sheets were produced by conventional techniques of arc melting and rolling (hereafter referred to as arc-melted). Tensile tests were carried out according to ASTM E-8 procedures. Elevated temperature tensile tests were performed under high vacuum. Constant load creep rupture tests were conducted in a vacuum of 10^{-4} MPa in the temperature range 1480 to 1590 K and stress range 14 to 70 MPa using apparatus described in detail elsewhere (2). The specimens were heated to test temperature by radiation from a concentric tantalum sheet

heater positioned within a water cooled chamber. The temperature was monitored with Pt/Pt-Rh thermocouples attached the reduced gage section of the specimens and to a digital temperature display. Creep strains were measured optically using a cathotometer to sight on fiducial marks initially placed on the gage length of the specimen. The precision of creep strain measurements is estimated to be • 0.02% for the gage length used. Creep strains were also monitored on a dial micrometer attached to the pull rods. This was particularly useful in monitoring large elongations for most tests.

RESULTS

The particle size analysis of the as-received powder given in Table II suggests that most of the powder particles had an average size of about 63 μm.

Table II

Particle Size Distribution Of As-Received WC-3009 Powder.

Size (μm)	88	63	35	25
Percent	4	60	15	11

Figure 1 shows the microstructure of as-received powder particles. Most of the powder particles were spherical and showed evidence of coring in the dendritic microstructure. This suggests that the particles had undergone high cooling rates of 10^{3-4} K/sec in the interdendritic areas.

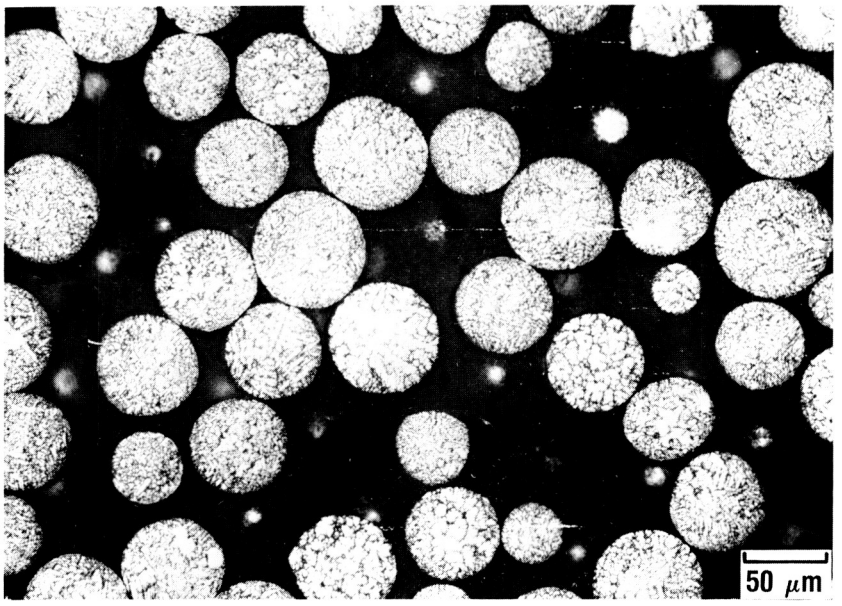


Figure 1: Microstructure of as-received powders of niobium alloy,WC-3009.

The consolidation of powders by hot pressing at 1590 K under a pressure of 200 MPa followed by swaging produced a fully dense rod. However, it was not possible to get a full densification when the hot pressing was carried out at 1590 K using a pressure of 150 MPa. Increasing the hot pressing temperature to 1810 K resulted in a severe reaction with the niobium pressing cans. The microstructure of the fully consolidated and annealed rod consisted of fine grains (Figure 2). The second phase particles present in Figure 2 were identified as hafnium oxide, formed due to the high oxygen content in the powder (Table I).

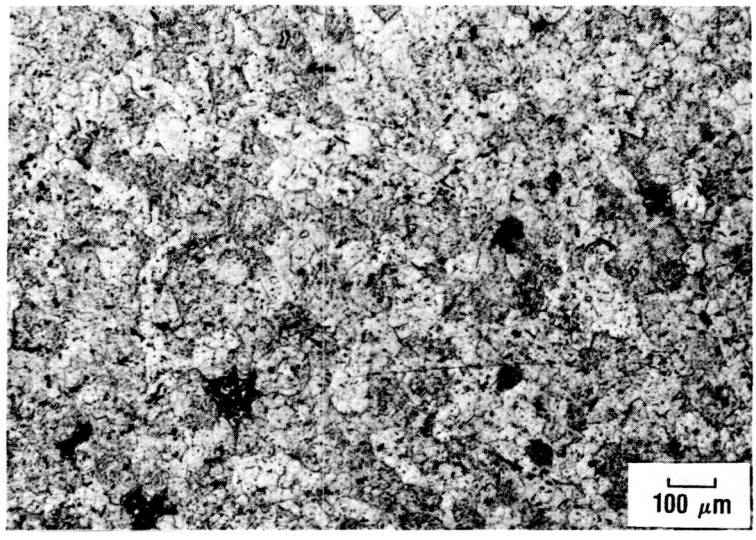


Figure 2: Optical micrograph of the fully consolidated and and 1 hour 1590 K vacuum annealed WC-3009 P/M alloy.

Figure 3 shows the results of tensile tests on both the arc-melted and P/M processed WC-3009. The values of strength and ductility shown in Figure 3 were the average of duplicate specimens tested under the identical conditions of temperature and strain rate. The range of tensile strengths levels observed in the P/M material at all test temperatures were within the normal range for arc-melted material. However, tensile elongations were affected by processing methods.The P/M material exhibited less tensile ductility than the arc-melted material at all test temperatures. Both arc-melted and P/M material showed a sharp rise in tensile elongation with temperature beyond 1480 K probably due to dynamic recrystallization. Wojcik (3) has observed a decrease in ductility in both forms of this alloy at 1380-1480 K. However this was not observed in the present investigation.

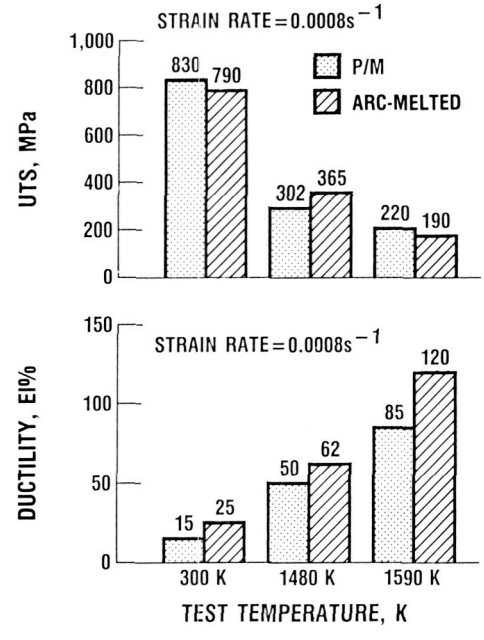


Figure 3: Comparison of tensile properties of arc-melted and P/M processed niobium alloy, WC-3009.

A typical creep curve of P/M processed WC-3009 tested at 1480 K under an initial stress of 55 MPa is shown in Figure 4. This creep curve shows that the material exhibited very little primary creep, a relatively short steady state region and a large amount of tertiary creep which resulted in high strains to fracture. This type of creep behavior was exhibited over the entire temperature and stress levels tested for both the arc-melted and P/M processed WC-3009 alloy.

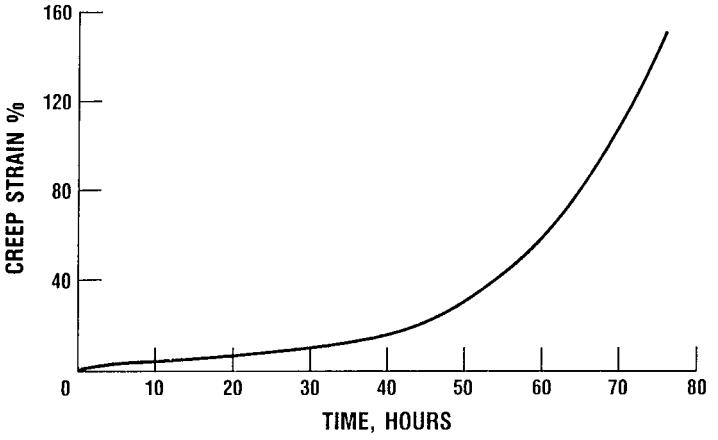


Figure 4: A typical creep curve of P/M processed niobium alloy, WC-3009 tested at 1480 K and 55 MPa.

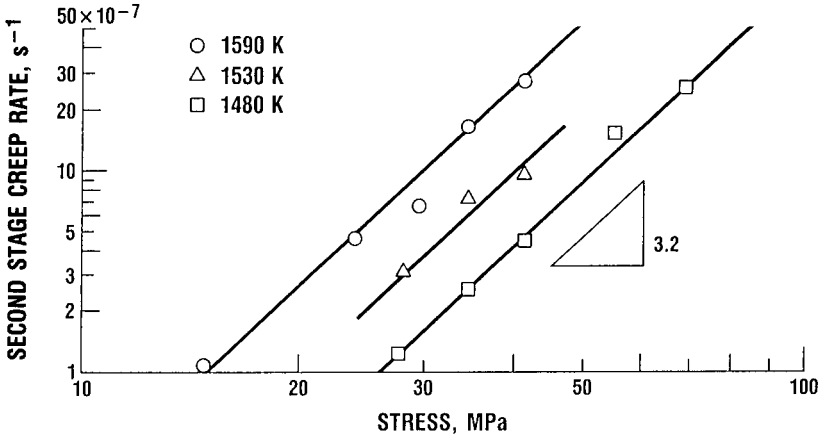


Figure 5: Steady state creep rate as a function of applied stress and temperature for niobium alloy, WC-3009.

The creep properties were characterized in terms of the stress and temperature dependence of the steady-state creep rate, $\dot{\epsilon}$, and the rupture life, t_f. The steady state creep rate can be described (4) by the following phenomenological equation:

$$\dot{\epsilon} = A\sigma^n \exp\left(-\frac{Q}{RT}\right) \qquad [1]$$

where A is a material constant, n is the stress exponent and Q is the apparent activation energy for creep, R is the universal gas constant, and T is the absolute temperature in K. This power law relation between steady-state creep rate and applied stress is illustrated in Figure 5 for P/M processed WC-3009 tested at 1480, 1530 and 1590 K. The values of n and Q were estimated by multiple regression analysis using all the creep data obtained at the three temperatures. The coefficients, n and Q, of the regression equation were found to be equal to 3.15 ± 0.21 and 270 ± 40 kJ/mol respectively.

In addition, the stress and temperature dependence of the creep rupture lives can be described accurately (5) by the following equation:

$$t_f = A\sigma^P \exp\left(-\frac{Q}{RT}\right) \qquad [2]$$

which is of the same form as eqn. [1]. The linear behavior of the log t_f vs log σ data is illustrated in Figure 6 in the temperature range 1480 and 1590 K.The coefficients, p and Q, determined by multiple regression were found to be equal to 3.9 ± 0.22 and 370 ± 32 kJ/mol, respectively.

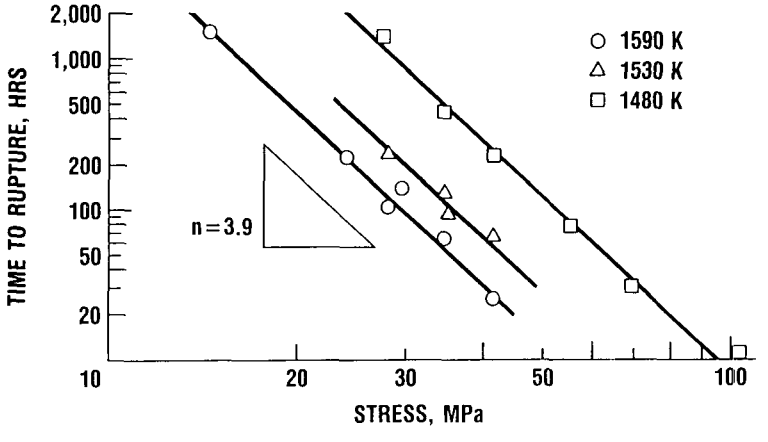


Figure 6: Time to rupture as a function of applied stress and temperature for niobium alloy, WC-3009.

The activation energies were also determined with the corrections for the temperature dependence of the elastic modulus, E. In this case, the temperature dependence of E for pure niobium from Reference 6 were used. Using the modulus correction in eqn. [1] and in eqn. [2] the apparent activation energies of 240 ± 30 and 338 ± 32 kJ/mol respectively were obtained. Thus the modulus correction did not significantly influence the values of activation energies determined for both creep rate and rupture life.

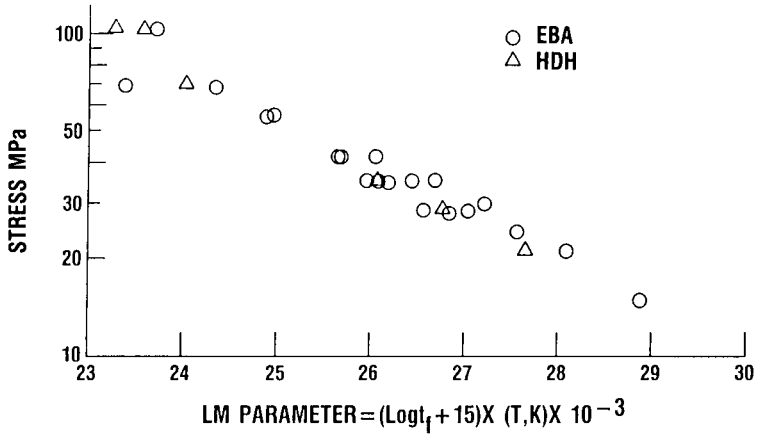
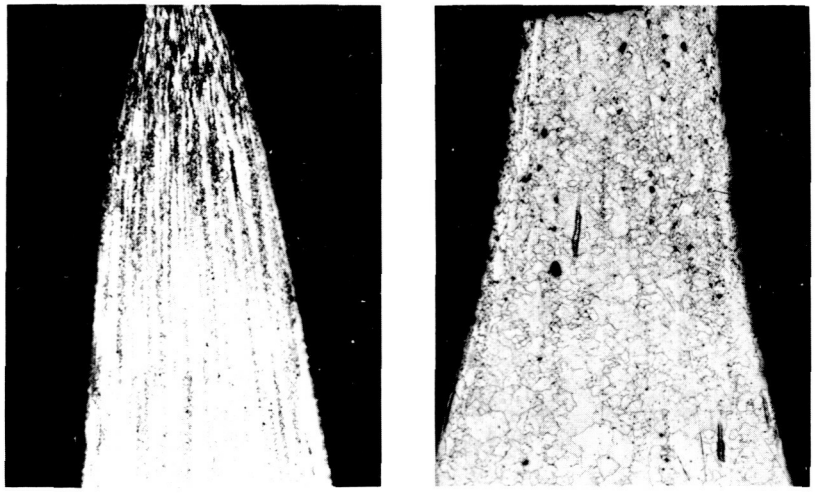


Figure 7: Larson-Miller plot showing the agreement between the atomized and hydride-dehydride powder processed WC-3009 alloy.

A summary of stress-rupture data of WC-3009 is shown as a Larson-Miller plot in Figure 7. The stress-rupture data on hydride-dehydride (HDH) powder processed WC-3009 reported in Reference 3 is also included in Figure 7. As with most niobium alloy data, a constant of 15 was used in the Larson-Miller equation. As shown in this figure, there was a good agreement between the rupture lives of the atomized and hydride-dehydride powder processed material. A fairly wide scatter band evident in Figure 7 may be due to scatter in tertiary creep which dominated most of the tests. It should also be recognized that considerable differences in total elongations were observed as evident in Figure 8 and no correlation between elongations and applied stress could be made. Figure 8 also indicates a larger amount of second phase particles present in a specimen tested at 14 MPa (which failed after 1465 hours) than in a specimen tested at 35 MPa (which failed after 64 hours) suggesting that these particles may be responsible for decreased ductility. The chemical analysis of the specimen tested at 14 MPa indicated an increase in oxygen content by about 60 ppm. Microprobe results confirmed the second phase particles to be hafnium oxide.

DISCUSSION

Although refractory metal powders have long been used by consolidating into sintered electrodes for subsequent melting into ingots for traditional metallurgical processing, application of powder metallurgy techniques as an alternate to the traditional techniques to produce a near-net-shape component is a recent one. Results of earlier work (3,7) as well as the present work suggest that P/M approach is a viable one for attaining properties equivalent to wrought material and is capable of directly producing near net shape components of the high strength niobium alloys which are otherwise difficult fabricate, thus, significantly saving the strategic refractory materials. The WC-3009 alloy is an excellent candidate since this alloy is very brittle in cast form, with a typical ductility < 1%. However, at least 15% tensile elongation can be achieved in a material consolidated from rapidly solidified powder without any subsequent hot or cold working (3). With the current



1590 K/35 MPa 1590 K/14 MPa

Figure 8: Comparison of cross-sections of fractured specimens tested at 35 and 14 MPa at 1590 K.

interest in niobium intermetallics for advanced aerospace applications due to their low density and good oxidation resistance, it is believed that only P/M methods will be capable of producing improved compositions.

The dendritic microstructure of the powder particles shown in Figure 1 is typical of PREP (Plasma Rotating Electrode Process) and EBA (Electron Beam Atomized) powders which undergo intermediate cooling rates of 10^{3-4} K/sec. For comparison, melt-spun products which undergo very high cooling rates ($\approx 10^6$ K/sec) and hence exhibit a more homogeneous and refined microstructure, resulted in improved strength and ductility (8). Wojcik (3) has shown that the WC-3009 powder produced by hydride-dehydride process was angular in shape and had the same structure observed in the as-cast ingot.

The tensile test data shown in Figure 3 indicate that P/M processed material results in strengths equivalent to wrought material at room and elevated temperatures. Similar results have been observed for niobium alloy C-103 (7). However, P/M alloy WC-3009 exhibited a decrease in ductility at all temperatures as compared to the arc-melted material. It is known that the distribution of oxides on the grain boundaries can drastically reduce tensile elongations (3). The P/M processed WC-3009 had higher amounts of oxygen (TableI) in the form of finely distributed hafnium oxide (Figure 2) which may be responsible for the somewhat lower ductility.

The shape of the creep curve shown in Figure 4 in which there is a very large region in which the strain rate is continuously increasing with the strain, is a typical of Class I solid solution strengthened alloy as opposed to pure metals and Class II or climb controlled alloys (9). This type of creep curve has been documented for another niobium alloy, C-103 (7). In contrast with pure metals and other alloys, Class I solid solution strengthened alloys exhibit subgrain formation only at very large strains and are not controlled by stacking fault energy (9,10).

The creep behavior of this alloy is similar to that of C-103 in which the values of n of 3.4 and Q of 316 kJ/mol, a value smaller than the activation energy for self-diffusion are observed (7). The creep behavior of Class I solid solution alloys is generally considered to be controlled by solute viscous drag on gliding dislocations (9-11). Therefore, the activation energy for creep might be expected to be related to the diffusion of either tungsten or hafnium in niobium. The diffusion of tungsten in niobium for up to 1473 K is only 38 kJ/mol (12) which makes it unlikely that this element controls creep. Unfortunately an activation energy value for the diffusion of hafnium in niobium is not found in literature.

The stress σ and strain rate $\dot{\epsilon}$ are related through the expression:

$$\sigma = B\dot{\epsilon}^{m} \qquad [3]$$

where m is the reciprocal of the stress exponent, n, in eqn. [1] and is termed the strain rate sensitivity, and B is a constant involving the temperature dependence. The values of m range from 0.004 for pure metals to 0.5 for superplastic materials and Class I solid solution alloys with m = 0.33 lie between the above two (13). An empirical correlation has been developed (14) between the total strains $\Delta L/L$ and the value of the strain rate sensitivity m ;

$$\frac{\Delta L}{L} = \exp\left(\frac{mK}{1-m}\right)^{-1} \qquad [4]$$

where K is typically 2-3. Using the data for WC-3009 and taking K = 2, the predicted strains from eqn.[4] agree quite well with the observed strains.

SUMMARY OF RESULTS

A commercial Nb-base alloy,WC-3009 has been successfully produced by P/M techniques. Results of the present investigation are as follows: (1) P/M alloy WC-3009 consolidated by hot pressing at 1590 K and 200 MPa pressure was fully dense. (2) Tensile strengths of the P/M alloy at 300-1590 K were similar to the arc-melted alloy. However P/M alloy showed less tensile elongation than the arc-melted alloy. (3) WC-3009 exhibited Class I solid solution behavior with n = 3.2 and Q = 270 kJ/mol. (4) WC-3009 exhibited high creep-rupture ductilities due to its high strain rate sensitivity (m = 0.3).

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15. Supplementary Notes

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