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Creep behaviour of hot isostatically pressed niobium alloy powder compacts

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A commercial (C103) niobium-base metal alloy (Nb + 10 wt% Hf + 1 wt% Ti) has been successfully manufactured from powders by a duplex hipping process. The microstructure, room temperature tensile properties and creep properties of this powder-source alloy compare favourably with wrought material. At high temperatures the creep behaviour of the alloy was found to be typical of that of Class 1 solid solution alloys (glide controlled), and over the temperature range 1400 to 1700° C it can be described accurately using the measured values of stress dependence and activation energy. The large ductility exhibited by this alloy at high temperature is discussed in terms of its strain-rate sensitivity.

1. Introduction

Advances in high-performance missile and space vehicle systems depend on the availability and effective use of strategic structural materials. Expenditures for such systems have increased significantly over the past several years primarily because of the escalating raw materials and manufacturing costs. A major factor in these systems relates directly to industrial limitations on producing metallic parts of close-to-final dimensions. In this regard, hardware fabrication invariably necessitates labour-intensive machining operations that result in large quantities of scrap. A relatively new processing approach which could reduce manufacturing costs encompasses the hot isostatic pressing (HIP) of net and near-net shapes from particulate material. In addition to minimizing machining, the method also has the capacity to improve microstructural and mechanical properties.

HIP fabrication procedures have been developed and demonstrated successfully for a wide variety of materials [1]. In recent times, with the ever increasing need to conserve critical metals, emphasis has been placed on the consolidation of titanium alloy [2, 3] and superalloy [4, 5] airframe and turbine engine components. Unfortunately, extension of this emerging technology to the strategically-important refractory metals and alloys has received little or no attention. These materials are employed extensively in missile and space vehicle hot-gas exhaust and attitude vector control systems in the form of forgings that are machined to precise dimensions. Like the superalloys, metal removal of refractory alloys in most cases is tedious and difficult. HIP processing is one technology that offers the potential both for reducing manufacturing costs and improving design performance capabilities.

In the present study, the potential of applying HIP processing techniques to a refractory metal, C103 alloy, has been examined. This alloy was manufactured from a powder-source and the creep properties at 1650° C were compared with those of conventionally produced material.

2. Experimental procedure

HIP rod segments were prepared from $-149\,\mu$m mesh hydride/dehydride powder employing a two-stage consolidation sequence. Initially, the powder was encapsulated in individual, 230 mm long, evacuated mild steel canisters. These canisters were subjected to a 3 h HIP cycle at 1260° C under an argon pressure of 103.4 MPa. This temperature was selected to promote a relatively high degree of

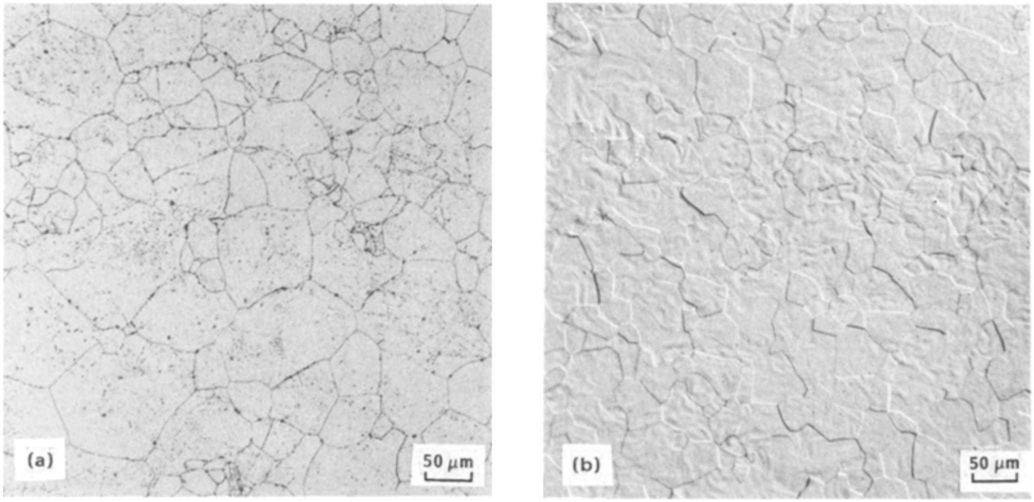

Figure 1. Optical photomicrographs from (a) a powder-source C103 alloy bar and (b) a wrought C103 alloy bar after recrystallization.

particle coalescence while simultaneously minimizing interdiffusion between the iron canisters and C103 powder. Care must be taken to avoid the Fe–Nb eutectic reaction which occurs at 1372° C. The mild steel canisters were removed in a 50 vol % HNO_3 + 50 vol % H_2O solution and the diffusion interaction zone (~ 0.64 mm thick) was dissolved in a bath composed of 50% HNO_3–5% HF–45% H_2O by volume. All rod segments thus produced were given a second 3 h HIP treatment at 1593° C under an argon pressure of 103.4 MPa to complete the consolidation process. The average density of the duplex HIP rods was measured to be 8.85 mg mm^{-3} (99.9% of theoretical), with a mean grain diameter of 75 μm. (Mean grain diameter, $\bar{D}$, was calculated from the mean linear intercept $\bar{L}$, using the equation $\bar{D} = 1.75\,\bar{L}$ [6].)

The wrought material was in the form of as-received, extruded bar stock. This material was in the fully-recrystallized condition and the mean grain diameter was measured to be ~ 50 μm.

The microstructures of the powder-source and wrought C103 alloys are compared in Fig. 1. Several minor differences can be observed. The quantity of second-phase particles is seen to be greater in the powder-source alloy than in the wrought alloy. These particles were qualitatively identified by Energy Dispersion Analysis by X-ray (EDAX) methods to be rich in hafnium. Similar phases have been identified elsewhere as hafnium oxide [7]. The chemical composition of the alloys, shown in Table I, supports this conclusion. It can be seen that the oxygen content is higher by a factor of about ten in the powder-source alloy (0.15% O_2) than in the wrought alloy (0.017% O_2). This is undoubtedly a result of the manufacturing process, as are the relatively high nitrogen and hydrogen contents in the powder-source alloy.

The increased levels of interstitial elements are not, however, especially detrimental to ambient temperature properties. In Table II the results of tensile tests on the powder-source and wrought C103 alloys are given. As may be seen, the powder-source alloy shows good ductility (26%) and is somewhat stronger than the wrought material after recrystallization. The properties of the wrought alloy after recrystallization are typical of those quoted in the literature [7].

Samples for creep testing were machined from the powder-source rod segments and from the wrought, bar-stock material. Samples machined from the powder-source material had a reduced

TABLE I Chemical compositions (wt %) of niobium C103 alloys

C103 alloy	Hf	Ti	W	Zr	Ta	C	O	N	H
Powder-source	9.4	0.96	0.25	0.37	0.25	0.005	0.15	0.07	0.01
Recrystallized bar-stock	10.3	0.95	0.15	0.24	0.23	0.005	0.017	0.005	< 0.0005

TABLE II Room temperature tensile properties of niobium C103 alloys

C103	Yield strength 0.2% offset (MPa)	Ultimate tensile strength (MPa)	Elongation (%)
Powder-source	430	530	26
Recrystallized bar-stock	311	414	40

section diameter of 3.8 mm and a gauge length of 20 mm. The wrought specimens were slightly larger, having a reduced section diameter of 6.35 mm and a gauge length of 29 mm.

Creep experiments were performed at temperatures ranging from 1593 to 1693° C, in a vacuum, using apparatus described in detail elsewhere [8]. Creep deformation was measured and recorded continuously by a d.c. linear displacement transducer attached to the loading assembly. The samples were heated to the test temperature by radiation from a concentric tantalum sheet heater positioned within a water-cooled chamber evacuated to 10^{-4} to 10^{-5} torr. The temperature was monitored with Pt/Pt–10 wt % Rd thermocouples attached to a multichannel digital temperature display.

3. Results and discussion

In this section the creep behaviour of the powder-source C103 alloy is described and compared with the wrought C103 alloy data. Creep tests were carried out at 1650° C for the powder-source alloy, and over the temperature range 1593 to 1693°C for the wrought alloy.

A typical creep curve from a powder-source C103 sample is shown in Fig. 2. The example chosen is from a test performed at 1650°C under an initial stress of 13.8 MPa. There are a number of features of interest to note in this creep curve. Typically, it was observed that the material exhibited little or no primary creep regime (decelerating creep rate). In addition, the secondary creep (constant creep rate) also was very limited. The creep curve can best be described as comprising mainly a tertiary creep regime (accelerating creep rate), although, a minimum creep rate usually could be detected in the early stage of deformation. Further, the ductility of the creep-tested samples was very high, over 100%. This type of creep behaviour was examined over the stress range 12.40 to 16.54 MPa at 1650° C and was found to be representative of both powder-source and wrought samples. It was also typical of wrought samples tested over the same stress range at 1593 to 1693° C.

Comparison of the data from powder-source C103 samples with data from wrought C103 alloy will now be made. The data, shown in Table III, are first presented in a log stress against log time format in Fig. 3 for the different strain levels 1, 2 and over 100%.* This type of plot is often used for presentation and comparison of short-time creep life data for components of rocket

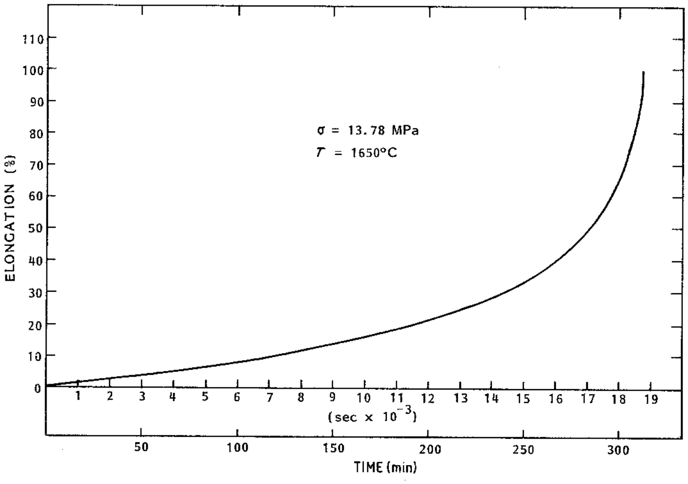

Figure 2. Typical creep curve for C103 alloy. The example shown is that for a powder-source C103 alloy sample tested at 1650° C at an initial stress of 13.8 MPa.

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*The degree of sample extension in most of the specimens tested was sufficiently great to exceed the available specimen traverse on the creep equipment used. Thus, tests often had to be terminated without final fracture being observed, although fracture was always imminent.

TABLE III Creep properties of wrought and powder-source niobium C103 alloys

Temperature (° C)	Initial stress (MPa)	Test duration (sec)	Creep ductility (%)	Minimum creep rate (10^6 sec^{-1})	$\frac{\sigma}{E}$* (10^4)	$\frac{\dot{\epsilon}}{D}$ (10^{-3} cm^{-2})
Wrought C103						
1593	12.40	34 920	> 114	7.20	1.229	5.52
1593	12.78	39 120	> 118	8.33	1.365	6.39
1593	16.54	17 100	> 116	16.7	1.638	12.80
1650	12.40	24 900	> 120	10.0	1.246	4.25
1650	13.78	18 360	> 100	19.2	1.384	8.06
1650	16.54	9 420	> 113	30.0	1.661	12.70
1693	12.40	13 920	> 125	17.9	1.256	4.85
1693	13.78	8 520	> 123	27.8	1.396	7.53
1693	16.54	3 810	112	55.0	1.675	14.90
Powder-source C103						
1650	12.40	30 134	> 125	11.6	1.246	4.89
1650	13.78	19 860	104	15.0	1.384	5.37
1650	16.54	10 420	98	30.0	1.661	12.60

*Modulus data are taken from [11] as follows: $E_{1593°C} = 101$ GPa, $E_{1650°C} = 99.6$ GPa, $E_{1693°C} = 98.7$ GPa.

propulsion and high-temperature, gas-control systems [8]. As may be seen, the powder-source C103 data is comparable to, or better than, that of the wrought C103 data. The equivalent creep properties between the powder-source C103 samples and the wrought C103 samples suggest that the powder metallurgy approach is a viable one for attaining equivalent high-temperature properties in this alloy. In particular, extremely good ductilities were obtained for all samples, as shown in Table III.

The shape of the creep curve shown in Fig. 2 is characteristic of solid-solution strengthened alloys that fall into the category of Class I solid solutions. The creep behaviour of these alloys, first described by Sherby and Burke [9], differs from that of pure metals and other solid-solution (Class II or climb-controlled) alloys in a number of ways. The continually increasing strain rate with strain, typified by the creep curve of Fig. 2, is one such feature of these alloys [9, 10]. Also, subgrain formation is generally only observed at very large strains for Class I solid solutions, while subgrains usually form during primary creep in pure metals and Class II solid solutions [9, 10]. In addition, whereas stacking fault energy is con-

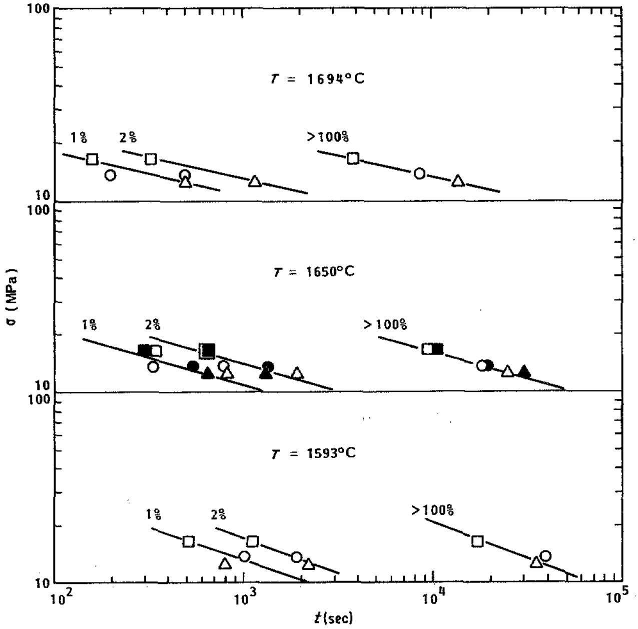

Figure 3. Log creep stress against log time creep data for strain levels of 1, 2 and > 100% from wrought (open symbols) and powder-source (closed symbols) C103 alloy over the temperature range 1593 to 1693° C and the stress range 12.4 to 16.5 MPa.

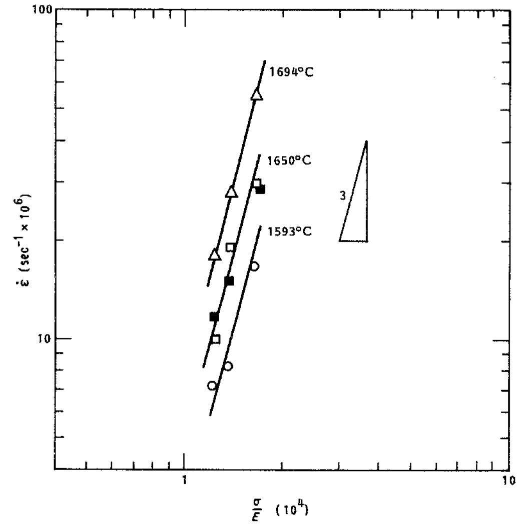

Figure 4. Creep data for wrought (open symbols) and powder-source (closed symbols) C103 in a log $\dot{\epsilon}$ against log σ/E format. Stress exponent values close to three are found at each of the three testing temperatures.

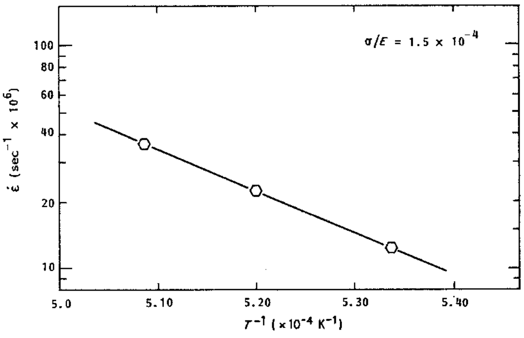

Figure 5. Creep strain rate, $\dot{\epsilon}$, against reciprocal absolute temperature at a fixed value of σ/E for determination of the activation energy for creep of C103 alloy. A value of 316 kJ mol^{-1} is calculated.

sidered an important variable in Class II solid solutions and pure metals, it is not believed to be a controlling factor in the creep behaviour of Class I solid solutions [9, 10]. Despite these differences between Class I and II solid solutions, an empirical equation frequently used to describe the creep behaviour of metals, Class I and Class II solid solutions is the following:

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

where $\dot{\epsilon}$ is the minimum creep rate, σ is the applied stress, E is the average dynamic unrelaxed Young's modulus, n is the stress exponent, Q is the apparent activation energy for creep, R is the gas constant, T is the absolute temperature and A is a constant. In this equation, the stress dependence, n, is found to be three for Class I solid solutions and five for pure metals and Class II solid solutions [9].

To examine the creep behaviour of C103 alloy with respect to this type of formulation, a plot of log σ/E against log $\dot{\epsilon}$ was made for the temperature and stress ranges examined and is presented in Fig. 4. It is often useful to present creep data using a creep stress compensated by the elastic modulus, σ/E, because E, the average dynamic unrelaxed Young's modulus, is known to vary with temperature [9]. In this case, the temperature dependence of E for pure niobium was employed for data analysis purposes [11]. Data for variations in E with temperature for pure niobium were used in lieu of C103 data, because the latter does not appear to exist over the temperature range of interest. It may be seen from Fig. 4 that (a) at each temperature a straight line relationship between log $\dot{\epsilon}$ and log σ/E can be drawn, (b) the slope of these lines is a constant of about three, and (c) the identical behaviour of the powder-source and wrought C103 is again demonstrated. It is possible using standard techniques, to calculate an apparent activation energy for creep, Q, from these results [9]. This is shown in Fig. 5. The value of Q determined in this manner was found to be 316 kJ mol^{-1} *. It may be expected, therefore, that a plot of log σ/E against log $\dot{\epsilon}/D$ (where $D = D_0 \exp(-Q/RT)$ and $D_0 \approx 1$ cm^2 sec^{-1}) would yield a straight line of slope ~ 3. Such a correlation is shown in Fig. 6 for the test data obtained from both wrought and powder-source samples as well as previously reported data in the pertinent temperature range [8, 15]. This latter data is also listed in Table IV.

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*It has been pointed out that in Class I solid solutions, a more precise measure of activation energy is given by [12]: $Q/R = (-\delta \ln \dot{\epsilon})/[\delta (1/T)] - T - 4(\delta \ln E)/[\delta (1/T)]$. This precise formulation is a result of the fact that physical models for deformation of Class I solid solutions incorporate a linear temperature dependence [12–14]. This term, in addition to the modulus correction term, should therefore be substracted from the apparent activation energy if the material behaviour is to be analyzed in the light of such models. Since the correction introduced by this term is small ($\sim$ 10 kJ mol^{-1}) and the present data can be adequately described using the more simple formulation of the empirical equation given in the text. this correction has not been incorporated in the present analysis.

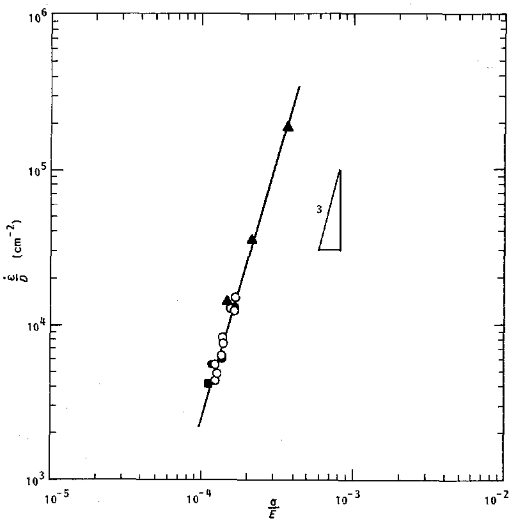

Figure 6. All available creep data on C103 alloy in the temperature range ~ 1400 to 1700° C is shown in a log $(\dot{\epsilon}/D)$ against log (σ/E) format. These data show a stress dependence of about three. The various symbols representing specific investigations are as follows: this work, powder-source (●) and wrought (○); Perkins and Wright (▲), and Klein and Metcalfe (■).

It can be seen that little work has been carried out on the C103 alloy at temperatures relevant to this investigation. Perkins and Wright [8] measured the time to 2% creep strain at several stress levels over the temperature range 1427 to 1650° C. For the purposes of the present investigation, a minimum value of creep rate was assumed to occur within the first 2% of creep strain. Their data, shown in Table IV and Fig. 6, agree well with the data from the present investigation. Klein and Metcalfe [15] have described the effects of various solutes on the creep strength of niobium. A value of creep strength at 1650° C was extracted from their raw data for a Nb-10% Hf binary alloy (which approximates the C103 alloy composition). This value is shown in Fig. 6 and is also found to agree well with the other available work.

TABLE IV Niobium C103 alloy creep data (previous investigations)

Temperature (° C)	Initial* stress (MPa)	σ/E† (10^4)	$\dot{\epsilon}/D$ (10^{-4} cm^{-2})
Perkins and Wright [8]			
1427	37.9	3.70	18.9
1566	21.6	2.14	3.48
1650	14.8	1.48	1.40
Klein and Metcalf [15] (Nb–10 % Hf alloy)			
1650	11.0	1.11	0.42

*Stress for 2% creep strain in ten minutes.

†Modulus data are taken from [11] as follows: $E_{1427°\text{C}} = 102.7$ GPa, $E_{1566°\text{C}} = 102.0$ GPa, $E_{1650°\text{C}} = 99.6$ GPa.

The line drawn through the data in Fig. 6 represents the empirical equation:

$$\dot{\epsilon} = 1.17\times 10^{17}\left(\frac{\sigma}{E}\right)^{3.4}\exp\left(\frac{-316\,\text{kJ}}{RT}\right). \tag{2}$$

The values of A ($1.17 \times 10^{17}\,\text{sec}^{-1}$) and n (3.4) were determined using a least-squares fit for all data. Thus, this equation can be used to describe very accurately the creep of C103 alloy over the stress and temperature range quoted.

The most comprehensive study of the creep behaviour of C103 alloy at low temperatures has been carried out recently by Klopp and Titran [16, 17]. This work covers the temperature range 827 to 1204°C and the stress range 6.89 to 138 MPa. Klopp and Titran thus studied the behaviour of C103 over a markedly different temperature range than that explored in the present work (1593 to 1693° C). Despite this, there is a great similarity to be found in the general creep behaviour of the alloy in the two investigations. For example, Klopp and Titran also found a stress dependence of about three and identified the C103 alloy as a Class I solid solution. In their first report [16], they quoted an activation energy of 315 ± 49 kJ mol^{-1} which is almost identical to that found in the present study (316 kJ mol^{-1}). In their final report, the value reported was either 374 kJ mol^{-1} or 336 kJ mol^{-1} depending upon the method of evaluation [17]. The values of activation energies calculated by Klopp and Titran [16, 17] and the present investigators are thus in reasonable agreement. The creep of Class I solid solutions is generally considered to be controlled by the velocity of dislocations subjected to solute drag [9, 10, 15–17]. Therefore, the activation energy for creep might be expected to be related to the diffusion of hafnium in niobium. Unfortunately, an activation energy value for the diffusion of hafnium in niobium does not appear to exist [18]. The experimentally determined values for activation energy are not inconsistent, however, with the suggestion that the diffusion of hafnium is rate-controlling. The activation energy for diffusion of tantalum in niobium (an element having an atomic weight and size similar to those for hafnium) has been determined to be 415 kJ mol^{-1} [19]. On the other hand, the value for

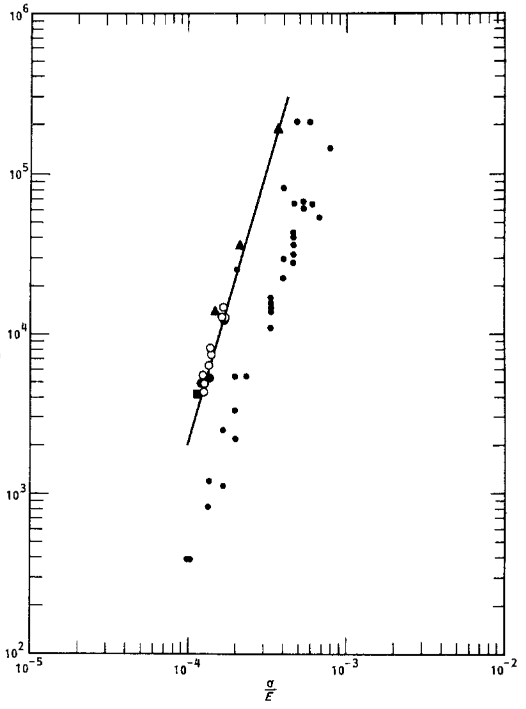

Figure 7. Creep data on alloy C103 in the temperature range 800 to 1700° C are plotted as log $(\dot{\epsilon}/D)$ against log (σ/E). The symbols are the same as described in Fig. 6, with the addition of the work of Titran and Klopp [17] (•).

titanium (of which there is about 1 wt% in C103 alloy) is only 145 kJ mol^{-1}, which makes it unlikely that this element controls creep.

Irrespective of the physical interpretation of Q, it is possible to use the experimentally derived value of Q with that of the stress exponent, n, to predict the behaviour of C103 alloy very satisfactorily as shown in Fig. 6. It is tempting to incorporate the extensive data of Klopp and Titran [16, 17] into the present formulation to see if the equation derived herein can describe the creep data of C103 alloy from 827 to 1693° C. Using the raw data of Klopp and Titran [16, 17], the data from this investigation, and that from the other sources depicted in Fig. 6 [8, 15], a plot of log $\dot{\epsilon}/D$ against log σ/E is shown in Fig. 7. It can be seen that in this case there is now more scatter in the data. The values measured in the present investigation fall within the scatter bands of the data of Klopp and Titran. Thus, it would appear that equation 2 can be used over the temperature range 827 to 1693° C and over the temperature-compensated strain rate $(\dot{\epsilon}/D)$, $\sim 10^2$ to 10^6 to predict the minimum creep rate of C103 alloy.

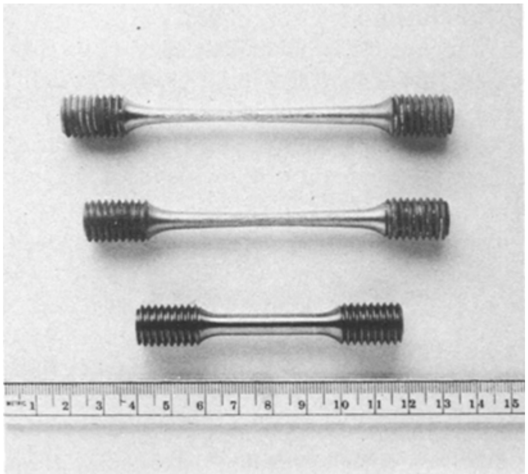

Figure 8. Appearance of wrought alloy C103 specimens before and after creep testing at 1650° C. The bottom sample is in the as-received condition; the centre sample was tested to 100% elongation, and the top sample achieved an elongation of 125% before termination of the test.

There is a final point concerning the behaviour of C103 alloy over this temperature and stress range. The reciprocal of the stress exponent, n, in Equation 2, is the strain rate sensitivity, m, where $\sigma = K\dot{\epsilon}^m$ (K is a constant). Woodford [20] has shown that at high temperatures tensile ductility is directly related to m (which controls the resistance to necking) for a wide range of metals and alloys. Values of m from ~ 0.004 to 0.5 were shown to relate to elongations from $\sim 4\%$ to 2000%. Most metals in creep show values of $n = 5$ ($m = 0.2$) [9] and elongations-to-failure of between 20 and 50%. Class I solid solutions, however, have $n = \sim 3.0$ with strain rate sensitivities of $m = \sim 0.33$, a value that lies between that for pure metals ($m = 0.2$) and superplastic metals ($m = 0.5$) [21]. The very good elongations-to-failure (> 125%) found in C103 alloy in the present study are a direct result of its high strain rate sensitivity. Further, the elongations are in agreement with those reported by Woodford [20] for other alloys having the same value of strain rate sensitivity. This gradual necking behaviour of the C103 alloy, which is a result of the high strain rate sensitivity and leads to high total elongation, is shown in Fig. 8. Uniform deformation was observed for specimen elongations approaching 100%. At about 125% elongation necking was in evidence but sufficient ductility remained such that the test had to be terminated prior to failure.

4. Conclusions

(a) A commercial niobium-base alloy, C103 (Nb + 10 wt% Hf + 1 wt% Ti), has been successfully manufactured from powders using a two-step HIP technique. The powder product is fully dense and has room temperature properties that are comparable to recrystallized wrought bar-stock.

(b) The creep behaviour of both powder-source and recrystallized C103 alloy has been studied over the temperature range 1593 to 1693° C and the stress range 12.4 to 16.5 MPa. The properties of the powder-source C103 were found to be as good as or better than the recrystallized product.

(c) The C103 alloy exhibits Class I solid solution creep behaviour that can be predicted by the empirical equation

$$\dot{\epsilon} = A\left(\frac{\sigma}{E}\right)^n \exp\left(-Q/RT\right).$$

The constants in this equation have been determined to be; $n = 3.4$, $Q = 316\,\mathrm{kJ\,mol^{-1}}$, and $A = 1.17 \times 10^{17}\,\mathrm{sec^{-1}}$.

(d) The high values of strain rate sensitivity ($m = \sim 0.33$) in C103 alloy lead to very high creep rupture ductility (> 125%).

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