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Abstract: The present study provides a feasible method to evaluate creep properties for a 9%Cr-Mo-Co-B power plant steel by comparing two sets of data obtained from small punch tests and conventional uniaxial creep tests. The method includes three steps: firstly, conduct a series of small punch tests and conventional creep tests in different load and temperature conditions; secondly, convert the load and central deflection data obtained from the small punch test to stress and strain data; thirdly, determinate the best fit correlation factor by comparing the two sets of data in selected creep models. It is found that two sets of data show a similar trend in stress–rupture time relation, stress–minimum strain rate relation and LMP–stress relation. The correlation factor, k_{sp} , can effectively bridge the gap between the load in small punch test and the stress in conventional creep test. For a high-Cr martensitic heat-resistant steel named as CB2, the k_{sp} value 1.4 can make a good prediction for rupture time, while for minimum creep rate and the Larson–Miller parameter, the k_{sp} value 1.4 will lead a conservative prediction in the low-stress range.

Keywords: creep test; small punch test; 9%Cr-Mo-Co-B steel

1. Introduction

In the background of increasing emphasis on energy conservation and pollution reduction all over the world, the operating temperature of components in steam power plants exceeds 600 °C and continues to rise in the ultra-supercritical (USC) units in order to achieve the desired overall efficiency and save fuel costs [1–3]. Materials development of creep-resistant steels for the plant applications at high temperatures has been a hot topic in the last several decades [4–8]. Steel CB2 is developed in the frame of the COST programs initiated from 1990s in Europe [9], and it has become the most promising candidates for production of cast turbine components working in USC steam conditions [10]. Steel CB2 belongs to the class of 9–12% Cr steels widely used in USC power plants. As a result of numerous trials in the COST programs, 9Cr-1Co-100ppm Boron composition is selected for the casting steel CB2 and forging steel FB2 that are suited for application at high temperatures up to 620 °C.

The selection of steels for power plant service is usually based on the requirement that creep failure should not occur under the prevailing operating conditions during plant lives of ~250,000 h (30 years). Design calculations generally are based on the stresses causing creep failure in 100,000 h [11]. Therefore, creep property assessment over the operating temperature range is crucial in the development of these steels. In order to evaluate the creep resistance of a material, a conventional creep test is commonly performed under specific stress and temperature. However, as we all know, the conventional creep test is time-consuming, and it takes a great deal of manpower and resources. In fact, creep test for steel CB2 is still in progress, as far as we know, so it may take decades for a power-plant steel to be put in actual use limited by the long period of creep test. Moreover, the



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conventional creep test needs a full-size specimen, which is not only material consuming but also impossible in many cases involving a remaining-life assessment of in-service components.

In recent years, the small punch (SP) method has been introduced to the creep test by many researchers all over the world [12-17]. It is a quick laboratory test that uses miniature-sized specimens, usually a small disc with a diameter of 8–12 mm and thickness of 0.5 mm. Its sampling does not cause any serious damages, and this is crucial for the assessment of components in service. However, the results of the small punch test cannot be applied to engineering designs directly in view of the different stress states between the SP test and conventional creep test [16]. Though several studies on the SP test have been performed, the relationship in a wide range of condition is not clarified. For the aim of applying the results from the SP test, it is important to evaluate the relationship between the two sets of test results in at least the following two relations: SP load and uniaxial creep stress; SP central deflection and strain. Many researchers have made efforts to determine the correlations between the SP load and uniaxial creep stress [18–20]. One of the most widely used methods is summarized in the standard CWA 15627: small punch test method for metallic materials [21], in which a correlation factor between SP load and uniaxial creep stress is introduced. However, the correlation factor varies with materials and conditions, and there is no practical method to get an accurate value of the correlation factor. On the other hand, steel CB2 is a potential alloy that will be widely used in USC plant components in China [22]; thus, it is urgent to discover a low-cost assessment method for creep properties that could replace the conventional creep test.

Therefore, this paper investigates the creep properties of steel CB2 both in the conventional uniaxial tensile condition and in small punch creep condition. By comparing the two sets of data in the creep models, a relationship between the two test methods can thus be established from the perspective of optimizing the correlation factor.

2. Materials and Methods

2.1. Materials

The steel CB2 used in the present work was supplied by Dongfang Turbine Co., Ltd., (Deyang, China) of Dongfang Electric Corporation in the state of cast parts. Its chemical composition is given in the Table 1. Heat treatments on the cast parts were conducted in the following condition: 1130 °C/10 h/FAC + 740 °C/10 h/FC (h, hour; FAC, forced air cooling; FC, furnace cooling). Metallographic examination was performed afterwards by optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM), according to the standard procedure, and several quintessential microstructures of steel CB2 on different scales are shown in Figure 1. As can be seen, the microstructure consists of blocks of tempered martensite laths which are distributed in the coarse prior austenitic grain or at the prior austenitic grain boundaries; and fine dispersed particles in the matrix, which are verified to be $M_{23}C_6$ and MX compounds (M—metal; X—C, N) [10,23]. Specimens for the small punch test and uniaxial creep test were cut from the cast parts.

Table 1. Chemical composition of steel CB2 for creep test (in weight, wt%).

Element	С	Si	Mn	Cr	Мо	Ni	Со	V	Nb
Wt%	0.13	0.27	0.93	9.1	1.49	0.13	0.93	0.21	0.076
Element	Ν	В	Al	Sn	Cu	As	Ti	Р	S
Wt%	0.022	0.0093	0.0044	0.0018	0.031	0.01	0.0017	0.012	0.006



Figure 1. Original microstructure of steel CB2 for creep tests: (**a**) an OM graph shows the boundaries between four coarse prior austenitic grains, (**b**) a SEM graph shows the local microstructural details and (**c**) a HRTEM graph shows dispersed particles no the nanoscale in the matrix.

2.2. Small Punch Creep Test

The small-punch-creep experimental setup was constructed according to standard CWA 15627: a code of practice for small punch creep testing. The whole setup includes a testing rig, loading system, displacement measurement system, heating system and argon shield system. Figure 2 schematically illustrates a cross-sectional view of the testing rig, which includes specimen holder (dies), a ceramic ball indenter of diameter 2.5 mm, the disc specimen that is 8 mm diameter and has an initial thickness of 0.5 mm, and a dilatometer quartz rod. Prior to the test, the holder was forced to clamp the disc specimen by pretightening bolts in order to limit specimen deformation in the region at the hole of the lower die. The receiving aperture of the radius, R, is 2 mm, with a 45° chamfer at R + 0.2 mm. A nickel-based superalloy was used to fabricate the upper and lower die to avoid excessive oxidation during the long test time. A spherical ceramic ball indenter was used to force the central portion of the test disc specimen through the aperture in the receiving die until the end of the test occurred. The diameter of the ceramic ball is 2.5 mm. The setup was loaded directly by different weights. During the test, a displacement sensor with a high precision of 0.001 mm worked in a continuous fashion. The disc central deflection can thus be recorded by a quartz rod directly contacted with the displacement sensor. For convenience of providing an inert environment, the whole test rig was put into a sealed quartz tube, which could be filled with argon during the test in order to avoid severe oxidation of the disc [24], and the tube was heated by an encircling furnace to provide a uniform temperature distribution throughout the test section of the disc. The small punch test was conducted under the condition of a temperature range of 580–650 °C and load range of 400-700 N.



Figure 2. Cross-sectional view of the testing rig in a small-punch-creep-test setup.

2.3. Uniaxial Tensile Creep Test

The uniaxial tensile creep test adopts conventional method according to Chinese Standard GB/T 2039-2012, which is equivalent to ISO 204:2009 metallic materials/uniaxial creep testing in tension. The uniaxial-creep-test specimen is 8 mm in diameter, and the gauge length is 50 mm. The test is conducted under the condition of temperature range 600-650 °C and stress range of 150–260 MPa.

3. Results and Discussion

3.1. Small Punch Creep Test

Figure 3 shows the relationship between the central deflection and loading time under different loads at 620 °C. The curves were plotted by using the data obtained directly from the small punch test. As can be seen, the time–deflection curves of steel CB2 are similar to those of typical uniaxial creep tests, which include three stages of deformation, i.e., the first attenuation stage, the second steady stage and the third acceleration stage, and a steady state deflection rate can also be observed in the secondary stage, which can be related to the minimum creep rate in uniaxial creep tests. Table 2 lists the rupture time (hours) both for uniaxial creep and for small punch creep at testing temperatures and stresses/loads, and this can be used for the discussion in the following parts.



Figure 3. Time-deflection curves of steel CB2 under different loads at 620 °C.

Table 2. Rupture time (hours) for uniaxial creep and for small punch creep at testing temperatures ($^{\circ}$ C) and stresses (MPa)/loads (N).

	Stress (MPa)									
Temperature	150	185	190		229	240	260			
600	-	2603	-		265.44	-	90.71			
620	1812	-	327.03		-	60.77	-			
650		-	60.17		-	-	8.55			
		Load (N)								
Temperature	400	450	500	550	600	650	700			
580	-	-	-	765.16	565.05	293.96	231.66			
600	-	-	698.93	298.58	133.19	102.78	81.23			
620	-	-	-	168.04	67.71	50.31	24.46			
650	240.72	40.53	18.91	-	-	-	-			

3.2. Comparative Study on the Two Sets of Data Both in Small Punch Tests and Uniaxial Tests

In this part, the relationship of the creep properties between the small punch test and uniaxial creep test are established by a comparative study on the two sets of data. In standard CWA 15627, the applied load in the small punch creep test has to be determined from geometrical factors and material properties in order that creep failure in the small punch test will occur at the same time as that in a conventional uniaxial creep test at the same temperature. For the case where there is no prior information on expected behavior, the ratio of SP test load (F) to the uniaxial creep stress (σ) is given by Equation (1):

$$F/\sigma = 3.33k_{sp}R^{-0.2}r^{1.2}h_0 \tag{1}$$

where *R* is the radius of the receiving hole, *r* is the radius of the punch indenter, h_0 is the test-piece thickness and k_{sp} is small punch creep test correlation factor. This relationship is derived from the Chakrabarty's stretch membrane theory and complicated regression analyses [25,26]. The empirical correlation factor, k_{sp} , is introduced in order to take into account the different creep ductilities of different materials. In the present study, R = 2 mm, r = 1.25 and $h_0 = 0.5$ mm, so $F/\sigma = 1.8945k_{sp}$.

By using the above formula, the dependence of the rupture time, t_r , on the loading stress (σ) both in the conventional uniaxial creep test and small punch test at 620 °C is given in Figure 4. By setting different values of k_{sp} between 1.0 and 1.4, five groups of SPT data are shown in the figure. As can be seen, the dependences in both sets of data can be described by the power-law relationships given by Equation (2):

t

$$r = A\sigma^n$$
 (2)

where *A* is temperature dependent factor. It is found by fitting the data that there is a good linear relationship between the rupture time and creep stress in both loading conditions in the double logarithmic coordinates. The absolute value of exponent *n* for small-punch-test data (n = -7.6) is slightly higher than that for uniaxial creep test data (n = -7.3) at 620 °C. By comparing the fitting lines between the two sets of data, it is found that the k_{sp} value of 1.4 predicts a good agreement in rupture time between the small punch creep tests and conventional uniaxial tests at 620 °C.



Figure 4. Dependence of rupture time on creep stress (or predicted stress for SP load) for uniaxial test and small punch test in different values of k_{sp} .

In conventional creep tests, the minimum creep rate is an important parameter that is always applied to investigate the residual creep life of key components [27]. However, in the small punch test, only the central deflection data are obtained. They should be converted into strain data, so as to compare them with those obtained from the conventional uniaxial creep test. References [28,29] provided a way to build the relationship between deflection and strain. By adopting the method in the references, we determined that the relationship between central deflection, δ , and strain, ε , in the present study is given by Equation (3):

$$\varepsilon = 0.23381\delta^2 + 0.40616\delta \tag{3}$$

By using the above formula, the minimum deflection rate, δ_m , can be related to the minimum creep rate, $\dot{\epsilon}_m$; thus, the dependence of the rupture time on the minimum creep rate (or converted creep rate for SP deflection) for the uniaxial test and small punch test at 620 °C can be plotted in Figure 5. It can be seen that there is a good linear relationship in the double logarithmic coordinates between rupture time and minimum creep rate for both sets of data, meaning that the creep data fit in well with the Monkman–Grant relation both in the uniaxial creep test and in the small punch creep test.



Figure 5. Dependence of rupture time on minimum creep rate (or converted creep rate for SP deflection) for uniaxial test (**a**) and small punch test (**b**) at 620 °C.

The dependence of \hat{e}_m on the creep stress or predicted stress, σ , in the conventional uniaxial creep test and small punch test at 620 °C is plotted in Figure 6. By setting different values of k_{sp} between 1.0 and 1.4, five groups of stress values predicted by SPT load data are shown in the Figure 6. As can be seen, the relationship between the minimum creep rate and creep stress shows a similar manner to those in Figure 3, and they can also be described by the power-law relationship given by Equation (4):



Figure 6. Dependence of minimum creep rate (or converted strain rate for SP deflection) on creep stress (or predicted stress for SP load) for uniaxial test and small punch test in different values of k_{sp} .

$$\dot{\varepsilon}_{mr} = A' \sigma^{n'} \tag{4}$$

As shown in Figure 6, in the double logarithmic coordinates, there is a good linear relationship between the minimum creep rate and creep stress in both loading conditions. However, there is a significant difference in the values of exponent n' for small-punch-test data (n = 9.3) and for uniaxial-creep-test data (n = 16.6), thus indicating that there would be a notable deviation in the wide stress range to predict the minimum creep rate by using small-punch deflection data for steel CB2, and that the creep deformation mechanism may be different. The difference in the mechanism is not discussed further here. Nonetheless, by comparing the fitting lines between the two sets of data, it is revealed that the minimum creep rates predicted by small punch tests using $k_{sp} = 1.4$ show a good agreement with that in conventional uniaxial tests in the range of middle-stress values (180~210 MPa). On the high-stress side, the minimum creep rate predicted by the small punch test using $k_{sp} = 1.4$ is slightly lower than that in the uniaxial tests, so a larger k_{sp} value should be used to make accurate predictions. Meanwhile, on the low-stress side (stress values below 180 MPa), using $k_{sp} = 1.4$ would lead to a conservative prediction from the perspective of structure safety, considering that the minimum creep rate predicted by the small punch test using $k_{sp} = 1.4$ is higher than that in the uniaxial tests.

3.3. Larson-Miller Parameter Method

One method of extrapolating short-time creep data to predict longtime life involves the use of a time-temperature parameter (TTP). In past decades, several TTPs have been proposed to investigate the relationship between the creep-rupture time and the testing temperature in metallic materials such as Larson–Miller parameter (LMP), Fisher–Dorn parameter, Manson–Haferd parameter, Orr–Sherby–Dorn parameter and Sub-Aviation parameter [30,31]. Such TTPs consider the combined effect of time and temperature, so all creep-rupture-time data for a given material can be correlated to produce a single curve wherein the stress (or log stress) is plotted against the TTPs. These parametric methods have the great advantage of requiring only a relatively small amount of data to establish the required curve. Therefore, TTPs are generally used to extrapolate or interpolate the available creep-rupture data. Among various TTPs, the LMP is famous for its simplicity and ease of use. Larson and Miller first introduced the concept of time–temperature grouping in 1952 [32], according to which the LMP, as a function of stress, is related to the time to fracture and test temperature in the form of Equation (5):

$$LMP = T(K + \log t_r)$$
(5)

where t_r and T are creep-rupture time in hours and test temperature in Kelvin, respectively. K is a material constant, and the initial value of K was used based on general suggestion of K = 20. In the following part, Larson–Miller parameter method is employed to evaluate the applicability of the relationship in Equation (1) for steel CB2 and also to determine the applicable value of correlation factor k_{sp} .

According to Equation (5), LMPs in each load level for small punch tests and uniaxial tests are calculated and plotted in Figure 7. It can be seen that LMPs increase linearly as creep stress decreases in both loading conditions, which means that TTPs method can also be applied to small punch test. By comparing the two sets of data, it is found that in range of high stress values there is a good agreement between the two sets of data using $k_{sp} = 1.3-1.4$, while $k_{sp} = 1.0-1.2$ can make a good prediction in low-stress values. In a wide range of creep stress, a conservative prediction can be obtained by using $k_{sp} = 1.4$.



Figure 7. Dependence of creep stress (or predicted stress for SP load) on Larson–Miller parameter for uniaxial test and small punch test in different values of k_{sp} .

4. Conclusions

(1) The methods of extrapolation of the creep results based on power-law relationships and the Larson–Miller parameter method can also be used for describing the data obtained by the small punch test, considering the following findings: The deflection curves of the small punch test are similar to those of the conventional creep test, which shows three typical creep stages. Both sets of data show a similar trend in stress–rupture time relation, stress–minimum strain rate relation and LMP–stress relation.

(2) The correlation factor, k_{sp} , can effectively bridge the gap between the load in the small punch test and the stress in the conventional creep test. For steel CB2, the k_{sp} value of 1.4 can make a good prediction for rupture time. For the minimum creep rate and Larson–Miller parameter, the k_{sp} value 1.4 will lead a conservative prediction in the low-stress range.

(3) It is a feasible method to evaluate the creep properties of steel CB2 by small punch tests. The method includes three steps: firstly, conduct a series of small punch tests and conventional creep tests in different load and temperature conditions; secondly, convert the load and central deflection data obtained from the small punch test to stress and strain data; thirdly, determinate the best fit correlation factor by comparing the two sets of data in creep models.

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