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A CREEP-PROPERTIES EVALUATION OF P91 STEEL WELDMENTS USING SHORT-TERM TESTING

OCENITEV LASTNOSTI LEZENJA VARA IZ JEKLA P91 S KRATKOTRAJNIMI PRESKUSI

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A key factor in any power station is the choice of materials designed to operate at the highest possible steam temperature consistent with reasonable component weights and thicknesses. Over the last two decades a new generation materials, particularly in the form of modified 9Cr-1Mo steels, have been widely used for replacement equipment and for new constructions.

This paper is concerned with the creep properties of modified P91 steel weldments, where the application of the small-punch test method for the assessment of material creep properties is used. Experiments have shown that the small-punch test can be used to describe the time-to-failure by means of an equation of the Dorn type, in which stress is replaced by load. The tests were carried out at temperatures from 560 °C to 640 °C and at loads from 460 to 610 N. The disc-shaped test specimens (ϕ8 mm x 0.5 mm) were machined from pure welds. There were three different states of welds: as-welded and with two different post-weld heat treatments (PWHTs). For small-punch tests less material is needed to establish creep activation energies and load exponents than with conventional creep tests. The conventional constant-load creep tests were performed at 600 °C and 620 °C under applied stresses ranging from 130 to 150 MPa. The microstructures of all the samples were also examined before and after the creep testing.

Key words: P91 steel, weld, small-punch, short term creep testing

V vsaki termoelektrarni je ključnega pomena izbor materiala, s katerim naj sistem pri razumnih težah in debelinah komponent deluje pri najvišjih možnih temperaturah. V zadnjih dveh desetletjih se za nove konstrukcije in zamenjave opreme veliko uporablja nova generacija materialov, največ v obliki modificiranih jekel 9Cr-1Mo.

V tem prispevku je narejena raziskava o lastnostih lezenja varov iz dodajnega materiala iz modificiranega jekla P91, pri čemer je bila za oceno lastnosti lezenja materiala uporabljena metoda merjenja z uporabo majhnega bata (small-punch). Eksperimenti so pokazali, da lahko s preskusi "small-punch" opišemo čas do loma z enačbo Dornovega tipa, v kateri napetost zamenjamo z obremenitvijo. Preskusi lezenja so bili narejeni pri temperaturah od 560 °C do 640 °C in pri obremenitvah od 460 N do 610 N. Preizkušanci v obliki diska (ϕ8 mm x 0,5 mm) so bili izrezani iz čistega vara. Zvari so bili v treh stanjih, in sicer v varjenem stanju in z dvema različnima toplotnima obdelavama. Pri preskušanju z majhnim batom potrebujemo mnogo manj materiala za ugotovitev aktivacijske energije lezenja in obremenitvenega koeficienta kot pa pri konvencionalnih preskusih lezenja. Poleg preskusov z majhnim batom smo izvedli tudi konvencionalne preskuse lezenja pri konstantni obremenitvi pri temperaturah 600 °C in 620 °C ter pri uporabljenih napetostih od 130 MPa do 150 MPa. Mikrostrukturo preizkušancev smo pregledali pred meritvami lezenja in po njih.

Ključne besede: jeklo P91, var, majhni bat, kratkotrajni preskusi lezenja

1 INTRODUCTION

A number of test techniques for extracting mechanical property data from small volume specimens are under development [1,2]. The small-punch creep method is a test technique originally developed for estimating the fracture-appearance transition temperature [3]. In the small-punch test a thin circular disc is supported over a receiver hole and forced to deform into the hole by a spherical penetrator [4-6]. When the evaluation of the observed dependence of the minimum displacement rate upon stress and temperature was performed, we found, that the load exponent and the measured activation energy were typical of the values for the investigated experimental steel. Over the last two decades a new generation of materials, particularly in the form of modified 9Cr-1Mo [7-10], have been widely used for replacement equipment and for new constructions. In the last few years a lot of effort was put into development of new welding consumables for high-temperature applications. We have tested an existing material for possible use in high-temperature applications.

2 EXPERIMENTAL PROCEDURE

For these experiments we used electrodes of modified P91 steel for the weldments. We also prepared buttered carbon steel plates 20 mm thick with 21 passes. The specimens for the small-punch creep measurements, with dimensions of ϕ8 mm × 0,5 mm, were machined from pure welds (**Figure 1a**). The relatively coarse martensitic microstructure of the experimental P91 steel weldment in the as-welded condition is shown in **Figure 1b**. The welds were in three different conditions, i.e. as-welded and with two different post-weld heat treatments: PWHT-1 (2h/760 °C, air); PWHT-2 (2h/760 °C, 150 °C/h till 300 °C, air) (**Figure 1c**). The chemical

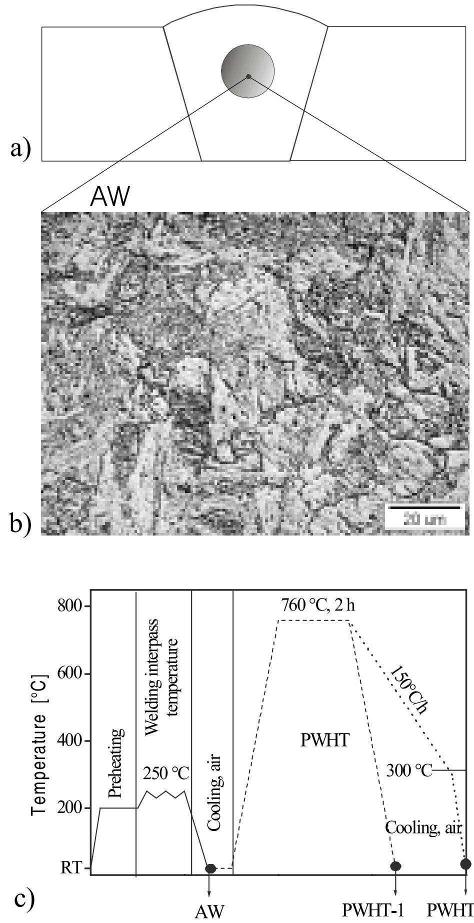

Figure 1: P91 steel weldment: a) Schematic presentation of pure weld and location of the specimens, b) The microstructure in the as-welded condition (AW), c) Welding parameters
Slika 1: Var iz jekla P91: a) shematski prikaz čistega vara in lokacija preizkušancev, b) mikrostruktura v varjenem stanju (AW), c) parametri varjenja

composition of the P91 steel in wt. % is as follows: C=0,06; Mo=1,0; Si=0,36; Mn=0,6; P=0,015; Cr=9,5; Ni=0,95 and V=0,21.

The experimental work consisted of tests on small-punch test equipment (**Figure 2**). The tests were carried out at temperatures from 560 °C to 640 °C, and at loads from 430 to 610 N. Under each test condition at least three tests were performed. The test specimens (disc specimens of diameter D = 8 mm and thickness t = 0,5 mm) were placed on the central axis of the lower die of the specimen holder and fixed by the upper die so that there was a loose fitting, i.e. neglecting friction between the upper die and the specimen. The ball and the puncher were inserted into the hole in the upper die of the holder. During the test a constant load acted on the specimen by means of a ceramic ball of diameter d = 2,5 mm. During the initial step of loading, the specimen rapidly deformed plastically into the hole in the lower die, the stresses in the disc were reduced, and further deformation of the disc occurred only as a result of creep. The diameter of the hole was a = 4 mm, and its shoulder radius was R = 0,2 mm. The temperature of the specimen was measured with a thermocouple in the direct vicinity of the specimen, the permissible variation in temperature being ± 1 °C. The displacement of the punch, i.e. the central deflection of the disk specimens, was measured using an inductive transducer with a high measuring accuracy (repeatability approximately 1 μm), and was recorded continuously by a computer.

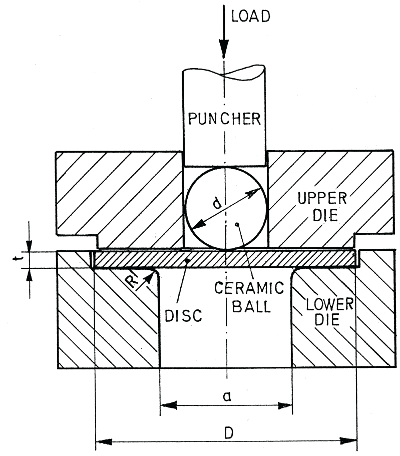

Figure 2: Schematic illustration of the dies in the small-punch test equipment
Slika 2: Shematični prikaz orodja majhnega bata

Before and after the creep testing of the specimens of the P91 steel weldments, high-resolution imaging and Auger-electron spectroscopy (HRAES) were performed using a static electron beam with 10 keV/1nA of a diameter of about 10 nm. For each specimen several characteristic regions of the microstructure were selected for multi-point analysis.

3 RESULTS

The mechanical properties of the experimental P91 all-welded steel in the as-welded condition and with two different PWHTs at different testing temperatures are listed in **Table 1**. From the mid section of the deposited all-weld metal Charpy V-notch test specimens were made for testing all three different welding conditions. The Charpy impact tests were performed over a temperature range in which the transition from tough to brittle behaviour is expected (**Figure 3**).

Short-term creep tests were performed on a small-punch test device. The dependence of the applied load P on the time-to-rupture t_r in the small-punch creep tests can be described by means of a modified form of the Dorn equation, i.e. an Arrhenius-type equation (4):

$$t_r = B \cdot P^{-n} \cdot \exp\left(\frac{Q}{RT}\right) \qquad (1)$$

where P is the load (N) acting on the disc, Q is the activation energy (kJ/mol), n is the load exponent, R is the universal gas constant (R=8,314 J/molK), T is the

Table 1: Mechanical properties of the P91 steel weldment at different temperatures
Tabela 1: Mehanske lastnosti vara iz jekla P91 pri različnih temperaturah

Welding conditions	Temp. (°C)	Yield stress (MPa) Measured		[11]	Tensile strength (MPa) Measured		[11]	Elongation (%)	Reduction of area (%)	Hardness HV3
As welded	20	844	>	550	1057	>	700	14	43	353
	500	750	>	380	944	>	440	8	32	
	620	343	>	280	358	>	330	17	77	
PWHT-1	20	601	>	550	708	>	700	20	66	229
	500	416	>	380	478	>	440	17	65	
	620	214	<	280	229	<	330	26	88	
PWHT-2	20	487	<	550	630	<	700	27	69	228
	500	366	<	380	439	<	440	19	69	
	620	215	<	280	225	<	330	24	88	

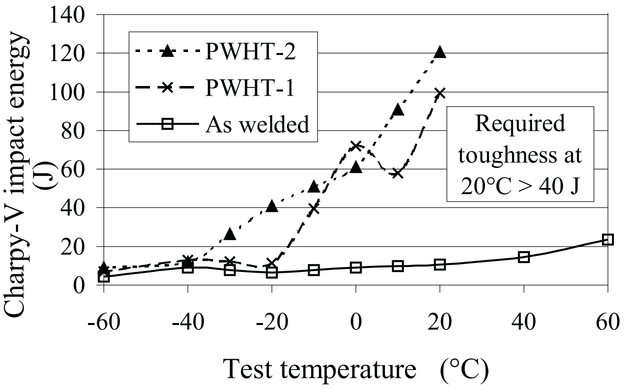

Figure 3: Charpy-V impact energy of P91 steel weldments
Slika 3: Udarna energija Charpy-V varov iz jekla P91

absolute temperature (K) and B is the constant of the modified Dorn equation.

Examples of the time dependence of the punch displacement, i.e. of the central deflection in the small-punch test at a load of 580 N and temperatures of 580, 600 and 620 °C, respectively (a); and at a temperature of 600 °C and loads of 550, 580 and 610 N, respectively (b); are given for the P91 steel weldment in the as-welded condition in **Figure 4**. From the tests under constant load with three different temperatures we can calculate the line slope in the diagram ln(t) vs. $1/RT$, which represents the activation energy Q (**Table 2**). In a similar way we can calculate the load exponent n (the line slope in the diagram $\ln(t)$ vs. $\ln(P)$) from tests at constant temperature with three different loads (**Figure 5**).

Table 2: The activation energy determined with the small-punch testing method
Tabela 2: Aktivacijska energija, določena z metodo preskušanja z majhnim batom

Material conditions of P91 steel weldment	Apparent activation energy Q (kJ/mol)
As welded	352 ± 4
PWHT-1	476 ± 151
PWHT-2	554 ± 24

The curves of the time dependence of the small-punch displacement, i.e. the central disc deflection at a load of 520 N and a temperature of 600 °C of the P91 steel weldment for the as-welded condition and for two different PWHTs, respectively, are given in **Figure 6**. It can be seen that the same general features of the curves can be observed as in conventional creep tests. However, the registered curves have a very pronounced stage of primary creep in which the deflection rate decreases by several orders of magnitude (initial hot

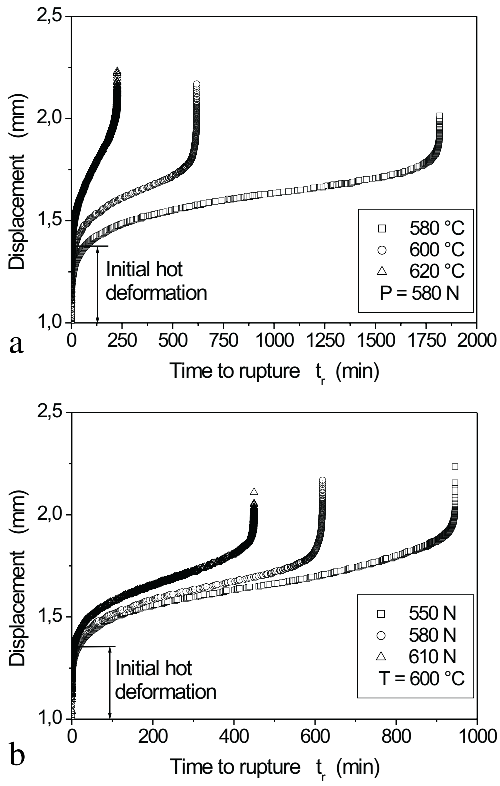

Figure 4: Central disc displacement vs. time in the small-punch test at different temperatures and loads. The P91 steel weldment is in the as-welded condition. a) P = const. = 580 N, b) T = const = 600 °C
Slika 4: Upogib diska v odvisnosti od časa pri preskusu z majhnim batom pri različnih temperaturah in obremenitvah, var iz jekla P91 v varjenem stanju: a) P = konst. = 580 N, b) T = konst = 600 °C

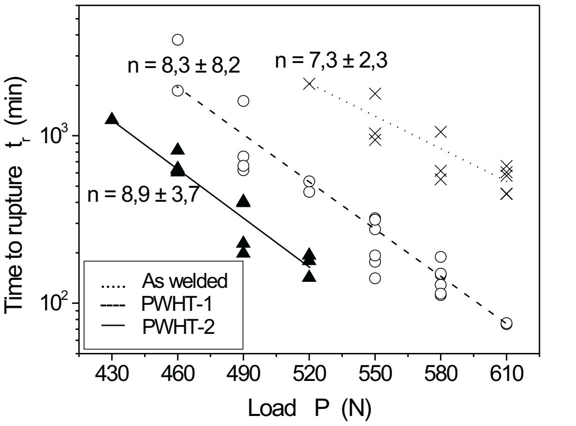

Figure 5: Load exponent *n* for P91 steel weldments in three different heat-treatment conditions

Slika 5: Obremenitveni koeficient *n* za var iz jekla P91 v treh različnih toplotnih obdelavah

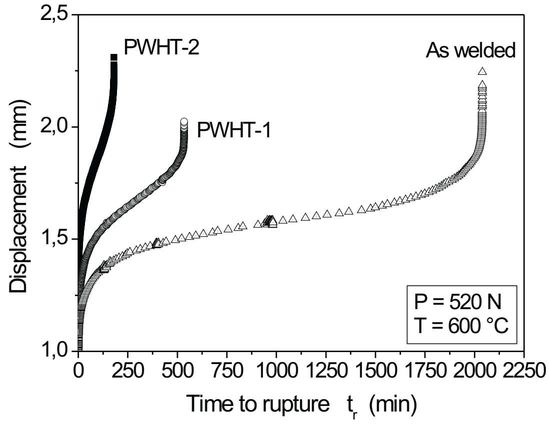

Figure 6: Time dependence of the central disc displacement in the small-punch test for the P91 steel weldment for three different heat-treatment conditions

Slika 6: Časovna odvisnost upogibanja diska pri preskusu z majhnim batom za var iz jekla P91 pri treh različnih toplotnih obdelavah

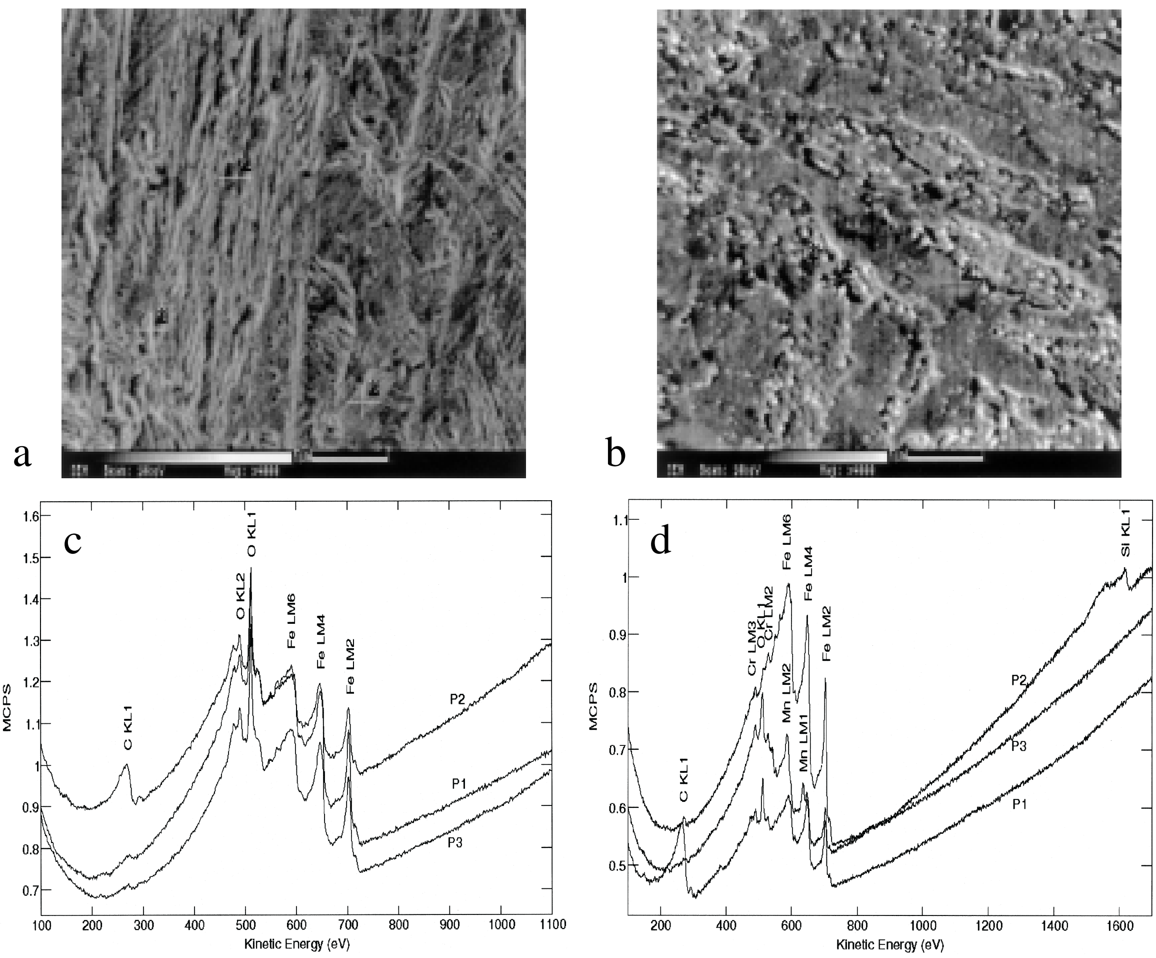

Figure 7: HRAES spectra of as-welded and crept specimens of P91 steel weldment shows chromium peaks that indicate a carbide precipitation process: a) SEM, as-welded, b) SEM, as-welded after 2040 min creep test, c) HRAES, as-welded, d) HRAES, as-welded after 2040 min creep test

Slika 7: HRAES-spekter varjenega in lezenega preizkušanca iz vara iz jekla P91 kaže kromove vrhove, ki indicirajo proces precipitacije karbidov: a) SEM, varjeno stanje, b) SEM, varjeno stanje po 2040-minutnem preskusu lezenja, c) HRAES, varjeno stanje, d) HRAES, varjeno stanje po 2040-minutnem preskusu lezenja deformation). The steady-state creep is missing but the minimum deflection rate can be evaluated.

In parallel with the small-punch tests we also performed conventional constant-load creep tests with some lower stresses. The results (time-to-rupture) we obtained were very different to the results of the small-punch creep tests. The results are presented in **Table 3**.

Table 3: Time to rupture for conventional constant-load creep tests
Tabela 3: Čas do loma pri konvencionalnih preskusih lezenja s konstantno obremenitvijo

Material conditions of P91 steel weldment	Time to rupture at σ = 130 MPa, T = 620 °C
As welded	381,6 h
PWHT-1	26,6 h
PWHT-2	30,6 h

4 DISCUSSION

The results of the Charpy-V impact energy (**Figure 3**) show that the P91 steel weldment has toughness in the as-welded condition that is too low for crack-free welding. Both PWHTs of the P91 steel weldments ensure toughness higher than 40 J, which is recommended for welding in high-temperature applications [11]. The mechanical properties of P91 (**Table 1**) in comparison with the producer's data [11] shows that at room temperature the yield and the tensile strength are high enough for as-welded and PWHT conditions. But at 620 °C only the as-welded microstructure has the required yield and tensile strength, while PWHT-1 and PWHT-2 do not reach the minimum required level.

The short-term creep tests show interesting results. The comparison of the calculated activation energies Q of the P91 all-welded steels for different heat-treatment conditions shows great variation in the values obtained. Because of this it is better to talk about apparent activation energies. We can also find differences in the calculated load exponents, but these are not so large. The comparison of average time-to-rupture for the same small-punch testing parameters (P=520 N, T=600 °C) shows that in the case of PWHT-1 the time to rupture is 498 minutes, and in the case of PWHT-2 it is 171 minutes. In other words, for PWHT-1 the time to rupture is almost three-times longer than in the case of PWHT-2.

However, the results are somewhat different for conventional constant-load creep testing using the same testing parameters (**Table 3**), where for both PWHTs very similar times to rupture are measured. In the case of the conventional creep testing the role of yield stress is shown in the time-to-rupture. The yield stress of the as-welded material is much higher than it is in the case of a different PWHT, and because of this the time-to-rupture is approximately 14 times longer than for both cases of PWHT.

The established discrepancy in the activation energy Q as well as in the power n between the as-welded and the PWHT microstructure obtained with the small-punch creep testing could also be partially explained by the unstable character of the as welded microstructure. Namely, the possible precipitation-hardening reactions (precipitation of vanadium carbides for instance [12]) in thermodynamically unstable microstructure of as-welded specimens for very short testing periods can coincide with the main creep reaction of the actual creep mechanism and can provoke some disturbances in the creep behavior. High-resolution imaging and analysis (HRAES) of the as-welded microstructure of the P91 steel before testing and after 2040 minutes of small-punch creep testing shows a large difference in precipitation-hardening kinetics (**Figure 7**). The HRAES spectra of the as-welded microstructure after 2040 minutes of creep testing (**Figure 7d**) shows chromium peaks that indicate a carbide precipitation process. According to these results the hypothesis of a disturbing effect of the precipitation-hardening reactions on the rupture time of small-punch creep testing of an as-welded microstructure is well established. Therefore, a high applied load, i.e. a short rupture time during creep measurement of an all-welded microstructure should be avoided.

5 CONCLUSIONS

We have investigated the possibilities of using the small-punch creep-testing method for the assessment of as-welded material properties. The main advantage of small-punch creep tests in comparison with conventional constant-load creep tests is the small amount of material required for testing to establish the creep activation energies and the load exponents. A large discrepancy between the results obtained for small-punch and conventional creep testing was observed. This discrepancy is caused by the precipitation-hardening reactions (the precipitation of chromium carbides) in the thermodynamically unstable microstructure of as-welded specimens during very short testing periods, which coincide with the main creep reaction of the actual creep mechanism and therefore induce disturbances in the creep behavior. Of course it was established that the material characteristics obtained during small-punch creep measurements are relevant if the times-to-rupture are not too short. Only when the stress in the specimen is low enough, the values of the activation energy approach the values of self-diffusion. A comparison of small-punch creep-testing results and conventional creep-testing results is only possible when very similar and sufficiently long times-to-rupture under both testing methods are used.

ACKNOWLEDGEMENTS

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