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

OCENITEV LASTNOSTI LEZENJA VARA IZ JEKLA P91 S KRATKOTRAJNI MI PRESKUSI

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1 INTRODUCTION

A number of test techniques for extracting mechanical property data from small volume specimens are under development. The small-punch creep method is a test technique originally developed for estimating the fracture-appearance transition temperature. 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. 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 steel. Over the last two decades a new generation of materials, particularly in the form of modified 9Cr-1Mo steels, have been widely used for replacement equipment and for new constructions.

1b 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 8 mm x 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 properties of modified 9Cr-1Mo steels, have been widely used for replacement equipment and for new constructions.
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 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.

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 \(t\) 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):

\[
t_r = B \cdot P^{-n} \cdot \exp \left( \frac{Q}{RT} \right)
\]

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
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 4. 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

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

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<th>Conditions of P91 steel weldment</th>
<th>Apparent activation energy (Q) (kJ/mol)</th>
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<td>As welded</td>
<td>352 ± 4</td>
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<td>PWHT-1</td>
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**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

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**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 preizkušanja 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

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<th>Material conditions of P91 steel weldment</th>
<th>Time to rupture at $\sigma = 130$ MPa, $T = 620$ °C</th>
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<tr>
<td>As welded</td>
<td>381.6 h</td>
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<tr>
<td>PWHT-1</td>
<td>26.6 h</td>
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<tr>
<td>PWHT-2</td>
<td>30.6 h</td>
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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. The mechanical properties of P91 (Table 1) in comparison with the producer’s data 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 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.

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ACKNOWLEDGEMENTS

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6 REFERENCES

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