Creep test with use of miniaturized specimens

E Chvostová, J Džugan
COMTES FHT a.s., Dobřany, Czech Republic, EU
E-mail: eva.chvostova@comtesfht.cz

Abstract. Application of the mini samples methods is very common especially for residual service life assessment of the components operating in the energy sector. Residual lifetime of operating device can be evaluated using standard tests, but these usually cannot be performed due to limited material amount that can be extracted from the components. It is possible to use in these cases a semi destructive sampling of materials and testing methods employing miniature specimens. This article deals with comparison of the creep results obtained with the use of on standard test bars and sub sized samples such as Small Punch Test and newly proposed miniature samples that are axially loaded. The experiment was performed on steam line steel CSN 15 128 after operation. Emphasis was put to practical use of the test results and therefore residual life was assessed with the use of standard and new mini specimens. The results obtained for standard and mini samples exhibit very good agreement without necessity of any correlation as in the case of SPT and thus much more straight forward approach can be applied even for “unknown materials”.

1 Introduction
Residual lifetime estimation and early detection of possible risks of mechanical equipment failure and structure is a key question for reliable operation of industrial plants. Currently, this issue is particularly critical for the power stations and petrochemical plants that are approaching their designed end of life. The remaining service life can be evaluated using standard tests, but also by using semi-destructive techniques using mini samples.

One of used semi-destructive methods is the Small Punch Test (SPT) [1]. SPT is usually based on the conversion of the obtained results into conventional mechanical properties [2]. The correlation parameters must be verified for each new material. There are also other available methods, where a small test sample is subjected to the same loading mode, as is the case of standard samples. For example the micro-tensile test [3-5], the miniature fracture toughness test [6-7], the miniature fatigue test [8] and mini-Charpy test for DBTT determination [9]. These methods maintain minimal material demands without requiring previously established correlations or at least they use a much more reliable type of correlation (without necessity to measure wide range materials). The above mentioned miniaturized testing techniques have been verified. These testing methods using miniaturized specimens maintaining testing loading mode are being developed and successfully applied and this paper shows results of miniaturized standard specimens testing in the field of creep tests.

2 Test samples
As a reference specimens standard creep test samples with diameter of 8mm and gauge length of 40mm were used here. The SPT test were done on disc shaped specimens with diameter of 8mm and
thickness of 0.500 mm ± 0.001 mm. The geometry of the newly designed mini specimen is shown in Figure 1. The specimen geometry comes from modified dog bone shape and dimensions are based on the shape of the sample with dimensions of 20x20x4 mm, that can be extracted from real in service components by semi-destructive device EDSE (the Electric Discharge Sampling Equipment) [10]. There can be produced at least 4 specimens of considered geometry out of the extracted experimental material. Figure 2 documents comparison of all specimens investigated in this study.

![Figure 1. Geometry of miniature creep test specimen.](image)

![Figure 2. Used specimens for testing creep.](image)

The experimental material was pipe line steel CSN 15 128 after certain service period. Chemical composition of the steel investigated is shown in Table 1.

| Table 1. Chemical composition of the experimental material. |
|-----------------|------|------|------|------|------|------|------|------|------|
| C    | Mn  | Si  | Cr   | Mo   | V    | Al   | P    | S    |
| 0.14 | 0.52| 0.36| 0.69 | 0.48 | 0.27 | 0.019| 0.02 | 0.02 |

3 The creep testing and evaluation

Creep tests on standard test bars were executed with the use of conventional creep machines fitted with furnace, strain and temperature measurement. Testing of the SPT creep specimens was carried out on specially adopted machines. In the case of miniaturized standard specimens testing, standard machines with modified grips were engaged.

Testing of SPTs on standard creep machine requires a special testing fixtures converting tension load into compression. The sample is inserted into the testing fixture and consequently positioned into the creep machine [1]. Testing set up can be seen in Figure 3. The test disc is placed on a special die No. 1 and then it is inserted into the fixture bottom part No. 2. The upper part No. 3 is subsequently screwed on the bottom part and the guide No. 4 is afterwards inserted followed by Al₂O₃ ball of diameter 2.5 mm and punch No. 5. The tip of the punch is tapered in order to centre the push ball during the test. After the testing fixture assembly, it is positioned in the creep stand.
and loaded. Load line displacement or strain is not measured in the current study. Time to fracture is evaluated here only.

Figure 3. SP Test product disassembled and SP Test preparation assembled [9].

Mini samples testing was done with newly designed and manufactured fixture which is screwed onto the rods of conventional creep machines, the machine itself does not require any alteration. In order to ensure consistent contact force for all tests, the testing fixture is tightened by torque of 25 Nm using a torque wrench. Testing set up for testing of mini specimens is depicted in Figure 4. The proposed fixture is intended only for time to fracture determination. Specimens of all considered geometries after creep tests are displayed in Figure 5.

Figure 4. Test arrangement creep - mini sample.

The creep tests were carried out according to ČSN EN ISO 204 [11], the results of creep tests are evaluated in the form of time to fracture. Tests were performed on 18 standard creep specimens, 8 mini specimens and 16 SPTs. Tests were carried out in temperature range from 500 to 600 °C. The longest time to fracture was 7,699 hours in the case of standard specimens. In the case of miniaturized specimens times to fracture were up to several hundreds of hours in the current study.
The test specimens after testing creep.

In the case of SPT creep tests there is difficulty with stress definition within the test-piece due to uneven stress distribution. There was derived expression (1) for stress calculation based on the theory of bending thin plates that is widely used in the publications for SPT creep test evaluation [13]. This expression was used here for stress calculation.

\[
\frac{F}{\sigma} = 3.33 k_{sp} R^{0.2} r^{1.2} h_0
\]

where \( R \) is die hole radius (2.2 mm), \( F \) is applied force in N, \( h_0 \) is initial specimen thickness (0.5 mm), \( r \) is ball radius (1.25 mm), \( k_{sp} \) is strain hardening coefficient (1.0) and \( \sigma \) is evaluated tensile stress.

Comparison of creep results for material 15 128 after service.

Standard and new mini-specimens were evaluated with the use of standard formulas such as e.g. stress calculation. In the case of SPT evaluation, a correlation for the material investigated was established on the basis of standard tests as a reference values and results of SPT. Comparison of results for all three groups of specimens was done with the use of Larson - Miller parameter defined as \( P_{LM} = T (20 + \log \tau) \), where \( T \) is temperature in K, and \( \tau \) is time in hours. The results are summarized in Figure 6. There are shown reference creep properties for the material investigated as specified by materials standard in virgin state [12] and results obtained with full sized specimens, new mini specimens and SPT. The values obtained clearly show material properties degradation due to service on difference between standard reference curve according to CSN 415128 and shift of presently measured values towards shorter times to fracture. The SPT
results exhibit significantly higher scatter in comparison with other results and also slightly lower times to fracture in comparison with full size specimens. However, if some of outlaying values around $P_{LM}$ of about 18 500 would be removed better agreement should be achieved, that can be expected as correlation based on current data population is applied. Comparison of full sized and axially loaded mini specimens yields excellent agreement within data scatter. This is very promising result as no correlation was applied here and thus this finding confirms of direct comparability of results obtained on specimens of different sizes.

The results obtained for full size and new mini samples are applied for residual service life estimate of the material that is investigated after period of service operation. The calculation was performed with the use of following parameters: internal gas pressure in pipe 18 MPa, operation temperature of 543 °C and the pipeline dimensions: diameter 324 mm, wall thickness 48 mm. Based on these input data, FEM simulation shown stress in the pipe at level of 122 MPa. These parameters were together with results from graph displayed in Figure 6 inserted into Larson-Miller relation shown below.

$$
\tau_Z = 10^{\left(\frac{P}{7} - 20\right)} = 10^{\left(\frac{18625}{543+27316} - 20\right)} = 661 \text{ [h] standard sample} \quad (2)
$$

$$
\tau_Z = 10^{\left(\frac{P}{7} - 20\right)} = 10^{\left(\frac{18726}{543+27316} - 20\right)} = 879 \text{ [h] mini sample} \quad (3)
$$

Calculation results shown residual service life estimate of 661 h for standard specimens’ data and 879 h for mini samples results. Both results are very close confirming applicability of mini-samples utilization for residual service life estimation of in-service components.

4 Conclusions

The paper deals with possibilities of creep properties determination with the use of miniaturized experimental specimens. Standard creep specimen results are compared with results obtained from SPT creep tests and newly proposed mini-creep specimens. The standard sized specimens provide reliable results, however it is usually impossible to machine them out of real in-service components. SPT utilize small size specimens that can be extracted from real components, but the evaluation procedure requiring known correlation relation for considered material and if possible verified also for the state investigated that is also rather difficult and in many cases impossible. Therefore new miniaturized creep specimens were proposed combining the advantage of maintaining the same loading mode as standard creep tests while material demand for specimens machining can be covered by volume that can be extracted from serviced component. The dimensions of creep mini-samples proposed here are based on material volume that can be extracted by available EDSE sampling device.

Creep tests were performed for all considered sample geometries for after service pipeline steel in temperature range between 500 and 600 °C. In the case of SPT tests correlation function was established for the material investigated based on relation between results of standard tests and SPT prior further comparison. Subsequently, all results were summarized for comparison in the form of Larson-Miller plot. There is visible slight offset of SPT results from the other two investigated data sets. This was caused by wider data scatter mainly. If some of the outlying results would be removed, the agreement with other results would be significantly better. This agreement would be expected as the correlation was established on the same data population. The results of SPT point out just inclination to higher data scatter and necessity to establish correlation specifically for the material investigated.

Results of mini creep specimens exhibited very good agreement with standard creep test results. This is very positive finding as the same evaluation was used for both specimens’ batches without any further adjustments for mini-specimens. The results confirm possibility of direct comparability of results attained for specimens of different sizes in loaded in the same mode.
The results of standard tests and mini creep tests were also used for residual service life estimation according to Larson-Miller approach. Very close residual service lives were obtained confirming previous statements about the mini specimens’ successful application to real components service life assessment.

More data with the miniaturized specimens have to be accumulated for more general conclusions and recommendations. Testing set up allowing also strain measurements is considered for future investigations.

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