# SMALL PUNCH TESTING OF THICK WALLED PIPE MADE OF THE STEEL P92 WITH HETEROGENEOUS MICROSTRUCTURE

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**https://doi.org/10.37904/metal.2020.3512**

## Abstract

The paper deals with the small punch testing of material properties including short-term creep resistance and microstructure analysis a thick-walled pipe made of P92 steel, where metallographic analysis revealed numerous internal defects, especially close to the inner surface of the pipe. These defects had their origin in the shrinkage porosity of the continuously cast slab and remained in the microstructure probably due to insufficient deformation during pipe rolling. The local material properties at the both inner and outer surfaces were compared by metallography, tensile tests, hardness tests, Charpy-V notch tests as well as small punch and small punch creep tests. The obtained results are compared with the material requirements for P92 steel and an attempt is made to evaluate the possible influence of the inhomogeneity of the microstructure and properties on the long-term service life of the pipe under creep conditions.

**Keywords:** Steel P92, heterogeneous microstructure, mechanical properties, internal defects, small punch creep tests

## 1. INTRODUCTION

In mid-2018 started the solution of the TK01020160 project " Complex procedures of material engineering to ensure the safe operation of innovated blocks of fossil power plants" within the THÉTA funding programme, in which participate four companies (MMV Ostrava, ČEZ, ÚAM Brno and UJP Praha). One of the main goals of this project is material research aimed at obtaining a database of properties of newly used materials with a special focus on creep, fatigue and brittle fracture of boiler tubes and steam pipes, their welds and other critical components of power boilers. Knowledge of these material data, together with the appropriate procedure for their evaluation, will enable to better manage the reliability and use of system resources.

One of these materials used mainly for USC boiler steam pipelines and headers is steel P92 (X10CrWMoVNb 9-2). It is one of the most commercially successful martensitic creep-resistant steels, which also achieves the highest long-term proven heat resistance among them [1]. However, for the demanding operating parameters of USC steam boilers, it means that the wall thickness of a steam pipe can even be about 100 mm. The most common metallurgical practice in thick-walled pipes production is piercing and pilgrim rolling from continuously cast slabs means that the initial dimensions of a slab are not very different from the final product. Martensitic heat-resistant steels are self-hardening, which means that the cooling rate and the choice of cooling medium do not have such a significant effect on the formation of martensite and its properties. Then, the final material characteristics of a very thick-walled pipe can be negatively affected by the inhomogeneity of the structure and the presence of internal defects due to the small degree of deformation, residues of the original dendritic structure, including dilutions.

## 2. EXPERIMENTAL MATERIAL AND PERFORMED ANALYSES

Verification of the material characteristics of the P92 steel steam pipe used in 660 MW USC boiler in Ledvice power plant was performed in the frame of the TK01020160 project. The nominal dimensions of the pipe were ID = 350 mm and WT = 80 mm and it was tested in the as-received state, i.e. after normalizing and tempering. Its chemical composition, including the nominal values specified in ČSN EN 10216 2 [2], is stated in **Table 1**.

**Table 1** Chemical composition of steel P92 (wt%)

| **Element** | **C** | **Mn** | **Si** | **P** | **S** | **Cr** | **W** | **Mo** | **V** | **Nb** | **N** | **Al** | **B** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Pipe** | 0.10 | 0.41 | 0.20 | 0.013 | 0.004 | 8.74 | 1.66 | 0.48 | 0.18 | 0.050 | 0.056 | 0.001 | 0.0020 |
| **P 92 (EN)** | 0.07 - 0.13 | 0.30 - 0.60 | ≤0.50 | max. 0.020 | max. 0.010 | 8.5 - 9.5 | 1.5 - 2.0 | 0.3 - 0.6 | 0.15 - 0.25 | 0.04 - 0.09 | 0.003 - 0.007 | ≤ 0.020 | 0.001 - 0.006 |

A complex analysis of the material properties of this pipe included verification of mechanical strength at laboratory and elevated temperatures, performance of Charpy V-notch tests in the temperature interval that enables to construct of the brittle- ductile transition curve, measurement of hardness profile over the wall thickness, microstructure analysis and also small punch creep tests.

## 3. MICROSTRUCTURE AND MECHANICAL PROPERTIES

Metallographic analysis confirmed the tempered martensitic structure of the pipe (see **Figure 1**), but also revealed that there were clusters of dilutes in nearly the entire cross section of the pipe wall. Their occurrence was more frequent at the inner surface of the pipe and, as can be seen in **Figures 2** and **3**, the size of these clusters was in the order of millimeters. In order to confirm that the suspected objects were indeed dilutes, they were studied by scanning electron microscope in topographic contrast (TOPO) in the mode of back scattered electrons. The rounded shapes of the original dendrites were clearly visible inside the individual dilutes as can be seen in **Figure 4**.

The results of the metallographic analysis inspired us to extend the testing program by comparative tensile tests performed at room temperature and at a temperature of 600 °C on test specimens located just below the outer and just below the inner surface. These results are summarized in **Table 2** together with a comparison with the requirements specified in ČSN EN 10216-2.

**Table 2** Mechanical properties at outer and inner surface of the pipe

| **Temperature** | **+20 °C** | | | | **600 °C** | | | |
|---|---|---|---|---|---|---|---|---|
| **Sample** | $R_{p0.2}$ (MPa) | $R_m$ (MPa) | $A_5$ (%) | **Z** (%) | $R_{p0.2}$ (MPa) | $R_m$ (MPa) | $A_5$ (%) | **Z** (%) |
| **Inner surface** | 481 | 651 | 27.4 | 69.6 | 280 | 294 | 22.1 | 89.5 |
| **Outer surface** | 484 | 645 | 26.6 | 72.2 | 292 | 306 | 21.9 | 89.5 |
| **P 92 (EN)** | ≥440 | 620 - 850 | ≥17.0 | - | ≥248 | - | - | - |

As can be seen from the results in **Table 2**, the difference between the mechanical properties of the two pipe surfaces at room temperature is only in megapascals and does not exceed 5 % at 600 °C either. Thus, it can be concluded that there is practically no difference in mechanical properties between the both surfaces. Nevertheless, when compared the strength of the tested pipe with the requirement for mechanical properties of P92 steel stated in the material standard, it is clear that particularly the value of the ultimate tensile strength of the evaluated pipe is very close to the lower limit of the required interval stated in [2].

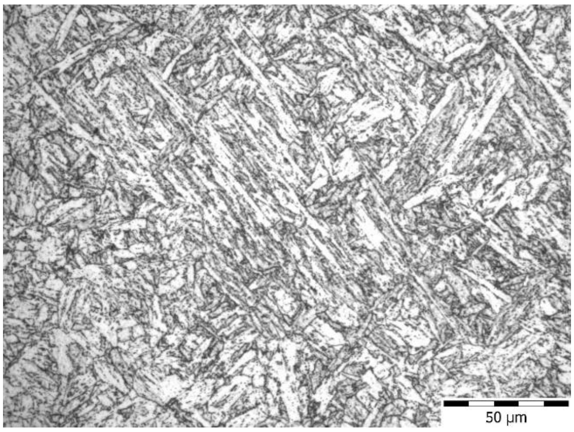

**Figure 1** Microstructure of the pipe

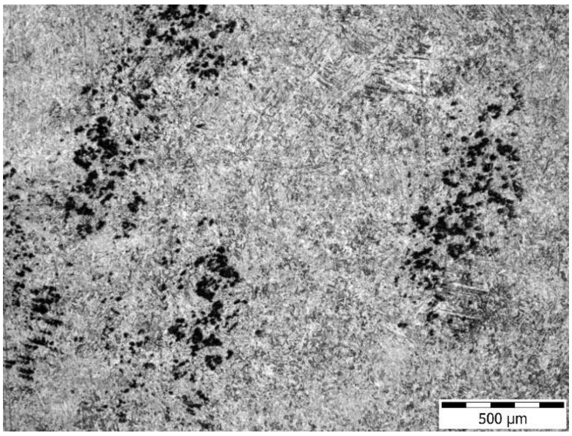

**Figure 2** Dilutes close to the inner surface of the pipe

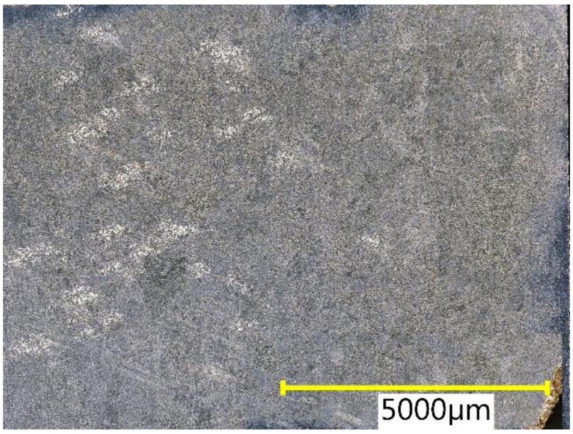

**Figure 3** Cluster of dilutes

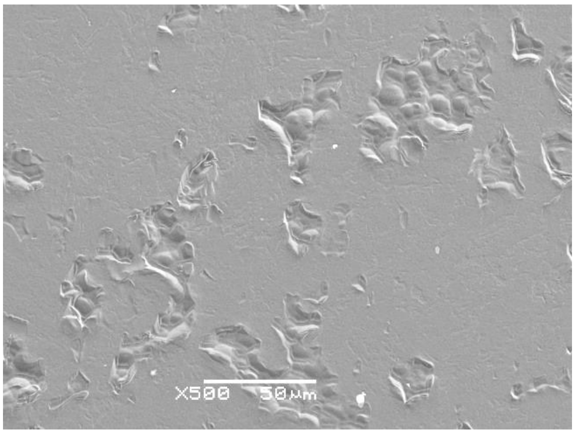

**Figure 4** Dilutes observed in TOPO contrast of SEM

The hardness does not change significantly from the outer to the inner surface, too, as is illustrated in **Figure 5**. The minimum hardness was measured near the outer surface of the pipe, then hardness increased again and the maximum was reached at a depth of about 55 mm below the outer surface. From this point it decreased again towards the inner surface, but the total difference in hardness throughout the whole wall thickness was less than 30 HV.

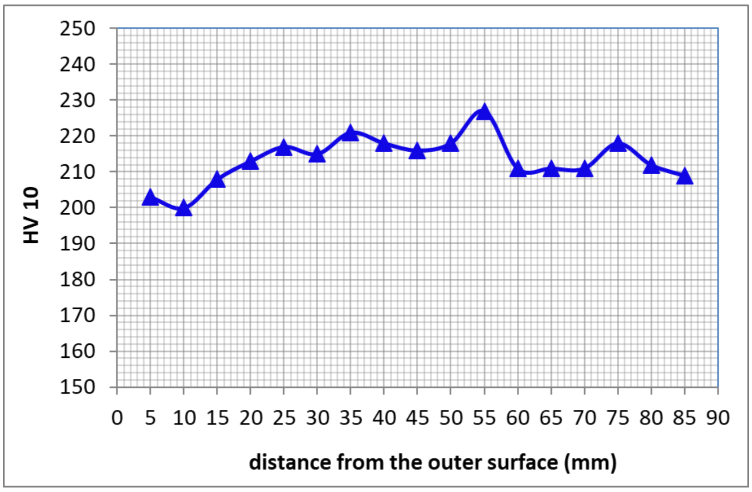

**Figure 5** Hardness profile HV 10 through the wall thickness

## 4. CHARPY V-NOTCH IMPACT TEST RESULTS

Charpy V-notch impact tests for determining brittle-to-ductile transition temperature were originally performed on test specimens with a V-notch in the transverse direction, and test specimens were machined from the area below the outer surface. After detecting defects in the structure, other test specimens were made to compare the properties of both surfaces. The orientation in the longitudinal direction was chosen with respect to the pipe geometry, where the plane of crack propagation in the test specimens corresponded to the expected direction of defect growth through the pipe wall thickness. The results of Charpy V-notch impact tests showed high values of impact energy of the pipe at room temperature, and did not confirm the expected difference in it between outer and inner surface, see **Table 3**. The brittle-to-ductile transition temperature (FATT) determined from test results in the transverse direction is also stated in **Table 3** and showed expected value under 0 °C. The fracture surface of all test specimens was mostly transgranular and ductile with sporadic islands of brittle intercrystalline fracture, see **Figure 6**.

**Table 3** Impact energy at room temperature and FATT temperature

| Pipe surface | Impact energy at +20 °C (J) | FATT (°C) |
|---|---|---|
| **Inner** | 169, 166, 169 | -9 |
| **Outer** | 160, 166, 197 | |

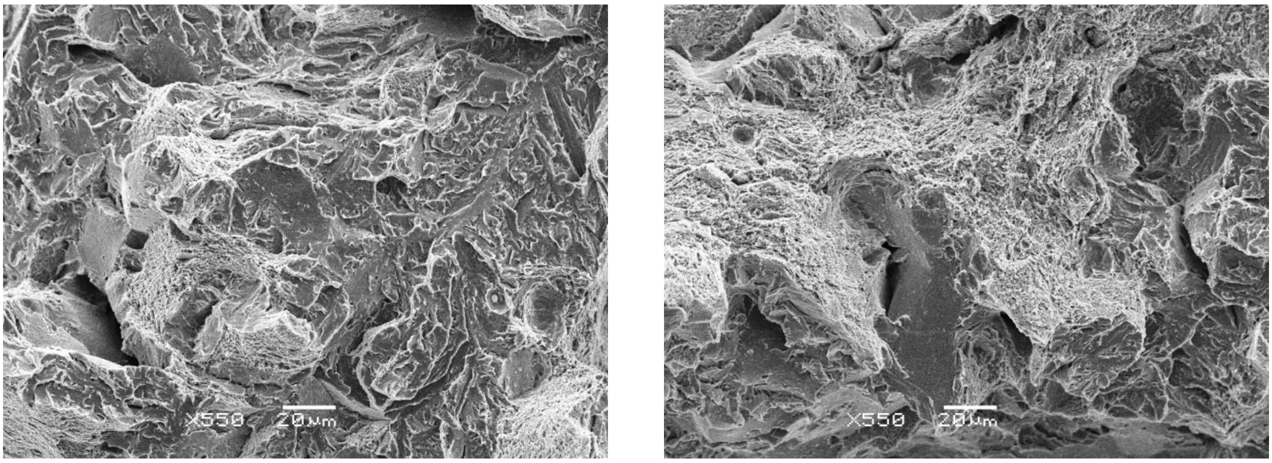

**Figure 6** Fracture surface of Charpy-V notch test in longitudinal direction close to inner surface of the pipe

## 5. CREEP RESISTANCE

With their size in the order of small tens of micrometers, the observed defects could be attributed to the interconnected cavities or chains of cavities. From the viewpoint of the safe use of this pipe it is of course very interesting to find out whether, when and how the presence of these a priori defects influences the creep resistance of the pipe. The original stress rupture testing program of conventional bulk test specimens was performed in order to verify whether the creep resistance of the pipe fulfils requirements specified in the material standard. Test specimens for these stress rupture tests were machined from the entire cross section of the pipe and tested at temperatures 600, 625 and 650 °C. The results of stress rupture tests are shown in **Figure 7**. As the fracture times of the finished tests are still relatively short, the Larson-Miller parametric method was used for the comparison of results and their interpretation in the form [3]:

$$P_{LM} = T \cdot (C + \log(t)) \qquad (1)$$

where T is the temperature in Kelvin, t is the time to fracture in hours.

The value of Larson-Miller constant C = 26.8 was calculated using the least squares method from the results reported for steel P92 in the tube/pipe standard ČSN EN 10 216-2. The experimental results of stress rupture tests are in **Figure 7** compared to the mean value of the creep rupture strength of steel P92 that is represented in this figure by the solid line, while the dashed line represents the allowed -20 % deviation from this mean value. This comparison clearly showed that the results of performed stress rupture tests of this steam pipe lay below the mean value, but practically all of them were in the deviation band -20 % around it. The empty symbols in this figure represent still running test specimens that will further refine the initial estimation of the long-term creep resistance of this pipe.

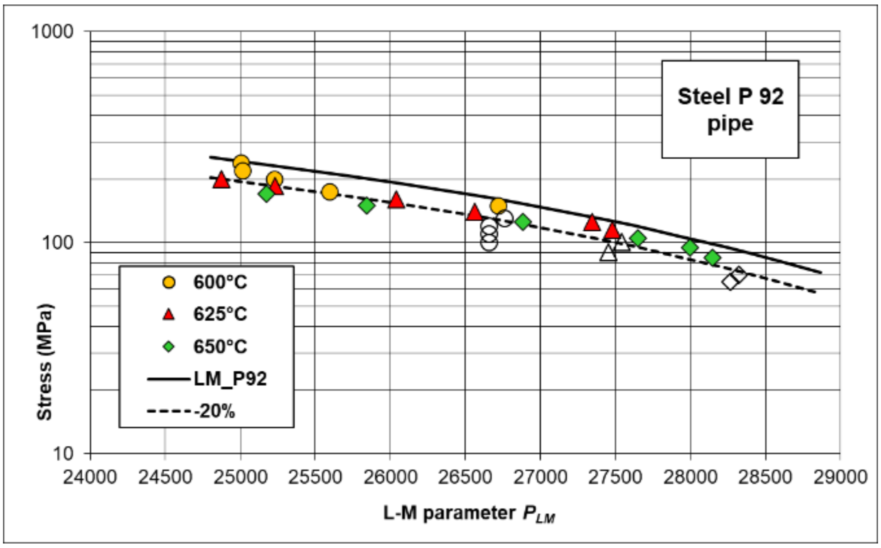

**Figure 7** Results of stress rupture tests shown as stress dependence on Larson-Miller parameter $P_{LM}$

Whereas by using the standardized stress rupture tests it is not possible to test narrowly located areas at pipe surfaces, small punch creep tests allow to prepare test specimens very close to the pipe surface. In order to assess the actual creep properties near both surfaces, this testing program was extended to small punch creep tests (SPC tests) [4, 5] for which test specimens were prepared close to both outer and inner surfaces. All SPC tests were performed at 650 °C and the results for either the inner either the outer surfaces are stated in **Figure 8** and **Figure 9** in the form of dependence of time to rupture and minimum deflection rate on load. It seems that material taken below the outer surface has a bit higher creep resistance compared to that from the inner surface. On the other hand, the load dependence of minimum deflection rate forms the straight single line regardless of the place from which the material was sampled.

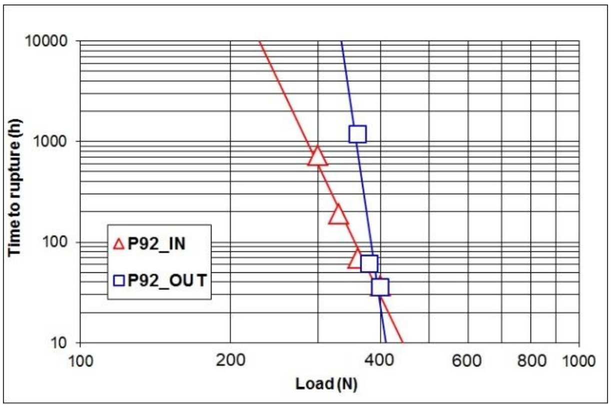

**Figure 8** Dependence of time to rupture on loading

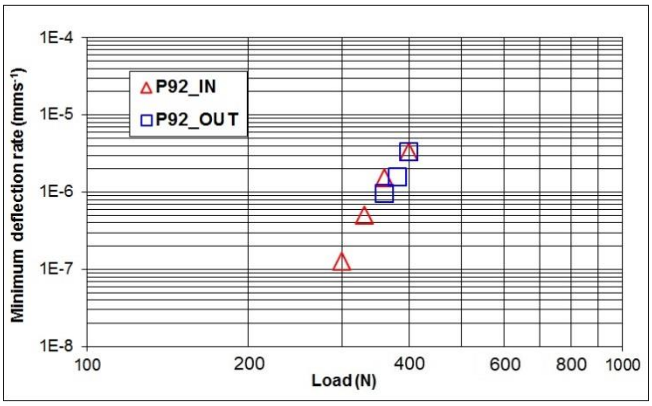

**Figure 9** Dependence of minimum deflection rate on load

## 6. DISCUSSION OF RESULTS

The presence of cluster of defects in the thick-walled pipe is most likely related to the low level of steel deformation during hot rolling of such a thick-walled pipe, especially in the case when the initial semi-finished product is a continuum casted slab. The occurrence of dilutions is usual and acceptable in the case of castings, but it is not common for them to occur in such numerous clusters, but they are typically randomly distributed in the structure, which eliminates their negative effect on the material properties of steel. In the monitored case, not only there were numerous dilutions detected in the wall of the pipe, but locally their accumulation was considerable and, in terms of size, these groups of defects have already reached the level of microcracks. The analysis of mechanical, fracture and short-term creep properties of a thick-walled pipe has not yet shown any influence of the existence of clusters of dilutions in the structure on any of the material characteristics. It is not yet possible to reliably determine whether and how much these dilutions represent a real danger, especially by a localized decrease in the heat resistance of the pipe, because the results of stress rupture tests completed so far reached only more than thousand hours. In such still short-term results the most significant role in creep deformation is still played by dislocation strengthening of the microstructure and the role of precipitation strengthening is suppressed. In the short-term creep tests, transgranular fracture also predominates, while in the long term tests, the character of the fracture changes into intergranular and creep damage is controlled by cavitation and is therefore concentrated at grain boundaries. Creep crack propagating through such a weakened region will be probably faster and thus will approach the critical size for the occurrence of a sudden unstable fracture sooner. In a softened and relaxed microstructure after long-term creep exposure, a similar defect can significantly accelerate the consumption of creep life and reduce the operation safety of a respective part of a boiler.

## 7. CONCLUSION

The identified clumps of diluents in the structure of the thick-walled pipe made of P92 steel may pose a potentially serious risk to the safety and reliability of the boiler operation in the future. Therefore, the diagnostic pipeline should be given increased diagnostic attention in the future, especially in the period approaching the end of the designed service life. At present, within the solution of the mentioned project, other works are being carried out focused on the determination of fracture toughness and conditions of crack growth under dynamic stress.

## ACKNOWLEDGEMENTS

***This paper was created with the financial support of the Technology Agency of the Czech Republic within the project THETA TK01020160 "Complex procedures of materials engineering to ensure safe operation of innovated units of conventional power plants".***

## REFERENCES

[1] KIMURA, K., TODA, Y., KUSHIMA, H., SAWADA, K. Creep strength of high chromium steel with ferrite matrix. *International Journal of Pressure Vessels and Piping*. 2010, vol. 87, pp. 282-288.

[2] EN 10216-2 Seamless steel tubes for pressure purposes - Technical delivery conditions Part 2: Non-alloy and alloy steel tubes with specified elevated temperature properties. 2013

[3] MONKMAN, F. C., GRANT, N. J. An empirical relationship between rupture life and minimum creep rate in creep-rupture tests. *Proc. Am. Soc. Test. Mater.* 1956, vol. 56, pp. 593-620.

[4] Lancaster, R.J., Jeffs, S. P. Small Punch Creep (Chapter 8), In: TANSKI, T. A. SROKA, M., ZIELINSKI, A., *eds. Creep*, London: InTech Open, 2018, pp. 151-169

[5] STEJSKALOVÁ, Š., KANDER, L., HERMANOVÁ, Š. The change of the structure and mechanical properties of the austenitic steels after exposure at the critical temperature. *Materials Science Forum.* 2017, Vol. 891, pp. 330-334.