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Industrial applications of small punch test

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Abstract: Small punch test is an advantageous method for evaluation of mechanical properties of components especially in cases, where it is technically difficult or even impossible to obtain enough bulk material for standard tests. Therefore, the method is very well applicable in power industry, for example in residual lifetime assessment of critical parts of components and structures after long-term operation. The testing material is sampled by using special sampling device that ensures no component damage and the amount of material being sampled is so small that obviously no component repair is necessary after sampling. Small punch testing is exploited not only for evaluation of mechanical properties, but also microstructural and chemical analyses can be performed from the obtained sample and complex actual material characteristic of component can be assessed. Company MATERIAL AND METALLURGICAL RESEARCH, Ltd., has more than 20 years of experience with small punch testing in industrial applications and several examples of its application for analysis of material properties and residual lifetime assessment are presented in this paper.

Keywords: small punch testing, material properties, residual lifetime, sampling

1. Introduction

Small punch test (SPT) is a useful method for evaluation of actual material properties of components in power industry. A big advantage of this method is that material can be sampled from studied component directly on site without negative influence on its function. This method can be also used in cases when it is necessary to obtain test specimen from very narrow layers of material, for example from decarburized layers, segregations, coatings, etc. Another typical example of utilization of SPT method is evaluation of material characteristics of specific areas of weld joints or deposit layers.

Mechanical properties, especially yield stress or transition temperature (FATT), are degraded during long-term operation at elevated temperature. Microstructure of operated components can be degraded by appearance of cavities. An approach based on specimen sampling from critical parts of components in periodic intervals (including first sampling from virgin state of newly produced component) with complex material analysis allows to determine material degradation for each period and subsequently to assess the residual lifetime of a component. MATERIAL AND METALLURGICAL RESEARCH, Ltd., has been performing long-time monitoring of components in power industry for about 20 years. It is an owner and also a producer of special scoop sampling machine and offers long-term monitoring of operated components behaviour in Europe. This paper summarizes some key results, emphasizes an importance of SPT in this field and shows new results of small punch testing of materials used in ultra-super critical (USC) boilers.

2. Materials and Methods

Wide range of materials used for production of components in power industry has been monitored in last years. Most typical are materials of rotors, rotor cases, steam pipelines, valves, boiler drums, etc. Monitoring of degradation of material properties during operation is carried out by method of scoop sampling of material for testing without component damage. These samples are subsequently prepared for complex material analyses, namely chemical analysis, microstructure analysis and small punch tests. Test results and assessed degree of material degradation is then compared with results of previous analyses and residual lifetime of component is then estimated. This approach has been successfully used for many components and some of the most interesting cases are stated below.

3. Determination of actual material characteristic of boiler drums

Monitoring of material behaviour was performed on 4 boiler drums operating in a brown-coal fired power plant and produced in 1940s and 1950s. Material degradation has been assessed in 5 years interval [1]. No material certificates describing types and properties in the as-received state has been preserved. Boiler drum has been exposed to temperature 300°C and pressure 7.8 MPa for approximately 300,000 hours. An example of specimen sampling on an inner surface of boiler drum is shown in Figure 1.

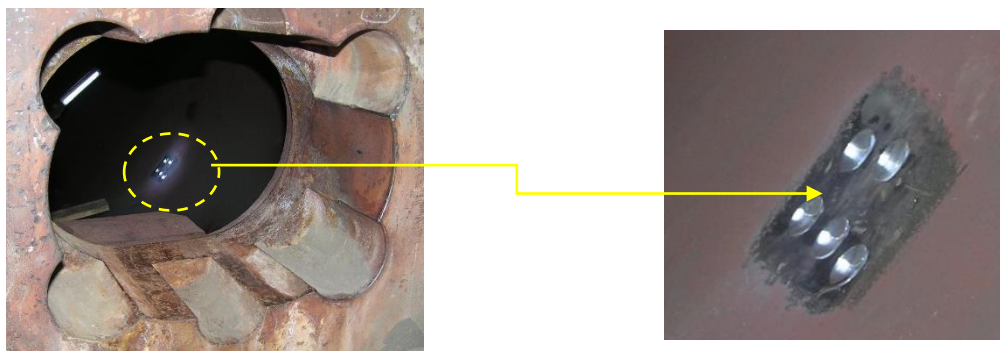


Figure 1. Specimen sampling on inner surface of boiler drum.

Results of chemical analyses performed on material of boiler drum No. 4 in 2009 and 2014 are stated in Table 1 and show the significant difference in concentration of some elements, especially high content of tramp elements (As, Sb, Sn) known for their embrittlement effect in the steel.

Table 1. Chemical analysis comparison of boiler No. 4 material (weight %).

Sampling Year	C	Mn	Si	As	Sb	Sn	Co
2009	0.34	0.75	0.019	0.010	0.016	0.003	0.009
2014	0.39	0.83	0.024	0.022	0.025	0.012	0.024

The results in Table 1 confirm low metallurgical quality of steel and also quite high scatter in concentration of some elements. This confirms the heterogeneity of material, because sampling locations were very close to each other. That fact is caused by small volume of sampling experimental material compared to the large size of analysed component.

Mechanical properties were tested using SPT [2]. Three specimens are usually used for tensile strength and yield stress determination at room temperature. A set of approximately 15-20 specimens is mostly used to test the transition temperature T_{SP} determined by SPT. Comparison of SPT records at room temperature performed in 2014 and 2009 is shown in Figure 2(a), calculation of T_{SP} is shown in Figure 2(b). Transition temperature FATT was subsequently calculated on the base of correlation.

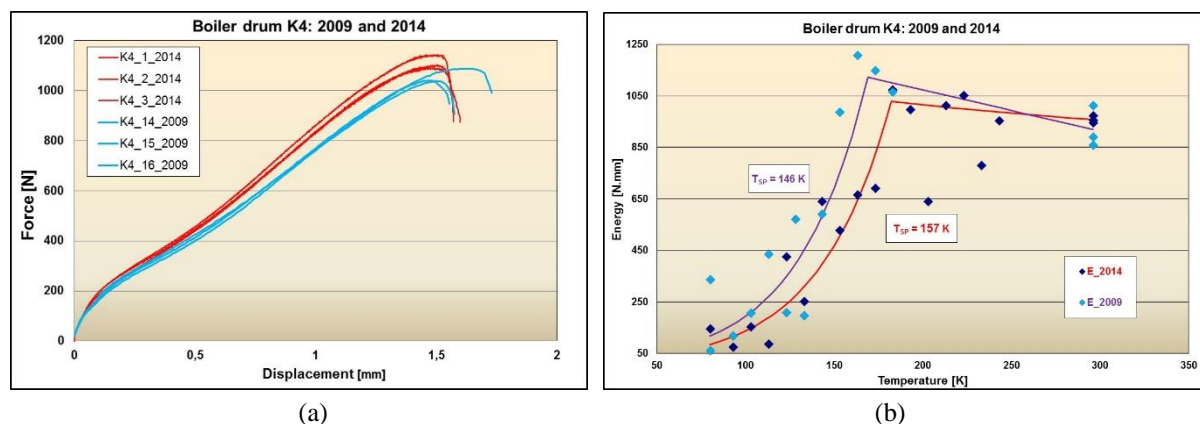


Figure 2. (a) Comparison of SPT records performed in 2009 and 2014, (b) Comparison of T_{SP} calculation using SPT performed in 2009 and 2014.

Figure 2(a) shows very small difference in force-displacement records between 2009 and 2014. Yield strength and tensile strength determined from these records are very well comparable. However, result of T_{SP} calculation increased by 11 K. T_{SP} value is used for determination of transition temperature (FATT) (see table 2). FATT increased by 26 °C between these two samples and it can be probably caused by locally increased concentration of tramp elements (Sn, As, Sb). But this change in FATT seems to be the only sign of material degradation after long-term operation at elevated temperature.

Table 2. Comparison of FATT values determined from SPT of boiler drum No. 4 in years 2009 and 2014.

Sampling year	T_{SP} [K]	FATT [K]	FATT [°C]
2009	146	340	66
2014	157	365	92

4. Determination of actual material characteristics of turbine case bodies at ethylene unit

Monitoring of material degradation of turbine casing using SPT was performed between years 2003 and 2014 [3]. Investigated components were approximately 37 years old in 2014. Several parts of turbine cases are exposed to the temperature about 500 °C and pressure 10.8 MPa (i.e. creep exposure), other parts are operated at maximum temperature 350°C and pressure 3.5 MPa. All these components were produced in Japan according to Japanese standards JIS. The main goal of performed analyses was to determine actual material properties in critical parts of components using chemical analysis, microstructure analysis and evaluation of mechanical properties, again by using SPT. Two parts of turbine case body GT201, made of the cast steel grade SCPH 21 (equivalent to GS 17CrMo 5-5 according to DIN 17 245) and working in the creep regime, were repeatedly analysed in 2003, 2008, 2012 and 2014, as can be seen in Figure 3, where are clearly visible sampling marks on the inner and outer parts of turbine case body GT201.



Figure 3. (a) Sampling marks on the inner surface of bottom part of turbine case body GT201, (b) Sampling marks on the outer surface of upper part of turbine case body GT201.

Comparison of mechanical properties of both part of the turbine case is stated in Table 3 and in Figure 4 and shows non-monotonic development of the both, proof stress and tensile strength. The both characteristics increased between the first two measurements, but then proof stress started to decrease, while tensile strength kept more or less the same values. An increase in tensile strength was in good accordance with the other analyses, namely metallographic analysis that confirmed higher concentration of carbides as well as the presence of annealing twins in microstructure in the respective sampling place.

Table 3. Change of mechanical properties of turbine case body GT201 determined by SPT.

Component	R _{p0.2}	R _m	R _{p0.2}	R _m	R _{p0.2}	R _m	R _{p0.2}	R _m
	[MPa]							
	2014		2012		2008		2003	
GT 201 bottom part of turbine case – inner surface	283	491	316	526	371	469	319	468
GT 201 upper part of turbine case – outer surface	261	445	358	520	410	473	351	465

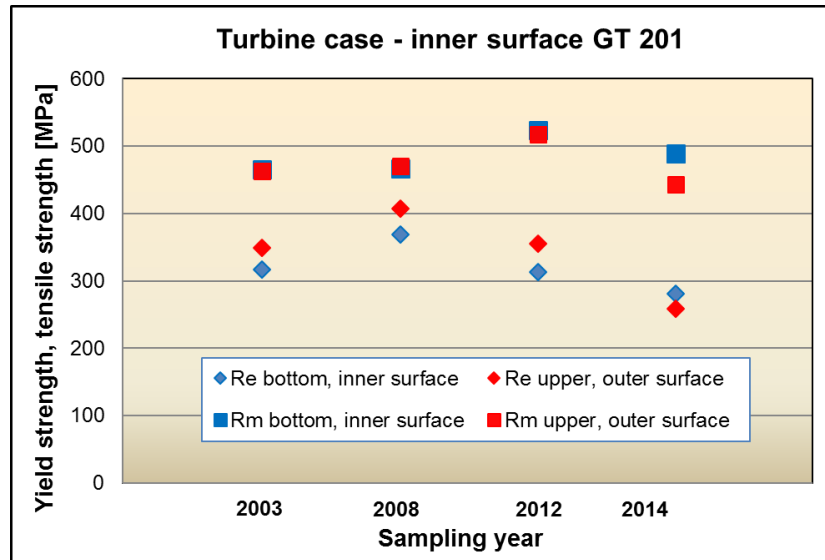


Figure 4. Trend of mechanical properties of GT201 turbine case between 2003 and 2014 determined by SPT.

5. Sigma phase precipitation and material properties of reheater tubes made of TP347HFG steel

Small punch testing of pressurised parts of USC boilers belongs to the last research activities focused on power industry. The development of the modern USC boiler is forced by demands on increasing the boiler efficiency altogether with the request for lowering CO₂ emissions. All these demands have to be accompanied by development of new steel grades with high creep strength and corrosion resistance. Due to relatively low price of iron-based materials in comparison with nickel-based superalloys the development was focused on new grades of austenitic heat resistant steels. Material properties and their testing by SPT tests were solved in the Czech Republic in the frame of a research project focused on three types of austenitic steels, namely Super 304H, HR3C and TP347HFG. Change of material properties due to necessary technological operations (bending, welding) and also the effect of long-term exposure at operating temperature were studied. Due to small dimensions of components (especially thin-walled tubes) SPT seems to be one of the best methods for evaluation of material properties. Small punch testing was also used to detect the effect of sigma phase on mechanical properties of TP347HFG steel after long-term operation.

The first research studies related to materials for USC applications were focused on the influence of sigma phase on material properties of TP347HFG [4] steel tubes used in an USC once-through Benson tower type boiler with double reheat in a Danish power plant.

Sigma phase is an intermetallic Fe-Cr phase that precipitates in stainless steel (especially ferritic and duplex) when the Cr content is above 20 wt.%. It precipitates also in austenitic stainless steels during long-term exposure at high temperatures and especially in steel grades with higher chromium content. The presence of sigma phase leads to embrittlement of the material at ambient temperature [5]. It is well-known that sigma phase represents a big problem in steels with higher Cr content and it was found that sigma phase developed in the AISI 316 steel within 1,000 hours at 700 °C and 50,000 hours at 650 °C [6]. However, its influence on toughness and plasticity of new

austenitic heat resistant steels has not been described in detail yet. The main goal of research work then was to quantify the influence of sigma phase on material properties including fracture behaviour using miniaturized test specimens and SPT.

Size and location of test specimens used for evaluation of effect of sigma phase on mechanical properties of TP347HFG steel are shown in Figure 5. Position 0° represents a tube orientation facing directly to the furnace (fire side) and thereby meeting the hottest flue gas, position 180° is an opposite side.

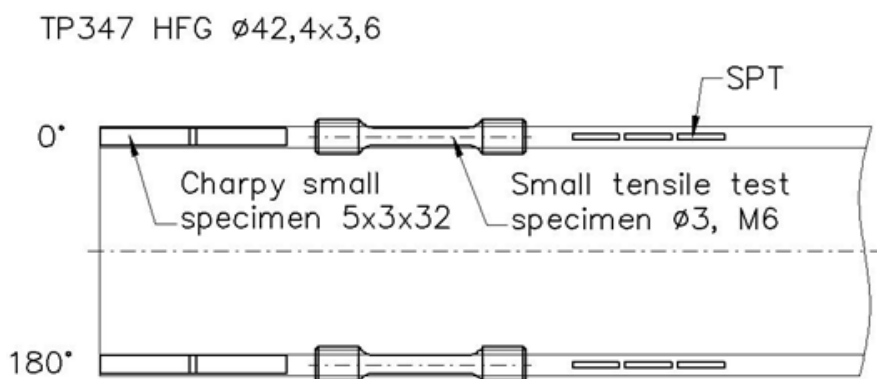


Figure 5. Size and location of test specimens machined from tube made of TP347HFG [4].

Microstructure of the steel and especially the presence and distribution of sigma phase particles was observed using LOM. Larger amount of sigma phase was observed in position 0° (fire side) than in position 180° (opposite side) and this fact can be clearly observed in Figure 6. The volume fraction of sigma phase at position 0° was 5.5 %, whereas at position 180° it was only 0.6 %.

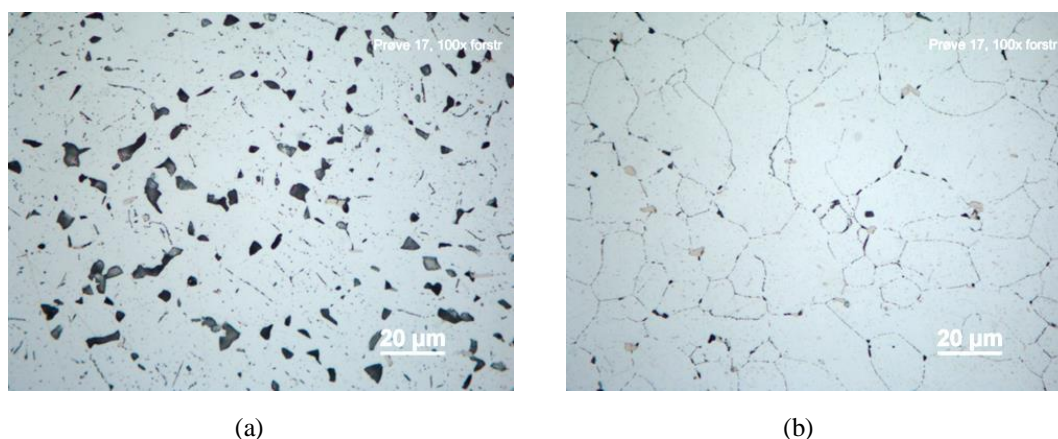


Figure 6. Microstructure analysis of TP347HFG with presence of sigma phase: (a) 5.5% in position 0°, (b) 0.6% in position 180° [4].

The both testing methods (miniaturized tensile test specimens and SPT) were subsequently used in order to compare the effect of sigma phase on mechanical properties. Tensile tests results performed on miniaturized specimens did not prove any significant difference in tensile strength on both sides of tube No. 17, whereas decrease of yield stress at the fire side of the tube was observed. Decreasing of plastic properties, elongation and reduction of area, also corresponded with increased sigma phase content.

Subsequently, five SPT specimens were cut from fire and opposite side of tubes in mid-thickness of the wall and testing was performed at room temperature. SPT is expected to reflect the changes in the microstructure more sensitively than tensile test. SPT records in the form force-elongation curves are compared in Figure 7 and clearly confirms the effect of sigma phase on mechanical properties. Fracture energy, calculated as an area under force–displacement curve, of specimens located in 0° position is 898 N·mm, which is significantly lower than fracture energy in 180° location (1408 N·mm).

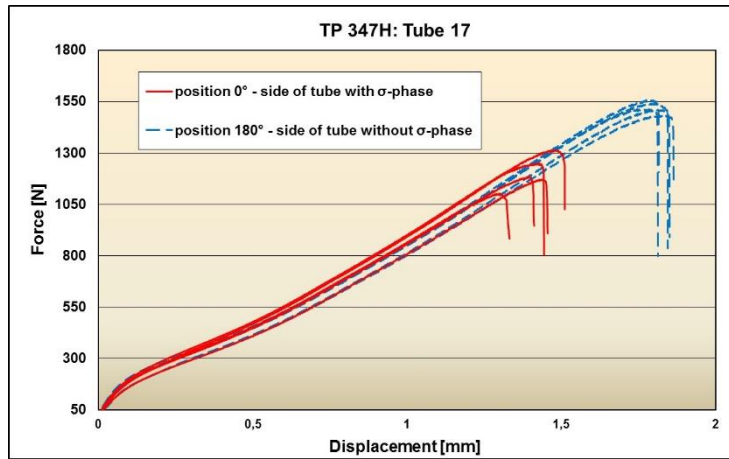


Figure 7. Comparison of small punch test results on fire and opposite sides of tube 17 with significant difference of sigma phase in the structure.

6. Material properties of welded joints of HR3C, TP347HFG and Super 304H steels

In this part of the research the attention was focused on material analysis of welded joints of HR3C, TP347HFG and Super 304H steels. Analyses were performed on circumferential welds of tubes with diameter 38 mm and wall thickness 6.3 mm. The main goal was to assess influence of one-year long exposure in the real operating environment of USC boiler on mechanical properties of welded joints. The samples had been exposed in the boiler without any loading at two working temperatures – one ranging from 635°C to 695°C and another ranging from 726°C to 775°C. Limited amount of material and low wall thickness are ideal conditions for using miniaturized test specimens or SPT. Altogether 21 combinations of base metals and welded joints of HR3C, TP347HFG, Super304H and P92 steels in as-welded state and after post-weld heat treatment were used for testing [7]. All material combinations were evaluated prior to exposure and subsequently after one-year exposure in the real USC boiler.

Evaluation of mechanical properties in as-received state was performed using miniaturized test specimens having 3 mm in diameter and 10 mm in length, absorbed energy was measured using miniaturized Charpy specimens and also SPT specimens were prepared. Location of test specimens inside the tubes is shown in Figure 8.

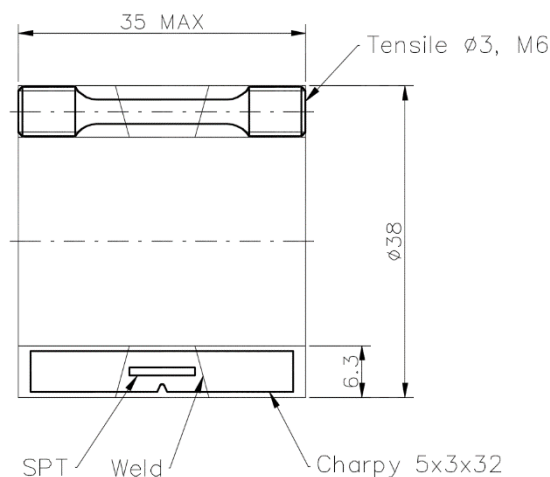


Figure 8. Location of test specimens in a welded joint.

Specimens for SPT were cut from welded joint and base material after exposure. Correlation established for these materials in 2011-2013 [8] was used for tensile strength and yield stress calculations of base materials. Charpy impact test was performed using specimens of welded joints in as-received state and after exposure.

Test results showed lack of sensitivity of tensile tests for assessment of material changes appearing in the locally restricted area of the studied welded joints. However, material degradation was very well detected and confirmed by using SPT method. Figure 9 shows significant influence of one-year exposure on material properties of TP347HFG-HR3C weld metal. Fracture energy was more than 50% lower compared to specimens after exposure.

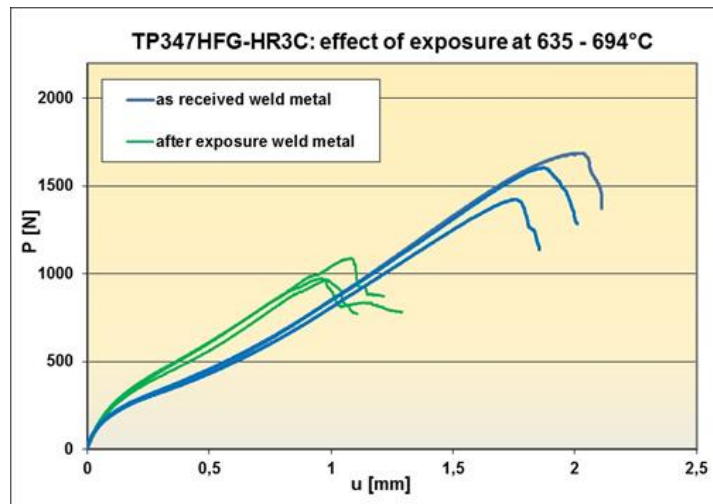


Figure 9. Influence of exposure on fracture energy of weld metal of TP347-HR3C weld joint.

Below is documented effect of PWHT and exposure at lower working temperature on fracture energy of the whole TP347HFG-HR3C weld joint (see Figure 10). It is evident that PWHT caused increasing of fracture energy of TP347HFG base material before high-temperature exposure (higher values of force and displacement in figures 10 (a) and (b)), but after high-temperature exposure values of fracture energy were nearly the same. On the contrary, HR3C material shows interesting behaviour. PWHT decreased its fracture energy moderately with bigger scatter of values in the as-received state. But fracture energy of HR3C base material after exposure shows drop to 50% of values in the as-received state independently of PWHT. Figure 10 shows that HR3C fracture energy after exposure corresponds to the level of weld metal.

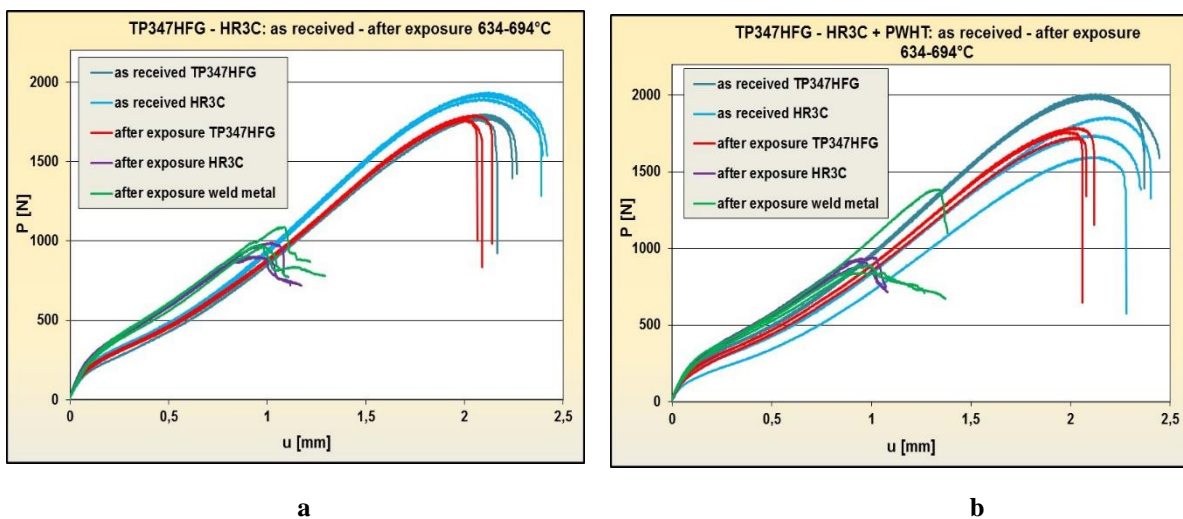


Figure 10. Comparison of SPT results of TP347HFG-HR3C welded joint in the as-received state and after exposure, (a) – as-welded state, (b) after PWHT.

The homogeneous welded joint was chosen as the other example of PWHT effect. The positive effect of PWHT especially on fracture energy of weld metal is evident in the Figure 11. In this case PWHT has a positive effect. Although fracture energy of TP347HFG base metal increased only moderately, increase of fracture energy of weld metal is evident.

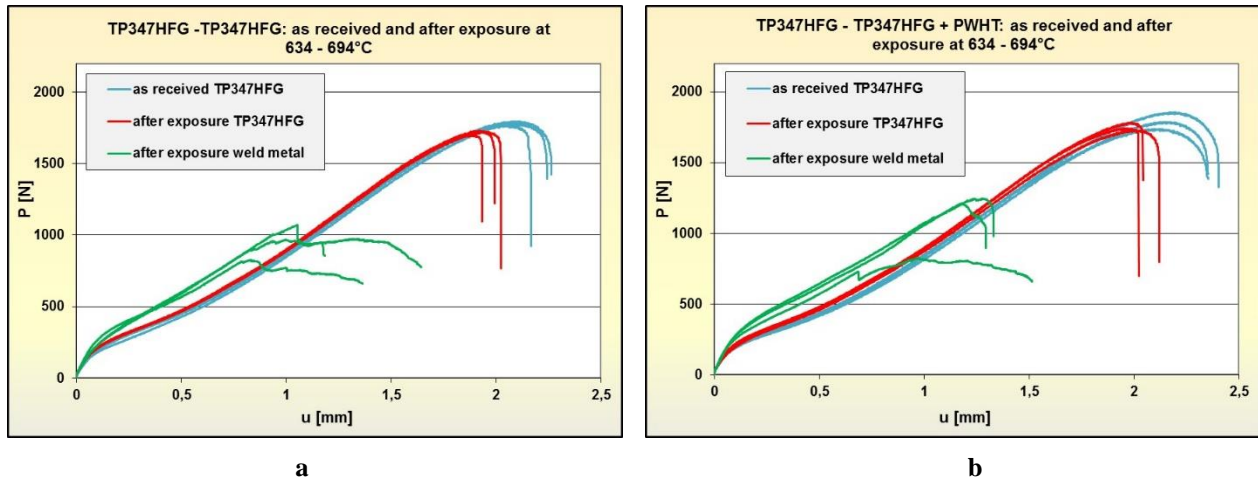


Figure 11. Comparison of SPT results of TP347HFG-TP347HFG in (a) as received state after exposure, (b) in as received state after heat treatment and exposure.

Degradation effects of high working temperature and corrosion environment of boiler was also demonstrated very well by Charpy test using miniaturized specimens. The absorbed energy of weld metal dropped down to 1/3 of the value in the as-received state (see Figure 12).

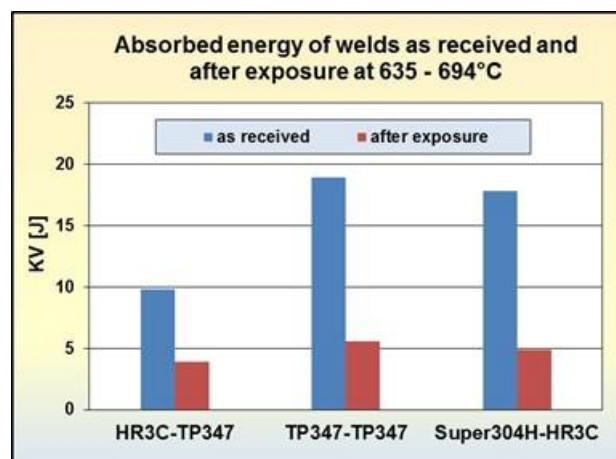


Figure 12. Effect of exposure on absorbed energy of chosen weld joints.

7. Mechanical properties of tube bends with different bend radii

In order to study the effect of plastic deformation on material properties of tubes, five tube bends of steel grades HR3C, TP347HFG and Super 304H with different bend radii were prepared by cold bending of tubes $\phi 38 \times 6.3$ mm [9]. R/D ratio as well as deformation of each bend was calculated for individual bend radii and is stated in Table 4. SPT specimens were also prepared in each bend from the area with corresponding deformation. In all tested bends were at disposal both results obtained by miniaturized tensile testing as well as by SPT, therefore it was possible put these data into correlation of force in SPT and stress in tensile tests. The correlation was further completed by the results of three tubes tested after long-term exposure (two tubes of grade TP347HFG exposed for 100,000 hours, one tube of grade Super 304H exposed for 13,000 hours). In Figure 13 is proved that data from SPT and tensile test, especially yield stress, can be very well correlated regardless deformation of material. The obtained results confirmed that all of the tested tubes and tube bends fitted the correlation line very well regardless of the bend radii (plastic deformation level) and/or the fact that they were tested in as-received condition or after long-term high temperature exposure.

Table 4. List of tested bends with calculated values of deformation.

Steel Grade/Radii	Deformation [%]				
	R50	R60	R80	R100	R120
▲ Super 304H	38	32	24	-	-
◆ HR3C	38	-	-	19	16
■ TP347HFG	38	32	-	19	16
R/D	1.32	1.47	2.10	2.63	3.16

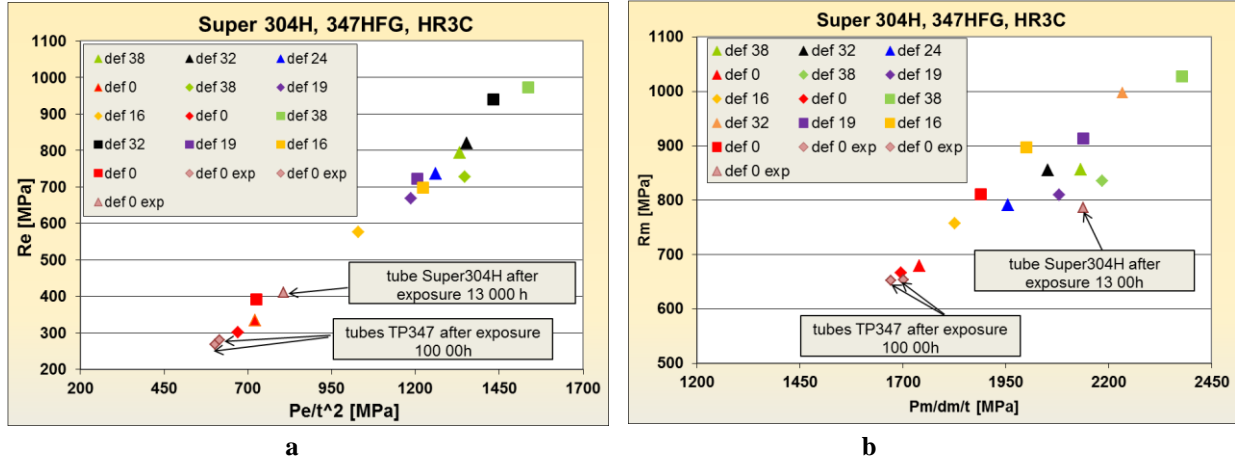


Figure 13. (a) SPT correlation of yield stress, (b) SPT correlation of tensile strength

8. Discussion

The presented paper summarized and reviewed the results of miniaturized tensile tests and small punch tests of various materials and components. It has proved the wide scale of use of SPT method, which seems to be a suitable method for evaluation materials not only in as-received state but also after long-term exposure at high temperature, e.g. after long-term degradation in power plant. It was demonstrated that the method is suitable for repeated assessment of material of boiler drums and it is capable of detecting little material changes manifested only by increasing of FATT, whereas yield stress and tensile strength stayed more or less constant. Differences in chemical composition of component correspond with heterogeneity of material and especially content of harmful tramp elements (Sb, Sn, Cu) influences fracture properties of material. Having in mind that the FATT is relatively high, it was recommended to pay attention to manipulation with this component during cold shutdown.

Another observation of material degradation was performed on turbine case bodies of ethylene unit. Periodically repeated analyses of mechanical properties confirmed decreasing tendency of the yield stress and also the yield stress/tensile strength ratio. Yield stress vs. tensile strength ratio is decreasing due to the process of grain boundary softening and decreasing of strain hardening and is one of the typical features of long-term high temperature exposure of metallic materials used in industrial components. On the other hand, perhaps all abrupt changes disturbing the trend of decreasing mechanical properties can be probably attributed either to the influence of material inhomogeneity of large castings or to a combination of other factors like decarburization on the surface of large castings accompanied by a change in strength. Therefore, it is also very important to machine small punch test specimens from the same distance from the outer surface of the tested component.

Big effort is devoted nowadays to the research of new materials for USC power plants. Small punch testing is capable to exactly analyse the material properties of thin-walled tubes as well as their welded joints and bends. The fracture behaviour of these tubes is significantly different in the fire-side and the opposite side of the tube due to precipitation of sigma phase in the microstructure. The embrittlement effect of sigma phase precipitating during long-term exposure at high temperatures is well-known [5], in the respective case the fracture energy determined using SPT was lowered by 50% in the fire- side tube.

Testing of welded joints of new austenitic heat resistant steels for USC application using SPT confirmed that weld metal was the weakest part of welded joints because its fracture energy was significantly lower compared to nearly all base materials except of steel HR3C after long-term exposure. The reason of this behaviour has not been reliably explained yet and could become a topic for further research focused on materials for USC application.

Within the study of change of mechanical properties of austenitic tube bends after technological operation the correlation between tensile test and SPT in as-received state was performed. The results of SP tests performed on tube bends with various plastic deformation (R/D ratio) fitted very well the correlation between conventional and SP tests for both yield stress and tensile strength, which means that the method can be used for the new austenitic heat resistant steel grades with wide range of strength, from solution annealed to deformation strengthened after cold plastic deformation. At the same time, the correlation can be used for materials analysed in as-received condition as well as after long-term exposure at high temperatures.

9. Conclusions

The paper presented some cases of exploiting small punch test method in analyses of mechanical properties, toughness and plasticity of materials used in the power industry and also evaluation of material degradation of power plant components after long-term exposure at high temperatures. Thanks to the minimized amount of sampled material the small punch test method is capable to perform repeated analyses in small area and to monitor changes of material properties and/or material degradation with prolonged time of exposure. Small punch testing programme performed on three newly developed austenitic heat resistant steel grades then confirmed that the method can be also used for evaluation of material properties and their changes due to both hardening caused by cold plastic deformation and degradation due to long-term exposure at high temperatures.

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