THE CHANGE OF THE STRUCTURE AND MECHANICAL PROPERTIES OF THE AUSTENITIC STEELS AFTER EXPOSURE AT THE CRITICAL TEMPERATURE

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Abstract

The paper deals with the change of the structure and mechanical properties of the austenitic steels after the exposure at the critical temperature. The effect of the bend radius of tubes on the mechanical properties and the structure was studied. The effect of solution annealing was studied too. The grades TP347 HFG, Super 304H and HR3C were chosen in the study. The bend radii R60, R80 and R100 were on the matter. The mechanical properties were studied using small punch test and miniaturized tensile tests. From the results can be concluded that the mechanical properties and the structure have been influenced due to one year exposure at the working temperature significantly. Various amounts of σ-phase were found in the pulled part of the bend side of the tubes even after only thermal exposure without any loading.

Keywords: σ-phase, stainless steel, small punch test

1. INTRODUCTION

The technical requirements of the properties of steels in many industrial segments, and especially in the power industry, are increased rapidly. The main demand is to support safety and long- live construction and design, and nowadays, this includes the change from the power plants material to the USC power plants materials. This change brings new material used in USC parameters. There is a growing interest in the so called advanced ultra-super critical materials used for coal-fired plants. The most used materials are nickel alloys and austenitic steels for that is typical high Cr content. Long term exposure of these steels at high temperatures leads to the precipitation of σ-phase. σ-phase in general is an intermetallic Cr, Fe phase that forms in austenitic steel during long term exposure at high temperatures. The presence of σ-phase leads to embrittlement of the material at ambient temperatures. This phase precipitates at elevated temperature above 400 - 500 °C and it is stable up to 700 °C. Formation of that intermetallic phase is influenced by C and Nb content too. **Figure 1** shows equilibrium phase diagram of σ-phase for TP347HFG steel [1].

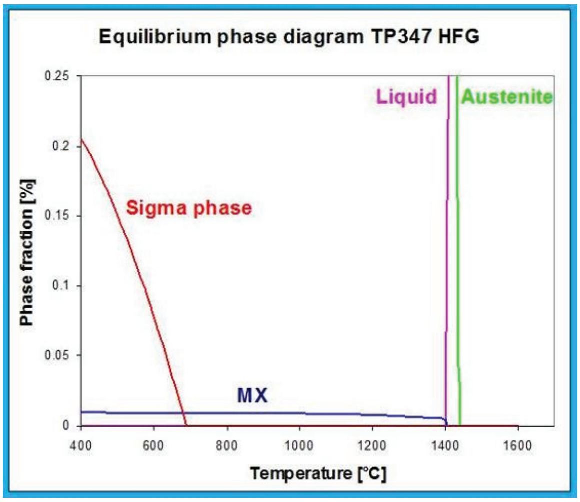


Figure 1 Equilibrium phase diagram TP347 HFG calculated with MatCalc software [1]

The main goal of this article was to quantify and qualify σ-phase precipitating in time and to assess the degradation of material properties of the tubes due to the presence of σ-phase.

2. EXPERIMENTAL MATERIAL

Experimental material consists of bend tubes as-received. The samples of the bend tubes ϕ 38 x 6.3 (app. 15 mm long) were cut out from bended part of tubes (see **Figure 2**) and located into the boiler without any loading for one year. The samples had been exposed at two working temperature range - one from 635 °C to 695 °C and another one from 726 °C to 775 °C.

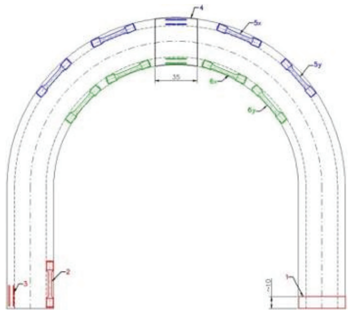


Figure 2 Samples under study and cutting plan

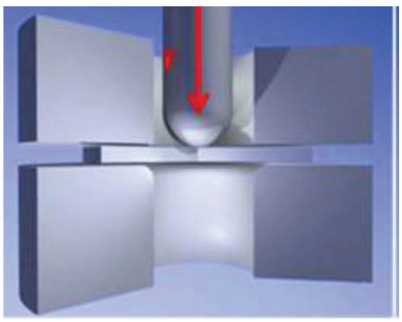
Grades TP347HFG, Super 304H and HR3C and the bend radii R60, R80 and R100 were on the matter. Almost every sample after one year exposure on the high temperature without any loading showed various amount of σ-phase. The effect of σ-phase on the mechanical properties and fracture behaviour was examined using miniaturized tensile tests and small punch test (SPT). The effect of plastic deformation applied during the bending of tubes on the structure and the mechanical properties was also studied. The positive effect of solution annealing on the structure was quantify too.

3. INVESTIGATION METHOD

To understand effect of σ-phase on material properties and structure, various testing methods based on miniaturized test techniques have been used including SPT method.

SPT were carried out according to recent version of draft of the standard for this test [2].

The SP testing technique utilises a small disc specimen, 8 mm in diameter and 0.5 mm in thickness, clamped around its circumference and indented by a spherical punch up to failure [2]. Monotonic load vs. displacement records are used to derive estimates of tensile and fracture toughness parameters (see **Figure 3**) as well as fracture energy evaluated as area under deflection curve.



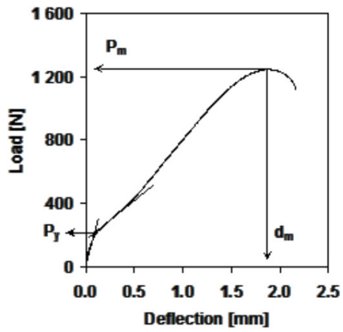


Figure 3 Principle of small punch test and typical SPT record

All tests presented in this work were performed at room temperature. All specimens prepared for metallographic examination from the examined tubes were prepared according to standard metallographic preparation techniques. Specimens were etched electrolytically with 10% oxalic acid to reveal microstructure. The microstructure of the all specimens was investigated with Light Optical Microscopy (LOM).

4. RESULTS

Unexpectedly, all specimens in as received condition that were exposed at the elevated temperature in the boiler without any loading, contains a various amount of σ-phase. Amount of σ-phase depends on temperature, bend radii and heat treatment after bending. The specimens of all examined grades with the smaller bend radii without heat treatment contain the most amount of σ-phase [3]. Logically specimens exposed at the higher temperature contain dramatically more σ-phase. The particle of σ-phase differs quantitatively and in size and even if the particles vary in occurrence in the wall of the bend tube. Specimens exposed at the lower temperature contain σ-phase mostly near the outer surface of extrados while σ-phase in specimens exposed at higher temperature occurs in the whole wall of extrados.

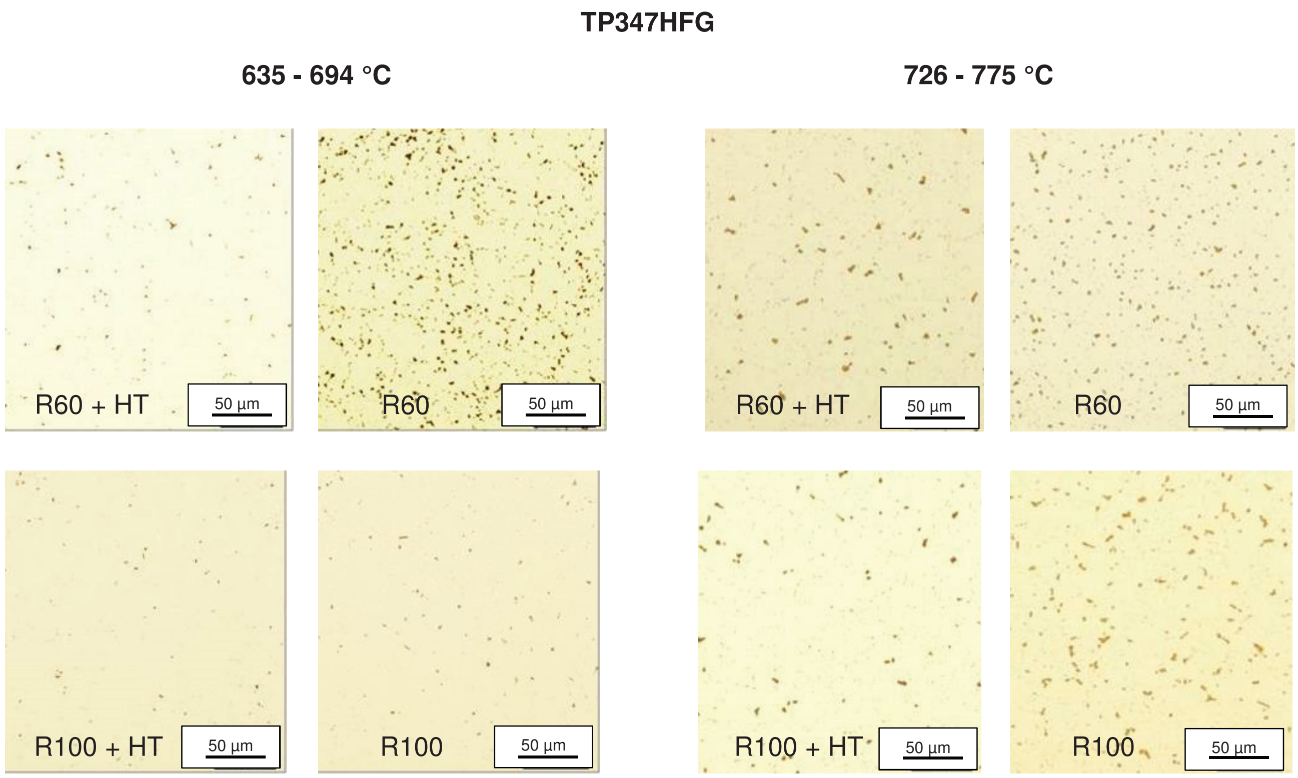


Figure 4 Comparison of the structure specimen TP347HFG as received after exposure in two location of boiler

To compare all specimens (all three steel grades under investigation) the higher exposure temperature affected the structure of TP347HFG specimens in the mildest way. Particles of σ-phase are equally distributed and they are relatively fine in all specimens. **Figure 4** shows the structures of TP347HFG specimens as received after the exposure in the boiler in two temperature range.

On the contrary the structure of HR3C specimens was affected by higher exposure temperature in more dramatically way - σ-phase particles are coarse and the biggest from all three grades. Moreover σ-phase in the specimens from bend tube without heat treatment creates almost lines at the grain border.

HR3C

635 - 694 °C **726 - 775 °C**

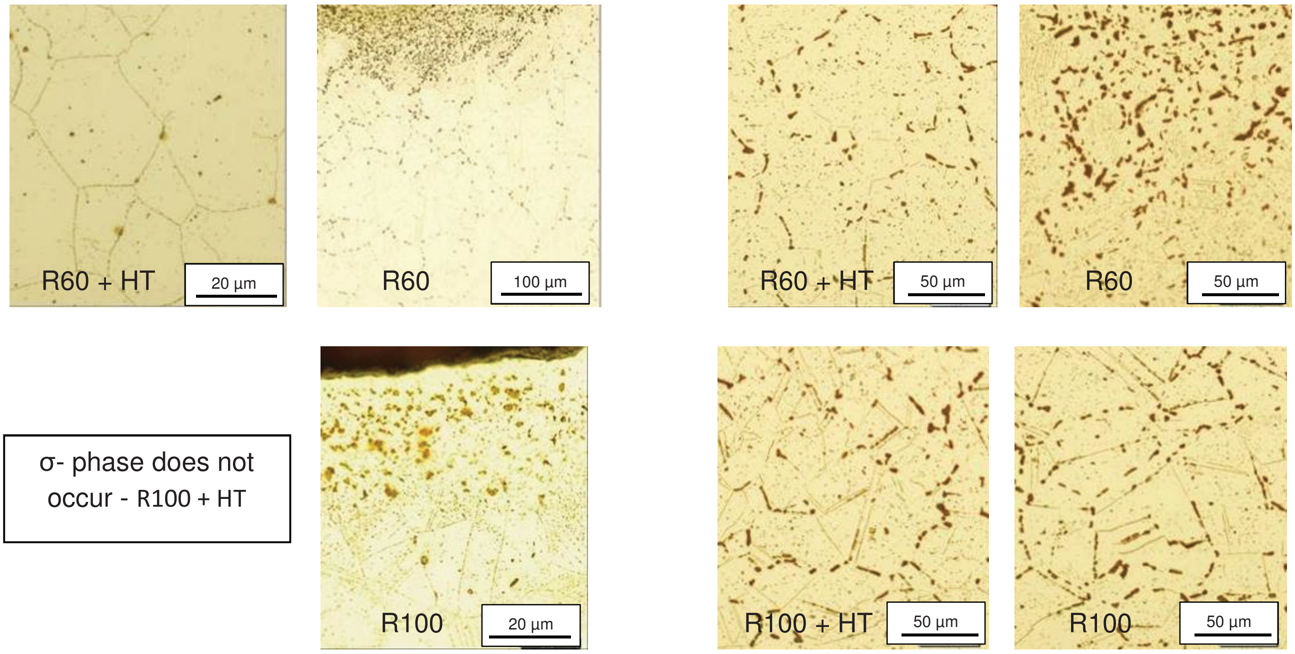


Figure 5 Comparison of the structure specimen HR3C as received after exposure in two location of boiler

Super304H

635 - 694 °C **726 - 775 °C**

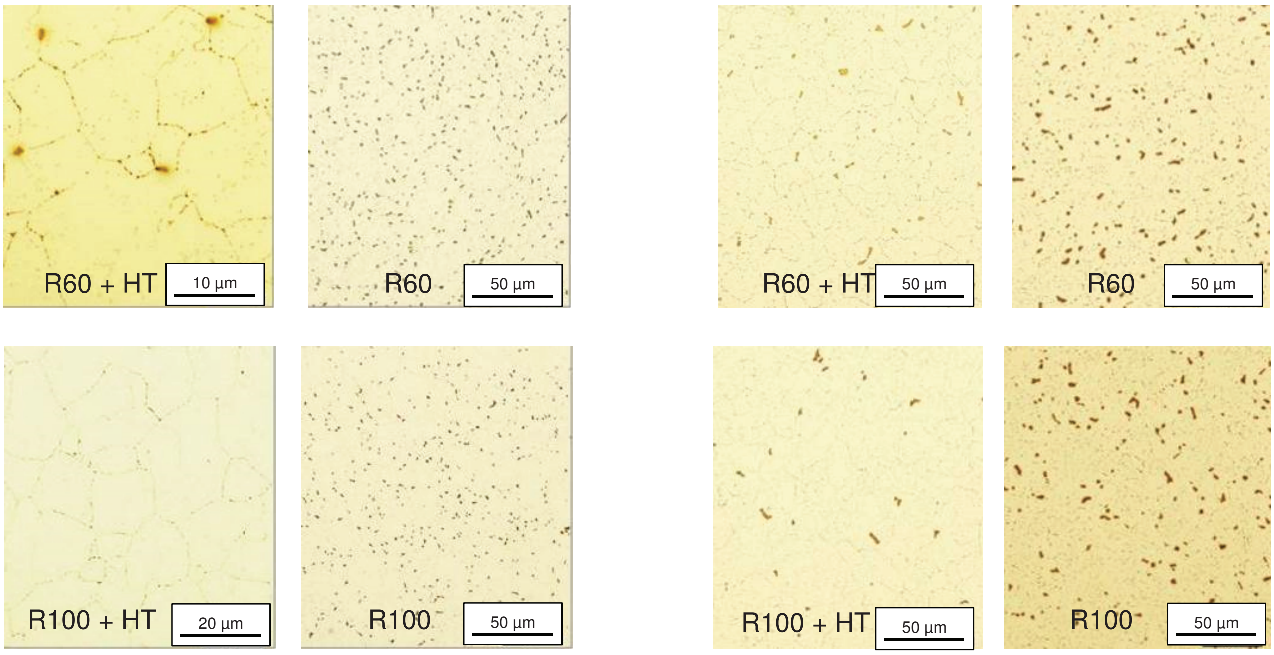


Figure 6 Comparison of the structure specimen Super 304H as received after exposure in two location of boiler

Reciprocally **Figure 5** shows the structure of HR3C specimens as received after the exposure in two locations in the boiler. The samples with heat treatment exposed at lower temperature showed very few σ-phase particles. Logically the samples without heat treatment contain dramatically more σ-phase (see **Figure 6**). The higher exposed temperature caused the coarsening of σ-phase in the samples without heat treatment and higher number of σ-phase particles in the samples with heat treatment. Presence even very small volume of σ-phase in the structure means drop in fracture energy that can be clearly detected from force - displacement record of the small punch test. The results in state as received and after the exposure (at lower temperature) were compared as well as the state with and without heat treatment after bending. **Figure 7** shows effect of σ-phase in the structure at TP347HFG samples. Effect of working temperature on the fracture energy represented by SPT method is the most evident at bend tube HR3C (see **Figure 8**). Probably another mechanism caused drop in force versus displacement after exposure compare to state as received. The mildest change of record force versus displacement due to σ-phase in the structure becomes evident at Super304H bends from all three examined grades (see **Figure 9**).

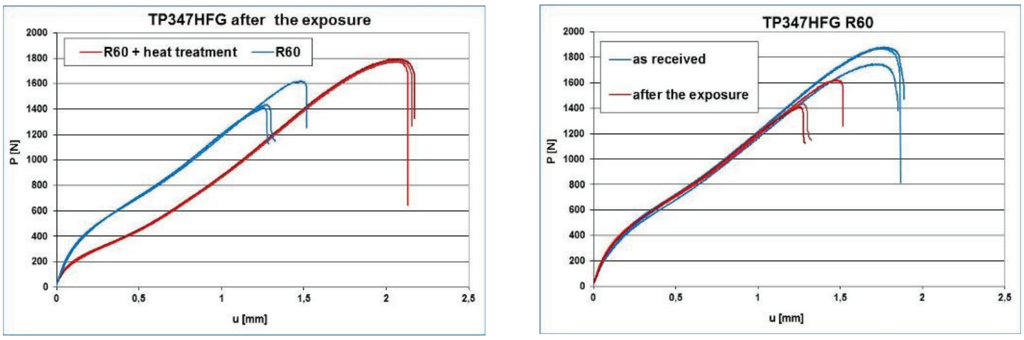


Figure 7 Force versus displacement of bend tube TP347HFG in state as received and after the exposure

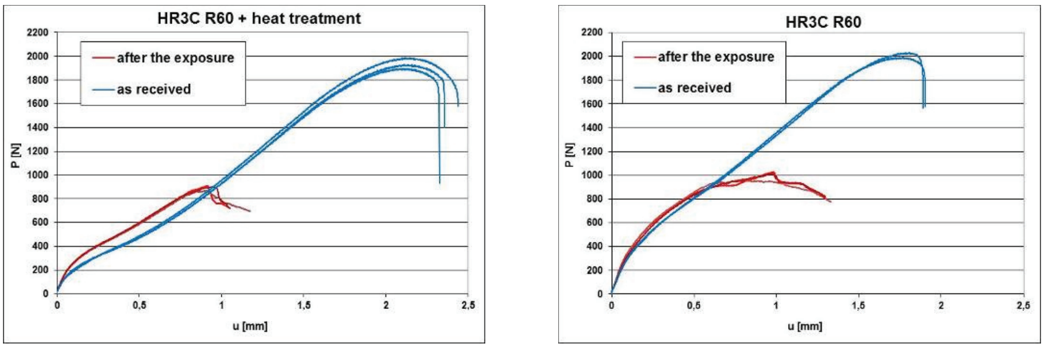


Figure 8 Force versus displacement of bend tube HR3C in state as received and after exposure

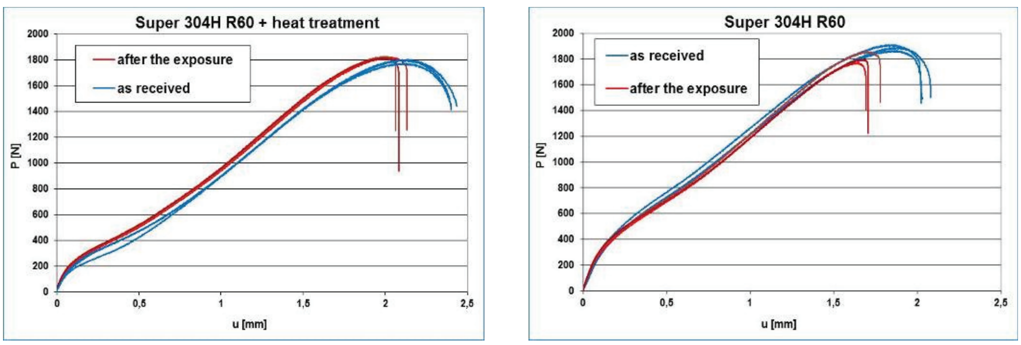


Figure 9 Force versus displacement of bend tube Super 304H in state as received and after exposure

5. DISCUSSION

Presented results indicate that σ-phase is a factor that is necessary to remember during the exposure in the USC plants. Detecting of σ-phase in the structure of bend tubes after one year exposure only at the high temperature without any loading is very important. It is necessary to point out that examined samples were bend tubes and σ-phase was detected just in the extrados at the outer surface (lower exposure temperature) or in the whole wall section of extrados (higher exposure temperature). It is evident that the amounts of σ-phase in the bend tubes is connected with the amount of deformation energy put into the wall of bend which decreases the activation energy of σ-phase precipitation. So samples with heat treatment after bending and with bigger bend radii show just low or any content of σ-phase. This fact is supported by another analysis made on the straight part of bend tubes after exposure (see **Figure 10**).

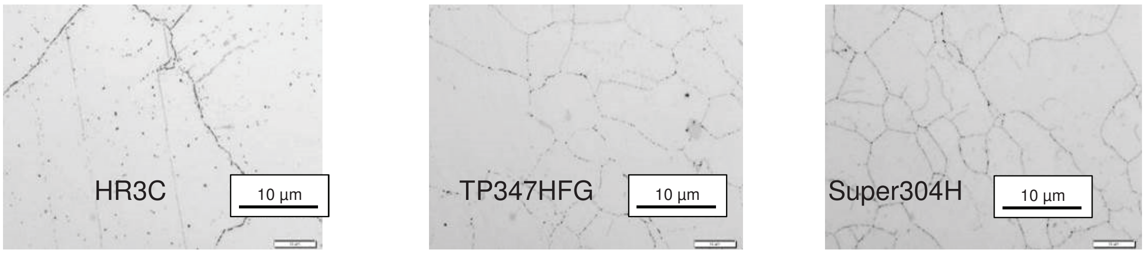


Figure 10 The structure without σ-phase in the straight part on bend tubes after the exposure

It means that the bend tubes with low bend radii (R60, R80) require heat treatment after bending to minimalize presence of σ-phase in the structure. If the exposure temperature is higher than 700 °C it is necessary to do heat treatment after bending even if for bend radii R100.

6. CONCLUSION

After exposure at temperature range 635 - 694 °C small amount of σ-phase has been found in all specimens near the surfaces, at higher temperature of exposure σ-phase has been found across the wall thickness of extrados. The effect of bend radii has been clearly identified - small bend radii contain more σ-phase compare to bigger one. To minimalize the volume of σ-phase in the structure of extrados with small bend radii it is necessary to carry out solution annealing after bending. SPT can be used for evaluation of effect of σ-phase in austenitic steels - high amount of σ-phase affects the fracture energy of SPT significantly.

ACKNOWLEDGEMENTS

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REFERENCES

[1] HSIEH, C. C., WU, W. Overview of Intermetallic Sigma (σ) Phase Precipitation in Stainless Steels. International Scholarly Research Network, ISRN. Metallurgy, 2012, vol. 2012, Article ID 732471, 16 p.

[2] Small Punch Test Method for Metallic Materials. CEN WORKSHOP AGREEMENT CWA 15627, December 2007.

[3] HORVATH J., KRAL P., JANOVEC J., SKLENICKA V. The effect of sigma-phase formation on long-term durability of Super 304h steel. In METAL 2015: 24rd International Conference on Metallurgy and Materials. Ostrava: TANGER, 2015, pp. 505 - 510.