

## RELATION OF MICROSTRUCTURE AND RESISTANCE TO SULPHIDE STRESS CRACKING OF 22/05 DUPLEX STEEL

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### ABSTRACT

*The paper is focused on evaluation of resistance of duplex austenite-ferrite stainless steel of grade 22/05 according to EURONORM 1.4462-X2CrNiMoN 22.5.3 to sulphide stress cracking (SSC). Steel was tested in two different conditions. First of all in as-received state after rolling and secondly after annealing by regime 675<sup>0</sup>C/ 5h/ air, which caused a precipitation of the phase  $\sigma$ . The aim of this work was to demonstrate that SSC resistance of steel is strongly dependent on microstructure and can't be derived only from material hardness, which is a common engineering approach.*

**Keywords:** Sulphide stress cracking (SSC), microstructure, mechanical properties, heat treatment

### 1. INTRODUCTION

Many structural materials, including steels, are exposed during their exploitation to impacts of aggressive environment. In order to ensure safety and long-term reliability of structures made of these materials, it is necessary to design these materials in such a manner that they resist to influences of aggressive environment. Hydrogen sulphide, which belongs to the most aggressive components, can be encountered at extraction, transport and storage of oil and natural gas, i.e. in petrochemical industry. Hydrogen can penetrate from liquid and gaseous environments, containing some portion of hydrogen sulphide, into materials and cause thus their degradation. One of the most dangerous types of damage is described as Sulphide Stress Cracking – SSC [1].

### 2. EXPERIMENT

We have used a duplex austenite-ferrite steel of grade 22/05 according to EURONORM 1.4462-X2CrNiMoN 22.5.3 (sheet with thickness of 12mm). The steel was tested in two different conditions – in as-received state after rolling and after annealing by regime 675<sup>0</sup>C/ 5h/ air, when there occurred decay of part of  $\delta$ -ferrite and  $\sigma$  phase was formed. Our investigation was aimed particularly at verification of dependence of SSC resistance of steel on micro-structural changes (precipitation of minority phases).

Chemical composition of duplex steel is given in the table 1.

Table 1. Chemical composition of duplex steel (mass %)

C	Cr	Ni	Mo	Mn	Si	P	S	N <sub>2</sub>
0.019	22.67	5.48	2.98	1.68	0.44	0.024	0.0002	0.17

Mechanical properties of duplex steel are given in the table 2., these values represents a mean value of 2 tensile tests.

Table 2. Mechanical properties of duplex steel (longitudinal direction)

steel condition	R <sub>p0.2</sub> (MPa)	R <sub>m</sub> (MPa)	A <sub>5</sub> (%)	R <sub>p0.2</sub> / R <sub>m</sub>
after rolling	540	741	33.5	0.73
after annealing	534	714	28.5	0.75

Microstructure of duplex steel in the as-rolled state is shown in fig. 1. It is an austenitic-ferritic structure, when both phases form predominantly rows oriented in rolling direction. Microstructure of duplex steel after annealing by regime 675<sup>0</sup>C/ 5h/ air is shown in fig. 2. It is also a mixed austenitic-ferritic structure, however, in part of δ-ferrite there was observed a decay accompanied by forming of phase σ. The steel was etching with 20% aqueous solution of NaOH.

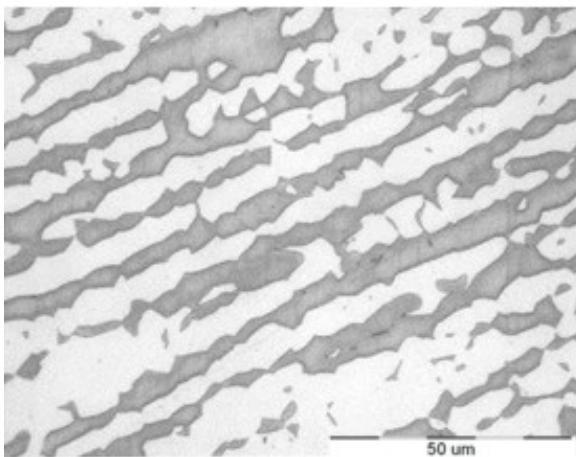


Figure 1. Microstructure of duplex steel in the as-rolled state, etching with NaOH.

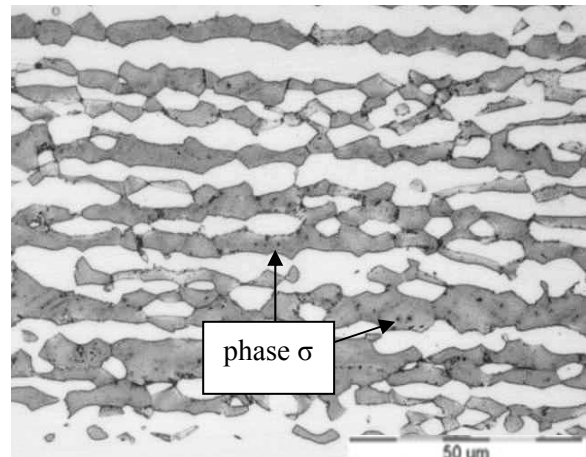


Figure 2. Microstructure of duplex steel after annealing 675<sup>0</sup>C /5h/ air, etching with NaOH.

### **Results of evaluation of SSC resistance of duplex steel**

Evaluation of resistance to SSC was made with use of round bars with nominal diameter of 3.81mm, completely in compliance with the directive NACE TM 0177 – 96, method A [2]. After filling of testing chambers with corrosion solution (we used a solution A), its degassing and saturation by hydrogen sulphide the samples were subjected to a stress, which corresponded to certain portion of ascertained yield strength, and they were exposed to the given environment. We tested 9 samples for each condition of steel, i.e. usually 2 test bars for the given load. Exposure duration was 720 hours, unless the test bar broke before elapsing of this time. The results are given in the fig. 3.

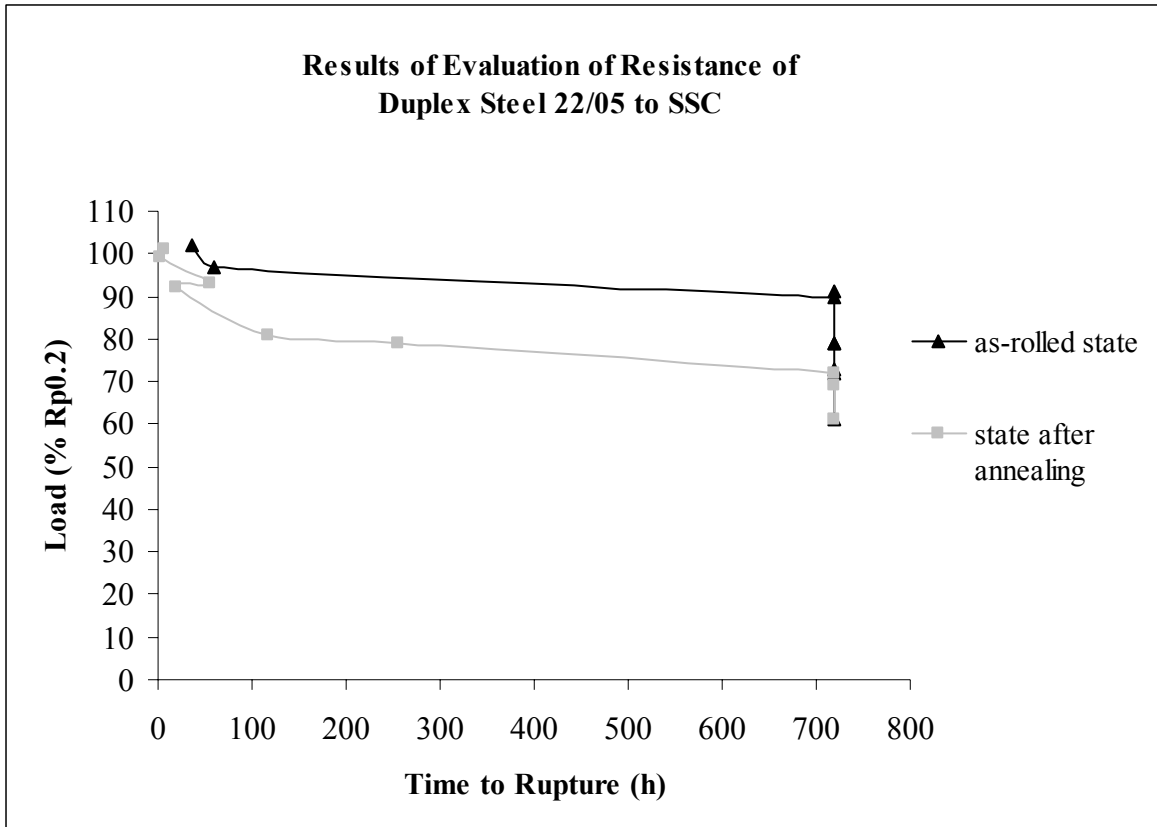


Figure 3. Results of evaluation of resistance of duplex steel to SSC.

It is obvious from the graph that in the as-rolled state after rolling the rupture has occurred only in two samples, which were exposed to the stress at the level of yield strength of the material. Due to concurrent presence of ferrite and austenite it can be presumed that yield strength could have been locally exceeded in ferrite and therefore there could have taken place a mechanism of interaction hydrogen-plastic deformation, which is characterised by important interactions between hydrogen and dislocations and transport of hydrogen together with them is possible. At the same time the steel slightly exceeds the value of ultimate strength recommended for ensuring good resistance to SSC. This is specified to be 690MPa. Load is in practice almost exclusively elastic, which means that there is no such hazard. Duplex steel of the type 22/05 with purely austenite-ferrite structure has therefore manifested very good resistance to SSC.

Situation was different in the state after annealing by regime 675<sup>0</sup>C/ 5h/ air, which had caused discontinuous precipitation of  $\sigma$ -phase. In this case ruptures occurred at stresses of approx. 80 % of yield strength, i.e. that there occurred distinct reduction of resistance to SSC.

### 3. CONCLUSION

The aim of this work was to test SSC resistance of duplex austenite-ferrite stainless steel of grade 22/05 according to EURONORM 1.4462-X2CrNiMoN 22.5.3. Steel was tested in the as-rolled state after rolling and also after annealing by regime 675<sup>0</sup>C/ 5h/ air. Tested duplex austenite-ferrite steel in the as-rolled state, when a precipitation of minority phases did not occur and structure was exclusively austenitic-ferritic, showed very good SSC resistance. In the state after annealing decomposition occurred of a part of  $\delta$ -ferrite accompanied by the precipitation of  $\sigma$ -phase, which resulted in considerable reduction of resistance of duplex austenite-ferrite steel to SSC.

It was established that SSC resistance of duplex austenite-ferrite steel of the grade 22/05 is dependent on structure and in our case it is deteriorated by presence of  $\sigma$ -phase.

#### **4. REFERENCES**

- [1] TIMMINS, P., F.: Solution to Hydrogen Attack in Steels, USA, 1997, 198 pp.
- [2] NACE Standard TM 0177-96: Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking in H<sub>2</sub>S Environments, NACE Int., Houston Texas, USA, 2003, 35 p.
- [3] SOJKA, J., JONŠTA, P., RYTÍŘOVÁ, L., SOZAŇSKA, M., JEROME, M. : Influence of Microstructure on Sulphide Stress Cracking of Hot Rolled Tubes, Acta Metallurgica Slovaca, vol. 11., 03/2006 p. 323-329, ISSN-1335-1532.

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