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IMPROVING THE RESISTANCE TO STRESS CORROSION CRACKING AND TO HYDROGEN EMBRITTLEMENT OF BAINITE HIGH STRENGTH STEEL

ABSTRACT

Susceptibility to pitting corrosion, hydrogen transport, hydrogen embrittlement and susceptibility to stress corrosion cracking have been studied under the "sea water" and the "acid rain" simulated conditions in order to evaluate the optimum chemical composition, heat treatment, microstructure and surface treatment of the high strength low carbon bainite 0.3C-1Cr-1Mn-1Si-1Ni type steel for replacement the airplane parts. Resistance to stress corrosion cracking under the open circuit conditions, associated with the resistance to pitting corrosion in Cl⁻ containing solution increased with decreasing carbon content and after application of shot peening. Resistance to hydrogen embrittlement was also higher for steel with the low carbon content. Shot peening increased the hydrogen trapping efficiency within deformed layer, but the presence of the shot peened layer decreased the hydrogen flux entering the core and thus decreased susceptibility of core to hydrogen embrittlement.

The optimal chemical composition, heat treatment and surface treatment parameters, mechanical properties and microstructure of steel providing the improvement of the resistance to SCC in Cl⁻ containing environments allowing the replacement of the standard 30HGSNAŽ steel at production of airplane parts have been evaluated.

Keywords: high strength steel, Cl⁻, pitting corrosion, stress corrosion cracking, hydrogen embrittlement

INTRODUCTION

Investigation of some failure cases of the 0.3C-1Cr-1Mn-1Si-1Ni (30HGSNAŽ according to Polish Standards) type high strength steel used for the parts of agricultural airplanes has shown that at exploitation in environment containing the aggressive ions, the fracture occurred due to the corrosion and mechanical action. The microcracks were initiated at corrosion pits and propagated according to the hydrogen embrittlement mechanism. Detailed analysis has shown that the material of failed parts contained the carbon amount close to the upper limit stated by the Standard.

In the laboratory tests [1], the different susceptibility to hydrogen induced cracking of steel in chlorides containing solutions has been observed, depending on the variation of heat treatment and the carbon content (both being in the accordance with the Standard). It has been also stated that the decrease in pH of Cl⁻ containing solution produced the detrimental effects.

From the results of failure case analysis and from the data reported in the literature, it has been concluded that the formation of the bainite structure and the decrease in the carbon content should improve the resistance to local corrosion and to hydrogen embrittlement, and thus to improve the resistance to stress corrosion cracking in Cl⁻ containing environment.

Since at the low carbon content, the strength required for the aircraft parts may be decreased, the shot peening (widely used in the aircraft industry [2]) has been proposed to be applied.

In present work, the evaluated optimum chemical composition and heat and surface treatment of the 30HGSNAŽ type steel, providing the improvement of susceptibility to pitting corrosion, to hydrogen embrittlement and to stress corrosion cracking in Cl⁻ ion containing environments have been presented.

MATERIALS AND EXPERIMENTAL PROCEDURE

Chemical compositions of the new experimental low carbon steel, the reference standard 30HGSNAŽ steel and those required by the Polish Standard are given in Table 1. The parameters of the heat treatment of the new steel were specially selected in order to obtain the structure and mechanical properties required by the corresponding standards for the airplane parts. Specimens of experimental steel were machined from the slabs, which were normalized at 900°C. After preparation, the specimens were isothermally quenched from 910°C in oil at 80°C and then aged at 190°C for 3 hr. The specimens of the standard 30HGSNAŽ steel, after normalization at 900°C and preparation were quenched from 900°C in oil at RT and then tempered at 210°C for 2 hr. After heat treatment, all the specimens were mechanically polished to attain the surface roughness lower than 0.32 μm (Polish Standard $0.32\sqrt{\text{ }}$) and additionally aged at 120°C in oil for 3 hr, according to airplane parts requirements.

Table 1. Chemical composition of studied materials

Material	C, %	Cr, %	Mn, %	Si, %	Ni, %	S, %	P, %
Experimental steel	0.253	1.12	1.13	1.04	1.65	0.004	0.017
30HGSNAŽ standard steel*	0.31	1.03	1.13	1.02	1.50	0.003	0.023
Polish Standard for 30HGSNA steel	0.27-0.34	0.90-1.20	1.00-1.30	0.90-1.20	1.40-1.80	max 0.25	

* Ž – in the steel code means the high purity of steel concerning the S and P amount and the nonmetallic inclusions

Heat treated experimental steel was subjected to shot peening by the cast iron shots (0.43 mm mesh and 470HV of hardness) pneumatically injected for 60s at the air pressure 0.5 MPa. Those parameters provided the coverage higher than 100%. Shot peened specimens were mechanically polished to remove the possible surface defects produced by the peening and to obtain the surface smoothness mentioned above.

The mechanical properties of experimental and standard steels after heat and surface treatment, as well as those required by the Polish Standard are presented in Table 2.

Table 2. Mechanical properties of studied steels and the requirements of the Polish Standard for 30HGSNA steel

Material	σ_Y , MPa	σ_{UTS} , MPa	ϵ_f	RA, %
Experimental steel, without deformation	1120	1590	0.11	47.6
Experimental steel, shot peened	1030	1700	0.116	57
30HGSNAŽ standard steel	1520	1740	0.26	46.2
Requirements according to Polish Standard	min. 1400	min. 1650	min. 0.09	min. 45

Susceptibility to stress corrosion cracking was investigated in two solutions. In order to compare the results obtained for experimental steels with the previously obtained ones and with the literature data, the 3% NaCl (pH=5.5). This solution may also simulate the conditions of the sea shore atmosphere and dew. Taking into account the increased aggressiveness of the environment, and especially the effects of the “acid rain” and the “acid dew”, the solution containing 30g/l NaCl + 71g/l Na₂SO₄ in distilled water (pH=3), simulating the “acid rain” [3], was used as the other test solution. Experiments were done at RT, in the non deaerated electrolytes.

Susceptibility to pitting corrosion was determined as the maximum depth of pits formed on the specimens surface after immersion in 0.1M FeCl₃ solution for 22 h [4].

Hydrogen permeation measurements were performed electrochemically using the double cell [5] divided by the studied membrane. The egress surface of the membrane was galvanostatically covered with the Pd film and exposed to the egress cell, filled with 0.1N NaOH solution. The potential +150 mV was applied to the egress side of a membrane and the anodic current being the measure of the hydrogen permeation rate was recorded in the egress cell. The ingress side of a membrane was exposed to the tests solution and was cathodically polarized at the constant potential -1500 mV. All potentials are given vs. the Hg/HgO reference electrode. The shot peened surface served as the ingress side of the membrane.

The build-up and decay hydrogen permeation transients were recorded in the egress cell at application and at cessation of cathodic polarization, respectively. The following parameters of hydrogen transport evaluated from the hydrogen permeation measurements of the studied steels were compared:

- J_{-1500}^{∞} – steady state hydrogen permeation current at cathodic polarization -1500 mV;
- D_{tb} – apparent hydrogen diffusivity calculated from the decay transient.

The values of hydrogen diffusivity were calculated according to equations [5, 6]:

$$D_{tb} = 0.05L^2/\tau_{tb}$$

where:

- L – the membrane thickness;
- τ_{tb} – break through time established on the decay transient.

The mean hydrogen content, left in the membrane (V) after the completing the hydrogen permeation test was calculated from the amount of hydrogen desorbed at vacuum extraction measurements at 400°C.

Stress corrosion cracking experiments were done at tensile straining of specimens (3 mm in diameter and 30 mm in gauge length). After pouring electrolyte into the cell with mounted specimen, it was kept for 30 min at open circuit potential (E_{ocp}) or at cathodic polarization (-1500 mV) and then stretched until the fracture at the strain rate $5.5 \times 10^{-6} \text{ s}^{-1}$ without changing polarization.

Susceptibility to stress corrosion cracking was determined as the ratio of the elongation to fracture of specimens tested in solution to that tested in air ($\epsilon_f^{E_{ocp}}/\epsilon_f^0$ and $\epsilon_f^{-1500}/\epsilon_f^0$).

The microstructure features of steels and the depth of the shot peened layer were estimated by quantitative metallography [7]. The side and fracture surfaces of the tensile specimens and the ingress surface of the membranes subjected to hydrogen permeation tests were observed by means of SEM. The presence of the retained austenite and the effect of the shot peening on the material state were checked by X-ray analysis with the Bruker AXS 8 diffractometer using Cu_{K α} radiation.

RESULTS AND DISCUSSION

Both steels exhibited the bainitic structure consisting of the acicular bainite ferrite laths and the cementite particles. X-ray investigation revealed no retained austenite. As follows from the comparison of the microstructure of non deformed part of the shot peened specimens of experimental steel with the microstructure of deformed layer (Figures 1a and 1b, respectively), the shot peening resulted in the refining of the bainite laths within the surface layer, 120 μ m deep. The X-ray analysis showed the broadening of the X-ray lines of shot peened material in comparison with the non peened one.

As seen in Figure 1c, after the shot peening procedure, the uneven surface and some surface defects can be traced. Since those defects may serve as the nucleation sites for pits and microcracks, they should be removed. For this reason, the mechanical polishing of shot peened specimens was done.

The results of pitting corrosion tests, stress corrosion cracking, hydrogen permeation tests, vacuum extraction measurements, and the hydrogen embrittlement tests of studied steels are collected in Tables 3, 4, 5 and 6.

Table 3. Susceptibility to pitting corrosion and to stress corrosion cracking at open circuit potential of studied steels

Material	Maximum depth of pits, h_{\max} , μ m	$\frac{E_{ocp}^0}{E_f^0}$ NaCl	$\frac{E_{ocp}^0}{E_f^0}$ NaCl + Na ₂ SO ₄
Experimental steel	78	1.0	0.74
Experimental, shot peened steel	0*	0.96	0.93
30HGSNAŽ standard steel	210	0.64	0.37

* – no pits has been observed

Table 4. Hydrogen diffusivity and the steady state hydrogen permeation rate estimated for studied steels.

Material	Hydrogen diffusivity D_{H_2} , cm ² /s	Steady state hydrogen permeation at polarization in 3% NaCl solution J_{-1500}^{∞} , μ A/cm ²	Steady state hydrogen permeation at polarization in NaCl + Na ₂ SO ₄ solution J_{-1500}^{∞} , μ A/cm ²
Experimental steel	$4.7 \cdot 10^{-7}$	1.3	1.8
Experimental, shot peened steel	$1.7 \cdot 10^{-7}$	1.0	0.43
30HGSNAŽ standard steel	$2.9 \cdot 10^{-7}$	1.5	1.1

Table 5. Residual hydrogen content as measured for studied steels by vacuum extraction from the membranes after completing the permeation tests

Material	Residual hydrogen content left after hydrogen permeation tests at -1500 mV in NaCl + Na ₂ SO ₄ , wt. ppm
Experimental steel	0.58
Experimental, shot peened steel	1.34
30HGSNAŽ standard steel	0.55

Table 6. Susceptibility of studied steel to hydrogen embrittlement (to stress corrosion cracking at cathodic polarization)

Material	$\frac{E_f^{-1500}}{E_f^0}$ NaCl	$\frac{E_f^{-1500}}{E_f^0}$ NaCl + Na ₂ SO ₄
Experimental steel	0.47	0.54
Experimental, shot peened steel	0.44	0.35
30HGSNAŽ standard steel	0.25	0.28

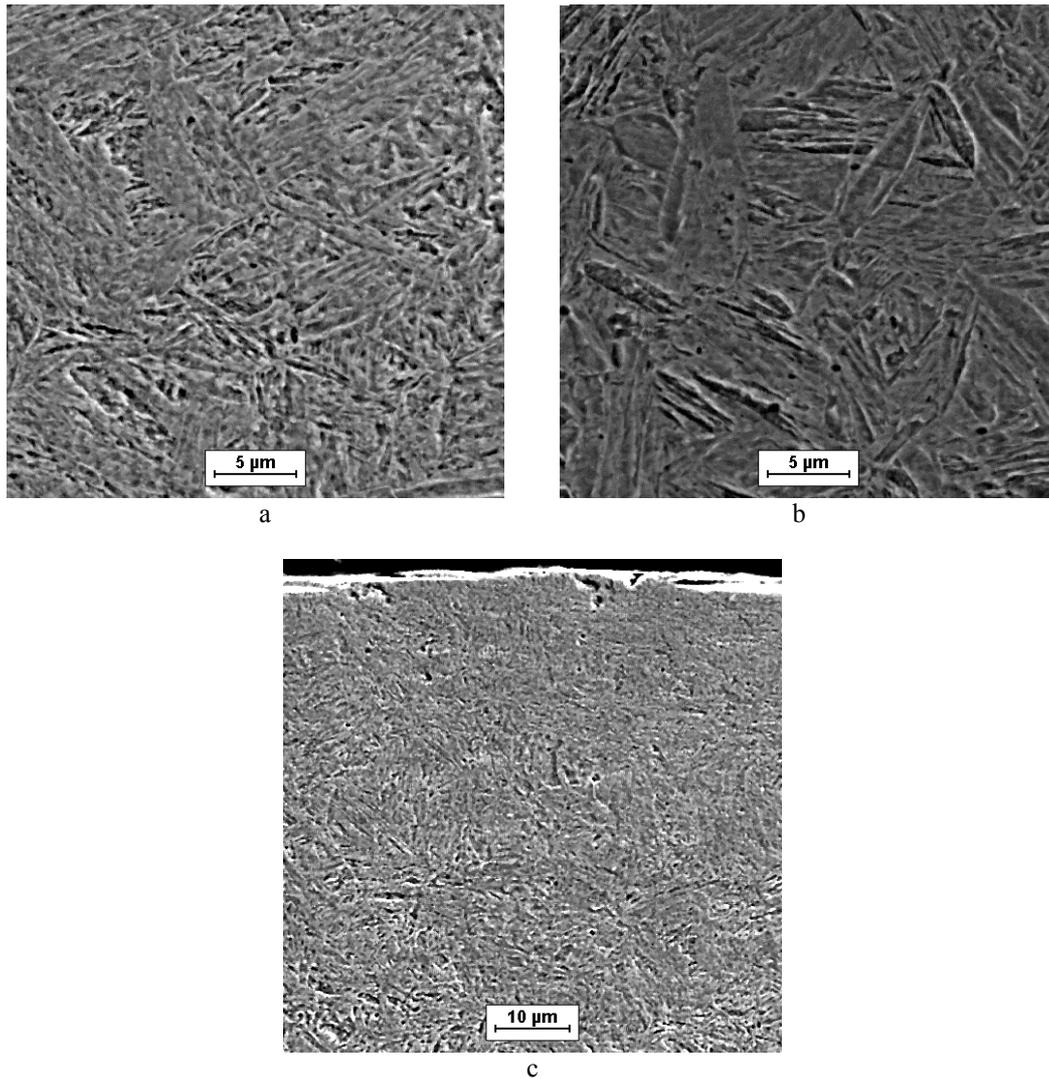


Figure 1. Microstructure of experimental steel (SEM).
(a) – in the core of shot peened steel
(b) – in the shot peened layer;
(c) – at the surface of shot peened steel, before removing defected surface layer.

As seen from Figure 2, in both solutions the cracks leading to the specimen fracture under the open circuit conditions were associated with the corrosion pits formed on the side surface of specimens. Therefore, the susceptibility to stress corrosion cracking of the steels in studied solutions should be accounted by the susceptibility to pitting corrosion. Indeed, as follows from the Table 3, the experimental shot peened steel exhibiting the lowest susceptibility to pitting corrosion as established in Cl⁻ containing solution, exhibited also the lowest susceptibility to stress corrosion cracking at the open circuit potential in NaCl and the “acid rain” simulated electrolytes.

The hydrogen induced cracking of the high strength steel has been affected by the hydrogen diffusivity, which determined the rate of hydrogen to reach the hydrogen traps, and by the

hydrogen entry flux, which determined the build-up the local concentration of trapped hydrogen to some critical level, necessary to initiate and propagate the cracking [8 - 10].

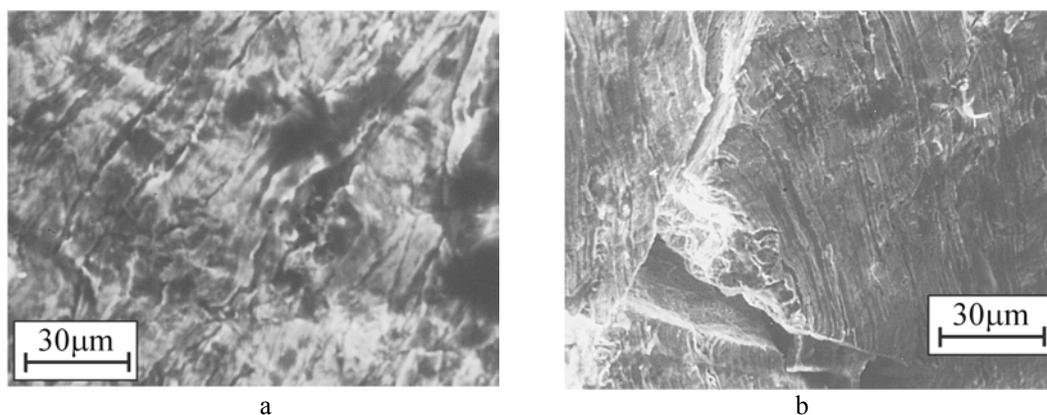


Figure 2. Side surface of 30HGSNAŽ specimens after stress corrosion cracking tests carried out under the open circuit conditions (E_{ocp}).
 (a) – after test in 3% NaCl solution;
 (b) after test in NaCl+Na₂SO₄ solution.

Under the similar polarization condition, the hydrogen entry flux could be similar for all the studied steels. Therefore, the observed difference in the steady state permeation (Table 4) reflected the hydrogen trapping within the steel. It has been known [11] that the higher the hydrogen trapping, the lower steady state hydrogen permeation current has been recorded in the egress cell, under the similar conditions. It is seen in Table 4, that the experimental shot peened steel exhibited the lowest value of the steady state hydrogen permeation. Therefore, the maximum hydrogen trapping may be assumed to occur in this steel. The hydrogen accumulation within the peened layer was supported by the data of vacuum extraction (Table 5). It is seen that under the similar condition, shot peened steel exhibited the maximum content of residual hydrogen. This was a result of intensification of hydrogen trapping due to the change of the microstructure produced by the shot peening (Figure 1).

As follows from the comparison of data in Tables 4 and 6, in both solutions the unpeened experimental steel exhibited higher resistance to hydrogen embrittlement (to SCC at cathodic polarization) and the higher steady state permeation than the 30HGSNAŽ one. This suggests that the susceptibility to hydrogen embrittlement has been associated with the trapping efficiency of steel: the higher the trapping efficiency, the lower the resistance to hydrogen embrittlement.

The shot peening of the experimental steel decreased its resistance to hydrogen embrittlement in comparison with the not peened one, cf. Table 6. This was accompanied by the increase in the amount of residual hydrogen (Table 4) and by the decrease in hydrogen permeation (Table 3). Therefore, the shot peening increased the hydrogen trapping efficiency within deformed layer, but the presence of the shot peened layer decreased the hydrogen flux entering the core and thus decreased susceptibility of core to hydrogen embrittlement.

It should be emphasized, however, that even though the shot peened experimental steel exhibited the lower resistance to hydrogen embrittlement than the non peened one, it still had the much higher resistance to stress corrosion cracking at cathodic polarization than the standard 30HGSNAŽ one, cf. Table 5.

CONCLUSIONS

The studied experimental steel (25HGSNAŽ) with the carbon content below the lower limit required for 30HGSNAŽ steel by the Polish Standards, subjected to surface modification by shot peening exhibited the required mechanical properties and the resistance to pitting corrosion, to stress corrosion cracking at open circuit potential and to hydrogen embrittlement higher than those established for the standard steel. Therefore, the 25HGSNAŽ steel may be applied for the production of aircraft parts, as the replacement of the 30HGSNAŽ steel.

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