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Study of stress corrosion cracking initiation of high alloy materials

Blahetová Marie[a*], **Čihal Vladimír**[a], **Lasek Stanislav**[a]

[a] VŠB – Technical university of Ostrava, Dep. of materials engineering, tř. 17. listopadu 15, 708 33 Ostrava – Poruba, Czech Republic, tel: +420596995205, fax: +420596664401

Abstract

The stainless steels and related alloys with sufficient resistance to a general corrosion can be susceptible to a localized corrosion (pitting, cracking, intergranular corrosion) in certain environment under specific conditions. The Drop Evaporation Test (DET) was developed for study of stainless materials resistance to stress corrosion cracking (SCC) at elevated temperatures 100 – 300 °C under constant external load using a chloride containing water solution. In the contribution the initiation and propagation of short cracks as well as pits were observed during the test. The crack initiation and/or propagation can be influenced by the cyclic thermal stresses, when the diluted water solution drops cool down the hot sample. The coordinates measurement of microscopic pits and sharp corrosion crack tips by the travelling microscope method allowed to derive the crack growth lengths and rates of short cracks.

Keywords : Stress corrosion cracking, pitting, intergranular corrosion.

Introduction

Stress corrosion cracking (SCC) is not well predictable and can cause a dangerous failure of pressure vessels, pipes, apparatus, etc. For testing of the material resistance to SCC at higher temperatures (100-300°C) in water environments were proposed several methods. The Drop Evaporation Test (DET) is useful for comparison and evaluation of resistance of stainless steels and related materials to SCC in chlorides containing water environments [1]. This test follows the results of failure analysis in cases, when evaporation of water environments (heating elements, boilers, steam generators) can increase the content of anions (chlorides) and there is possibility to form a salt deposition (crevice) on material surface.

The relatively new DET can replace older SCC tests in boiling solution of $MgCl_2$ and also DET can partially replace expensive and complicated tests in autoclaves at higher temperature (200 – 300˚C) and pressures [2]. The advantage of DET is possibility of testing at higher temperature in the range 100~500°C using a lower Cl^- water content (0-100 ppm) in convenient gas around the specimen. During DET there is a good possibility to observe a sample surface, initiation and propagation of pitting as well as cracking [3].

The process of thermal and corrosion fatigue is superposed on SCC mainly in the stage of initiation and propagation of short cracks especially in case of a higher difference of temperature between drops and heated sample. The DET simulates also the influence of heat transfer from material to environment on localized corrosion under evaporating conditions.

The aim of this contribution is the study of initiation and short corrosion cracks propagation on duplex steel of type X2CrNiMoN 22-5-3 (1.4462) by means of travelling microscope method under DET conditions.

Experimental

Materials and specimens

The chemical composition of tested duplex steel X2CrNiMoN 22-5-3 (1.4462) is given in Table 1. The austenitic and ferritic phases are approximately 50% (Fig. 6).

Table 1: Chemical composition (% wt.)

element	Ni	Cr	Mo	Si	Mn	N	C
wt. %	6,0	23	3,0	0,8	2,0	0,08	0,02

The specimens in the form of small rods (overall length 100 mm, cross section ϕ 4 mm, central circular section ϕ1,8 x 10 mm for corrosion exposition) were made from plates by water jet cutting with addition of erosive particles, followed by precision turning and finish grinding of circular section in parallel direction with sample axis (SiC papers P2000, P4000, surface roughness $R_a \leq 0{,}1$ μm). Before testing the specimen diameters were precisely measured by micrometer and small circular sections were cleaned and degreased.
The tensile tests were also performed on these small specimens for comparison purposes and for determination of relative stresses σ/R_m, $\sigma/R_p0{,}2$, where σ is applied external tensile stress. The main mechanical properties (R_m - tensile strength, $R_p0{,}2$ - yield strength, A – elongation, Z – contraction are given in Table 2.

Table 2: Mechanical properties (at room temperature)

Material	R_m [MPa]	$R_p0{,}2$ [MPa]	A [%]	Z [%]
Cr22Ni5Mo3N	742	511	37	33
Small specimens	770	589	41	36

Testing procedure

The susceptibility to corrosion cracking for selected materials was determined according to the proposed standard of DET [3] at a constant external load. The specimens, stressed horizontally by lever mechanism, were resistively heated by passing current (27-30 A) up to 200˚C in the exposed section under dropping of 0,1 M NaCl or 0,1 M NH_4Cl water solution with frequency about 0,1 Hz. The specimen temperature was controlled by the thermocouple.

The short or small cracks length was observed by the method of travelling light microscope (magnification 50-100x). On the specimen surfaces were measured coordinates of crack initiation points or pits.
When the specimen has broken, time to fracture was registered by electromechanical clock and number of cycles i.e. drops to fracture ($N_f = f.t_f$, f – dropping frequency) was also calculated. If the specimen had not broken within 1500 hours, the test was interrupted and the specimen was taken out. After testing the exposed sections of specimens were cut off and fracture surfaces were examined by the light and scanning electron microscope. Then specimens were prepared for metallographic study of structure and cracks in the cross section.

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Description and discussion of results

The initiation of small pits or short cracks was accompanied by small elliptical spots (max. ϕ 0,5 mm) of a bright colour (Fig.3), different from the rest of an exposed area of sample with dark grey and black colour after longer test time. The localized change of the surface colour is the result of intensive electrochemical corrosion reactions – cyclic anodic dissolution in the pits or crack tips and a cathodic reduction in their vicinity (active-passive galvanic cells). The time to the first micropits initiation was observed within 5-50 minutes and in case microcracks: 0,2-2 hours. The example of short (small) cracks is shown in Fig. 1

Figure 1 Monitoring of microcracks propagation "in situ" on the sample in the NH_4Cl solution (digital fotocamera, mag. ≈ 80 x)

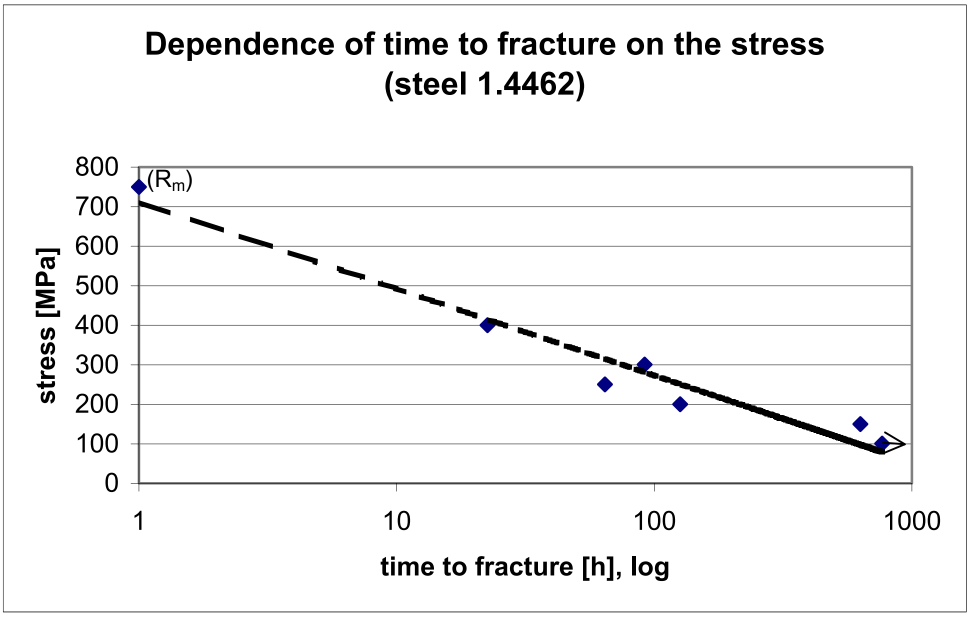

Figure 2. Dependence of time to fracture on applied stress. (0,1M NaCl)

The time to fracture was shorter for 0,1M NH_4Cl solution than 0,1M NaCl.

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Figure 3. Typical corrosion cracks and fracture of tested material (NaCl (stereomicroscopy 16x)

The SCC crack initiation and propagation accompanied by uniform corrosion or thermal fatigue means that it does not exists the marked threshold stress σ_{th} ($\lim \sigma \rightarrow \sigma_{th}$, $t_f \rightarrow \infty$), but the conventional threshold stress can be set up, e.g. for time 500 hours (σ_{th500}) [2]. According to trend in Fig. 2, for the threshold value can be estimated $\sigma_{th500} \approx 200$ MPa .
Metallographic observation was aimed at the mechanism of cracking, geometry and number of corrosion cracks. The numbers of cracks on exposed surfaces were higher at upper stress and also they are higher in NH_4Cl solution than in NaCl (appr. 5 times). The interaction of close cracks can influence their kinetics, for comparison see Fig. 1 and 3.
The example of corrosion crack growth is shown in Fig. 4, 5 including logarithmic trend for crack kinetic
The typical corrosion cracks with transgranular (transphase) brittle mechanismus of cracks propagation, accompanied by limited branching. Are documented on Fig. 6.

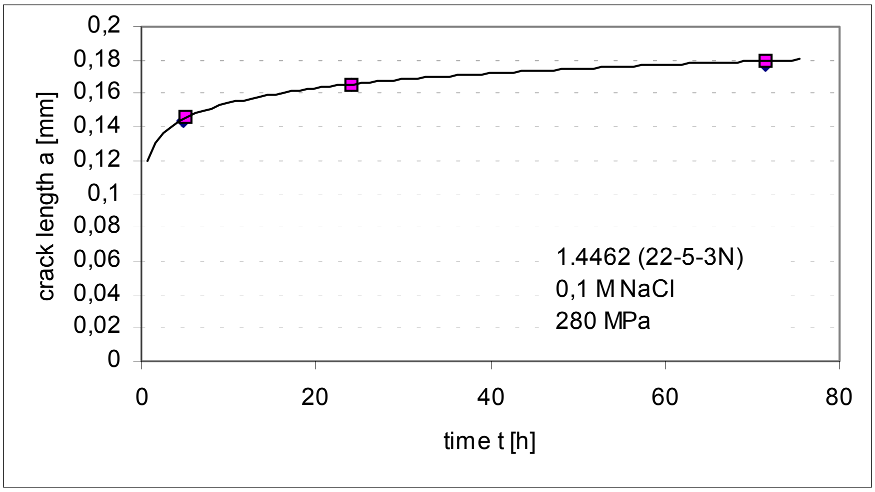

Figure 4. Dependence of crack length of time to fracture in 0,1 M NaCl

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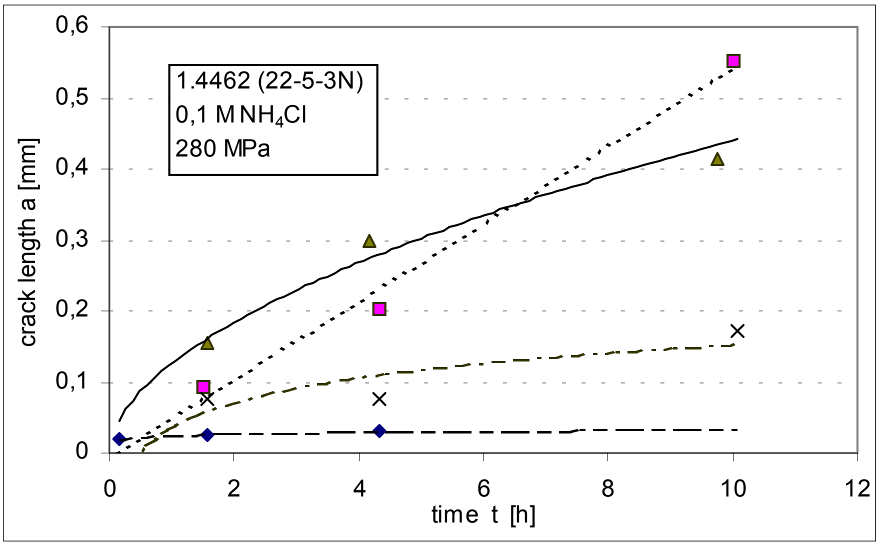
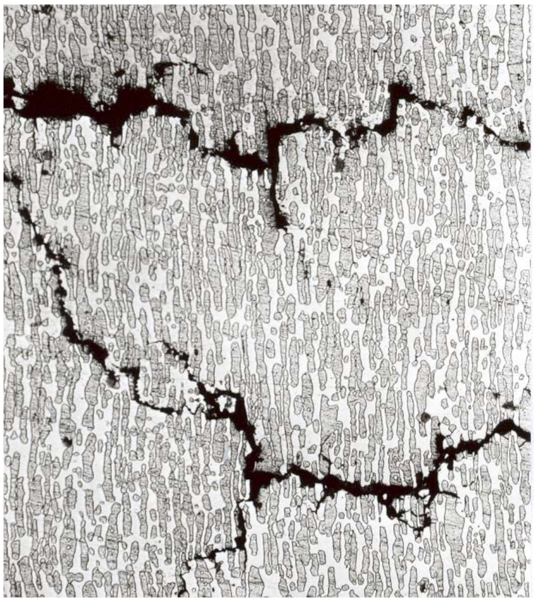


Figure 5. Dependence of crack length of time to fracture in 0,1 M NH_4Cl.

a) Magn. 50 x



b) Magn. 200 x

Figure 6. Stress corrosion cracks and structure of tested material (0,1M NaCl).

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Conclusions

The resistance of duplex steel of type Cr22Ni5Mo3N (1.4462) to the stress corrosion cracking is evaluated in the contribution. The results of our research confirm the suitability of the DET method for the investigation and comparison of sensitivity of metal materials to the corrosion cracking. The drop evaporation test (DET) is convenient method for observation of short corrosion cracks under evaporative conditions at elevated temperature and can be also used for experimental study of pitting or thermal fatigue corrosion.

The possibility of monitoring of microcrack initiations and propagation of short cracks "in situ "on the specimen is big advantage of this method. From the performed tests, we found that the sensitivity to the corrosion cracking is lower in case the duplex steel Cr22Ni5Mo3N (1.4462) in comparison with the austenitic steels [4]. The resistance of the materials to the corrosion cracking was evaluated mainly according to the corrosion cracks rates and dependence of the time to fracture on the stress in the tested specimens.

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