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Influence of Design and Initial Surface Preparation of The Samples on SSC-A Test Result

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Abstract

The influence of several initial surface preparations on NACE SSC-A tests results was analysed in this study. The effect of scale layer, machining and polishing level on corrosion rate and cracking susceptibility has been experimented. These experiments were done on steel wires used in oil industry. Experiments have proved that the machining and polishing level, characterized by surface roughness, is critical on both corrosion and cracking susceptibility of samples. Performance index was defined to characterize and take in account corrosion and cracking phenomena during a SSC-A test in sour service conditions.

Introduction

Industrial operating in oil & gas install or subcontract the installation of devices for collection. Whether in "off-shore", "on-shore" or "sub-sea", the collection devices are subjected to aggressive atmospheres and various mechanical stresses. Therefore, to ensure the design and reliability of the installed devices, industrial owe to previously qualify the materials used.
For obvious reasons of environmental protection and difficulty to repair at depths up to 3000 meters today, these materials must not present failures in service. Qualifications testing of these materials are various, but the most common perform on the materials some static tests 3 or 4 bending, DCB (Double Cantilever Beam) or uni-axial tensile as the method A.

The existing standards (as for example NACE TM0177 for the most commonly used) needed to perform these tests on samples with an initial specific surface preparation. Industrial will then be based on this standard either on their own operating method for surface preparation before testing. One test consists of a uniaxial tensile test and is referred to SSC-A, for Sulphide Stress Cracking Method A. The design of test samples obtained by machining is standardized. Surface preparation by milling, grinding and final polishing are also described. However, the question may arise about the representativeness of a sample machined compared to a non-machined sample or with different grade of surface roughness. NACE standard TM 0177 specifies a roughness Ra <0.81μm. Are the corrosion phenomena and the strength of the material the same during SSC-A test for various surfaces [2]?

To provide some answers, the SSC-A tests should be performed on samples for various surface preparations in strictly similar environmental conditions. In addition, the material must be from the same batch and the same casting to be similar for metallurgical point of view.

Experimental

Materials

Samples used for this study from steel wires that can be used in oil field. The tests are performed on sample from the same batch number and the same heat reference. The samples are metallurgical similar. This low alloyed steel was produced from casting, cold working and stress relieving. The chemical composition of the steel samples and its mechanical properties are summarised in Table 1. The chemical composition was analysed by inductively coupled plasma (ICP) and by a carbon/sulphur analysis. Observations from optical microscopy and scanning electron microscopy (SEM) have shown an acicular structure that corresponds to the martensitic structure.

Table 1: Chemical composition & mechanical properties of the steel performed

|		
Chemical composition (wt %)	C	0.63 +/- 0.03
	Mn	0.76 +/- 0.05
	P	0.014 +/- 0.002
	S	0.006 +/- 0.001
	Si	0.24 +/- 0.02
	Al	0.028 +/- 0.003
	Cu	0.010 +/- 0.002
	Cr	0.053 +/- 0.006
	Ni	0.017 +/- 0.002
	Sn	< 0.002
Mechanical properties	UTS (MPa)	1 224
	Ys (MPa)	1 151
	A_{100} (%)	10.9

Samples preparation

The test samples were variously prepared. Five surface preparations have been tested. With or without surface preparation, every sample was degreased with a specific cleaning solution. Surface preparations of samples are described in the Table 2. The non-machined samples had a brown-black color representative to

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the surface scale layer. The polishing was performed by a rotating device under lubricant with SiC paper; the grades of the SiC paper were in accordance to achieve the Ra required. The machining was performed by milling and grinding according to requirements of NACE TM0177. Surfaces exposed to the test were parallelepipedal shape and not cylindrical shape as required in NACE TM0177. Useful lengths exposed to the solution, the length without curvature radius and the radius of curvature are in accordance with the NACE TM0177 requirements.

Table 2: Sample preparation description

Kind of sample preparation	Number of sample tested	Design (mm)	Ra (average) (µm)	Ra standard deviation
As-received			0.89	0.015
As-received & rough polished		9x3	0.31	0.012
Machined & not polished	6		0.31	0.019
Machined & polished		6x2.5	0.18	0.022
Machined & fine polished			0.05	0.018

The roughnesses were measured by a contact profilometer with 8mm stroke length. The roughnesses were measured in longitudinal and transverse directions on the test samples. The specimens were machined dumbbell shape. The specimens were again degreased and immediately embedded in cells under continuous flow of nitrogen. This is to prevent any oxidation of the surface before the test. The Photograph and micrographics of the five sample preparations have been shown in Fig. 1.
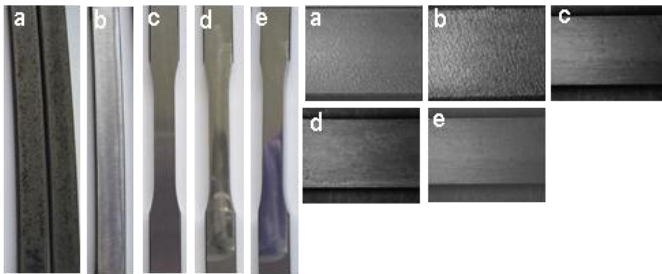
Figure 1: Photography's and micrographics of the five sample preparations : a) for as-received b) for as-received + polished c) for machined and no polished d) for machined and rough polished e) for machined and fine polished

The measured roughnesses are different according to the surface preparation. With the exception of non-machined and polished samples and samples only machined. In order to also consider the influence that can have machining on the NACE SSC-A test result, the polishing of the non-machined test was performed to achieve a Ra similar to the sample machined and not polished. Due to the qualification of Ra, it is the influence of design, of material removal and of surface roughness will be investigated [3].

SSC A tests & environmental conditions

The tests were performed with a specific device developed into the LNE to ensure uniaxial tensile up to 100kN in a corrosive and sour environment (Fig. 2). The stress level is controlled by calibrated load cell. Cells containing the samples under nitrogen are stressed and filled by the de-aerated corrosive solution. Tests parameters are monitored and recorded daily, such as the stress of the sample, solution pH and temperature. Dissolved sulfide rate is controlled during starting test and at the end. The oxygen level is checked after the de-aeration, before the test begins. The filling of

the cells is done by circulation of the solution from a saturated sulfide reactor.

The duration of the tests was 30 days corresponding to 720 hours at 24±3°C. The oxygen dissolved rate was about 10ppb. The solution pH was 2.7 and the monitoring wasn't free but corresponding to a pH maintaining at ±0.1 pH unit. If necessary, pH was adjusted with hydrochloride acid or sodium hydroxide. The solution was prepared with dissolved water, $50gr.L^{-1}$ of sodium chloride and $5gr.L^{-1}$ of sodium hydrogeno-carbonate. The solution was saturated from gas mixture H_2S-CO_2 at 10% H_2S.

Figure 2: Sulfide Stress Cracking device to stress the sample up to 100kN with an uniaxial strength

The samples embedded into cells were then placed in uniaxial stress corresponding to 0.9xYS, then at 1036MPa. The surface to volume ratio was defined as:

$$\frac{\text{Total volume of solution}}{\text{Sample area exposed to the solution}}$$

This ratio depending on samples areas. For 9x3mm and 6x2.5mm samples areas, this ratio was respectively of 46 and $64ml.cm^{-2}$.

Assessing the level of corrosion

The corrosion level of the samples is first measured from the sample integrity. The macrographic analysis can reveal cracks, crack initiations, indications or indentations on the samples. Second, this is the kind of corrosion which is evaluated with the presence of pitting or no, low or severe corrosion, generalized corrosion or not. Then, the mass loss is measured after the test. Finally, the corrosion is quantified by determining for each sample the corrosion rate as defined below:

$$T_{corr} = e_{corr} / t = m_{corr} / (\rho * S * t) \qquad (1)$$

where: T_{corr}: corrosion rate or loss of thickness steel by unit time (µm/d or µm/y); e_{corr}: steel thickness lost by corrosion (cm or µm); m_{corr}: steel weight lost by corrosion (g); ρ: material weight density (g/cm3); S: exposed area of the sample (cm²); t: duration of the sample exposition (d).

A performance index is then introduced taking into account the presence of crack, the number of days remaining before the end of the test if crack is observed and the corrosion rate calculated initially.

$$PI\ (\%) = K/((T_{Corr} + (NSCR\ x\ NMHMFT)) \qquad (2)$$

where: PI (%): performance index in % compared to the reference sample; K: performance index for reference sample; T_{corr}: corrosion rate or loss of thickness steel by unit time (μm/d or μm/y); NSCR: number of sample cracked; NMHMFT: number of maximum hours missing to finish the test.

Results and Discussion

The tests were performed on 6 samples for each kind of initial surface preparation. The corrosion rate and performance index were evaluated for 6 samples and the average was calculated.

Part A: Influence of initial surface preparation on corrosion rate

The corrosion rate was evaluated after the test from cleaned sample (Fig. 3). The samples were cleaned by using a scraping sponge to remove contamination and corrosion products without more damaged the samples. After cleaning, the samples were rinsed with ethanol and dried. The corrosion rates vs initial sample preparations are described below.
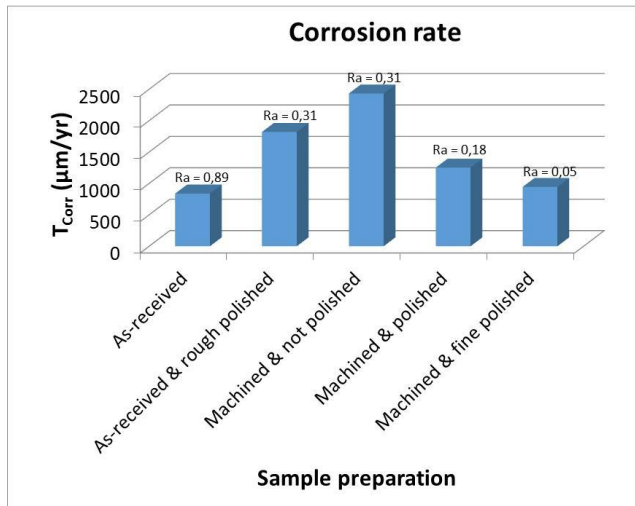

Figure 3: Corrosion rate vs initial sample preparation

Table 3: Corrosion rate results with standard deviation associated

Kind of sample preparation	Ra average (μm)	Ra standard deviation	Corrosion rate (μm/yr)	T_{corr} standard deviation
As-received	0,89	0,015	840	19
As-received & rough polished	0,31	0,012	1821	19
Machined & not polished	0,31	0,019	2433	7
Machined & polished	0,18	0,022	1253	57
Machined & fine polished	0,05	0,018	945	43

Table 3 shows that the standard deviations associated to Ra and corrosion rate and recorded over 6 measurements are low and allow comparing the results without overlap by initial surface preparation categories.

The first remark concerns the corrosion rate twice or more comparing polished and unpolished samples (as-received samples). It is important to note that in most cases the unpolished state corresponds to the operating conditions. This initial surface is representative of the future material operating conditions. We can correlate this difference with the layer of scale which is on the surface of the sample for unpolished condition. This oxide scale

layer, which we analysed, contains carbonaceous components and oxides. This scale layer represents a protective barrier against corrosion that protects the material of aggressive environments. This oxide scale layer is directly related to the manufacturing process of the material and can therefore differ from one process to another in terms of chemical and structural compositions and thickness [4].

The second point relates to the influence of machining. Our machining process leads to roughness about 0.3μm after grinding. So we wrote surface polishing of un-machined samples corresponding to 0.3μm roughness. This allows us to compare samples with a similar roughness but with different metallurgical surfaces. We wanted to highlight any difference related to the metallurgical nature of the material. According to the material either on the surface or more to core, its metallurgical structure is different. This can almost be likened to two different materials. This is also shown by the large difference in corrosion rate. It is therefore highly damaging to machine this material compared to a sample un-machined and polished. In this case, the corrosion rate increased more than 30%.

Last point concerns the influence of polishing. If we take a sample of the same metallurgical state on the surface, corresponding to the machined sample to the core, the more polishing leads to a low Ra, so more polishing is thinner and the corrosion rate decreases. Surface preparation by polishing is capital and strongly influences the corrosion rate for this material during SSC-A test. Note that the thickness removed from rough polishing and fine is negligible, about 20μm.

Part B: Influence of initial surface preparation on performance index

The performance index is absolutely not a scientific term recognized in the field of corrosion. It is established only in our case and in our industry for material qualification requirements. However, this term allows us, taking into account the corrosion rate, to complete our analysis by including the aspects related to the way of corrosion and damages.

Table 4: Performance index results associated with initial sample preparation

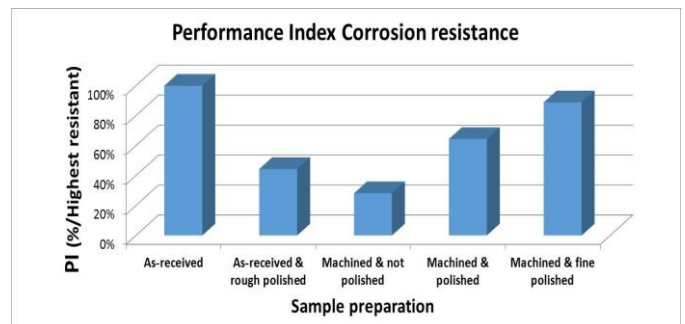| Samples	T_{corr} (μm/yr)	Number of sample cracked or highly damaged	Number of maximum hours missing to finish the test	IP Corrosion resistance
As-received	840	0	0	100%
As-received & rough polished	1821	1	72	44%
Machined & not polished	2433	2	272	28%
Machined & polished	1253	1	51	64%
Machined & fine polished	945	0	0	89%

Figure 4: Performance index vs initial sample preparation

We can see that the term associated with damage following the corrosion rate. Indeed, the greater the corrosion rate and the greater the damage are observed (Fig. 4 and Table 4). This is not every time the case, as a high corrosion rates may correspond to a generalized corrosion while the cracking phenomena is more often associated with localized corrosion. The highest performance index is considered for the reference material, i.e. as-received material. This material, in most cases, is representative of the operating conditions. The lowest performance index corresponds to the material being machined and un-polished. That one presented two cracks on 6 samples tested. If we consider the characterization laboratory point of view, this performance index allows us to say that to describe this kind of material in SSC-A test and if machining is necessary, polishing as thin as possible is recommended. The performance index is about 90% for this material with a fine polishing process. We can hypothesize that the fine polishing can be reduced or even annihilate some phenomena associated with machining such as hardening and surface stresses. These surface effects can occur even if precautions are taken during machining operations such as lubrication, cooling etc.

Conclusions

This study compares the corrosion rate and performance index of samples during SSC-A tests. The tests were performed on 6 samples for each initial surface preparation and the standard deviations of the results are low.

We have noticed both the generalized corrosion aspects associated with corrosion rate and in terms of damage that the most resistant material was the un-machined and no polished material, i.e. as-received material. This material is representative of the operating conditions. It has a surface scale layer that protects it against corrosion in this test environmental condition. This layer is correlated with the process of material manufacturing.

Considering a similar surface roughness, the machining has a great influence on the corrosion phenomena. The corrosion resistance is different from the material surface compared to the material core for SSC-A test in our environmental conditions.

The performance index associated with the machined material is the highest for fine polished samples. This index is close to un-machined material. This highlights the importance of polish mastering before to perform a SSC-A test.

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