EXPERIMENTAL ASSESSMENT OF THE SUSCEPTIBILITY TO STRESS-CORROSION CRACKING OF Ti-6Al-4V ALLOY EXPOSED TO MON-1 PROPELLANT TANK ENVIRONMENT – BACKGROUND AND TEST DESIGN

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ABSTRACT

Due to the Rosetta satellite launch postponement, the possibility of subjecting the titanium propellant tanks to off-loading, decontamination and subsequent refilling was considered by the European Space Agency. In this light, it was unknown whether, during this process, alterations of chemical composition of residuals in the tanks could cause unacceptable stress-corrosion damage of the tank material. So far, the susceptibility of Ti-6Al-4V alloy to stress-corrosion cracking in MON-1 environment has been assessed in nominal conditions of stress and chemical environment which are not sufficiently representative of real cases such as the off-loading, decontamination and refilling of tanks. The test programme that will be described in the paper has the objective of assessing the possibility of stress-corrosion cracking in Ti-6Al-4V propellant tanks in the case of tank off-loading, decontamination and refilling. The assessment involves the experimental determination of crack propagation stress intensity threshold values of parent plate and weld material exposed to the propellant tank stress and chemical environment expected during off-loading and decontamination. Based on these test results, the Agency will generate requirements on the reusability of titanium propellant tanks and recommendations to future projects on how to design tanks to avoid stress-corrosion damage. Since the test activity is currently in progress, in this paper the discussion will be focused on the background and test design phase.

1. PREVIOUS WORK

Stress-corrosion cracking (SCC) is a failure phenomenon which occurs in engineering materials, typically metals but also ceramics and polymers, by slow environmentally induced crack propagation. The crack propagation is the result of the combined synergetic interaction of mechanical stress and corrosion [1]. SCC occurs in the presence of a chemically aggressive corrosive environment and under sustained tensile stresses. Water condensation, exposure to coastal environment and the presence of chemical substances such as propellant or accidentally released substances e.g. nitrogen tetroxide, hydrazine, cleaning solvents and hydraulic fluids can promote stress-corrosion cracking.

SCC of Ti-6Al-4V in NO-free N_2O_4 has been reported for stress levels between 275 and 620 MPa. Testing of pre-cracked specimens showed SCC stress intensity factor threshold (K_{ISCC}) values of 70-80 % of the materials toughness (K_{IC}), [2]. Increasing temperature results in decreased time to failure of smooth specimens and in K_{ISCC}. Excesses of NO above 0.7-0.8 % in liquid N_2O_4 prevent SCC. The addition of water further inhibits SCC by reducing the oxygen level and producing NO species, however it causes the formation of nitric acid with risks of corrosion damage, [3]. In the absence of sustained stress, titanium alloys show almost no corrosion in liquid or gaseous N_2O_4 in tests at temperatures up to 68º C, [4].

The effect of NO concentration levels in N_2O_4 on SCC of Ti-6Al-4V STA was investigated in [5]. Tests were performed on pre-cracked specimens under sustained loading while being exposed to N_2O_4 with various concentrations of NO and at various temperatures for a period of 22-72 hours. Results indicated that at room temperature for a 0.09 % NO concentration the reduction factor K_{ISCC} /K_{IC} was less than 0.53. No additional tests were performed at room temperature. Tests carried out at temperatures up to 65°C indicated that K_{ISCC} decreases sharply with NO concentrations less than 0.18 %, the lowest measured K_{ISCC} /K_{IC} value being 0.41. The effect of NO concentration levels on SCC of Ti-6Al-4V welds was not investigated. These test results are in contrast with those reported in [2] where pre-cracked samples exposed to pure N_2O_4 showed a reduction in K_{ISCC} corresponding only to 20-30 % K_{IC}.

Tests on Ti-6Al-4V full-scale tanks pressurised to an equivalent wall stress of 620 MPa were carried out at different temperatures and N_2O_4 NO mixtures, [4]. Table 1 shows a summary of the results. Tanks filled with N_2O_4 which did not contain NO failed after 200 hours or less, the time-to-failure decreasing with increasing temperature. Subjecting the tank internal surface to shot-peening was beneficial in increasing the time-to-failure to 23 days. The use of a Teflon membrane between the tank walls and the propellant did not prevent tank failure from happening since Teflon is permeable to N_2O_4. No failure was observed in tanks filled with mixtures of N_2O_4 with 1 % NO and NTO with 0.3 % NO and 0.8 % Cl.

2. ESA EXPERIENCE WITH ROSETTA

Rosetta is an ESA scientific satellite for deep space exploration (comet observation), Figure 1. More information about the mission and the propulsion system of the spacecraft can be found in [6]. Due to launch in January 2003, its launch was put on hold after the loss of an Ariane 5 in December 2002. During this stand-by period the spacecraft underwent its maintenance programme and preparation for the second launch campaign with its Ti-6Al-4V alloy tanks filled with MON-1 (N_2O_4 with 0.8-0.9 % NO). For safety reasons it was decided that the N_2O_4 tank should be offloaded, if the new launch date had to be further postponed beyond March 2004. However, on 2 March 2004 Rosetta was successfully launched on an Ariane 5G+ dedicated launch.

Although no further launch delay occurred and the N_2O_4 tank was never offloaded, the first launch postponement highlighted the following problems: if the tanks had to be offloaded, inevitable chemical modifications of the propellant residuals would have exposed the titanium alloy walls to the risk of SCC. This risk was associated with the possible formation of NO-lean N_2O_4 and HNO_3, the latter caused by the contamination with water during N_2 flushing. In addition, as it emerged during literature review, no sufficient and reliable materials data were available to allow the realistic assessment of such a risk.

Following this experience, a SCC test programme was initiated at ESA with the main objectives of:

a) Simulating the Rosetta tank environment by subjecting tank material samples to a representative chemical exposure and stress history (sample conditioning).
b) Assessing any reduction in the mechanical properties (K_{ISCC} /K_{Ith}, where K_{Ith} is the materials toughness under sustained loading conditions, and K_{ISCC}/K_{IC}) of the tank material by the SCC testing of conditioned samples.
c) Producing SCC reliable materials data for both Ti-6Al-4V parent plate and welds.

3. TEST PROGRAMME

Table 2 gives an extract of the text matrix foreseen for the programme. The test plan includes toughness K_{Ith} tests as well as K_{IC}. K_{Ith} is a measure of the material resistance to crack propagation under sustained loading, this condition being representative of the pressurisation stresses present in a propellant tank. K_{IC} is generally higher than K_{Ith} particularly for titanium alloys where stable crack propagation, hydrogen embrittlement and creep crack growth can take place.

In the Rosetta representative SCC test, fatigue pre-cracked samples of parent material and weld are subjected to a conditioning phase during which they undergo an exposure and load history representative of the Rosetta tank material. This includes tank off-loading, N_2 purging and re-filling process. The pre-conditioning phase has the objective of reproducing specific conditions in terms of chemical concentrations, sustained stresses (including weld residual stresses and pressurisation), substance retention in cracks and surface defects that are expected to influence the stress-corrosion and corrosion behaviour of the Rosetta tank material. Conditioned samples are then subjected to SCC test by exposing them to MON-1. During this test, sustained tensile stress levels are applied to the samples for 72 hours. After testing, the samples are subjected to detailed scanning electron microscopy (SEM) for evidence of stress corrosion crack initiation. K_{ISCC} values determination based on growth-no-growth criteria.

SCC tests of samples in N_2O_4 and HNO_3 represent two possible worst cases for the propellant tanks since during off-loading and N_2 purging, the formation of HNO_3 and NO-lean NTO cannot be excluded. During these tests pre-cracked samples are subjected to sustained tensile stress levels for 72 hours. As in the previously described SCC test K_{ISCC} values are determined via scanning electron microscopy.

The following main assumptions were made:

a) Real tank material defects are simulated by fatigue thumbnail surface cracks on flat samples.
b) A SCC test duration of 72 hours is sufficient to initiate and propagate an existing crack. This

assumption is based on previous test experience, [5].

c) In order to obtain simplified test exposure sequences which are representative of the real case with respect to SCC damage, complex decontamination procedures are represented in terms of blocks of constant tensile stress and exposure to a specific chemical environment. Possible history effects are also taken into account by respecting the chronological order of exposure and loading sequence of the real case.

3. TEST DESIGN AND SET-UP

Titanium alloy Ti-6Al-4V 2 mm thick surface crack tension (SCT) samples were manufactured. These samples have same form, temper, grain orientation and thickness as the Rosetta propellant tank material (for welds the same welding process). Thumbnail surface cracks were introduced by fatigue cycling to obtain a 1 mm deep crack.

One of the major challenges in the programme consisted of designing an environmental test facility to expose the SCC samples. It was decided to use environmental cells individually mounted on each sample. This allowed an easier control of the chemical environment for the samples. Figure 2 shows the environmental cell design concept. The cells are mounted on the samples so that an orifice at the bottom of the cells allows the surface cracks to be exposed to the substances introduced in the cells. Cells are filled and empted using inlet and outlet tubes. The cells are made of materials such as stainless steel and glass chemically compatible with N_2O_4.

Up to 12 environmental cells are connected with Teflon tubes to an environmental control system specifically designed and developed for this programme. The control system consists of a series of valves, meters, and lines made of materials chemically compatible with the N_2O_4 and connected to N_2, He and N_2O_4 reservoirs. The system allows carrying out in a controllable and reproducible way N_2 and He flushing, decontamination procedures using water or IPA, and SCC exposure of 12 samples. The cells can be filled or emptied in parallel or individually. When required, sampling lines allows the extraction of defined quantities of the cell content for chemical analysis. This is normally carried out before and after each exposure test.

During exposure samples are subjected to tensile stresses. Stresses are applied using loading frames. Figure 3 shows the schematic of a loading frame. After a sample is mounted in the frame, a tensile load is applied by gradually compressing a series of leaf springs to reach the desired stress level. The applied stress level is measured by processing the electric signal of a calibrated strain gauge. Signals from strain gauges are monitored and recorded using a data acquisition system. Since the loading frames are not in contact with the exposure media they can be safely accessed during testing in order to vary the samples stress levels as needed to simulate any change in tank pressurisation.

Figure 4 shows the test set-up. The photo shows the general arrangement of the load cells and environmental frames. The environmental cells are mounted vertically on the samples, and the loading frames are fixed on a support structure. Pressure gauges mounted on the feed lines are also visible in the photo. In the case of a sample failure, feed lines of the affected environmental cell can be disconnected and the sample dismounted and stored in inert environment. A sample failure is detected by monitoring the strain gauge signals.

4. SIGNIFICANCE AND USE OF TEST RESULTS

The results of this test programme will have the following significance and use:

a) Produce homogeneous and reliable set of materials data on parent plate and welds to be used in propellant tank design and damage tolerance verification. In current tank design practice the effects of stress-corrosion are taken into account using a rather empirical approach which consists of applying reduction factors typically between 0.6 and 0.75 to the K_{IC} values of the parent and weld material. The availability of test results in terms of K_{ISCC} /K_{Ith}, and K_{ISCC} /K_{IC} will reveal whether such factors are realistic or too conservative.

b) Test results will be used for the validation of tank decontamination procedures designed to prevent the occurrence of SCC.

c) Based on operational reasons and by the fact that a tank replacement likely leads to unacceptable damage to the satellite, the next generation of metallic propellant tanks will have to be reusable. The reusability requirements will have to be defined compatibly with the tank resistance to stress-corrosion cracking. The test results will help in defining the reusability requirements of the next generation of metallic tanks.

d) The developed test facility, currently the only one in Europe to be specifically designed for SCC testing in N_2O_4, can be used to perform additional SCC tests in support to technology development programmes and space projects. Since no SCC test standards are currently available, the developed test procedures will be a reference point for future standardisation activities.

5. CONCLUSIONS

a) Ti-6Al-4V is susceptible to SCC in N_2O_4 if exposed to N_2O_4 with NO levels of less than 0.7 %. K_{ISCC} /K_{IE} values between 0.8 and 0.3 were reported for test samples under various test conditions. The addition of 1.5-2.0 % water inhibits SCC, however it causes the formation of HNO_3.
b) No reliable SCC test data for Ti-6Al-4V parent plate and welds in N_2O_4 are currently available from open literature.
c) ESA initiated a test programme aimed at generating reliable SCC materials data to be used in tank design, validation of tank decontamination procedures and in defining the requirements of the next generation of metallic tanks.
d) A test facility was built by ESA to allow SCC testing in N_2O_4, HNO_3 and IPA, and the validation of tank decontamination procedures.
e) The test activity is in progress and test results will be published in open literature.

6. REFERENCES

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3. Schultz, R. W., *Stress-corrosion cracking of titanium alloys*, ASM International, 1993
4. Jackson, J. D. and Boyd, W. K., *Corrosion of Titanium*, Defense metals Information Center, Battelle Memorial Insitute, Sept. 1966
5. Bixler, W. D., *Flaw growth of 6Al-4V Titanium in nitrogen tetroxide with low nitric oxide contents*, NASA-CR-16094
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Material	**Propellant**	**Temperature**	**Time to failure**
Ti-6Al-4V	Red N_2O_4	30º C	8 days
Ti-6Al-4V	Red N_2O_4	40º C	3 days
Ti-6Al-4V Tank with Teflon bladder	Red N_2O_4	40º C	23 days
Ti-6Al-4V Shot peened tank	Red N_2O_4	40º C	No failure after 30 days
Ti-6Al-4V	N_2O_4 with 1% NO	40º C	No failure after 30 days

Table 1. Summary of SCC test results carried out on full scale Ti-6Al-4V tanks, [4].

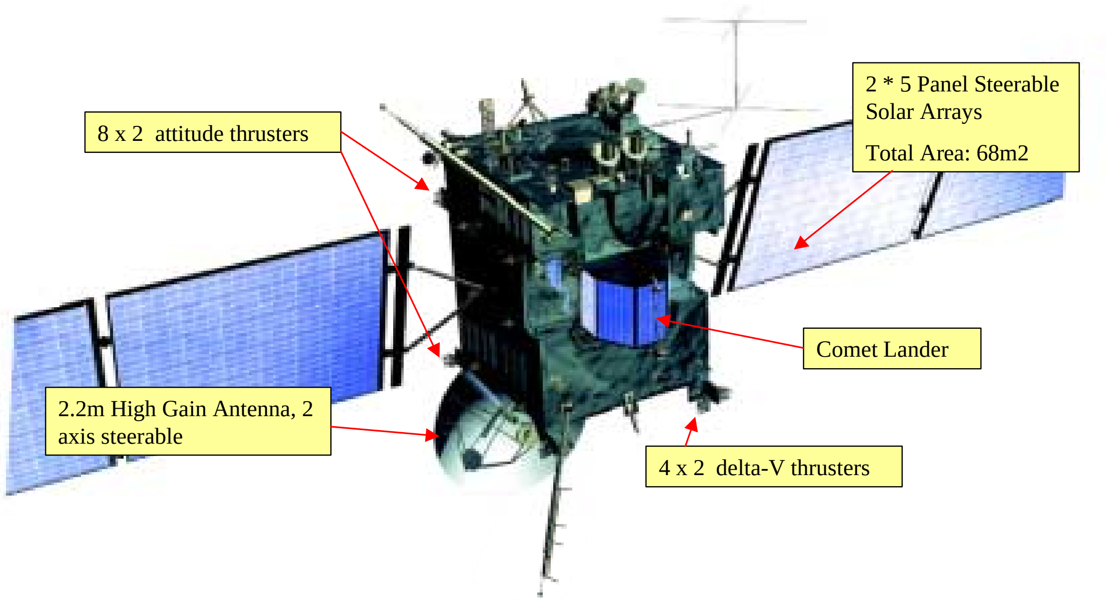

Figure 1. Artist's impression of the Rosetta satellite.

Test description	**Material (min. number of tests)**	**Test results**	**Use of results**
Toughness test	Parent metal (3) and weld (3)	K_{IC} in air	To verify material toughness requirements and provide term of comparison air to medium
Crack propagation under sustained loading	Parent metal (6) and weld (6)	Toughness (K_{Ith}) under sustained loading in air	To determine material toughness under sustained loading (representative of pressurized tanks) and provide term of comparison air to medium
Stress-corrosion test representative of Rosetta tank	Pre-conditioned parent metal (6) and weld (6)	Threshold stress-corrosion intensity factor (K_{ISCC})	To assess possible reductions in toughness caused by the off-loading, decontamination and refilling of MON-1 tanks
Stress-corrosion test in HNO_3	Parent metal (5) and weld (5)	Threshold stress-corrosion intensity factor (K_{ISCC})	Possible worst case during tank off-loading. Data will be of general applicability and will be compared with materials data
Stress-corrosion test in NTO with low NO content	Parent metal (5) and weld (5)	Threshold stress-corrosion intensity factor (K_{ISCC})	Possible worst case during tank off-loading. Data will be of general applicability and will be compared with materials data
Stress-corrosion test of welded smooth samples	Weld (6)	Threshold stress-corrosion stress level	To asses the effect of typical weld surface defects on SCC stress levels

Table 2. Extract of the text matrix foreseen for the SCC programme.

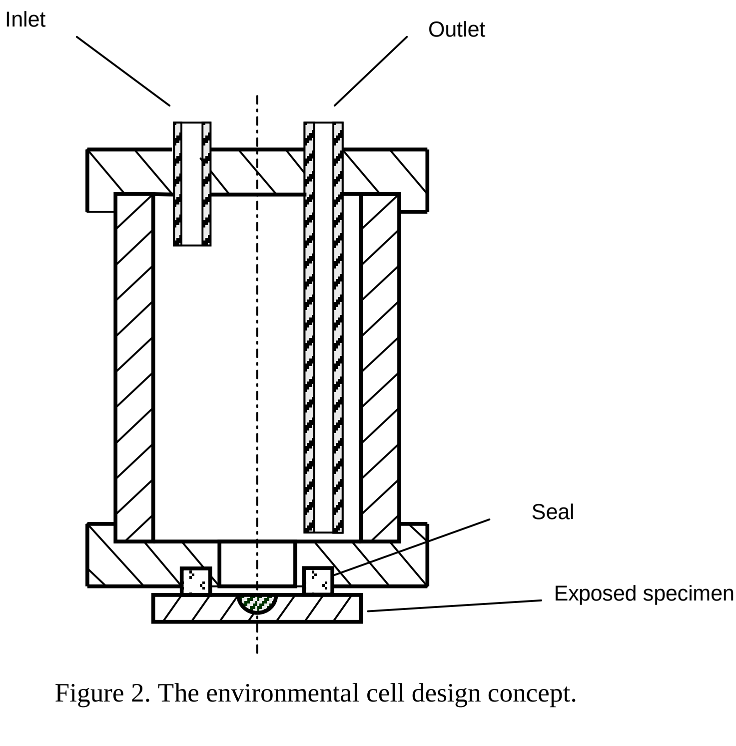

Figure 2. The environmental cell design concept.

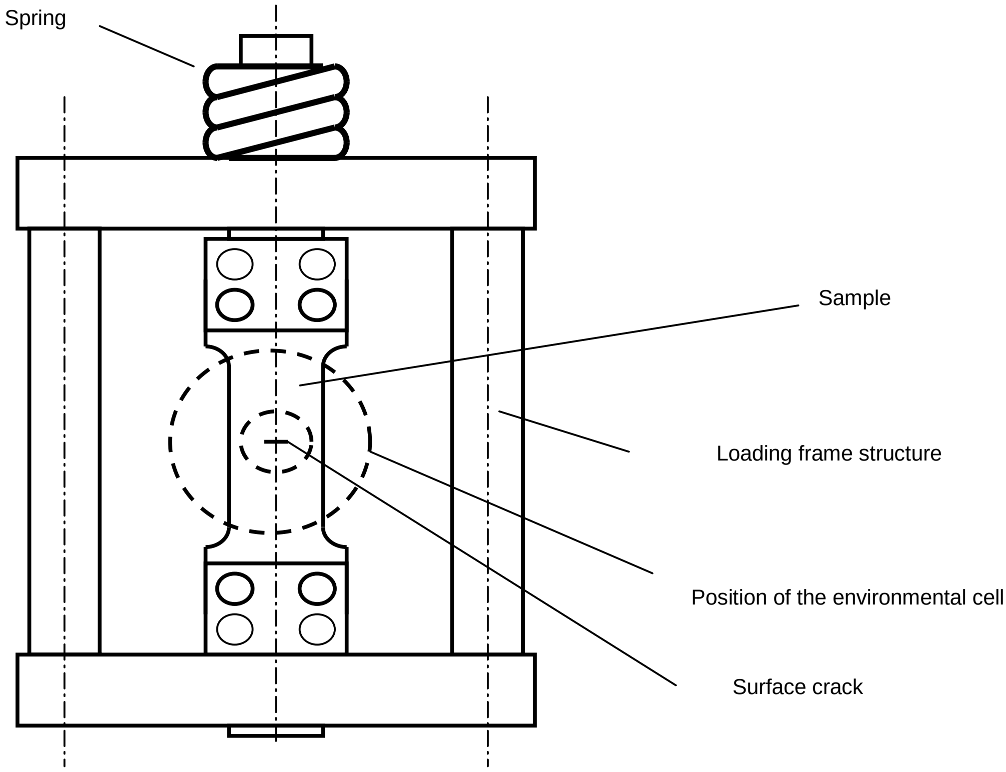

Figure 3. Schematic of the loading frame used to load the samples.

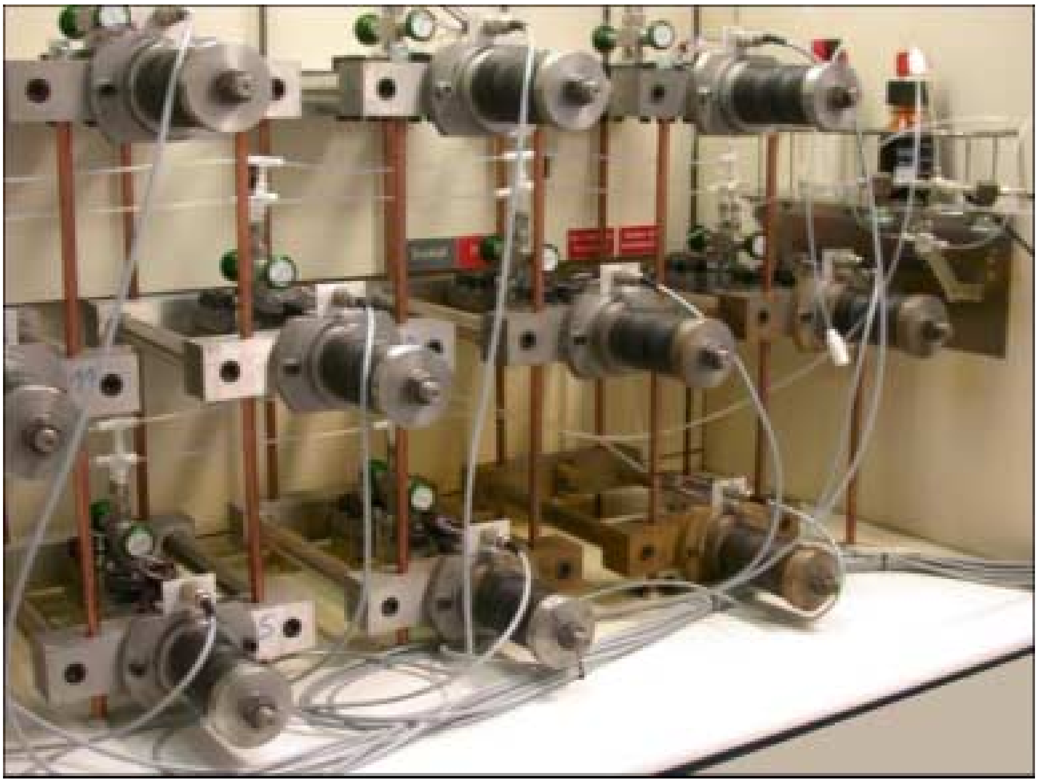

Figure 4. Photo of the SCC test set-up.