ACOUSTIC EMISSION MONITORING OF HYDRIDE CRACKING IN ZIRCONIUM

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ABSTRACT

This paper describes the use of acoustic emission (AE) to detect the onset of delayed hydride cracking (DHC) in hydrogen damaged zirconium laboratory notched specimens. The onset of the DHC is an important factor in the integrity of an in-service component. The testing method described will help to quantify the number of cycles required to initiate the DHC and determine the critical section of the thermo-mechanical duty cycle, at which the DHC mechanism would initiate. The DHC mechanism and the experimental method of AE monitoring during thermo-mechanical cyclic loading are discussed. The development of on-line linear location via wave-guides proved to be vital to eliminate extraneous noise because of the test environment. The advantage of AE over the previous use of the DC potential drop technique is shown and the detection of DHC on the negative temperature slope of the thermo-mechanical cycle is demonstrated.

INTRODUCTION

General Background

In service, zirconium alloy components may be subjected to loads induced from thermal warm-up and cool-down cycles, causing cyclic stresses and strains, which are potentially a significant factor for the integrity of these components due to the possible onset of delayed hydride cracking.

Zirconium alloy components can accumulate high levels of hydrogen pick-up during their service lifetime. In zirconium and its alloys, excess dissolved hydrogen precipitates as zirconium hydrides. These zirconium hydrides are brittle, and tend to precipitate preferentially at high stress points such as discontinuities or notches in stressed components subjected to thermal cycles. If failure of either the hydride or the hydride interface occurs the surrounding ductile material matrix will arrest crack growth. As hydride formation is enhanced by the application of stress, another and possibly larger hydride can then form at the newly generated crack tip. Further crack propagation can then occur, caused by the repeated hydride formation and failure cycle until total fracture of the component occurs. This mechanism of crack initiation and slow propagation is called delayed hydride cracking (DHC).

The Hydride and Thermo-Mechanical Problem

As part of the design justification for DHC of certain zirconium alloy components, there is a need to characterise the hydride behaviour of the zirconium alloy material under simulated service conditions. To create a realistic, although worst case scenario, notched laboratory specimens were subjected to a thermo-mechanical cycle, which represents the major, and most damaging, events in the service cycle.

The definition of a thermo-mechanical cycle is when the stress intensity at a stress concentration changes simultaneously with the temperature. The combination of stress and temperature is the key factor. Hydrogen is concentrated by the stress field below the notch, and on cool down, hydrides precipitate locally in this area. These brittle hydrides can then be ruptured by either an increase in the stress intensity on cooling or by the local stresses applied as the brittle hydride phase precipitates.
Careful manipulation of the hydrogen level in the material and the thermo-mechanical cycle applied can hopefully mimic (in test specimens) the actual service conditions experienced when the material is in its deteriorated condition. A specially developed pulsed DC potential drop (DCPD) system had been used previously to monitor fatigue crack initiation on similar notched bend specimens under isothermal test conditions [1]. The early detection of hydride cracking, rather than fatigue cracking at its very initial stages, is essential if laboratory techniques are to be used in characterising the damaging thermo-mechanical hydride formation fracture process. The use of acoustic emission (AE) was therefore considered necessary as it was thought that the temperature changes in the thermo-mechanical cycles would render the potential drop technique impractical. There is also a possibility that the hydride cracking may initiate sub-surface, and of a localised nature, which would prove difficult to detect with DCPD.

**Literature Review**

Work on material more susceptible to hydride precipitates [2] observed that the temperature at the onset of AE production during hydride formation in Nb, Ta and V was dependant upon the level of hydrogen concentration. It was concluded that the AE detected was due to cracking of precipitate particles rather than phase transformation or plastic deformation phenomena.

Investigation into brittle crack growth in metals [3] concluded that in some materials a one-to-one relationship exists between macroscopic crack movements and AE. A correlation was also derived between the amplitudes of the emissions and the energy release or size of the cracking event.

A study of stress corrosion cracking in high strength steels [4] used an electrical resistance technique (DCPD) and AE for simultaneous measurement of the stress corrosion cracking. No particular problems were highlighted in operating the two systems, although the testing was all performed at room temperature. The AE system was found to be more sensitive to the initiation of the stress corrosion cracking than the DCPD system and this was attributed to non-uniform crack propagation during the early stages of cracking. Initial cracking took the form of localised tunnelling, but eventually the crack front broadens across the test piece thickness and then propagates as a whole. As the DCPD technique effectively averages the crack length under its field of view it will therefore underestimate the length of the crack growth in the early stages. The AE technique, being sensitive to the elastic stress waves generated by the initial cracking, will therefore be better at detecting crack initiation in the brittle material.

Research work has been performed on the influence of hydrides on the ductile fracture of zirconium alloy [5]. Using AE techniques and static tension tests, two types of hydride induced embrittlement were identified. The first is the delayed hydride cracking process caused by stresses below the yield stress of the material and at comparatively low temperatures (<100ºC). The other is the whole scale reduction of the alloy toughness due to the precipitation of a large number of hydride platelets. The fracture toughness (K<sub>IC</sub>) of the zirconium hydride was found to be 1 MPa√m at room temperature and 3 - 4 MPa√m at 300ºC.

Fracture initiation studies on zirconium hydrides [6], again using AE and static tension tests, showed that if, at room temperature, the average hydride platelet length was in excess of approximately 50 - 100 µm that a critical applied stress was the governing factor in the failure initiation of the hydride. Subsequent investigation [7] studied the initiation behaviour at elevated temperatures. Although the static tension tests performed in this study were under isothermal conditions this is the first reference to the use of AE at significant elevated temperature (300ºC) to measure hydride cracking.
The results from the specimens showed that up to 100°C to 150°C cracking at the hydrides was initiated at slightly below the yield stress of the material. When the hydrides fractured, bursts of AE events occurred in large numbers. However, at higher temperatures, the hydrides were able to flow with the ductile matrix without cracking, and hence very few AE events were recorded.

All the reviewed work used AE techniques either at room temperature or at elevated isothermal conditions, recording cumulative AE data to display damage trend analysis. The work reported in this paper involves AE techniques to detect the individual failure initiation of reoriented hydrides, in notched specimens, using thermo-mechanical cycling to create a delayed hydride cracking mechanism.

OBJECTIVES

- To develop laboratory techniques to allow the AE activity from the initiation of delayed hydride crack formation in Zr-2.5Nb alloy to be detected during thermo-mechanical loading.
- To develop laboratory techniques to allow the simultaneous use of AE and DCPD crack detection techniques during thermo-mechanical loading.

TEST ARRANGEMENT

The notch specimen (see Fig. 1) was loaded by a cantilever configuration and mounted in an insulated hot chamber. Wave-guides and the DCPD are attached to the specimen as shown in Fig. 3, showing the AE sensors outside the hot chamber. The heating method was a forced air circulation system, where heating and cooling rates of at least 1°C/min. was achieved to perform the thermo-mechanical cycle.

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Fig. 1: Notch specimen.
Fig. 2: Location calibration test rig.

The AE sensors (model Pancom P15W) are resonant at 150 kHz. To avoid coupling interface problems, the element of the sensor is embedded into the conical head and was screwed on to the 6 mm mild steel wave-guide to provide easy removal and a consistent interface. Likewise, the junctions of the specimen and wave-guide are also threaded joints. The AE system provided on line location analysis of the AE activity; this was very important because of the high background noise from the rollers, PD and thermocouple wiring, plus differential expansion activity at the test rig interfaces. The test arrangement was also designed with different lengths of wave-guides on either side of the test specimen. This arrangement was used to shift the null point of the specimen and wave guide assembly to some point remote to the notched area on the test sample, thereby any spurious electrical noise hitting both AE sensors simultaneously would then not resemble an AE event at the notch. To verify the ability to locate signals coming from the notch during the feasibility study, a calibration specimen was used which had three wave-guides directly attached to the specimen, see Fig. 2.

Two test parameters were fed to the Vallen AE instrument AMSY4; the load output from the servo hydraulic testing machine and the temperature from a ‘K’ type thermocouple, the latter being connected directly onto the specimen. The preamplifier gain was 34 dB and the threshold of AMSY4 set to 50 dB.
A typical example of the thermo-mechanical cycle is shown in Fig. 4, noting the initial temperature cycles at no load to provide 'shakedown' of some of the mechanical elements in the system.
RESULTS

Location calibrations were performed using pencil-lead breaks (2H, 0.7 mm diameter x 2 mm length) on the special external wave-guides (see Fig. 2), at room temperature and various elevated temperatures up to 290°C. The results of the calibration showed that location could be obtained around the notch to an accuracy of ±6 mm. This correlates well within the maximum theoretical accuracy expected when taking into consideration:

1. the depth of the specimen (4 mm)
2. the two different velocities of mild steel and zirconium.
3. wave mode changes introduced at the material/structure interfaces.

Sources of erroneous noise found during the initial thermo-mechanical trials included:

a) the interface between the jig and the hot box.
b) the high temperature ceramic based adhesive to secure the DCPD current pick up leads.
c) wave-guide and specimen junctions
d) reaction points of the specimen.

The noise from a) was greatly reduced by transferring the main rig reaction point outside the hot box, then it was not subjected to the thermal cycling as shown in Fig. 2. Extensively modifying the method by which the specimen wiring was restrained removed the noise condition of b). The remaining noise from c) and d) was overcome by using on line linear location, which allowed analysis of the data without stopping the test. Calibration using pencil-lead breaks were made at the beginning and the end of the test at the notch. A repeatability of ±4 mm was obtained.

Initial trials using thermal cycling at a high constant load showed that the DCPD changed linearly with temperature at an approximate rate of 1 µV/°C. There was a correlation between the AE output characteristic of hydride failure and a change in the DCPD output. Although the DCPD system used was stable and sensitive (typically 5 µV change for 50 µm fatigue crack growth) it became apparent the AE was more sensitive, being able to detect isolated hydride failure.

The following results show a typical thermo-mechanical test. Figure 5 shows the total energy against the load and temperature applied to a specimen over nine full thermo-mechanical cycles. It can be seen from Fig. 5 that there are two main regions of AE activity;

1) combination of low temperature/high load and
2) combination of high temperature/low load.

The result of filtering the data on those two criteria is shown in Figs. 6 and 7, respectively. Figure 6 shows that the cracking at the notch, occurring on the rising slope of the load and whilst the temperature is falling. Whilst from Fig. 7 there is no activity at the notch. All the data in Fig. 7 is erroneous noise from the reaction points and the wave-guide specimen interface, generally associated with fatigue cycling during the steady state section.

Hydride cracking was first found in the 4th cycle as shown by Fig. 8, with the load rising (196 N) and the temperature falling (100°C). This demonstrates the effectiveness of parametric filtering of the data, which enhanced the ability to show clearly an individual hydride cracking at the notch.

Performing a series of these tests has allowed the interaction of the crack tip stress intensity (K) and the number of cycles to the initiation of the DHC mechanism to be investigated. The critical section of the thermo-mechanical duty cycle, at which the DHC mechanism would initiate has also been identified.
CONCLUSIONS

The background level of AE associated with temperature and load cycling was such that a twin sensor, linear location method of AE testing was necessary. The linear location method enabled AE activity from the notch area of the specimen to be distinguished from the rest of the AE activity. This linear location method was not sensitive to temperature cycling.

Two crack detection systems, AE and DCPD, were completely compatible. The instrumentation associated with each technique did not influence the results obtained from the other. This was achieved for thermo-mechanical cycling conditions. There is a high degree of confidence that an AE signature associated with hydride failure for this specimen configuration has been identified. This characteristic signature was found to be extremely repeatable.

The conventional DCPD technique failed to discriminate individual hydride failures and ultimately was unable to find small hydride cracking. The conclusion is that a stage has been reached when the AE system can detected hydride cracking, which cannot be found using conventional DCPD or metallurgical techniques.

The test program showed that manipulation of the crack-tip stress intensity during the thermo-mechanical cycle influenced the time to initiation of the DHC and the subsequent rate of DHC mechanism. Under static load conditions, the initiation of hydride failure was consistent with expected ductile-to-brittle transition temperature. The developed AE technique was able to establish the thermo-mechanical conditions, at which the DHC mechanism would initiate.

It can be concluded that this programme of development has produced an AE testing technique, which is capable of detecting the initial stages of spontaneous hydride cracking characteristic of the DHC. The sensitivity of the system is such that localised hydride failure can be detected at a stage not normally possible using existing techniques, which are compatible with thermo-mechanical cycling.

REFERENCES

Fig. 5: Summation of AE energy from nine thermo-mechanical cycles, showing two regions of activity; 1) combination of low temp/high load & 2) combination of high temp/low load.

Fig. 6: Showing cracking at the notch with rising load and falling temperature.
Fig. 7: Showing erroneous noise at low loads and high temperatures.

Fig. 8: Showing the initial crack at the notch during the fourth thermo-mechanical cycle.