

Measurement of Fracture Strain in Oxide Scales and Coatings

Summary

This measurement note is concerned with the techniques required to measure fracture strain in thermally-grown oxide scales and protective coatings. The procedure addresses loading modes, strain measurement techniques, methods for crack detection, high temperature testing and reporting of results.

Background:

Components that operate at high temperature are invariably subjected to environmental attack in addition to mechanical and thermal stresses. The materials used in these situations are chosen such that they form a protective scale or alternatively a coating is applied to prevent metal loss due to corrosive attack. This approach is satisfactory as long as the scale or coating remains intact. After mechanical failure of the scale or coating its protective nature may be reduced or even negated. Measurement of the fracture strain of scales and coatings is therefore an important step in the determination of component lifetimes at high temperature.

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Introduction

At room temperature oxide scales are fundamentally brittle and fracture at low strain. Failure strains have been measured at typically $<0.5\%$ for scales on alloys with industrial relevance and decrease with increasing scale thickness [1]. This behaviour imparts particular problems to the measurement of failure strain where the requirements for precise strain measurement limit the choice of measurement technique.

Principle of Test

This test procedure covers the measurement of failure strain in thermally-grown oxide scales subject to mechanical stress at ambient or elevated temperature. Specimens of appropriate geometry are submitted to a mechanical stress; the subsequent strain is measured and the onset of scale failure is detected. The test draws upon the expertise of standard tensile tests but requires additional care due to the precision required of the measurements. The applied stress may be tensile or compressive and may be applied directly or in flexure.

The test should be carried out to satisfy the requirements of accepted Standards for mechanical testing of materials under the selected method of loading [2,3]

Equipment Required

In simplest terms the equipment required is a mechanism to apply load to the specimen; extensometry to measure the strain; and apparatus to detect/monitor fracture of the surface layer. Load is normally applied continuously through servo-electric testing machines; the load capacity of the frame should be sufficient to allow straining of the specimen to beyond the yield point of the substrate material, although continuation of the test to complete separation of the specimen is not normally required. For flexural testing a suitable test jig is required -4pt bending is recommended as this applies more uniform bending moment over the gauge length. A suitable jig is shown in [Figure 1](#).

Extensometry should be sufficiently precise to measure strain at a resolution of 0.01%

Crack detection in the scales may be performed visually or by monitoring acoustic emission (AE). Visual inspection requires suitable long focal length video facilities with access to the gauge length. At high temperatures the availability of a cool path to the video camera is also required to avoid shimmer of the image. Commercial acoustic emission equipment suitable for this task is readily available.

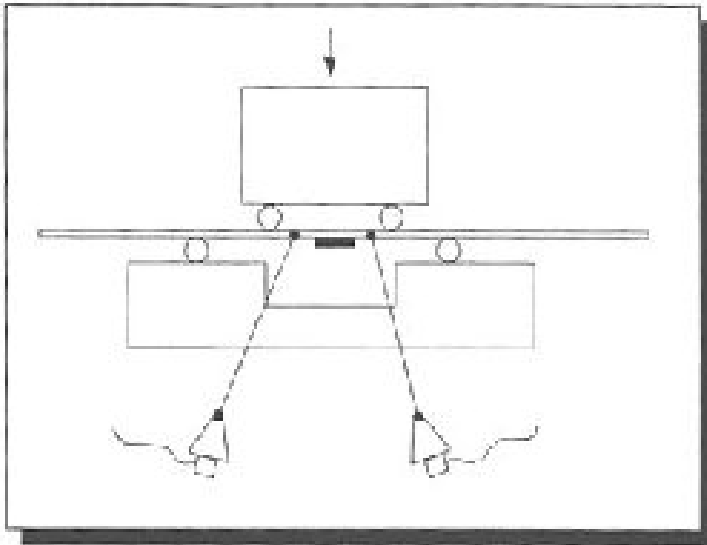


Figure 1 Flexural testing jig with acoustic emission sensors

Specimen Preparation

It is inefficient in terms of occupying equipment to grow scales in situ in the test rigs. Therefore it is necessary to prepare specimens in a separate furnace prior to testing. Specimens with identical exposures should be exposed as a single batch and cooled to room temperature for storage and subsequent testing. Exposure temperatures should be controlled to within the limits specified for normal high temperature corrosion testing [4] viz.

$\pm 3^{\circ}\text{C}$ for temperatures up to 600°C

$\pm 4^{\circ}\text{C}$ for temperatures between 600°C and 800°C

$\pm 5^{\circ}\text{C}$ for temperatures between 800°C and 1000°C

$\pm 8^{\circ}\text{C}$ for temperatures between 1000°C and 1500°C

$\pm 10^{\circ}\text{C}$ for temperatures above 1500°C

Care must be taken to ensure that the oxide scale remains intact during the cooling cycle. Failure may occur due to stress imposed by the thermal mismatch between scale and substrate. This stress may be partially relieved by controlling the cooling rate after the scale growth exposure to allow stress relaxation by creep processes in the substrate. If it is not possible to retain an intact oxide in this manner then this becomes an absolute limit to the thickness of oxide scale that can be characterised.

The strain that is measured during this technique represents a summation of the inherent fracture strain of the surface layer and the residual strain present at the test temperature. For most purposes it is the inherent fracture strain that is required, therefore it is recommended that the residual strain in the oxide is measured at the test temperature by an appropriate technique eg X-ray diffraction. This may be carried out on each test specimen but it is normally sufficient to only measure one specimen under each oxidation condition (temperature and time).

Specimens for testing under flexural loading require that the oxide scale is removed from one face in order to avoid detection of failure events from both tension and compression. In addition it is also recommended that scale is removed from the region where contact is made in the test jig. This precaution reduces the amount of extraneous signal arising from local fracture of scales under high point loading. For testing at elevated temperatures it is also recommended that the specimen is coated with a corrosion-resistant coating (suitable proprietary coatings eg Berkatekt 12 are readily available).

Specimen Geometry

Specimens for tests under flexural loading are simple beams with dimensions suitable for the test jig. Typical specimens are 50 x 5 x 2 mm, but are dependent upon the strength of the material at the test temperature. Care must be taken to ensure that the specimens have sufficient thickness that uniform straining is achieved -the onset of localised deformation with specimen thickness is material and temperature dependent; the specimen design for each material of interest should therefore be reviewed prior to starting the measurement programme.

Specimens for testing under direct tensile loading may be planar or circular cross- section. The choice is governed mainly by the form of material available as both specimen types have advantages and disadvantages, with neither geometry showing sufficient superiority over the other to present a definitive case for its use. In both cases care should be taken to minimise the introduction of stress concentrations which would induce early failure of the oxide scale.

For testing under direct compression, specimens with circular gauge cross section should be used. Care must be taken in the choice of gauge length and cross-section to avoid buckling and barrelling of the specimen during compressive loading.

Load Application

The application of load may be performed under load, displacement or strain control. The rate of loading is not critical but should conform to the rates specified for tensile testing of metals at ambient [2] or elevated temperatures [3]. Load should be applied smoothly in order to avoid impact loading of the oxide or coating which may cause premature cracking. The loading mechanism should also ensure that bending stresses due, for example, to twisting of a flexural beam or by misalignment of uniaxial specimens, are not introduced.

Strain Measurement

For testing under 4pt -flexure, strain, ε , is calculated from the dimensions of the test jig and the relative displacement of the upper and lower rollers according to the expression:-

$$\varepsilon = \frac{3\delta d}{u(3s - 4u)}$$

where δ is the relative displacement between the upper and lower rollers

d is the testpiece thickness

u is distance between outer and inner rollers

s is the spacing of the outer rollers

This expression is valid for strains $\leq 0.3\%$ [5]. The spacing of the rollers is fixed and therefore does not vary from test to test; the thickness of the specimen is measured by micrometer to an accuracy of 0.01 mm; the relative displacement of the upper and lower rollers is measured by transducer and an accuracy of 0.01 mm can readily be achieved. This leads to a repeatability for the measurement of $\sim 1.5\%$ of the value and a reproducibility of 8% if the difference in roller spacing between test jigs is 0.5 mm.

To achieve the same repeatability in direct loading extensometry must be used that will provide an uncertainty of $< 1.5\%$ the measured strain at the strain level of scale fracture. This can be achieved by extensometry of Grade 0.2 in the Standard BS EN 10002 pt 4 [6] when the gaugelength of the specimen exceeds 2.7 mm. A grade 0.5 extensometer requires a minimum gaugelength of 6.8 mm.

Detection of Scale Fracture

Continuous monitoring of the acoustic emission from the specimen under test may provide enough information to determine the onset on scale cracking in many cases but care must always be taken to ensure that the signals detected originate due to cracking events and not from other sources. The use of a two- sensor system with position location software is strongly recommended to identify the AE signals coming from within the gauge length

of the specimen. It is also recommended that a sample of the substrate material is tested without the presence of an oxide scale in order to determine if any AE originates within the bulk material under strain -if this proves to be the case then AE cannot be used as a reliable detection method for scale cracking.

Acoustic emission signals usually show the behaviour illustrated schematically in [Figure 2](#) and require careful interpretation to derive the failure strain of the oxide scale. This failure strain is defined by the back extrapolation of the region where the number of events is rising rapidly with strain. Events occurring before this region may be attributed to cracking events that do not propagate through the entire thickness of the scale or to general 'noise' in the system. Events occurring in the upper plateau of the trace are attributed to repeated cracking of the oxide scale leading to a decrease in crack spacing.

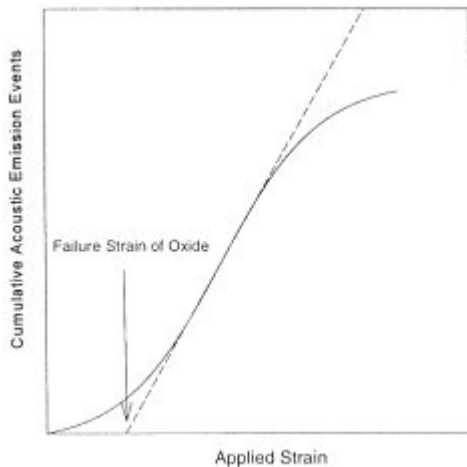


Figure 2 Analysis procedure for acoustic emission signals

Visual inspection of the specimens during the test is an attractive alternative to AE. Video equipment with long focal length is available to enable direct observation during the test. Problems may be encountered during flexural loading but these can be overcome by the use of fibre optics and suitable jig design.

An alternative technique for room temperature testing is periodically to produce plastic replicas of the specimen surface for subsequent examination using Scanning Electron Microscopy [\[7\]](#). This method will only give intermittent strain monitoring.

Temperature Control

Temperature control during these tests should be consistent with that required for normal short-term mechanical testing [\[3\]](#) viz

- ±3°C for temperatures up to 600°C
- ±4°C for temperatures between 600°C and 800°C
- ±5°C for temperatures between 800°C and 1000°C

At temperatures above 1000°C temperature limits are not specified but it is stated that they should be agreed with the customer. It is suggested that the same limits should be used as for the exposure of the specimen to grow the oxide scale.

Temperature measurements should have a resolution of 1°C and an accuracy of ±2°C.

Reporting of Results

The report of the measurement should contain as a minimum the following information:

- Identification and source of the test specimens
- Oxidation conditions/ coating parameters for the test specimens

- Identification of all equipment used in the test
- Test parameters: Temperature, loading rate etc.
- Test results
- Any deviation from established procedures or any unusual behaviour of the test specimens

Case Study - Fracture Strain of Oxides grown on Mild Steel

Specimens of mild steel, in the form of beams with dimensions 50 x 5 x 3 mm, were oxidised at 550°C to produce oxide scales up to 60 J.1m in thickness. The scales consisted of two layers - an outer haematite layer which normally spalled on cooling to room temperature and an inner magnetite layer which remained intact ([Figure 3](#)). Oxide thicknesses were measured on polished cross sections.

The oxidised specimens were prepared for mechanical testing by partial removal of the oxide scale, leaving a small island within the region between the inner rollers. The specimens were tested in flexure using the jig shown in [Figure 1](#). The start of cracking was detected by acoustic emission using a Mistras~ system. Typical output from the Mistras system is shown in [Figure 4](#) where it can be seen that the number of AE events increases with strain (Parameter #1) in an approximately sigmoidal manner as expected.

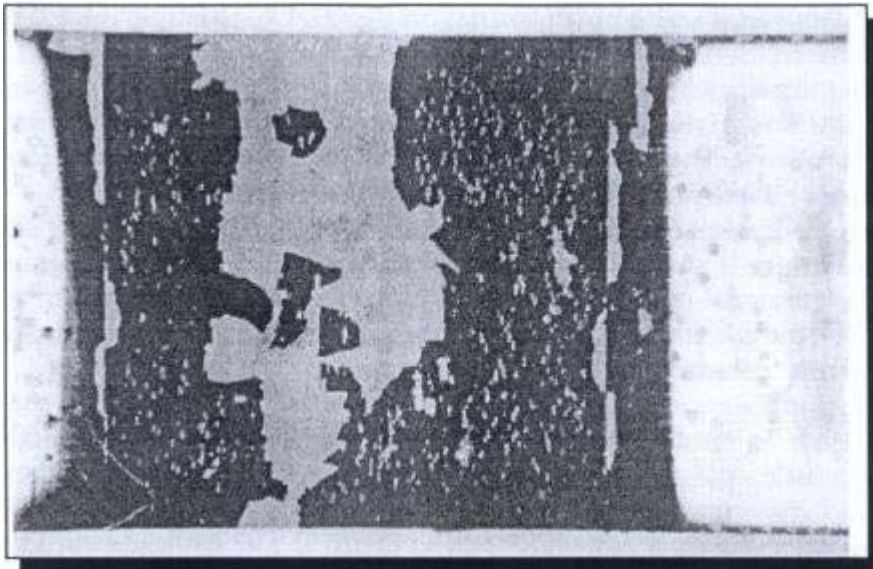
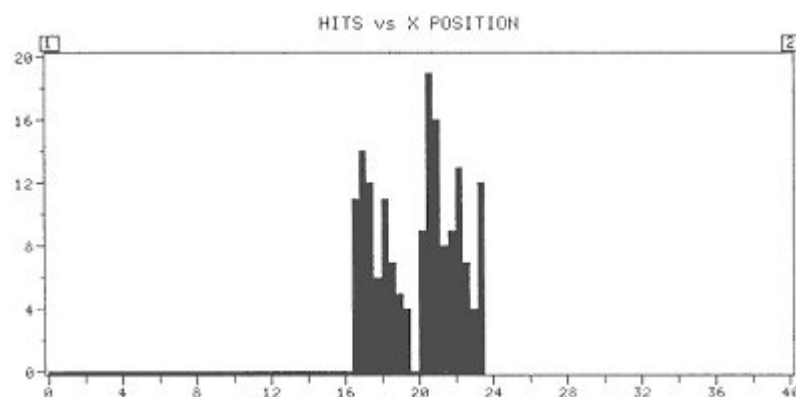


Figure 3 Spallation of haematite (with intact magnetite) on mild steel after cooling from 550°C after oxidation for 96 h



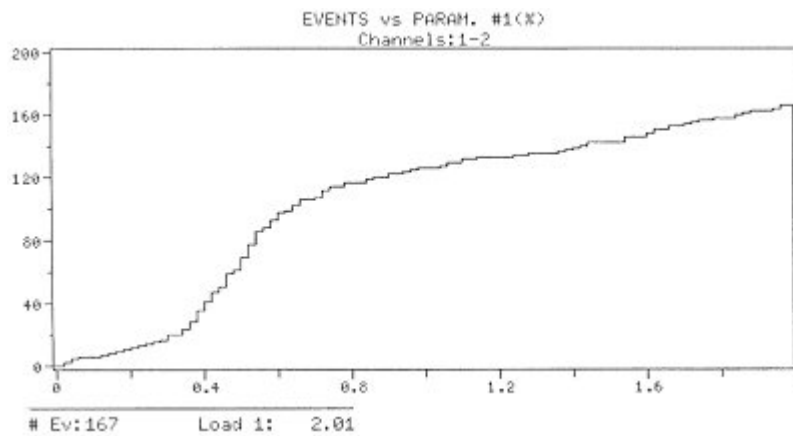


Figure 4 Output signal from Mistras Acoustic Emission Equipment (a) AE hits vs Position (b) Cumulative Events vs Strain

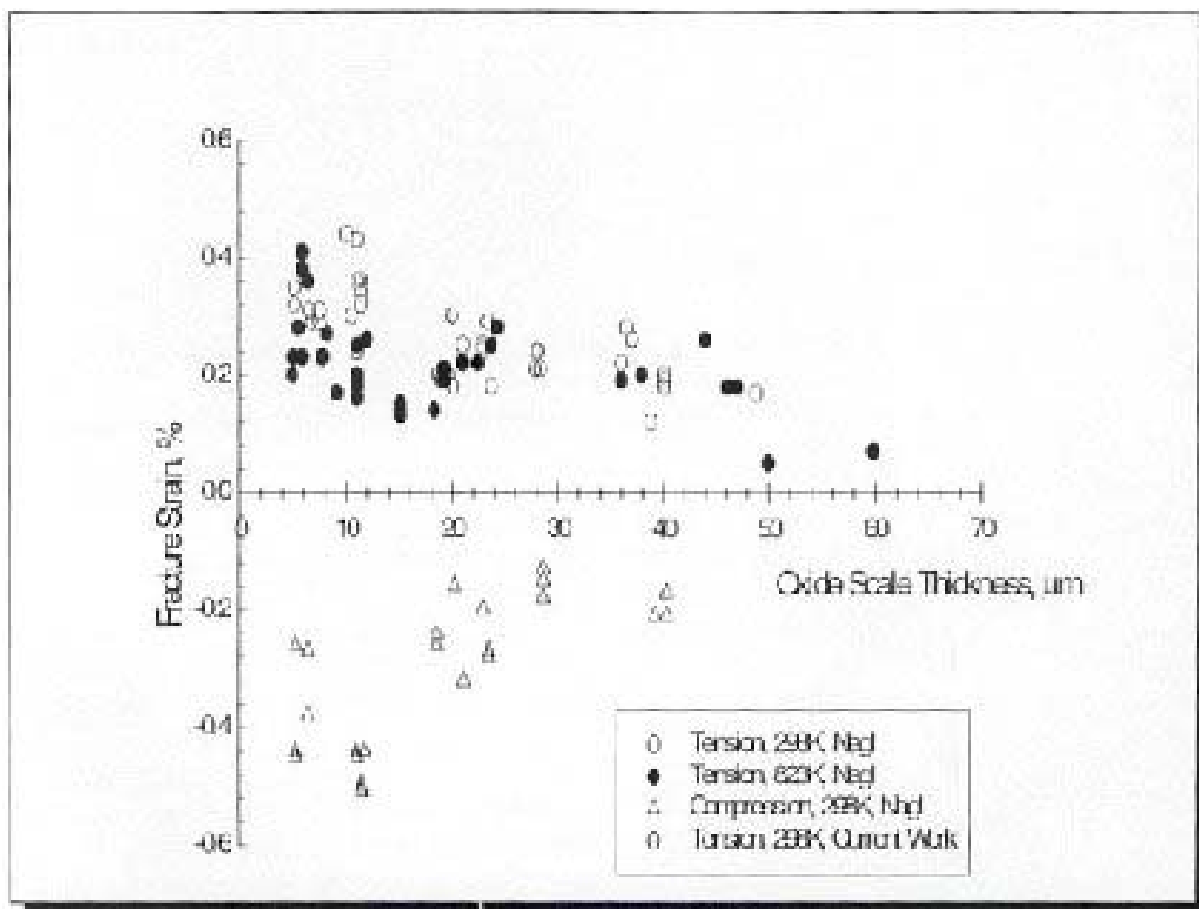


Figure 5 Oxide Failure Strain Data for Mild Steel, Oxidised in Air at 550°C

The overall results are summarised and compared to previous work [5] in Figure 5. Although there is considerable scatter in the results some broad conclusions can be drawn.

- Results from the two batches of material tested in tension at 298K are consistent
- There is no apparent influence of test temperature on the failure strain in tension. However it must be emphasised that the residual stress in the oxide scale at the two test temperatures will be different - the scale at room temperature will have a significant compressive residual strain whilst the scales at 823 K will be effectively free from residual strains.
- Failure strains in compression are apparently similar to those in tension. However the influence of residual strain must be considered here also.

Acknowledgements

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