MONITORING FRACTURE TOUGHNESS USING MINIATURE SAMPLES

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ABSTRACT

This paper describes the development of a miniaturised disk test technique capable of measuring load-displacement and fracture properties. Reproducible data have been produced over a wide range of conditions for a normalised and tempered low alloy steel. As the test temperature was reduced, a transition in failure mode from ductile to brittle rupture occurred. Fractographic studies have been performed to evaluate the behaviour through this transition.

INTRODUCTION

To prevent catastrophic failure of high energy components, design approaches involve structural integrity assessment. These assessments consider loading conditions, material properties and the presence of defects. Typically the appropriate component stresses are determined from established analytical techniques, with the size and location of defects estimated using non-destructive testing procedures. Measurement of appropriate properties also can be performed following fabrication since batches of representative material should be available. In general, the necessary material characterisation involves tensile testing to establish modulus, yield stress, ultimate strength and ductility, also assessment of fracture toughness by compact tension and/or Charpy impact testing.

Since many industrial installations operate for periods up to and even exceeding 30 years, it is frequently necessary to repeat an assessment of structural integrity at appropriate intervals. Any changes in loading conditions can usually be adequately evaluated using the same analysis involved during design. Furthermore, in the majority of situations, repeated non-destructive testing can be carried out to ensure that defects have not developed. However, a major difficulty in undertaking this type of assessment arises because of uncertainty regarding appropriate material properties. This will be a particular problem in facilities that operate at elevated temperatures, since, in many cases the materials of construction are susceptible to time dependent changes in properties, eg reductions in fracture properties may occur due to temper embrittlement[1]. Thus, for these components, realistic assessments require that additional tests are performed to measure actual properties. Problems may then be encountered since standard procedures for tensile or fracture testing require relatively large specimens. In some cases the component geometry may be insufficient for the manufacture of samples with the appropriate geometry. Furthermore, even when component dimensions would allow the fabrication of relatively large test pieces, the act of material removal and subsequent repair may be deleterious to continued operation, either by modifying the properties and/or due to the development of welding residual stresses. Thus, in many cases structural integrity after a period of service can only be estimated based on assumed properties.

The process of assuming properties is clearly unsatisfactory. If the assumptions are too conservative components will be replaced unnecessarily, alternatively if too little change is considered, catastrophic failure may occur. The ability to measure actual properties from plant without affecting future service performance is therefore vital to accurate condition assessment. Recently significant advances have been made in techniques for the removal of material samples. Thus, equipment has been developed which will allow the removal of button shaped samples in an effectively non-destructive manner[2].

Programmes to utilise these samples to measure bulk properties using miniature test pieces are in progress. Much of this work is based on disc-bend tests, initially proposed to monitor irradiation embrittlement in nuclear reactor materials[3]. The present study was initiated to evaluate the deformation and fracture behaviour of 2¼Cr1Mo steel. This paper describes the test technique developed, presents details of the results obtained and outlines the correlation of data from the miniature tests, with those from standard approaches.

MATERIAL AND EXPERIMENTAL TECHNIQUES.

The present programme considered a section of normalised and tempered 2¼Cr1Mo steel thick walled pipe. This material was selected since it is widely used for piping and pressure vessels operating at high temperature and stress. Furthermore, this alloy is known to be susceptible to temper embrittlement[1]. Details of the composition are given in Table 1.

Table 1. Composition of the 2¼Cr1Mo steel.

ELEMENT	C	Cr	Mo	Mn	Si	S	P
COMPOSITION %	0.10	2.10	0.89	0.52	0.27	0.016	0.017

Metallographic preparation and evaluation revealed that the structure of the pipe consisted of predominantly ferrite, average grain size 17μm, with about 5% of transformation product. The average Vickers hardness was determined to be 135 DPN. Detailed examination of the microstructure was carried out using optical and scanning electron microscopy supported by a Graftek Optilab image analysis system. This evaluation was carried out to document the size and distribution of inclusions present, and to identify the inclusion types. Examination of 90 fields revealed that the average volume fraction of inclusions was 0.6%, with the average number of inclusions in a field size of 0.0124mm^2 being about 18. However, a few fields contained relatively high densities of inclusions, ie greater than about 40. Moreover, whilst most inclusions were less than 2μm in diameter, several fields were examined where the average size was about 5μm. Compositional analysis, performed using an energy dispersive x-ray analysis system, revealed that the inclusions were either manganese sulphide or silicate type.

To date 70 disk tests have been performed over a range of temperatures, from -196°C to room temperature. The temperature was controlled to an accuracy of ±1°C using an environmental chamber around the test apparatus. Disk shaped specimens were machined with a diameter of 9mm. The samples were tested with thicknesses in the range 0.25mm to 0.75mm. In all cases the necessary thickness and surface finish were ensured by hand lapping with a purpose built jig. The metallographic grinding papers used, allowed a final thickness tolerance of ±1μm. Testing was carried out using equipment similar to that reported in earlier studies[3,4,5,]. In general, the test piece was located in a recessed specimen holder and was clamped at the edges by applying appropriate pressure to the upper die. The hemispherical punch was accurately positioned in the centre of the disk, and the equipment installed in the load train of a computer controlled servo-hydraulic testing machine. This machine then applied a specified displacement rate to the punch, with the data acquisition system recording load:displacement results at a predetermined rate. The testing arrangement was designed and constructed to ensure that the alignment of the punch at the centre of the disk was facilitated with the clearance between the punch and lower die adjusted for a given disk thickness.

Charpy impact tests were performed on standard notched bars to evaluate fracture behaviour over a range of temperature. The bars were taken from the pipe such that their orientation was the same as that of the disks. Duplicate tests were performed at six test temperatures from -32°C to +140°C. Heating and cooling media such as a sand bath, hot water and ice were used to achieve the correct temperatures. The results produced, were in the form of fracture energies, measured in joules. Post test examination was performed to estimate the percentage of observable ductility on each fracture surfaces. Plots of fracture energy and % ductility versus temperature were produced to calculate the 50% fracture appearance transition temperature (50%FATT).

Fractured disk test specimens from the complete test temperature range, were prepared, cleaned in an ultrasonic bath, and examined using a Jeol scanning electron microscope. Detailed examination was also performed on sets of samples tested under nominally identical conditions.

RESULTS

The results from the disk test program are presented and analyzed in the following section.

Load Vs. displacement behaviour

Tests were performed at selected test temperatures over the range -196°C to +25°C. The load:displacement data observed showed that the pattern of results changed as the test temperature was reduced. Thus at relatively high temperatures, above about -110°C, yielding was followed by a significant period of deformation until eventually a maximum load was noted. The load then gradually decreased until failure occurred. Typical curves for four room temperature tests are shown in Figure 1a. Clearly the same general pattern of behaviour was observed in each test. Furthermore, the reproducibility of specific results was good, ie the yield load was about 0.194 kN in each case, with the ultimate load and displacement to failure being 0.95 to 1.18 kN and 1.35 to 1.70 mm respectively. In tests at low temperature the yield load increased and the level of deformation decreased compared to the room temperature results. Indeed as shown in Figure 1b at -196°C very little plastic deformation was measured with failures occurring close to the yield region. Thus for these tests the total deformation was about 0.6mm with values of yield load and ultimate load being 0.6 kN and 0.8 kN respectively. Between these limits a transitional pattern of load:displacement results was observed.

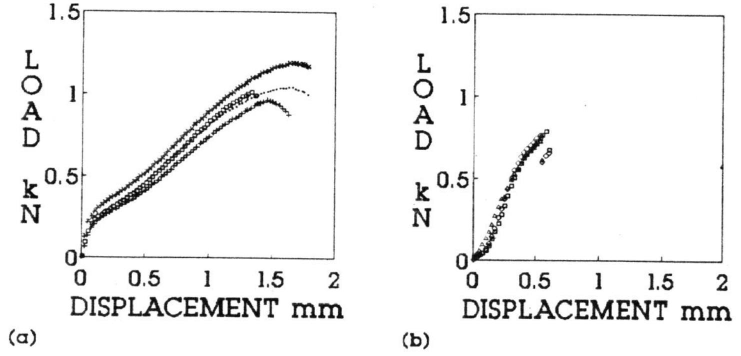

Figure 1. Variation of load-displacement behaviour for four tests performed at +25°C(a) and three tests performed at -196°C (b).

Fracture surface studies

The fractured Charpy V-notch specimens were examined macroscopically to estimate the percentage of ductile fracture. In general, the broken specimens exhibited a ductile region directly behind the notch, ductile fracture then changed to brittle fracture at a point on the fracture surface that was dependent on the test temperature. It was found that at low test temperatures ie below 0 °C the percentage of ductility observed behind the notch was below 15%. A gradual increase in ductility and conversely decrease in brittleness occurred as the temperature increased, until above +140°C 100% ductility was observed.

Details of the fracture behaviour of the disk samples were established using the scanning electron microscope. Particular emphasis was given to identifying the location of crack initiation as well as studying the degree of ductile and brittle fracture on each sample. It was clear that cracking invariably started on the bottom surface of the disk. However, the position of crack initiation changed with temperature. Below -180°C, cracking started in the middle of the disk under the centre of the punch and propagated radially, as well as through the disk, until macroscopic failure occurred. At temperatures above -120°C the cracking initiated circumferentially approximately in line with the edge of the spherical punch. This circular region continued to deform with the final failure being completely ductile. Between these temperatures a mixture of these two situations was observed.

The amount of ductility on each fracture surface was recorded. Below -180°C no ductility was observed, with cleavage facets evident over the whole of the fracture surfaces. Above -120°C purely dimpled ductile fracture occurred. Between these temperatures mixed mode failures took place. Thus as the temperature increased between -180°C and -120°C the amount of ductility increased. This is illustrated in Figures 2a and 2b which show the fracture surfaces of samples tested at -130°C and -150°C respectively. Clearly the -130°C test exhibits more ductility than is present in the -150°C test.

To assess the reproducibility of the fractographic observations, the behaviour of disks tested under similar conditions were studied. It was found that in the fully brittle and fully ductile conditions the appearance of the fracture surfaces was very reproducible. However, this was not the case for samples with mixed failure modes. Thus, for tests undertaken at one temperature within the transition range variations in the degree of ductility were observed. Detailed fractography suggested that these variations were a consequence of local differences in inclusion size and distribution. Thus, in samples which contained relatively large amounts of ductile fracture many of the dimples contained non metallic inclusions, Figure 3. Chemical analysis of the inclusions showed them to be of two types; Manganese sulphide and Silicates. The observed variation in fracture behaviour therefore appears to be a consequence of changes in the distribution of inclusions as noted from the initial metallographic characterisation.

Fracture energy

Fracture energies produced from Charpy tests over a temperature range of -32°C to +140°C, exhibited a change from low to high energy as the temperature increased. Below 0°C energies of less than 45J were recorded. As the temperature increased, from 0°C to 85°C the fracture energies increased from 45 to 150J. At higher temperatures the fracture energy continued to increase but at a slower rate until eventually at +140°C a maximum fracture energy was reached of 160J. Over the whole test range the fracture energy varied from 2.5 to 160 J, Figure 4.

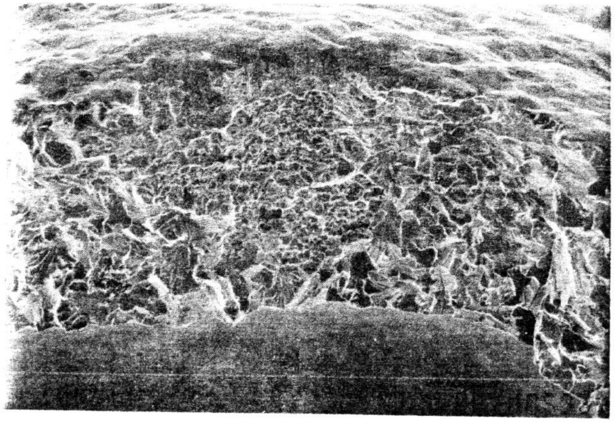

Figure 2a. Typical micrograph of the fracture surface for tests at -130 °C.

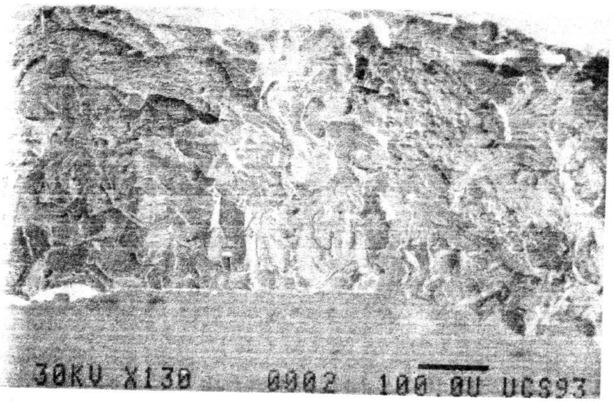

Figure 2b. Typical micrograph of the fracture surface for tests at -150 °C.

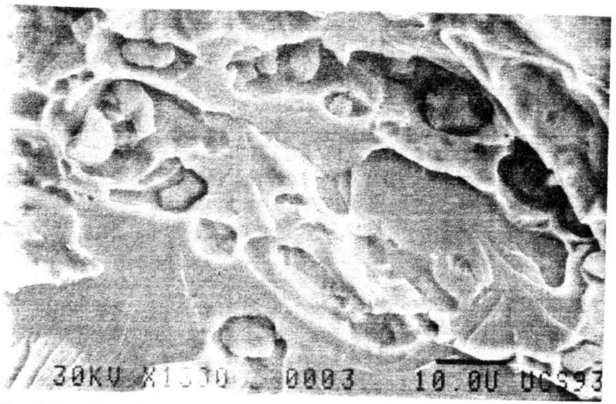

Figure 3. Detail of the dimpled fracture and associated inclusions

For each disk test, an estimate of the fracture energy was made from the load displacement curves. The fracture energy was taken to be the area under an individual load displacement curve up to the point of maximum load. Average fracture energy values were then calculated for groups of individual tests and plotted against the relevant temperature, Figure 4. It was found that at very low temperatures, ie the brittle failure region below -180°C, low fracture energies were produced, 0.3Nm or less. As the temperature increased between -180°C and -120°C, ie the transition region, a sharp increase in the fracture energies was observed. Thus an increase from about 0.3 to 1.4Nm was noted over this temperature range. Above -120°C, where the disks behaved in a fully ductile manner, the average fracture energies were fairly constant. Over the whole test temperature range the fracture energy changed by approximately 1.2 Nm.

For each group of four or five tests performed under similar test conditions, a certain amount of data scatter was observed, Figure 4. The scatter in the fracture energies was most marked in the high energy region of the graph, ie above -120°C, where energy differentials of up to 0.6 Nm were evident. In the transition region, between -120°C and -180°C data scatter was significantly reduced to 0.3 to 0.4 Nm. Below -180°C data were reproducible such that in this fully brittle fracture region, differences in energy were only 0.1 to 0.2 Nm.

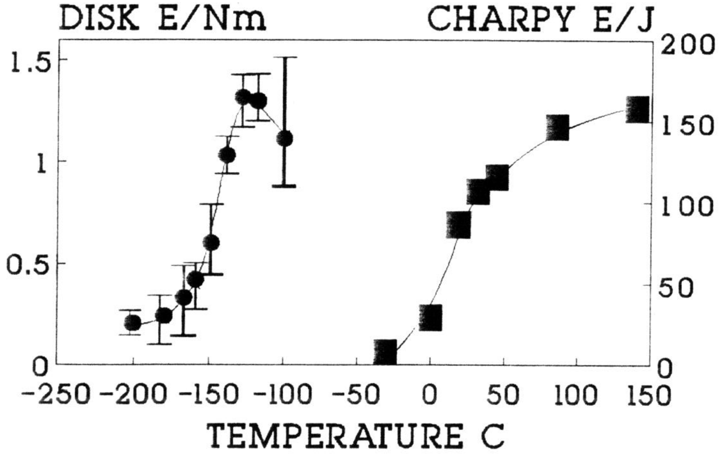

Figure 4. Variation of disk and Charpy fracture energy against test temperature.

DISCUSSION

The load displacement behaviour observed was dependent on a balance between deformation and fracture characteristics. Thus, variations in behaviour can be explained in terms of test temperature and details of the constituent microstructure. Tests at room temperature showed a large amount of ductility, with three distinct regions of behaviour. Initially, the disk deformed elastically until yielding occurred. Due to a non uniform stress distribution at the punch tip, yielding occurred gradually. The yield front then moved through the thickness of the disk and also radially outwards. A smooth transition from elastic to plastic behaviour therefore occurred. From this point plastic deformation predominated, with work hardening leading to a steady load increase. This steady

period is known as 'membrane stretching'[5], continued until the maximum load was reached. Finally the specimen became dimensionally unstable and fracture, indicated by a significant load drop, followed.

At very low temperature, below -180°C, macro ductility was not observed on the fracture surfaces. Under these conditions the stress required for plastic deformation is high so that initiation of a microdefect resulted in rapid brittle crack propagation through the ferrite matrix. Non metallic inclusions appeared to play no part in this process. As the test temperature increased the critical crack size became larger. The stress field ahead of the cracks caused plasticity and the zone of plastic deformation interacted with non metallic inclusions to form voids. Deformation then caused the ligaments between the newly formed voids to neck down and link up leading to regions of dimpled rupture[6]. Within the transition zone ductile fracture extended until a critical defect size was attained, leading to rapid fracture. At the highest test temperatures the relatively large critical crack size combined with a relatively low yield stress, resulted in significant plasticity through the thickness and a fully ductile fracture. It appears that the presence of inclusions within the process zone of an individual defect was therefore responsible for the development of local dimpling and promoting crack blunting. Thus, it appears that the scatter in fracture energy at a given test temperature was related to the level of inclusions present within the zone of plasticity at a crack tip. For a series of tests at the same temperature the material in each disk did not have identical inclusion profiles, as shown by the metallographic characterisation. For one such test, if the advancing crack encountered no inclusions in it's stress field, the crack remained relatively sharp and on reaching a critical size, rapid brittle fracture occurred. If, on the other hand the crack encountered a region of inclusions inside it's stress field, the ductile process of void formation took place. This effectively blunted the advancing crack and increased the amount of energy required to make the crack grow further. Scatter increased with temperature as a consequence of the decrease in the yield strength, in view of the increase in the plasticity at relatively high temperatures a greater proportion of the disk, had a bearing on the fracture process. As the inclusion profiles were far from uniform throughout the material a larger volume of material involved in fracture increased the probability of scatter.

The variation of fracture energy with test temperature for the disk samples and from the Charpy tests are shown in Figure 4. It is clear immediately that the transition from high to low fracture energy for the disk tests occurred at a much lower test temperatures than for the V-notch impact test. What is also clear is that the transition in the disk specimens occurs over a much narrower temperature range, in the region of 60°C, compared with a transition over approximately 120°C for the Charpy test. From these curves it is possible to quantify the respective transition temperature. For the disk tests, a transition temperature corresponding to a point at 50% of the energy range, was estimated -146°C. The 50 % FATT value calculated from the Charpy tests was 34 °C.

The results have shown that the 50% FATT produced by the impact tests was much higher than the 50% DBTT produced by the disk samples. The test techniques varied in two distinct areas, and this variation appears to be responsible for the differences in the observed data. Firstly, the v-notch manufactured in the impact bars produced triaxial constraint in the material behind the notch. This effectively reduced the ability of the material in this region to deform plastically, minimizing the size of the plastic zone size that could form under loading and promoted brittleness. Secondly the impact specimen, in particular the constrained notch root, experienced a very high loading rate due to the nature of the test.these rapid loading rates also promoted brittleness. The disk test however, produced a biaxial loading system. There was no constraint in the thickness direction of the disk due to it's small dimension and the absence of a notch. A very much lower loading rate was also used for the disk test. These two differing factors meant that material tested via the disk test route exhibited ductility at much lower temperatures than material tested via the impact test route.

CONCLUSIONS

The miniaturised disk bend test described has great potential for examining the deformation and fracture behaviour of materials used in structurally critical components. The test program has demonstrated that credible load:displacement data can be produced over a wide range of temperatures. As the temperature is changed, sensible variations in deformation and fracture behaviour were observed. The energy and displacement to fracture underwent a sharp transition from high to low values, as the temperature is decreased. This indicated a sharp change in the fracture behaviour of the disk specimens. The variation of fracture energy with temperature, Figure 4, has been shown to be similar in form to a Charpy transition curve. Due to differences in geometry and loading conditions, the DBTT is much lower than the FATT. However, it is possible to relate the data from the two different test techniques, and a number of possible correlations are being investigated.

Sensible trends from the fracture surface studies, have shown that the amount of observed ductility increases with the temperature of the tests and, therefore the energy of the tests. Fracture energy scatter associated with the disk test has also been explained using the fracture surface studies.

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