ACCURATE DETERMINATION OF THE FRACTURE TOUGHNESS AT LOW TEMPERATURE BY ULTRASONIC MEANS

Daniel De Vadder & Nicolas Roubier
Laboratoire MSSMAT / UMR 8579 CNRS, École Centrale Paris
Grande voie des vignes, F-92295 Châtenay-Malabry CEDEX

ABSTRACT

Various techniques were tested to extent to low temperatures the detection of initiation of ductile tearing in a three point bend specimen of ferrito-pearlitic steel by diffraction of ultrasonic waves by the crack tip. The most successful one was found by the use of high viscosity grease to glue the transducers to the specimen. Tests were performed to determine their best position. By destructive examination of specimens after interrupted tests at room temperature and at -100 to -140°C, the amount of ductile tearing was determined near the initiation point as detected by the ultrasonic technique.

INTRODUCTION

For an accurate determination of the fracture toughness of metals, it is necessary to detect at an early stage the initiation of ductile tearing in fatigue precracked specimens. In particular this is needed in the brittle to ductile transition temperature region. That determination is known to be difficult at low temperature. Unfortunately the methods commonly used to detect the initiation of ductile tearing present a lack of precision and reproduceability at low temperature. That is why we were asked by IRSID (Institut de Recherche de la Sidérurgie) to adapt our knowledge on crack tip diffraction of ultrasonic waves to three-point bending specimens at temperatures between room temperature and –140°C for early detection of the initiation of ductile tearing. Our previous experience using the so called MU3F method had shown that such an early detection of the initiation of ductile tearing was possible using CT specimens at room temperature.

The MU3F method [1] applies to a CT25 specimen, the transducer being positioned so as to “see” the tip of the crack as the “far tip” position. When an increasing load is subsequently applied, the amplitude of the echo continuously decreases as the crack tip is progressively blunted. The relationship between the amplitude of the echo and the radius of the crack tip had been previously studied theoretically and experimentally. As soon as ductile tearing is initiated, the amplitude of the echo rapidly increases. The change in the curve is not an inflexion point but a cusp, making it very easy to determine the load at which the crack initiates.

Two challenges were to be simultaneously fulfilled. The first was to convert the MU3F method to a new method, applicable to three-point bending specimens. The second challenge was to make this new method usable at low temperature.

POSITION OF THE PROBLEM

The specimens used for three point bending test had a size of 10 x 20 x 80 mm. From a machined notch, a crack was propagated by fatigue to a total length of approximately 10 mm. The specimen was then submitted to an increasing load, producing blunting of the crack tip, and then ductile tearing. The problem is to detect as soon as possible the initiation of ductile tearing. Previously an electrical method was used. In spite of using improved
equipment, this lead to unsatisfactory results. This was due to very low signal to noise ratio, and the fact that the initiation of a crack produces only a shallow minimum on the curve.

**PROBLEMS DUE TO LOW TEMPERATURE AND SOLUTIONS**

To apply ultrasonic techniques to metallic pieces, the most common way is to use manufactured probes, in which the active element, a ceramic piezo-electric piece, is coated with front and back plastic pieces. The backing plays an important role as an absorber (damping) to shorten the length of the ultrasonic pulse. When rapidly lowering the temperature of the probe, the difference in the thermal expansion coefficients between ceramic and plastics produces delamination between one or the other coatings and the piezo-electric element. This puts the probe out of service.

Thus, the only available way was to use the piezo-electric element directly fixed onto the piece under test, without any backing material. The electrical ground of the ultrasonic device being connected to the specimen, the back surface of the piezo-electric disk is connected to the ultrasonic device by means of a small diameter copper wire. When using manufactured probes, it is possible to use angle probes, from which the ultrasonic beam is inclined to the normal to the interface. When using the piezo-electric element directly fixed onto the piece, the axis of the ultrasonic beam is normal to the interface. So, to point the beam towards the crack tip, it was necessary to machine notches in the test piece to place the piezo-electric element; see Fig. 1. The useful surface of each milling is placed in such a way that its axis is directed toward the theoretical position of the crack tip.

![Figure 1: Notches machined in the test piece to manage four locations to place the piezo-electric elements.](image)

Taking into account the geometrical constraints, the angles $A$ and $B$ can be chosen according to table 1.

<table>
<thead>
<tr>
<th>angle A (degrees)</th>
<th>angle B (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>55</td>
</tr>
<tr>
<td>25</td>
<td>55</td>
</tr>
<tr>
<td>20</td>
<td>55</td>
</tr>
<tr>
<td>25</td>
<td>50</td>
</tr>
<tr>
<td>20</td>
<td>50</td>
</tr>
<tr>
<td>20</td>
<td>45</td>
</tr>
</tbody>
</table>
For the main part of the experiments the angles were $A=25^\circ$ and $B=50^\circ$.

It was verified, by means of numerical computation, that the stress field in the vicinity of the crack tip due to the applied load was not affected by the notches. Only the overall compliance of the test piece was modified.

Two sorts of problems then remained to be solved: the fastening of the disk onto the specimen with electrical and acoustical transmission and the lack of any backing material.

The first solution to fasten the piezo-electric elements onto the test piece was to glue them. But this is very expensive as the piezo-electric elements cannot be re-used. It was decided to fasten the piezo-electric elements by means of grease of high viscosity. Several kinds of grease for fastening the disk onto the specimen were tested. It was found that an electrical connection was necessary between the grounded specimen and the metallized front surface of the disk. This was probably due to the major drop of the electrical potential in the grease layer, however thin it may be. Four solutions were tested. In the first an electrically conducting grease was used, but the unavoidable surplus of grease, at the periphery of the thin disk (0.17 mm) caused electrical connection with the opposite electrode.

*Figure 2:* Solutions tested to fasten the piezo-electric disk onto the test piece with electrical contact between the front face of the disk and the grounded test piece.

To avoid these electrical connections, a thin plastic coating was laid down on the upper face, the lower face being preserved by means of a rubber mould (solution 2). This solution was discontinued because the mechanical properties of the plastic material changed dramatically when lowering the temperature, making the acoustical response of the whole too variable.

As a third solution, specially designed piezo-electric disks were manufactured, as indicated in Fig. 2-sketch 3, where both connections were accessible on the free side of the disk. This solution proved to be effective, although the efficient diameter of the disk was reduced. This solution was not, however, adopted because the special design made the piezo-electric disk too expensive.

Finally, a non-conducting grease was chosen. It was thus necessary to rub the disk on the specimen, while pushing on it (slightly because of its brittleness) so as to reduce the thickness of the grease layer until a constant electrical contact was obtained. Several kinds of high viscosity grease were tested for the evolution of their mechanical properties at low temperature. Although during the blunting and tearing test the temperature was kept constant, any rapid evolution of the properties of the grease with changing temperature had to be avoided. When the temperature falls, the grease that was chosen (Apiezon ©) hardens progressively, as shown by the increase in the echo amplitude.
POSITION OF THE TRANSDUCERS

During the first experiments, we used common PZT piezo-electric disks. As these disks are not self-damped it was necessary to use two separate disks, one as a transmitter and one as a receiver. These two transducers can be placed either on the same side of the crack (configuration 1—2), or on different sides of the crack (configuration 1—4). The configuration 2—3 produces too low a signal, and the configuration 1—3 is of no interest.

The first experiments were carried out at room temperature. Previous experience using the MU3F method, [1,2] proved that the configuration 1—2 would be successful. No information was found in the literature on the behaviour of the echo during the blunting of the crack tip and the initiation of ductile tearing for a configuration analogous to 1—4. Nevertheless we tested this configuration in which no diffusion echoes from the rough surface of the crack could interfere with the echo diffracted from the crack tip.

The results appear in Fig. 3.

The behaviour of the echo below 1 kN is due to the opening of the crack tip. At about 6 kN the blunting increases which can be determined on both curves. The initiation of ductile tearing is determined on the 1—2 curve by a cusp, making it easy to measure the load at which this phenomenon occurs. On the contrary, no significant change occurs on the 1—4 curve at the same load. It is clear that the configuration where two transducers are placed one on each side of the crack is inadequate to detect the initiation of ductile tearing.

By replacing conventional PZT piezo-electric disks with lead metaniobate ones it was possible to use a method where the same transducer was the transmitter and the receiver (named configuration 1—1). The comparison of the results obtained at the same time with configurations 1— and 1—2 appears in Fig.4.

Figure 3: Comparison of the configurations 1—2 and 1—4 of the transducers for the detection of the initiation of ductile tearing

Figure 4: Comparison of the configurations 1—1 and 1—2 of the transducers for the detection of the initiation of the ductile tearing.
It is clear that the results are the same. The small difference in the position of each cusp is due to the manual recording of the experimental points during an experiment evolving rapidly during the critical phase. The number of experimental points is insufficient to precisely describe the phenomenon. It is most probable that this difference would completely disappear if an automatic recording of the data were used.

It must not be forgotten that the signal to noise ratio is significantly lower when one transducer only is used. But during our experiments, this signal to noise ratio was always sufficiently high (S/N > 3 dB). One time only (out of 12 experiments) during the last phase of the blunting the amplitude of the signal fell under the noise level; it quickly reappeared as soon as the ductile tearing was initiated.

**Alignment of the beam axis towards the crack tip**

The useful surface of each milling is placed in such a way that its axis is directed toward the theoretical position of the crack tip. The dimension of the surface being 6x10 mm and the diameter of the disk 4 mm, there is a possibility of adaptation of the position of the disk. It was found that to get the best result, it was necessary to apply a pre-load to the specimen, so as to nullify the closure of the crack.

**DETERMINATION OF THE BEST ANGLES**

In this particular case, it was very difficult to determine by means of theory the angle at which the amplitude of the diffracted echo would be the larger. From theory and experiment the smaller the angle, the larger the amplitude when the distance between the transducer and the crack tip is constant. Here, the distance depends on the angle, making the incident beam amplitude vary as a function of the absorption in the material and the beam shape. A set of specimens was machined with different combination of angles A and B so as to experimentally determinate the best set of angles. For these specimens the crack tip was replaced with a machined notch. It was found to be impossible to perform this experiment with the small piezo-electric disks previously used. It was also found that the variability in the results depends more on the way the disk was set against the specimen than on the angle. It is well known that when using the contact technique, the transmission through the interface strongly depends on the transducer pressure on the piece. We then performed experiments by the immersion technique with miniature probes. It was shown that the influence of the distance is not preponderant. The recommended angles are A= 20° and B=45°.

**EXPERIMENTAL VERIFICATIONS**

There was no doubt for us that the decrease in the echo amplitude was due to the blunting, and that its rapid increase was due to the initiation of ductile tearing, but this was to be proven. This was possible and simpler to perform at room temperature. Some tests were stopped at steps just before and just after the cusp in the echo amplitude. The test pieces were cut, then the techniques of metallography were used to display cut views of the crack tip.

**Detection of blunting and ductile tearing**

In Fig. 5 it can be seen that the decrease in the amplitude of the echo corresponds to the blunting of the crack tip. No initiation of a tearing appears.
**Figure 5:** Comparison of the echo amplitude versus load curves and cut views of the specimen AD09. In the left hand photograph mid range graduations are millimetres. In the right hand micrograph we can see the blunting of the crack tip.

In Fig. 6 it can be seen that the cusp in the amplitude of the echo corresponds to the initiation of ductile tearing.

**Figure 6:** Comparison of the echo amplitude versus load curves and cut views of the specimen AD18 (room temperature). In the left hand photograph mid range graduations are millimetres. In the right hand micrograph we can see the large blunting of the crack tip and the initiation of the ductile tearing. The load at which the initiation occurs is 5.9 kN.
EXPERIMENTATIONS AT LOW TEMPERATURE

A series of experiments was then performed at low temperature, in the range of -100°C to -140°C. These experiments confirmed the results obtained at room temperature. Moreover, an increase was observed in the amplitude of the echo diffracted by the as-yet non-blunted crack tip as the temperature decreased. Thus we conclude that the signal to noise ratio is generally higher at low temperature than it is at room temperature. This is probably due to the increase in the viscosity of the coupling grease at low temperature.

The first goal of our experiments was to demonstrate the feasibility of the method and its robustness. Among the whole set of tests there was a perfect equivalence between the increase in the echo amplitude and the initiation of ductile tearing: no initiation missed, no false alarm.

![Comparison of the echo amplitude versus load curves and cut views through the width of the specimen AFE4. The temperature was -120°C.](image)

It can be seen that, as expected, the initiation of ductile tearing appears in the middle part of the specimen. The length of the tearing is estimated to be less than 50µm. The load at which the initiation occurs is 9.16 kN.
The second goal of our experiments was to determine the sensitivity of the method, i.e. the minimum length of ductile tearing that causes a significant increase of the amplitude of the echo. To estimate this sensitivity we performed experiments during which the loading was stopped very early, as soon as the amplitude of the echo was significantly increased.

One example of the results can be seen in Fig. 7. After the experiment, the test piece was cut in thin slices so as to display 6 cut views approximately equally distributed among the 10 mm width of the specimen.

From the curve the load at which the initiation occurred should be 9.16 ± 0.03 kN. This precision was beyond the precision of the measuring instrument. It should be sufficient and reasonable to say that this load was between 9.1 and 9.2 kN.

The length of the ductile tearing that produces a non-ambiguous increase in the echo amplitude was found to be less than 50 µm.

**CONCLUSION**

The ultrasonic diffraction technique to detect ductile tearing initiation in a three point bend specimen of steel was extended for use at low temperature by a proper choice of transducer and of the adhesion technique on the test piece. It was shown that a ductile tear as short as 50µm could be detected unambiguously at a temperature as low as -140°C.

**ACKNOWLEDGEMENTS**

This study was supported by Institut de Recherche de la Sidérurgie (IRSID) France.
The authors gratefully acknowledge that the suggestions of Prof. D. François were helpful to them in writing the text.

**REFERENCES**