ICF100550OR

ROUGHNESS WAVES AS DETECTED ON GLASSY MATERIALS BY DCDC EXPERIMENTS IN THE LOW SPEED REGIME: THE ROLE OF MICROSTRUCTURE.

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ABSTRACT

We studied the low speed fracturation regime (10^{-4} – 10^{-9} $m.s^{-1}$) in different glassy materials (soda-lime glass, glass-ceramics) with variable but controlled length scale of heterogeneity. The chosen mechanical system enabled us to work in pure mode I (tensile) and at a fixed load on DCDC (double cleavage drilled compression) specimen. The internal residual stresses of studied samples were carefully relaxed by appropriate thermal treatment. By means of optical and atomic force (AFM) microscopy techniques fracture surfaces were examined. We evidenced for the first time that the crack front line underwent an oscillating behavior - with a wavelength in the micrometer range - as a result of a reproducible sequence of instabilities. This new phenomenon was observed for different glassy materials providing that their typical length scale was lower than a threshold limit estimated to few nanometers.

KEYWORDS

Fracture, double cleavage drilled compression, surface, crack front waves, AFM.

INTRODUCTION

Quasistatic brittle fracture is a subject of importance for both practical and fundamental reasons. Mainly two classes of experimental set-up have been developed in order to propagate cracks in a stable and controllable manner. The first one is based on a thermally induced stress field [1,2]. According to values of thermal gradient and width of the glass plate three types of crack propagation regime can develop from the small initial notch made to ensure the nucleation of a single propagating crack : no propagation, straight propagation and wavy propagation. Such propagating cracks were observed for velocities between $10^{-2}m.s^{-1}$ and 10^{-5} $m.s^{-1}$. An attempt of explanation was done [1] by using the Cotterell and Rice criterion [3] but partially failed when the real experimental temperature profile was measured [2].

An other classical way to propagate slow cracks is to use double cleavage drilled compression DCDC specimen. This method initially developed by Janssen [4] has numerous advantages : compression

loading, mid plane crack stability and auto precracking. Though the associated stress intensity factor (in mode I) K_I and the average crack speed are at the same order of magnitude as for the "thermal" set-up, to our knowledge no evidence of crack instability has been reported till now.

By working at a fixed load on DCDC specimen, the internal residual stresses of which were carefully relaxed by appropriate thermal treatment, we evidenced - for the first time- that the crack front line underwent an oscillating behavior as a result of a reproducible sequence of instabilities. The wavelength was in the micrometer range.

EXPERIMENTAL

DCDC samples were parallelepipedic ($4x4x40mm^3$) with a central hole of 1mm in diameter (nominal) drilled perpendicularly to two of large parallel faces. Details of experiments have been reported in a previous paper [5]. The sample was then placed in equilibrium on the horizontal basis of our experimental set-up with its large faces vertical. By means of a freely moving piston the upper face of the specimen was then loaded by a variable weight at a rate of about $25N.s^{-1}$ till the crack was initiated. Then the sample was loaded at a constant weight of typically 1500N. These experiments were done at a constant temperature of 22 ± 1°C and at a relative humidity of 50 ± 5%.

The crack front position was measured in the middle of the specimen (at equal distances of the lateral surfaces) by i) a standard video-recorder system for crack speed between 10^{-4} $m.s^{-1}$ and 10^{-6} $m.s^{-1}$ and ii) an optical system with a magnification of x40 for lower crack speeds (10^{-6} $m.s^{-1}$ - 10^{-9} $m.s^{-1}$). In that last case the position of crack front relatively to the center of the specimen was measured with an accuracy of 0.01mm. The stress intensity factor, K_I, was deduced from the measured crack length, using the equation of reference [6]. The $v = f(K_I)$ was then plotted. When the crack speed reached a value of 10^{-9} $m.s^{-1}$ the samples were reloaded in order to gently fracture the sample all over its length. Special care was taken in order to preserve the two halves of the specimen from eventual damages.

Then the fracture surfaces were analyzed by an experimental system (Veeco, D3100®) combining optical microscopy and atomic force microscopy (AFM). Both methods could be simultaneously performed. The samples were glued on the sample holder. This one was moveable under the fixed AFM-optical system thanks to a step-by-step motor. The position of the center of the studied portion of surface relatively to the center of the specimen was measured with an accuracy of less than 10μm. As a matter of fact it was thus possible to virtually know for every point of the AFM or optical images the value of K_I and the speed the crack had when it ran through this point.

AFM experiments were done in a high amplitude resonant mode (« tapping » mode). Experiments were performed in ambient conditions at a relative humidity of 35± 5% and a temperature of 22.0 ± 0.5°C. More details are given in reference [7].

Experiments were performed on two kinds of materials. The first one was a soda-lime silicate glass. A thermal treatment (530°C) was done before fracture experiment in order to remove residual stresses. The second set of samples was made from a lithium alumino-silicate glass-ceramics. By modifying thermal treatments three types of structure were observed : the first one (sample A) corresponds to a pure glassy state as controlled by X-ray diffraction (660°C). The second one (sample B) is related to a slightly unglassy state where small crystals of β-quartz phase were nucleated. This structure was got after a two-steps thermal treatment at plateau temperatures of $T_1 = 750°C$ and $T_2 = 900°C$. Samples C are related to a more unglassy state (increased number of crystallites of larger size) due to a higher temperature (950°C) for the second step of thermal treatment.

RESULTS

In the Figure 1 are plotted typical $v = f(K_I)$ curves as obtained with the different types of samples we studied. A noticeable feature is that for the glass ceramics (open symbols) the curves are shifted towards higher K_I values when the thermal treatment is enforced. The reproducibility of these experiments is revealed by the very good superimposition of the two $v = f(K_I)$ curves performed on two different samples (B1 and B2: up and down triangles) which underwent the same thermal treatment.

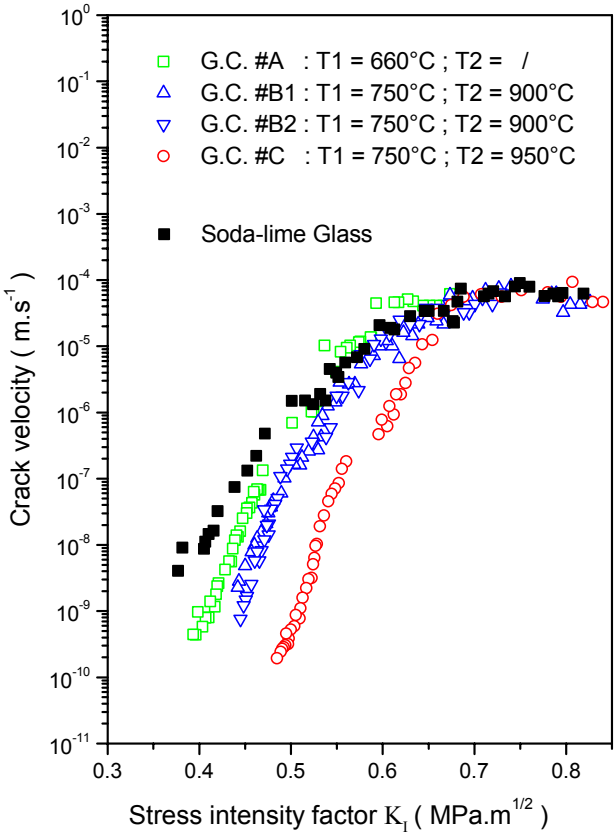

Figure 1: Crack speed versus K_I plots for different materials (G.C. : Glass-ceramics).

The fracture surfaces were then systematically characterized by our optical-AFM system. We put a special attention to the part of the surface fracture corresponding to crack speed reachable (10^{-4}m.s^{-1} – 10^{-9}m.s^{-1}) with our experimental mechanical set-up. We firstly concentrated on the lowest speed regime. The RMS roughness values were characterized by AFM on 10µmx10µm surfaces where crack speed was in the range of 10^{-8} m.s^{-1}. The results are reported on Table 1.

TABLE 1
RMS ROUGHNESS ON 10µmx10µm SURFACES FOR DIFFERENT SUBSTRATES

Soda-lime glass	1 nm
Glass ceramics : sample A	1 nm
Glass ceramics : sample B	5 nm
Glass ceramics : sample C	10 nm

These RMS roughness values for pure glassy materials are very similar whatever the chemical composition of glasses. But for the glass ceramics with an increasing number of larger β-quartz nanocrystals an important enhancement of the RMS roughness is observed.

For higher crack speeds (except for glass-ceramics sample C; see below) an original phenomenon was observed. The study of the fracture surface revealed the presence of periodical variations of its height (in the direction normal to the mean crack plane) along the crack propagation direction. These roughness waves were observed all along the virtual line followed by the crack front of the specimen on both fracture surfaces and for the two symmetrical parts of each of them in relation to the axis of the 1mm diameter hole. An example (soda-lime glass, average crack speed $\approx 2.10^{-5}$ m.s^{-1} and $K_I \approx 0.6$MPa.m$^{1/2}$) is shown on Figure 2. The 70µmx70µm AFM scan of Figure 2.a shows alternate black (deep) and white (high) bands parallel to the crack front. The sine-like shape of these roughness oscillations is better evidenced in Figure 2.b where all the lines parallel to the crack propagation direction (i.e. perpendicular to the black and white bands) of an independent 10µmx10µm AFM scan (not shown) are averaged.

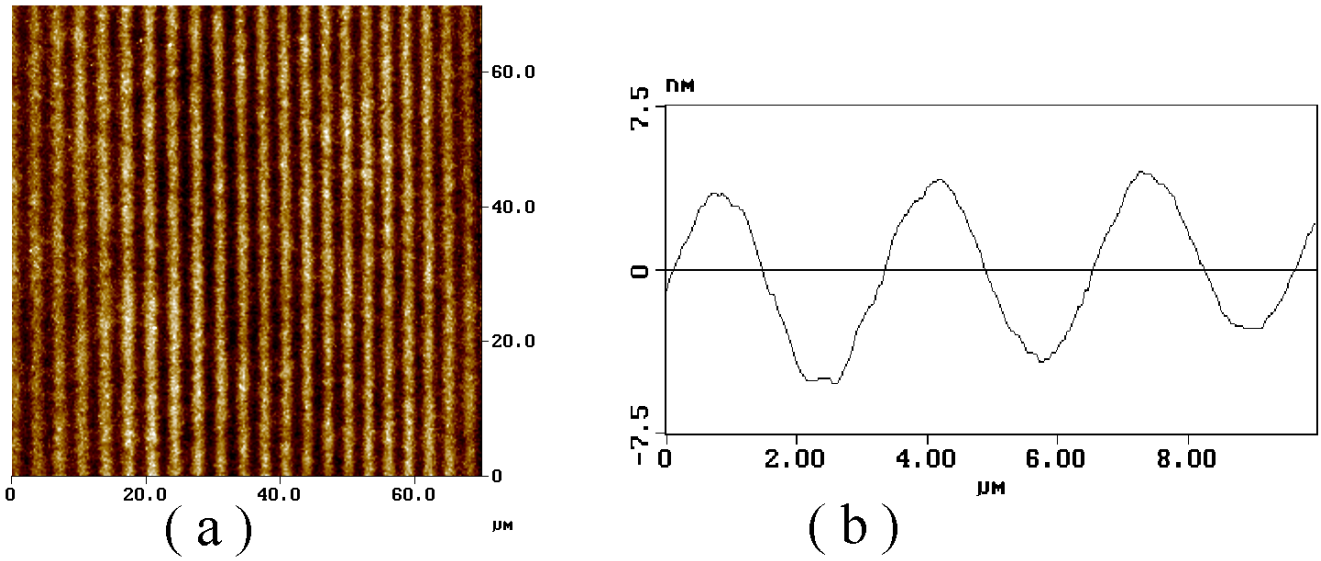

Figure 2: AFM data of fracture surface of soda-lime glass; the vertical scale corresponds to 15 nm. a) 70µmx70µm scan; b) averaged profile of roughness oscillations as obtained on an independent 10µmx10µm scan (not shown)

The occurrence (or not) of such a phenomenon was followed for all the previously described samples. The following common features have been found:

a) Samples of materials in pure glassy state (soda-lime and glass ceramics) revealed this wavy behavior for a large domain of K_I values. For instance, in the case of soda-lime glass, that K_I domain was delimited by following values: $K_I = 0.8$ MPa.m$^{1/2}$ and $K_I = 0.5$ MPa.m$^{1/2}$ respectively corresponding to crack front speeds of 10^{-4} m.s^{-1} and $1.5\ 10^{-6}$ m.s^{-1}. Between these two limiting values the crack ran over a distance of 9mm in approximately 9 minutes. However for samples of glass ceramics –in the pure glassy state- the oscillating domain is slightly narrower $\Delta K_I = 0.17$ MPa.m$^{1/2}$.

b) One striking feature is that the K_I-width of occurrence of the roughness waves is strongly reduced for samples in intermediate unglassy state (sample B): $\Delta K_I = 0.08$ MPa.m$^{1/2}$. No such oscillations were observed on highly unglassy samples (sample C);

c) The scaling of the wavelength of roughness waves with K_I values was systematically studied. Figure 3 shows the typical variation as observed in the case of soda-lime samples. It evidences a large decrease of the wavelength with decreasing values of K_I. Similar variations – in the same range of wavelength - were observed for glassy or slightly unglassy samples.

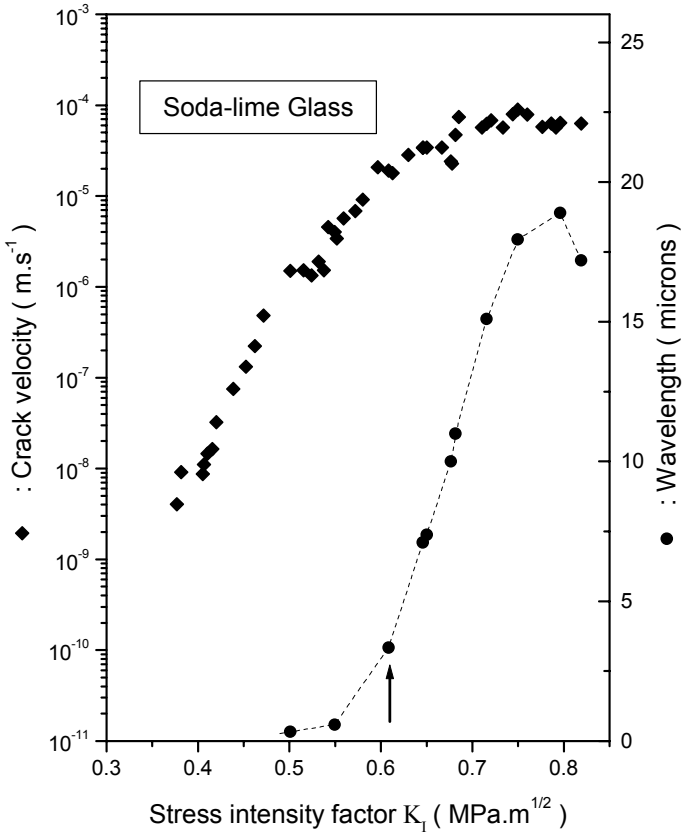

Figure 3: Crack speed (diamonds) and roughness oscillations wavelength (circles) versus K_I for soda-lime glass. The arrow indicates the case corresponding of AFM data of Figure 2.

DISCUSSION

The experimental study presented in this paper revealed that fracture surfaces of various glassy materials are modulated by roughness waves the wave vector of which is parallel to the crack propagation direction. These roughness waves were observed for various heterogeneous materials providing that length scale of the heterogeneities - which can be very roughly estimated by the RMS roughness of surface created by crack propagating at low speed (lower than 10^{-8} m.s^{-1}), see table I – is lower than 10 nm. It means that this phenomenon is directly correlated to the length scale of microstructural heterogeneities inside the broken materials. The wavelength of these ripples is in the micrometer range.

It must be emphasized that, even if the aspect of these roughness ripples presents similarities with patterns observed in stress wave fractography, such an explanation is not relevant for the observations reported in this paper. Indeed no external transverse stress wave generator [8] was used. Furthermore the experimental set-up was working in a very quiet acoustical environment. Moreover the role of hypothetical residual external mechanical vibrations of the DCDC apparatus may be excluded. The resonance frequency and the quality factor of the equivalent mechanical system was indeed estimated to be higher than 70Hz and 6.10^4 respectively. Consequently the hypothetical residual vibrations would have a negligible amplitude at the frequency range (between 0.1Hz to 1Hz) related to the observed roughness waves and calculated by the ratio between the crack front speed and the related wavelength.

Similar results were previously reported by Yuse *et al.* [1] and Ronsin *et al.* [2]. These authors studied the steady propagation of a crack localized within a thermal gradient and observed an oscillatory behavior for given values of crack speed and glass plate width, *W*. Furthermore, Yuse's experiments [1] showed that the related wavelength scaled linearly with *W*. This last result can be presented in an

equivalent manner by plotting (curve not shown) the roughness wavelength versus the stress intensity factor K_I related to this mechanical system using thermal stress according to calculation of Marder [9]. We found that i) this plot reveals similar variations as those deduced from our own experiments and ii) K_I values for wavy behavior are at the same order of magnitude for the two types of experiments (thermal and DCDC). As the wavelength of oscillatory behavior induced by thermal stress was in the millimeter range such as the width of the region of rapid temperature change, it was assumed [1,2] that the related instability mechanisms were only due to 'external' parameters such as stresses induced by the thermal field, independently with the microstructure of broken materials.

Then a very likely explanation to the occurrence of such oscillations in our DCDC experiments is that the crack front similarly undergoes a reproducible sequence of instabilities. As the wavelength is in the micrometer range, the destabilizing factor –in the sense of Cotterell and Rice criterion [3] - from a straight propagation of the crack is likely due to the microstructure of broken materials. When the length scale of heterogeneity is low enough such wavy propagating behavior is observed. On the opposite, materials with larger heterogeneities the fracture surface only presents an incoherent structure (with a much higher roughness). The origin of this instability is perhaps to find in the recently predicted crack front waves [10] which can be generated when fracture occurs in mode I. As experimentally observed [11] the *local* crack speed may exceed the mean crack speed by several order of magnitudes. Therefore it can be forecast that even in the low (mean) speed regime crack front waves may be generated.

CONCLUSION

Experiments were done on a DCDC mechanical set-up working at a fixed load. The internal residual stresses of specimen were carefully relaxed by appropriate thermal treatment. We evidenced for the first time that the crack front line underwent an oscillating behavior along the direction of crack propagation. The wavelength was in the micrometer range and the peak-to-peak amplitude in the nanometer's one. This phenomenon was observed for different glassy materials providing that their typical length scale was lower than a threshold limit estimated to few nanometers. Studies are now performed at sub-micrometer scale in order to understand how this reproducible sequence of instabilities is developing.

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