Experimental Analysis of River Patterns in Silicon Brittle Fractures

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Abstract. — During propagation of brittle cracks in silicon single crystal, steps parallel to the direction of the crack and separated by terraces, are often created. As the crack further propagates, these steps gradually coalesce. This produces the characteristic surface morphology usually identified as a river pattern. An experimental analysis of these river patterns appearing during fractures of silicon single crystals at room temperature is presented. The coalescence of the steps and the coarsening of the terrace widths reveal an effective interaction between the propagating steps. With an interaction between steps supposed to be determined by the size of the terraces, the observed river patterns are numerically reproduced.

The fracture process is one of the most important behavior of solids observed in everyday life. In many circumstances, preventing cracks to appear and to propagate in materials is an absolute requirement. Therefore, numerous studies deal with this problem, as shown in reference [1]. One of the major concepts is the distinction between the brittle fracture and the ductile fracture. The brittle fracture is thought to create two new surfaces and to leave the bulk structure without new defects, whereas the ductile fracture is associated with the plastic deformation of the solids and the appearance of defects like dislocations in the bulk. The brittle fracture is then expected to be essentially a surface effect. The opening of the crack with the creation of new surfaces is the main mechanism which dissipates the elastic energy of the applied stress. This has been pointed out and developed by Griffith [2]. In his calculation of the equilibrium of a crack, the energy balance between the surface energy and the stored elastic energy is clearly stated (see also Ref. [3]). A second important point concerns the subsequent propagation of a crack. Under the application of an external stress large enough to induce a crack, a brittle fracture should propagate with an increasing velocity. It has been realized long ago that a speed limit must be encountered and that the specific effects due to a crack velocity comparable to the speed of sound should be taken into account to describe the

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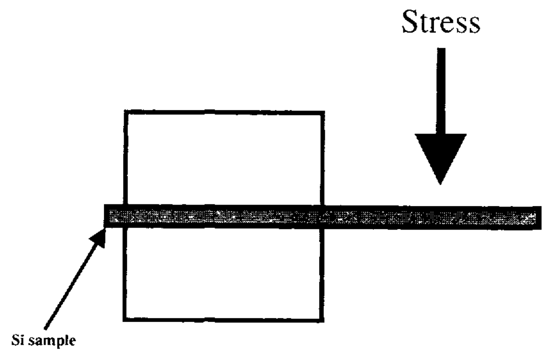

Fig. 1. — Schematic representation of the method used to induce a fracture in silicon single crystals by bending. The silicon single crystal is hold between two metal blocks. A stress is applied on the free part which induces the fracture.

crack path [4,5]. The surface morphology produced by the brittle fracture has indeed been investigated long ago both experimentally [6,7] and theoretically [8].

However, despite significant results, the empirical description of the surface morphology shows that a detailed understanding of the propagation of a brittle fracture and of the morphology of the created surfaces, is not well-established. One major example is the description of a commonly observed surface morphology issued from the brittle fracture with the well-known "mirror-mist-hackle" scenario [6,7]. This refers to the general change of the surface from an apparent optical mirror state to an extremely rough surface with characteristic lengths in the micrometer range or above. This often reported observation shows the evolution towards an important surface roughness with the appearance of increasingly larger characteristic lengths during the crack propagation. These observations have recently triggered new experimental work [9]. The propagation of a brittle crack has been specifically investigated in order to identify the basic mechanisms which induce the appearance of this increasing roughness of the created surface and at the same time the unstable behavior of the crack velocity. These results [9] strongly suggest the existence of a dynamical instability during the propagation of a brittle fracture. This conclusion is further reinforced by recent numerical studies which reproduce the essential experimental features [10]. The existence of such an instability is also discussed in recent theoretical descriptions [11].

The fracture of silicon at room temperature is usually presented as a prototype of the brittle fracture (see for example Refs. [12,13]). In this covalent material, cleavages along the $\{111\}$ and the $\{110\}$ atomic planes are often observed. Also, it has been repeatedly reported [14,15] that cleavage or fracture of a silicon single crystal induced by bending as described in Figure 1, can produce the typical surface morphology shown in Figure 2.

Shortly after the initiation of the crack, its propagation produces a mirror surface (such a surface is thought to have a residual roughness in the nanometer range [14,15]) During further propagation, one observes the simultaneous appearance of many steps which run in the direction parallel to the crack propagation [14,15]. During the subsequent development of the crack, this set of steps and terraces, which forms a "zigzag" crack front, evolves to form what is called in the fracture literature a river pattern. The mechanism which produces this pattern is quite clear: two (or occasionally more) steps join to form a single higher step. In Figure 2, we present a particularly well-developed and ramified example of such a pattern: the steps appear white and they are separated by flat terraces in black. The word *terrace*

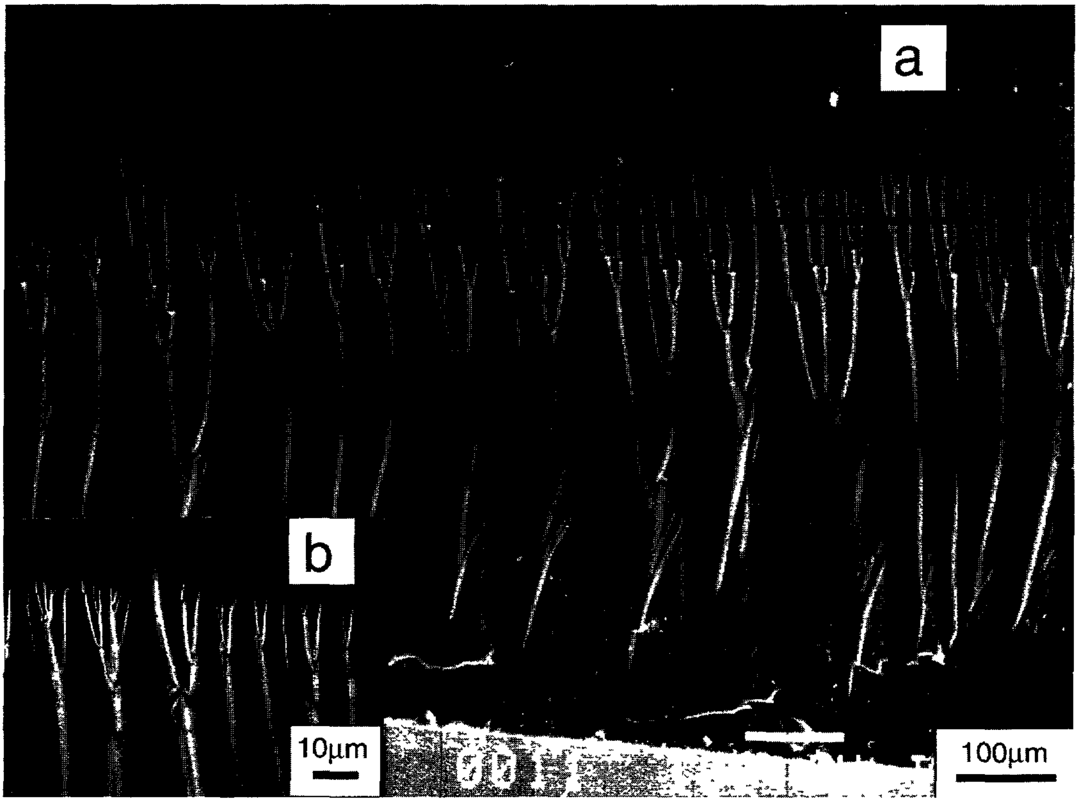

Fig. 2. — a) Observation by Scanning Electron Microscopy of the silicon surface produced at room temperature by a fracture induced by bending as shown in Figure 1. The X-axis shows the direction of the fracture propagation. The Y-axis is parallel to the crack front. The black lines indicate the part of the pattern which is simulated in Figure 5. b) Observation of the same sample by Optical Microscopy in polarized light.

used here does not imply that these terraces are atomically flat well-defined terraces. At the resolution of the SEM and of the Optical Microscope, they experimentally appear flat, i.e, with no observable contrast. Beside the "mirror-mist-hackle" structure, the river pattern is a well-organized structure developed by the brittle fracture. The observation of these river patterns is by no means limited to the aforementioned references and to silicon. This is a generic structure often observed in the brittle fracture of other materials including glass. It is sometimes reported that these structures can be empirically used to identify the local direction of the crack propagation [16]. Gilman [17] has experimentally investigated the formation of the initial steps at the beginning of a river pattern. He has provided experimental evidence that the appearing steps during crack propagation in LiF single crystals originated at screw dislocations. However, although analysis and descriptions have been reported by different authors [18], the river pattern structure does not appear to be completely and satisfactorily understood. Several basic experimental points are still open. In the case of a silicon single crystal with no pre-existent dislocation, the creation of numerous steps which simultaneously appear along the crack front over macroscopic distances much larger than one millimeter certainly calls for further experiments [19]. As the set of steps and terraces is created, the moving front invariably evolves towards a similar but coarsened structure with increasing characteristic lengths (i.e., the step heights and the terrace widths increase). Mechanisms at the origin of this behavior have not received, to our knowledge, a satisfactory experimental explanation.

In this article, we experimentally describe the river patterns observed after the fracture of silicon single crystals. The river patterns show that the crak front presents a scale invariant structure produced by the propagation of interacting steps. An experimental evidence for an effective interaction between the steps will be deduced from the curved path of joining steps. On the basis of this experimental result and taking into account an interaction related to the terrace widths between steps, we shall describe how it is possible to numerically generate river patterns. In particular, this simulation reproduces the observed step structures and the coarsening of the characteristic lengths.

We have used silicon single crystals which are 2 mm thick (111) silicon wafers 2 inches in diameter. One face of the sample was optically polished. The experiments have been performed as described in Figure 1 in air at ambient temperature. The samples were bent in such a way that the fracture has always been initiated in this polished face. No further surface preparation has been made (especially no initial notch made on purpose to localize the fracture). Before the fracture, the silicon wafers presented a circular shape with no indication of crystallographic orientation. No attempt has been made to determine the orientation of the original crystals and then to initiate cracks along well-defined cleavage directions. This lack of orientation control does not seem to impinge on the reproducibility of the experimental results here presented. However, a precise control may become necessary for further quantitative measurements like the precise investigation of the transition between the mirror and the faceted regions. Our observations are very much comparable to the morphologies observed after fracture in vacuum by bending [14, 15]. This also shows that working in air does not seem to introduce noticeable changes. Optical Microscopy and Scanning Electron Microscopy (SEM) have been used to analyze the surface morphology. Using an interferometric technique with polarized light, the perpendicular resolution of the Optical Microscopy is improved to about few nanometers. In order to increase the contrast between the steps and the terraces in electron microscopy and thus to specifically reveal the structure of the river pattern, we have used a large angle of incidence between the steps and the electron beam in SEM investigations. Figure 2a has been acquired in those conditions. A mechanical profilometer has been used to quantitatively analyze the surface morphology (Fig. 3). The lateral resolution of this instrument is limited to the micrometer range, whereas a perpendicular resolution around 10 nm can be achieved. Furthermore, Laue diffraction, together with the optical reflection of a Laser beam, has been used to identify the orientation of the terraces. The accuracy of this measurement is not better than few degrees ($< 5°$). This is essentially due to the small size of the terraces.

The fracture of a silicon single crystal, induced by the method described in Figure 1, gives rise to the surface morphology shown in Figure 2 [14, 15]. In the mirror region, no significant contrast is observed, either by Optical Microscopy ($\times 1000$), or by SEM (even at the largest magnifications $\times 5000 - \times 10000$). Optical Microscopy at small magnification shows that the mirror region of the sample presented in Figure 2, is curved in the propagation direction, i.e., the fracture does not follow a cleavage plane in the mirror region. During the crack propagation, the initial mirror region disappears. Instead terraces and steps are simultaneously nucleated along the crack front. This transition is observed in Figures 2a and 2b.

Perpendicular to the propagation direction (i.e., along the crack front), the morphology of the mirror region investigated by means of the mechanical profilometer is shown in Figure 3a. Figure 3b shows a profile along the crack front acquired using the same technique in the river pattern. Within the precision of the profilometer, the terraces are parallel and present a roughness much smaller than the typical step height between two terraces. Optical Microscopy

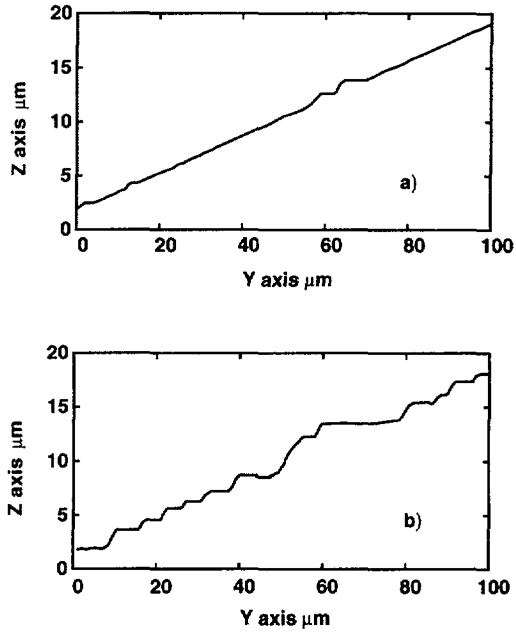

Fig. 3. — Profiles measured by a mechanical profilometer in the direction parallel to the Y-axis. a) in the mirror region, but very close to the frontier between the mirror and the river pattern regions. Most of the profile is characteristic of the mirror region, however some terraces have already been created. b) in the river pattern region. The terraces have been made parallel to Y-axis on purpose by orientation of the sample.

with polarized light also shows a unique orientation of the terraces and no clear variation of contrast within one terrace, which is consistent with a limited roughness. The combination of the Laue diffraction and of the reflection of a Laser beam on the surface of this sample indicates that these terraces exhibit a (111) orientation. In contrast, a step between two terraces is clearly not flat but can be a rather heavily distorted surface. This can be seen on Figure 2a.

As the crack front moves away from the origin of the steps, the steps gradually coalesce, which induces the formation of the river pattern. At any point of the pattern, the crack front is made of alternated steps and terraces. Indeed, by changing the scale in Figure 3b, it is possible to have a description of the crack front at different stages of the river pattern. This is an experimental evidence for the scale invariance of the crack front. This pattern invariance on scales varying from 1 to 100 μm is also supported by the comparison of Figures 2a and 2b.

Appearance of new steps and terraces is not observed during the crack propagation after the initial creation of steps at the interface with the mirror region. Also the crack front appears as a staircase in the presentation of Figure 3: all terraces are parallel to the Y-axis and all steps are up-steps (i.e., with a positive slope). However during the propagation of the crack in the X-direction, these steps can turn, either to the left, or to the right, as seen in Figure 2. Therefore, although the crack front is clearly asymmetric (i.e., no down-step), this asymmetry is not a property of the whole river pattern.

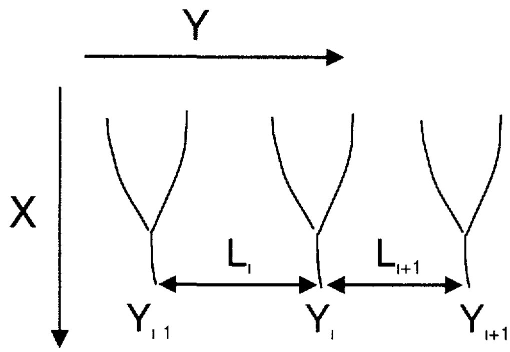

Fig. 4. — Schematic definition of L_i, the width of a terrace and of Y_i, the position of a step.

The steps do not propagate as straight lines. To the contrary, a step curves in the direction of the step it is going to join. One quite systematically observes the increase of this curvature as the intersection is getting closer. Furthermore when a small terrace is located between two larger terraces, the two steps on the side of the small terrace usually join and the small terrace disappears. These two points provide some evidence that the propagating steps do not meet at random but that there exists an effective interaction between the steps which seems to be determined by the size of the neighboring terraces. This statement can be formalized by the following equation:

$$\frac{\mathrm{d}Y_i}{\mathrm{d}X} = C\frac{L_i - L_{i+1}}{L_i + L_{i+1}} \tag{1}$$

where L_i and L_{i+1} are the terrace widths on sides of a step. The position of the step along the crack front is given by Y_i (see Fig. 4). C is a coupling constant and X is the axis along the direction of crack propagation. High steps close to the end of the crack propagation in Figure 2a do not appear strongly curved although they can be separated by large terraces of various sizes. Therefore, that $\mathrm{d}Y_i/\mathrm{d}X$ can be directly proportional to the difference of terrace widths, appears rather unlikely. The denominator $(L_i + L_{i+1})$ is introduced in equation (1) to prevent an extremely strong interaction between steps separated by large neighboring terraces of different sizes [20].

After measurement of the step positions at one stage of the river pattern, equation (1) enables one to numerically calculate the number and the position of steps at any further stage. Figure 5 presents the result of the numerical simulation based on equation (1). The initial positions of steps have been measured along the black line parallel to the Y-axis in Figure 2a. Straight steps have been imposed at both edges (black lines parallel to the X-axis in Fig. 2a). It is then possible to directly compare the simulation (Fig. 5) with the experimental results (Fig. 2a). The path of each step is only approximately described. However the hierarchy of the steps in the region analyzed is reproduced. Therefore Figure 5 shows that the main features of river patterns observed in the brittle fracture can be reproduced by this simple algorithm although this description based on equation (1) is only empirical. This also indicates that a precisely defined initial distribution of terrace widths can completely determine the subsequent evolution of this pattern.

A river pattern can be further characterized by the change of the density of steps as the crack propagates. This is the result presented in Figure 6. The step density has been measured on SEM pictures with magnification ranging from 300 to 16000. Together with the direct observation of a pattern (Figs. 2a and 2b), Figure 6 shows that the density of steps can continuously

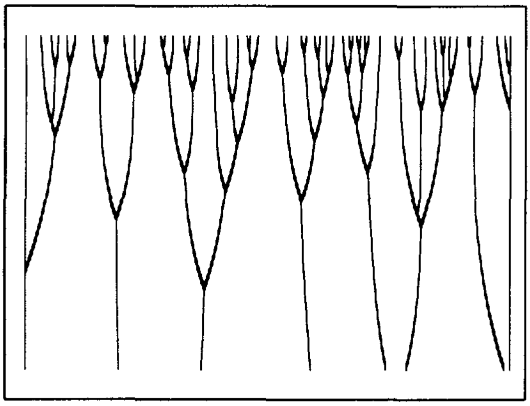

Fig. 5. — Calculation of the step pattern produced during the propagation of the crack. This numerical simulation is based on equation (1) (see text). This figure should be directly compared to the area defined by black lines in Figure 2a. The initial positions of steps used in the calculation have been measured in Figure 2a along the black line parallel to the Y-axis.

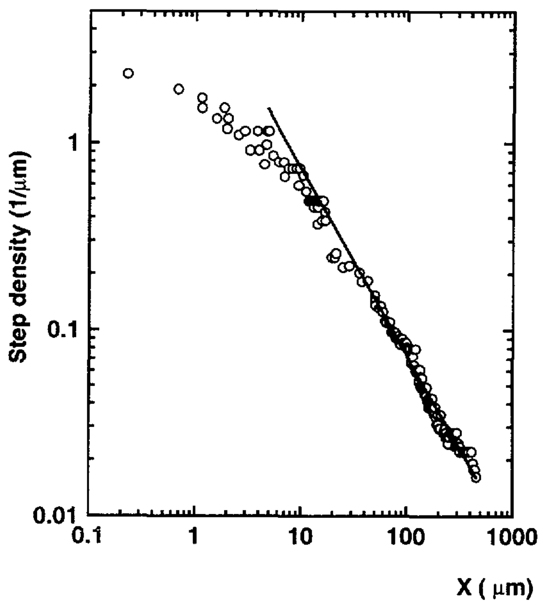

Fig. 6. — Variations of the step density along the direction of the fracture propagation (X-axis). The origin of the curve ($X = 0$) is at the interface between the mirror and the stepped regions. This density of steps has been extracted from SEM pictures with different magnifications ($\times 300$, $\times 500$, $\times 3000$, $\times 5000$, $\times 16000$). The full line (-) is calculated using $N(x) = A/x$.

decrease by two orders of magnitude during propagation of the crack. As the pattern develops away from the mirror region ($X > 10$ μm), the density of steps appears in Figure 6 to be described by a power law with an exponent close to -1. However measurements on different samples indicate that other values of this exponent can be found. Further experiments with improved control of the crack growth parameters are needed to clearly establish what determines the interaction between steps and this exponent [21].

In conclusion, a detailed investigation of the river patterns observed in the brittle fracture of silicon shows that they can be analyzed as the propagation of interacting interfaces. Although its origin is not experimentally identified, this effective interaction favors the large distances between the steps and thus leads to a coarsening of the terrace widths. By using the simplest interaction that one can deduce from the experimental results, the main features of river patterns produced by brittle fractures of silicon can be numerically reproduced. The calculated pattern appears completely determined by the interaction between steps and by the initial terrace width distribution.

We have not directly addressed the transition between the mirror and the stepped regions. A relevant experimental description of this transition in silicon certainly needs to show whether or not a plastic deformation is present. Also the characterization of the surface morphology close to this transition calls for further experimental work with better spatial resolution. Interesting aspects of these river patterns may be: i) they are examples of surface morphologies produced by brittle fracture which can be quantitatively analyzed by means of direct space techniques like Atomic Force Microscopy, ii) previous works on brittle fracture [6, 17, 22] suggest that it may be possible to induce and to control the appearance of steps. To some extent, this means that these patterns can be seen as a quantitative tool to study the propagation of brittle cracks. Although the present work remains preliminary in view of the difficulty of this problem, it is an attempt in this direction.

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[19] In references [14,15], it is noticed that the mirror surface is associated with the part of the sample initially in extension and that the part of the sample, which usually exhibits the river pattern, is at the beginning in compression. This is experimentally correct. However, to our knowledge, it has not been shown how this remark could be relevant to describe the transition between the two regimes.

[20] Changes in step heigths as the crack propagates should certainly appear in equation (1). However no direct measurement of the step heigths is here presented. The necessary introduction of $(L_i + L_{i+1})$ in equation (1) could appear as a way to represent changes in the step heigths and the influence of the step heigth on the step propagation.

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