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An improved technique for characterizing the fracture toughness via scratch test experiments

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ABSTRACT

The scratch test consists in pushing a tool across the surface of a weaker material at a given penetration depth; and it has several applications in Science and Engineering including strength testing of rocks and ceramics, damage of polymers and metals and quality control of thin films and coatings. Despite numerous attempts in the scientific literature, the application of scratch tests to the characterization of fracture properties remains a challenge and a heavily controversial topic. Therefore, this investigation aims at articulating a rigorous theoretical and experimental framework in order to assess the fracture toughness at both the macroscopic and the microscopic length scales, using scratch tests. First, we apply optical microscopy and scanning electron microscopy to investigate the physical evidence of crack initiation, crack propagation and material removal mechanisms during scratch tests. Then, we employ Finite Element simulations of crack growth during macroscopic scratch tests to assess the influence of the blade back-rake angle, the friction coefficient between the blade and the material and the wear flat of the blade on the scratching forces, thus testing the robustness of our Linear Elastic Fracture Mechanics scratch model. Finally, at the microscopic scale, a meticulous scratch probe calibration procedure is described to improve the accuracy of the fracture properties determination by addressing important issues such as moisture content, specimen surface cleanliness and choice of reference material. In summary, we bring forward a robust, convenient and accurate method that is applied to polymers, ceramics and metals and can be further applied to the multi-scale study of fracture processes in complex and challenging materials such as gas shale, cement paste and cortical bone.

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1. Introduction

The scratch test consists in pulling a probe across the surface of a softer material as illustrated in Fig. 1. Although scratch tests are relevant nowadays to several fields of science and engineering, ranging from strength characterization of ceramics [9,24,10] to coating and adhesion of thin films [16–18,21] and wear and damage of metals [2] and polymers [14,15,35], the underlying failure mechanisms are still not fully understood.

Early attempts were made in the late 90s to quantify the scratch deformation mechanisms in polymers as a function of the applied load, and of the scratching probe geometry. For large values of the scratch probe half-apex angle, $\phi > 45^\circ$, Briscoe et al. [14] reported the creation of an elastic reversible groove, for low normal force levels, $F_V < 2$ N, followed by ductile ploughing as the normal load was raised up to 20 N. On the other hand, beyond 20 N and for sharper angles, $\phi < 45^\circ$, micro-cutting processes were observed. The transition from elastic to plastic behavior in scratch testing has been extensively studied by Schirrer and coworkers et al. [15], who developed numerical and analytical solutions to link the residual groove recovery angle to the prescribed strain [30], the initial contact radius, and the friction coefficient between the probe and the material [26]. However, the work of Kurkcu et al. [25] suggests that these models are not valid at a larger scale ($d > 40$ μm or $F_V = 15$ N).

In contrast to ductile deformation mechanisms, little attention has been given to brittle fracture in scratch testing. Wong et al. [35] reported the presence of semi-circular cracks on the residual groove for scratch tests carried out on polypropylene with a spherical probe and with vertical force values greater than 18 N; however, they did not incorporate the fracture properties into their analysis of the scratch response. Williams [34] suggested the existence of a linear relationship between the vertical force and the cube of the groove size, based on the equilibrium crack dimension model developed by Lawn and Fuller [27] for indentation fracture; however, he concluded that micro-cutting in scratch

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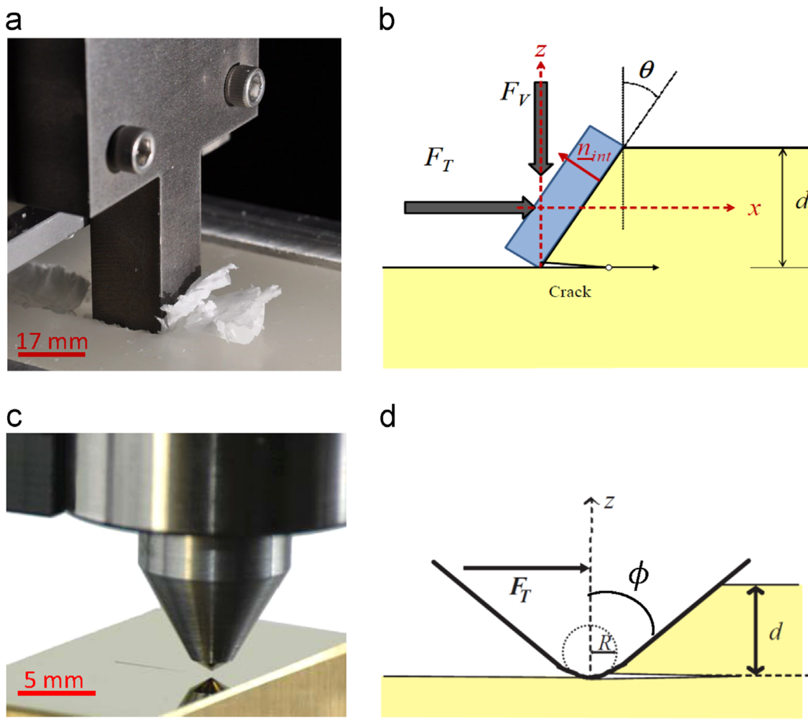

Fig. 1. (color online) (a) Macroscopic scratch tests experiments on paraffin wax with a straight parallelepiped steel blade. (b) Idealized 2-D geometry of a macroscopic scratch test: a rectangular blade, making an angle θ with the vertical axis, is pushed horizontally against an elastic material, at a penetration depth d, by applying a vertical force F_V and a horizontal force F_T. (c) 200-μm Rockwell C diamond probe commonly used in microscopic scratch testing applications [9,19]. (d) Idealized representation of a scratch test at the microscopic scale: an axi-symmetric probe, of tip radius R and half-apex angle ϕ, is pulled across the surface of a softer material at a penetration depth d, resulting in a horizontal force F_T.

tests could only be predominant at very large scales (with groove dimensions in tens of millimeters).

The challenge lies in connecting the scratch response to quantifiable material fracture properties while integrating different levels of complexity inherent to the test such as:

- several length scales (the penetration depth, d, can range from nanometers to tenths of centimeter) and force ranges (the vertical force, F_V, can range from millinewtons to thousands of newtons);
- a three-dimensional probe shape (parallelepiped, cylindrical, conical or spherical);
- a variety of deformation patterns (reversible groove, permanent depression or debris generation).

In recent works [4–6], through the application of physical arguments such as Dimensional Analysis [11] and Fracture Energy [22] to scratch testing we demonstrated that the mode of failure (fracture vs. plastic yielding) is influenced by the material properties as well as the geometry of the scratching tool. Moreover, an analytical model based on Linear Elastic Fracture mechanics was constructed to rationalize scratch tests at both the macroscopic (cf. Fig. 1(a)) and microscopic (Fig. 1(c)) length scales. Consider then a horizontal crack emanating from the tip of the scratch probe during a scratch test; the scratch probe being a parallelepiped (cf. Fig. 1 (b)) or axi-symmetric (cf. Fig. 1(d)) and inclined with an angle θ with respect to the vertical axis. Using an Airy stress function $\varphi(x,z)$ under plane conditions, the stress components read

$$\sigma_{xx} = \frac{\partial^2 \varphi}{\partial z^2} = -6bxz + c \tag{1}$$

$$\sigma_{xz} = \frac{\partial^2 \varphi}{\partial x \partial z} = b\left(3z^2 - \frac{3}{4}d^2\right) \tag{2}$$

$$\sigma_{zz} = \frac{\partial^2 \varphi}{\partial x^2} = 0 \tag{3}$$

where the constants (b,c) are chosen so as to satisfy the stress boundary conditions at the material-probe interface (S):

$$\int_{(S)} \underline{\underline{\sigma}} \cdot \underline{n}\, dS = F_T \underline{e}_x - F_V \underline{e}_z \tag{4}$$

Applying the energetic contour-independent J-integral [32] the strain energy release rate, which is the energy required to create a unit fracture surface, reads

$$\mathcal{G} = \frac{1-\nu^2}{E} \frac{F_{eq}^2}{2pA} \tag{5}$$

where $E^* = E/(1-\nu^2)$ is the plane strain elastic modulus; E being Young's modulus and ν being Poisson's ratio. 2 pA is the *scratch probe shape function* equal to $2(w+2d)wd$ for a parallelepiped blade of out-of-plane width w and to $4(\tan\phi/\cos\phi)\, d^3$ for a cone of half-apex angle ϕ; d being the penetration depth. Finally, F_{eq} includes the contributions of both the vertical, F_V, and the horizontal, F_T, forces. In particular, the contribution of the vertical force F_T to the fracture process is significant only when the probe is inclined, i.e. $\theta > 0$. In other words, for a parallelepiped tool or a conical probe, the equivalent force F_{eq} reads as follows:

$$F_{eq} = \begin{cases} F_T & \text{if } \theta = 0 \\ \sqrt{F_T^2 + \frac{3}{5}F_V^2} & \text{if } \theta > 0 \end{cases} \tag{6}$$

By entering the expression of the strain energy release rate in the fracture criterion $(\mathcal{G} = \mathcal{G}_f)$, which is enforced at the onset of crack propagation, it then becomes possible to link the forces and the