Crack Velocity Thresholds and Healing in Mica

Kinetic crack effects in mica in nitrogen, air, and water environments are investigated. There are applied loading thresholds below which the cracks close up and heal. Significant hysteresis is observed during load–unload–reload cycles. The results are interpreted in terms of the restraining influence of oscillatory attractive surface forces at the crack interface. © 1986 Academic Press, Inc.

Recently there has been renewed interest in the crack velocity and healing threshold behavior in brittle solids (1–4). The importance of this research area is twofold: first, it bears on the fundamental nature of fracture and the associated "crack laws" (5); second, it offers the prospect of gaining information on "surface forces" (6) acting at newly formed, narrow interfaces.

In this communication we describe some preliminary results of threshold experiments on a muscovite mica. Our technique is a derivative of that used first by Obreimoff (7) and later (in somewhat more sophisticated forms) by several others (8–12). Mica is chosen as a test material for its ideal, molecularly smooth cleavage (13), providing optimal conditions for crack retraction at unloading. Also, mica has been the focus of attention in the extensive and delicate intermolecular force studies by Israelachvili and co-workers (6, 14), where surface–surface interactions have been measured directly down to atomic separations. Thus the opportunity exists for correlating the fracture results with independently determined surface force characteristics.

The experimental set-up is shown schematically in Fig. 1. A wedge is inserted into one end of a mica flake, thus propagating a stable crack (7, 15). The crack can be made to run forward or backward in a controlled manner by simply advancing or retracting the wedge via a micrometer drive. In this way we can investigate threshold properties, including some hitherto unexplored kinetic aspects. The crack system is conveniently monitored by observing the Fizeau fringe system (13) at the open interface through the microscope objective. Using beam theory, the stress intensity factor K_a associated with the applied (wedge) loading is readily evaluated in terms of the measured crack length, c (Fig. 1).[1] The motion of the fringe system can be used to determine the crack velocity, v. Hence a characteristic $v(K_a)$ response can be generated for any given test conditions.

In our experiments we ran cyclic tests by adjusting the stationary wedge position and following the subsequent crack motion. The test sequence was as follows: (i) *load* (L) by advancing the wedge, so that the crack ran through virgin material; (ii) *unload* (U) by retracting the wedge, to allow the crack to close and heal; (iii) *reload* (R) by reinserting the wedge (but not quite to the first position), thus driving the crack through the healed interface. In each of these three stages the crack (by virtue of its highly stable configuration) (15) at first moved rapidly in response to the disturbance from its old equilibrium, and progressively slowed down as it approached its new equilibrium. With this arrangement several cycles could be run on one specimen, by advancing the crack further into virgin material at the beginning of each new cycle, thereby avoiding specimen-to-specimen data scatter. This was particularly useful in data comparisons for different environments.

The results of a typical experiment are shown in Fig. 2. The data are for runs in a nitrogen stream, air (50–60% relative humidity) and distilled water. Each L–U–R sequence is designated by arrow indicators for forward and backward crack motion. The individual data branches represent data accumulations over static loading periods of about one hour. Chronologically, the progression of data is downward (i.e., toward zero velocity) on all these branches.

Certain features of the data plots in Fig. 2 warrant comment. First, the $v(K_a)$ plots are very steep (much steeper than, for instance, in glass) (16), indicative of sharp thresholds on the K_a axis. (The fact that the cracks reverse direction between stages of the loading cycle is confirmation enough that the $v(K_a)$ curves must extrapolate to zero velocity at *nonzero* K_a levels.) Second, these zero velocity thresholds displace to lower K_a in more moist environments, demonstrating that the presence of water molecules must play an important role in the equilibrium, as well as the kinetic (16), crack states. Third, for a given environment, the $v(K_a)$ curves exhibit some *hysteresis,* suggesting that the thresholds may be representative of metastable rather than true equilibrium. Finally, of the two stages U and R which pertain to the healed crack interface, it is the former which appears to contribute more strongly to the phase lag between crack motion and driving force; this is reflected in Fig. 2, notably in the air data, by a somewhat less abrupt approach to threshold in the U curve.

[1] The stress intensity factor K_a defines the strength of the concentrated stress field at the crack tip, and is thus used by the fracture mechanics community as a measure of the crack driving force. In terms of the dimensions c, δ, and d shown in Fig. 1, $K_a = 3^{1/2}\delta d^{3/2}E/2c^2$, where E is Young's modulus (15).

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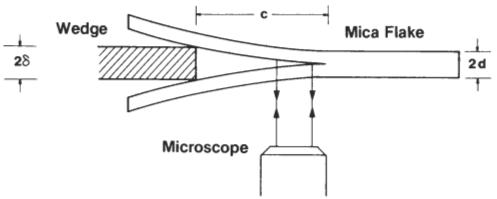

FIG. 1. Set-up for observing crack motion in wedged mica flake. The crack is subjected to a load–unload–reload (L–U–R) cycle in specified environments.

We assert that these phenomena have strong implications concerning surface forces. Clearly, there must be significant attractive forces at the crack interface in order for the healing to occur in the first place (11, 17, 18). In our case the healing can not be due to reversible remaking of the virgin "bonds" across the mica cleavage surfaces since, if it were, the R curves in Fig. 2 would simply retrace the original L curves. This is not entirely unexpected, for the inevitable presence of water and other environmental species will surely "contaminate" the interface. Indeed, the forward crack motion through the virgin material almost certainly involves some chemical adsorption reaction at the tip bonds; (10, 16, 19) true reversibility in the crack growth would require these environmental molecular species to be desorbed and ejected from the tip on closure. The presence of such trapped molecular layers has in fact been confirmed directly by Bailey (20), who used optical interference techniques to compare the thickness of mica specimens before and after partial cleavage: she found a difference of 0.6 ± 0.2 nm (which corresponds closely to two water molecule diameters). The attractive surface forces are therefore believed to be a measure of the strength of interaction between the adsorbed species and the narrowly confined mica walls.

We are now in a position to account for the features observed in Fig. 2. The thresholds arise because the attractive surface forces generate a negative stress intensity factor, K_i, at the crack tip (2, 4, 5). Zero velocity configurations then correspond to balance points of zero *net* driving force, $K = K_a + K_i = 0$. The interfacial contribution K_i is expressible in terms of the work to separate the healed crack walls, i.e., the interfacial energy (4), explaining the environmental dependence in the threshold levels.

This brings us to perhaps the most fascinating aspects of the results, namely the hysteresis in the crack velocity cycle and the relatively pronounced backward creep during the unloading stage U. Now if the thresholds were to represent states of true mechanical equilibrium at $K = 0$, consistent with the notion of a well-defined interfacial energy term K_i, the healing should occur spontaneously as the loading wedge is withdrawn (for otherwise K_a would diminish below K_i, corresponding to "forbidden compressive states," $K < 0$), and the zero velocity configurations for any given environment should be identical for all branches of the $v(K_a)$ curves. It seems, therefore, that there must exist some kind of energy barrier to the crack motion in the healing region. This is consistent with an oscillatory component in the $K_i(c)$ function (in certain analogy to the periodic, "lattice trapping" surface energy functions described by Thomson (21) in his theories of activated crack-tip bond rupture), reflecting some element of "discreteness" in the interface structure.

Such discreteness has in fact been demonstrated, in dramatic fashion, by Israelachvili *et al.* (6, 14) in their surface force experiments. They measure oscillations in the interaction energy vs distance functions for liquid-immersed mica sheets (Fig. 3): the strength of the oscillations increases rapidly as the separation diminishes, and the periodicity is close to the diameter of the intervening molecules. These deviations from the classical conception of a monotonic potential function are attributed to near-surface "ordering" of water or other environmental molecules (6). Thus to bring opposing mica surfaces together one has to traverse the energy barriers in the potential curve, by squeezing out the intervening fluid layer by layer. There can be significant rate effects associated with this molecular-scale drainage process, as shown in the mica–mica squeezing experiments in organic liquids by Chan and Horn (22). This would explain why the *backward* crack motion in the healed region is comparatively sluggish. It also provides an explanation for the hysteresis, since we would expect the crack to become "trapped" at the secondary minima in Fig. 3 at various points along the interface, giving rise

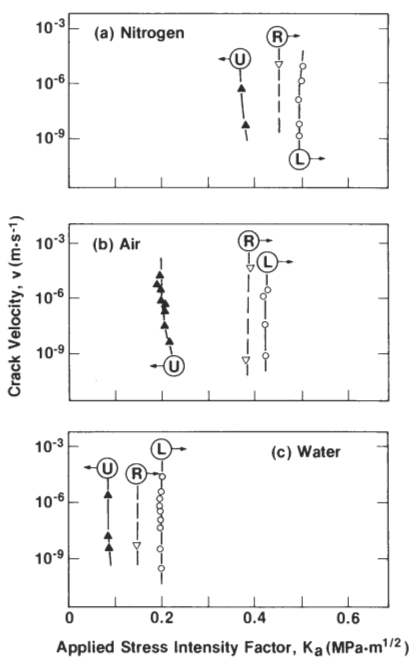

FIG. 2. Crack velocity responses in mica for cyclic (L–U–R) loading in three environments. Arrows to the right denote crack advance, to the left crack retreat.

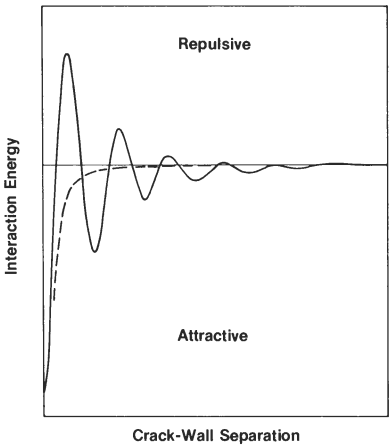

FIG. 3. Schematic of interaction potential function for mica–mica surfaces in fluid environment. Oscillation distance corresponds to molecular diameter of environmental species. Broken curve is the continuum van der Waals function [after Ref. (6)].

to quasi-equilibrium configurations. It would appear, from our earlier reference to Bailey's optical interference measurements, that the barriers to drainage are sufficiently high as to preclude ejection of the final two water layers.

It is well to emphasize that the surface force influence described here exists whether the crack is growing in the healed or virgin regions. The crack velocity in the latter region must be determined by the *net* driving force K (not just K_a, as is often implicitly assumed) (4), and provided the environment continues to have access to the interface we must expect the K_i contribution to reflect strongly in the measured $v(K_a)$ response (5).

Our experiments are in their early stages of development. There are outstanding questions concerning the specific roles of different environmental species, the potential influence of capillary condensation in gaseous environments, the effects of crack history (e.g., aging time between cyclic loading stages), etc., on the strength of the interfacial interaction. Nevertheless, the preliminary findings go far enough to suggest that surface forces may be of considerably greater significance in brittle fracture than generally supposed. Conversely, controlled fracture testing may serve as a useful adjunct to the more direct techniques of surface force measurement.

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