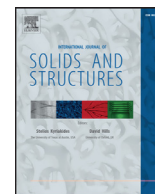




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Kinetics of channeling cracks in polymeric coatings

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ABSTRACT

Techniques to measure the relationship between crack velocity and energy-release rate for channeling cracks in thin films and coatings are presented in this paper. The approach uses linear-elastic fracture mechanics to relate the crack spacing, material properties and geometry to the energy-release rate of a channeling crack, by means of detailed finite-element calculations. The velocities of individual cracks are monitored and related to their distance to nearest neighbors as part of these calculations. Other parameters that were identified as being important in determining the energy-release rate were the depth below the surface to which the crack penetrated, and the residual strain in the cracked layer. A technique to determine the crack depth was developed which involved focused-ion beam milling of the specimen, followed by electron microscopy. The residual strain in the crack layer was determined by measuring the crack-mouth opening using atomic-force microscopy, and comparing this to numerical analyses.

These approaches were used to analyze a multi-layer system consisting of a polymeric, colloidal-silica nano-composite layer and a primer layer coating a polycarbonate substrate. The crack growth showed a threshold value of energy-release rate corresponding to about 6.6 J/m², with a minimum detectable crack velocity of about 3 nm/s. When the energy-release rate was increased to about 25 J/m², the resultant crack velocity was 10⁻² m/s. The channeling cracks in this system exhibited the characteristics of a thermally-activated rupture process, but showed no evidence of fatigue or stress-corrosion cracking under the conditions studied.

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1. Introduction

The replacement of glass by transparent polymers, such as polycarbonates, has many advantages for automotive applications. Lower densities permit lighter-weight, energy-efficient structures. Alternative processing routes allow for more creative designs. There are, however, two major limitations with the use of polymers for automotive glazing. The first is their vulnerability to ultra-violet light, which can cause embrittlement and discoloration. The second is their relative softness, which results in them being very easy to scratch. Both of these limitations can be overcome by the application of nano-composite coatings containing a hard wear-resistant phase and ultra-violet absorbers. However, the intrinsic hardness of these coatings makes them vulnerable to failure by fracture over prolonged service conditions (Nichols and Peters, 2002). Therefore, the motivation for this paper was to explore the phenomenon of

time-dependent fracture in nano-composite coatings on polymer substrates.

Cracking induced by uniaxial tensile stresses in a coating on a substrate occurs by a mechanism known as channeling; this results in a series of parallel cracks propagating across the coating. The basic fracture mechanics for this problem in an elastic system is well understood. For example, the energy-release rate for a single crack channeling across a coating of thickness h_c on a very tough substrate of thickness H , and subjected to a uniform tensile strain of ε_0 , is of the form (Beuth, 1992; Vlassak, 2003)

$$\mathcal{G} = \varepsilon_0^2 \bar{E}_c h_c f_D(\tilde{\alpha}, \tilde{\beta}, h_c/H). \quad (1)$$

In this equation, $\bar{E}_c = E_c/(1 - \nu_c^2)$, is the plane-strain Young's modulus of the coating, ν_c is Poisson's ratio, and $f_D(\tilde{\alpha}, \tilde{\beta}, h_c/H)$ is a function of the two Dundurs' parameters, $\tilde{\alpha}$ and $\tilde{\beta}$, which are measures of the elastic mismatch between the coating and substrate, and the ratio of coating and substrate thicknesses. The Dundurs' parameters are given by Dundurs (1969)

$$\tilde{\alpha} = \frac{\bar{E}_c - \bar{E}_s}{\bar{E}_c + \bar{E}_s} \quad (2)$$

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and

$$\tilde{\beta} = \frac{\bar{E}_c(1 - \bar{\nu}_s) - \bar{E}_s(1 - \bar{\nu}_c)}{2(\bar{E}_c + \bar{E}_s)} \quad (3)$$

where, $\bar{\nu}_c = \nu_c/(1 - \nu_c)$ is the plane-strain Poisson's ratio for the coating, and \bar{E}_s and $\bar{\nu}_s$ are the plane-strain modulus and the plane-strain Poisson's ratio, respectively, for the substrate. A single crack propagates if the energy-release rate exceeds the toughness of the coating, Γ_c . Additional cracks can grow at higher levels of the energy-release rate, forming patterns of parallel cracks. The density of the cracks increases with applied strain (Thouless, 1990; Thouless et al., 1992; Hutchinson and Suo, 1992); it also depends on the modulus ratio (Shenoy et al., 2000) and toughness (Thouless et al., 2011) mismatch between the coating and substrate, and on the statistics of the initial flaw population (Takayama et al., 2014).

A coating will remain crack-free during service, provided the value of the energy-release rate (Eqn. (1)) is always less than the toughness of the coating. However, cracks can develop during service for a variety of reasons. A build up of stress could increase the energy-release rate. Degradation of the coating could decrease the toughness. Cyclic loading could result in sub-critical fatigue-crack initiation and growth. Stress-corrosion, creep, or visco-elastic effects could induce time-dependent crack growth. It is the time-dependent and fatigue aspects of the problem that were explored in this study.

Time-dependent crack growth can occur if the constraint on a coating is gradually relaxed by creep of the substrate, or of the primer layer (Prévost and Suo, 2002; Liang et al., 2003). This is because both creep and visco-elastic relaxation lower the effective modulus of a material, and the energy-release rate for channeling in a coating increases with the compliance of the material beneath it (Beuth, 1992). For example, if one were to model the substrate as a standard-linear solid, one could consider two limiting values for its modulus (Wineman and Rajagopal, 2000): a higher, unrelaxed modulus at short time scales, and a lower, relaxed modulus at long time scales. If the energy-release rate for channeling associated with the unrelaxed substrate modulus exceeds the coating toughness, Γ_c , spontaneous fracture occurs. If the energy-release rate for channeling associated with the relaxed substrate modulus is less than Γ_c , fracture will never occur. In between this range of energy-release rates, the crack will channel across the coating at a steady-state velocity associated with a time scale corresponding to the effective modulus that makes $\mathcal{G} = \Gamma_c$.

Time-dependent crack growth associated with visco-elastic effects in the coating itself would not seem to be possible, although it does occur in visco-elastic materials under load control. Under these conditions, a lower threshold for crack growth is associated with the fully-relaxed modulus of the coating, and an upper threshold for crack growth is associated with the unrelaxed modulus (Wang et al., 2016). However, under the strain-controlled conditions of a visco-elastic coating bonded to an elastic substrate, the drop in modulus with time would not result in such behavior, as it would cause the energy-release rate to decrease with a drop in crack velocity.

Stress-corrosion cracking can cause time-dependent crack growth (Wiederhorn, 1967). This has been shown to occur in silica-containing coatings (McKerrow and Vlassak, 2005) and in silicon-nitride films (Kim et al., 2016; Graham and Pierron, 2017). It has also been shown to be an issue for delamination of silica-containing films (Lane et al., 2001; Vlassak et al., 2005; Guyer and Dauskardt, 2004). Like visco-elastic crack growth, stress-corrosion cracking occurs within a limited range of energy-release rate. There is an upper threshold when the crack grows so quickly that there is no time for the crack tip to interact with the environment (such as water vapor). There is a lower threshold when the crack grows so slowly that the crack tip is essentially in equilibrium with the

environment, corresponding to slow crack growth in which the environment (such as water vapor) can interact with the bonds at the crack tip, essentially lowering the toughness of the coating. Between these limits the crack velocity depends either on how quickly the environment can reach the crack tip, or on the rate of reaction between the environment and the atoms or molecules at the crack tip (Cook and Liniger, 1993; Lawn, 1993). There is also a higher threshold, corresponding to crack growth in a vacuum. Stress-corrosion cracking can be identified by any observed effect of the environment on crack velocity, and by the existence of these upper and lower thresholds.

Crack growth in solids is always associated with the rupture of bonds or the pullout of molecules. These are thermally-activated processes, so crack growth is inherently a time-dependent phenomenon, even in the absence of environmental effects¹. For example, while stress-corrosion cracking is a thermally-activated process, so is crack growth in a vacuum (Wiederhorn et al., 1974). However, the activation energies and other parameters may not always be in a range for the time-dependent nature of crack growth to be manifested. A thermodynamic model of fracture has been developed by Lawn (1975); this relates the crack velocity, v , to the energy-release rate, \mathcal{G} , and the toughness of the coating, Γ_c . In this model, the driving force for thermally-activated rupture at the crack tip is the difference between the energy-release rate and the intrinsic toughness of the material. At equilibrium, the energy-release rate equals the toughness, and the rate of bond rupture equals the rate of bond healing, so no crack propagation occurs. This corresponds to a threshold energy-release rate for fracture. Above this threshold, the driving force for rupture increases, and the crack velocity increases exponentially with energy-release rate. Thermal-activation of rupture also results in an exponential increase in velocity with temperature. The crack velocity is given by

$$v = A \exp(-Q/kT) \sinh[B(\mathcal{G} - \Gamma_c)/kT], \quad (4)$$

where k is Boltzmann's constant, T is the absolute temperature, Q is the activation energy for the rupture process. The parameters A and B are related to the discrete increments of length over which rupture can occur, such as atomic spacing, both in the direction of crack advance and perpendicular to it. The parameter A is given by $A \approx kT\Delta_x/\pi\hbar$, where Δ_x is the characteristic length in the direction of crack growth and \hbar is the Dirac constant. The parameter B is given by $B \approx \Delta_x\Delta_y/2$, where Δ_y is the characteristic length parallel to the crack tip. In the absence of environmental effects, this kinetic model exhibits only a lower threshold associated with equilibrium.

In this paper, we explore channel cracking in a nano-composite coating on polycarbonate. Empirical observations had shown that exposure to service conditions resulted in the formation of such cracks after a period of time. This provided the original motivation behind the study: to try and elucidate what might be the nature of any time-dependent crack growth. Therefore, we developed a technique, based on fracture mechanics, to measure crack velocities as a function of energy-release rate in coatings. We showed that the crack velocity had the form expected from Lawn's kinetic model, for which we could obtain material parameters associated with the fracture process. From a practical perspective, we also demonstrated that there was no fatigue or stress-corrosion cracking in the particular system described in this paper.

¹ Although not discussed specifically, there seems to be evidence of thermally-activated crack growth in the absence of environmental effects in the studies of Refs. Kim et al. (2016) and Graham and Pierron (2017).

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