

Creep induced rate effects on radial cracks in multilayered structures

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Abstract This paper considers foundation and epoxy creep induced loading rate effects on radial cracks in multilayered structures. These include top layers of glass or silicon that are bonded to polycarbonate foundations with epoxy. The creep properties of the epoxy join and the polycarbonate foundation are determined using compression experiments and spring-dashpot models. The measured creep parameters are then incorporated into an analytical mechanics model, and finite element simulations are used to predict the effects of creep on the critical loads for radial cracking at different loading rates. The models suggest that the combined effects of creep and slow crack growth must be considered in the predictions of the critical loads required for radial cracking in the systems containing glass top layers. Since slow crack growth does not occur in silicon, the model considering the creep effect is used to predict the critical loads for radial cracking in the systems containing silicon top layers. In both of the structures, analytical solutions are obtained for bi-layer structures and finite element simulations are used for tri-layer structures. Our results show that the analytical solutions obtained by bi-layer structures provide good estimations for tri-layer structures when the epoxy thickness is less than 100 μm . The predictions obtained for both systems are shown to provide improved predictions by comparing with experimental results reported by Lee et al. [*J. Am. Ceram. Soc.*, 2002, 85(8), 2019–2024]. In both systems, the modeling of join/substrate

creep is shown to be important for the accurate prediction of loading rate effects on radial cracking.

1 Introduction

In a recent paper, Lee et al. [1] showed that the modeling of slow crack growth in the glass layer of glass/epoxy/polycarbonate multilayers is required for the prediction of rate effects on the critical loads for radial cracking under Hertzian indentation loading. However, since slow crack growth does not occur in bulk silicon, they were unable to fully explain the loading rate effects that were observed in their studies of contact-induced deformation in silicon/epoxy/polycarbonate multi-layers. Lee et al. [1] suggested that the differences between the measured and predicted critical loads that were obtained from these systems were due to substrate creep effects. However, these effects were not considered in the mechanics models that they used to estimate the effects of loading rate on the critical loads for radial cracking in the top layers (silicon or glass).

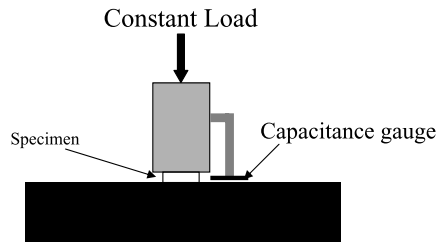
In this paper, we consider the combined effects of foundation/join creep and slow crack growth on the radial cracking in the top layers. The join/foundation creep properties are measured using compression creep experiments. The measured creep properties are then fitted to a spring-dashpot model, which is incorporated into analytical models and finite element simulations of multilayer deformation at different loading rates. The models show that considerations of substrate and join creep are required for the prediction of the critical loads for radial cracking in the two systems considered in this study. In the case of the systems containing glass layers, the prediction of the radial cracking requires the combined modeling of substrate/join creep and slow crack growth. The role of slow crack growth is not considered in

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Table 1 Creep properties of polycarbonate and epoxy

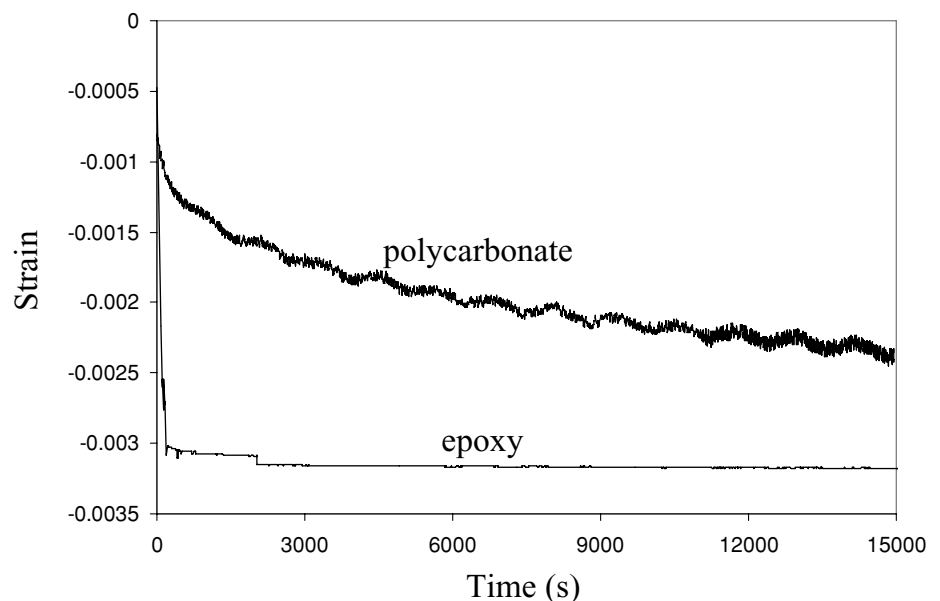
Time t (s)	Polycarbonate			Epoxy		
	k_1 (GPa)	k_2 (GPa)	η (GPa · s)	k_1 (GPa)	k_2 (GPa)	η (GPa · s)
$0 < t < 150$	1.32	0.98	150	0.63	2.57	0.032
$150 < t < 1500$	1.15	1.15	410	0.50	2.70	5.15
$1500 < t < 15000$	0.74	1.56	2510	0.48	2.72	7.80

**Fig. 1** Schematic of creep test set-up

the systems containing silicon top layers, since bulk silicon does not undergo sub-critical crack growth.

2 Creep measurements and results

The creep experiments were performed on a loading system (Fig. 1) that was designed and built at Princeton University. A constant stress of 1.73 MPa was applied to polycarbonate samples with dimensions of $11.67 \times 5.10 \times 4.40$ mm, or epoxy specimens with a diameter of 21 mm and a thickness of 8.42 mm. The loads were applied for durations for 5 h. During loading, the time-dependent deformation of the specimen was measured using a capacitance gauge (Fig. 1), and stored in a computer data acquisition system. Further details

Fig. 2 Strain increment under constant stress of polycarbonate and epoxy (compressive stress is 1.73 MPa)

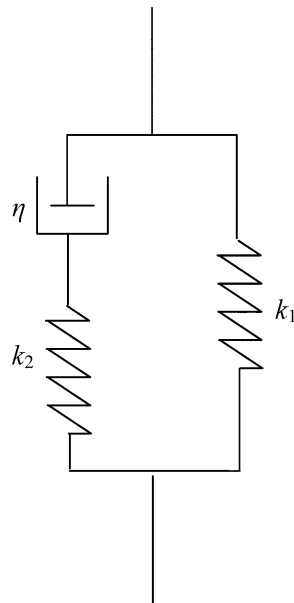
on the creep measurement techniques are provided in Ref. [2].

The results are shown in Fig. 2. These show that epoxy and polycarbonate will creep under constant stress. The experimental data is fitted with a 3-parameter spring-dashpot model (Zener Model) shown in Fig. 3. The parameters were found to be time-dependent, as shown in Table 1. Hence, a more complex model is needed to accurately describe the creep deformation of the polycarbonate and epoxy join materials. Nevertheless, for simplicity, in this paper, time-step material properties are used to model creep deformation for the two materials, as shown in Table 1.

3 Creep induced stress increment under different loading rate

Bulk silicon does not exhibit slow crack growth [1, 2]. Hence, the rate-dependence of the critical load in the multilayered systems containing silicon top layers is thought to be due to the viscoelastic deformation of the substrate and join layers. In this section, the possible effects of polycarbonate foundation creep and epoxy join creep will be evaluated. We will first derive the analytical solutions for bi-layer structures, and then compare it with the finite element simulations of tri-layer structures with different epoxy thickness.

Fig. 3 3-parameter spring-dashpot solid model (Zener model)



If the thickness of the epoxy layer is less than 10 microns, the whole structure can be treated as a bi-layer system. By stress analysis of plates on elastic foundation with center-point loading on the top coating layer, the maximum stress in the top layer can be obtained by a coating/substrate bi-layer model as [4, 5]:

$$P = \frac{B\sigma d^2}{\log(CE_c/E_f)}, \tag{1}$$

where P is the load applied on the indenter. σ is the maximum tensile stress in the top layer near the interface of the top layer and the foundation. d is the top layer thickness, E_c and E_f are Young’s moduli of the top layer and foundation, respectively. B and C are dimensionless coefficients. The quantity C is expected to be close to 1, because the tensile stresses concentrated in the top coating must vanish when $E_c = E_f$ [4]. For simplicity, C is taken to be 1 in this paper. Also, B was estimated from finite element simulations to be 1.455.

Since the foundation is a viscous material, the Young’s modulus of the foundation, E_f , decreases with time (Fig. 2). Equation (1) shows that the load will decrease with time to keep the same maximum tensile stress in the top layer near the interface. Therefore, the critical load will decrease with decreasing loading rate. To obtain the explicit solution for the estimation of the critical load, we model the substrate as a 3-parameter spring-dashpot solid (Zener model), as shown in Fig. 3. The Young’s modulus at a constant loading rate is then given by [6]:

$$\frac{1}{E_f} = \frac{1}{k_1} \left\{ 1 - \frac{k_2}{k_1 + k_2} \exp \left[- \frac{k_1 k_2 t}{\eta(k_1 + k_2)} \right] \right\}, \tag{2}$$

Table 2 Material properties used in calculation

	E (GPa)	Poisson’s ratio
Glass	70	0.2
Silicon	110	0.2
Epoxy	3.2	0.4
Polycarbonate	2.3	0.4

where t is the time, which can be expressed as: $t_R = P_m/\dot{P}$ under constant loading rates; k_1 , k_2 and η are the parameters used in the Zener viscoelastic model (Fig. 3), which are constant in certain time regime (Table 1). Substituting Eq. (2) into Eq. (1), and using relationship $t_R = P_m/\dot{P}$, gives the relationship between the critical load P_m and loading rate \dot{P} as

$$P_m \log \left(\frac{CE_c}{k_1} \left\{ 1 - \frac{k_2}{k_1 + k_2} \exp \left[- \frac{k_1 k_2 P_m}{\eta(k_1 + k_2) \dot{P}} \right] \right\} \right) = B\sigma_m d^2, \tag{3}$$

where σ_m is the tensile stress required for radial crack formation in the top layer. It is determined by the initial flaw size and the fracture toughness. By fitting Eq. (3) with the experimental data of Lee et al. [1], σ_m is estimated to be 170 MPa. Substituting the appropriate parameters (Tables 1 and 2) into Eq. (3) gives estimated critical load as a function of loading rate.

The results of the analyses are presented in Fig. 4. For comparison, we also plot the previously reported experimental results by Lee et al. [1] and the finite element simulations of tri-layer structures with different epoxy thickness. The finite element simulations were carried out using the commercial finite element software, ABAQUS (Version 6.3, Hibbit, Karlsson & Sorensen, Inc., Pawtucket, RI, 2002), adopting

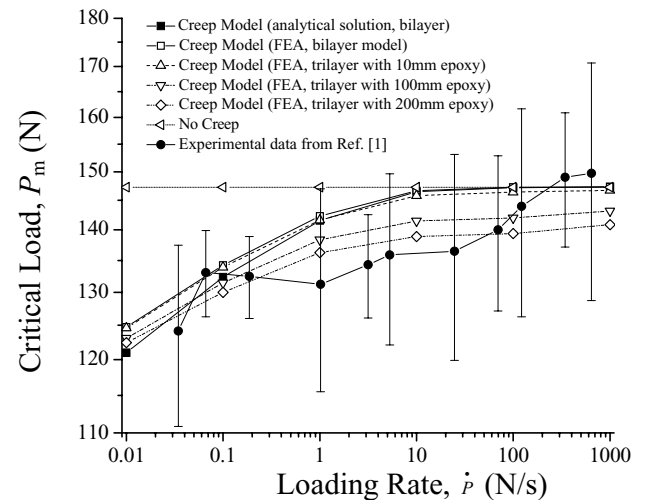


Fig. 4 Critical load as a function of loading rate in the multiplayer system containing silicon top layer

four-node axisymmetric elements. We can see that when the epoxy thickness is less than $100\ \mu\text{m}$, the difference between the predictions of the analytical solution of bi-layer structures and those of the finite element simulations of tri-layer structures is less than 3%. The epoxy thickness for the structures tested by Lee et al. is around $10\ \mu\text{m}$ [1]. Our model predicts the right range of the experimental results. It is interesting to note that the fitted critical tensile stress for radial cracking was about 70% greater than the strength measured in the four-point bending experiments by Lee et al. [1]. This may be due to the absence of compliant substrate effects in the four-point bending experiments of Lee et al. [1]. Hence, the differences are attributed largely to compliant substrate creep effects in the current experiments.

4 Creep-assisted slow crack growth model (CASCg)

Since glass is highly susceptible to Slow Crack Growth (SCG) [6, 8], SCG may contribute significantly to the loading rate effects reported previously by Lee et al. [1]. Furthermore, the stresses in the top glass layers may be increased by the creep of the foundation and join layers. This section will, therefore, explore the possible effects of creep-assisted slow crack growth. Small flaws at the bottom surface of the top coating can be treated as the initial cracks. The stress intensity factor of the initial crack is induced by the Herzian indentation loading. Due to the creep of the epoxy and foundation, the stress at the bottom surface increases with time as shown in the previous section, thus the stress intensity factor also increases. This expedites the slow crack growth process. When the crack grows to the final crack length and the stress intensity factor reaches the fracture toughness, the radial crack forms. To accurately simulate this process, one needs to simulate the crack propagation, because the crack growth will change the stress state. However, for the cases of cracking in ceramics coatings, the initial crack length is very small, so the stress intensity factor can be estimated by the stress on the bottom surface of the top coating without cracking. Under this assumption, the condition to cause the radial crack can be obtained by the standard power law slow crack growth theory [1, 8, 9] under the condition that final crack length is much greater than the initial flaw size:

$$\int_0^{t_R} \sigma^N dt = D, \quad (4)$$

where $D \approx \frac{K_{IC}^N}{(N/2-1)v_0\psi^N a_i^{N/2-1}}$, independent of load and time. N and v_0 are crack velocity exponent and coefficient, respectively; ψ is a crack geometry coefficient, which is 1.12 for edge crack; a_i is the initial flaw size in the lower surface of the glass layer, and K_{IC} is the fracture toughness. $t_R = P_m/\dot{P}$, is the time required to form the radial crack. σ is the stress

at the bottom of the top coating. As the epoxy join and polycarbonate foundation will creep, the stress, σ , will vary with time, t . If we know the expression for $\sigma(t)$ and the fitting parameter D , we can obtain the critical load, P_m , as a function of \dot{P} .

For tri-layer structures, $\sigma(t)$ can be obtained by finite element simulations. However, when the epoxy layer is thin, its effects on the stress in coating layer are very small. Under this condition, the stress, $\sigma(t)$ in a tri-layer structure can be estimated by that in a bi-layer structure, which can be obtained analytically. We will first derive the analytical solution of creep-assisted slow crack growth for a bi-layer structure, and then compare it with the finite element simulations of tri-layer structures with different epoxy thickness.

Substituting Eq. (1) into Eq. (4) gives

$$\int_0^{t_R} [P \log(CE_c/E_f(t))]^N dt = A^{N+1}/(N+1), \quad (5)$$

where $A = (N+1)^{1/(N+1)} D^{1/(N+1)} (Bd^2)^{N/(N+1)}$, is independent of load and time. As the foundation creeps, the Young's modulus, E_f , varies with time. It is difficult to obtain an explicit solution from the integration of Eq. (5). Hence, we will consider a simple worst scenario, in which $E_f = E_f(t_R)$. In this case, Eq. (5) can be integrated analytically to give:

$$\frac{P_m}{\dot{P}^{1/(N+1)}} [\log(CE_c/E_f(t_R))]^{N/(N+1)} = A. \quad (6)$$

Substituting Eq. (2) into Eq. (6) leads to:

$$\frac{P_m}{\dot{P}^{1/(N+1)}} \left[\log \left(\frac{CE_c}{k_1} \left\{ 1 - \frac{k_2}{k_1 + k_2} \right. \right. \right. \\ \left. \left. \left. \times \exp \left[-\frac{k_1 k_2 P_m}{\eta(k_1 + K_2) \dot{P}} \right] \right\} \right) \right]^{N/(N+1)} = A. \quad (7)$$

Equation (7) can be used to estimate the critical load for radial cracking due to creep-assisted slow crack growth. Two special cases are considered here. One is a condition without slow crack growth, where $N \approx \infty$. In this case, noticing that $K_{IC} = \psi \sigma_m a_f^{1/2}$, Eq. (7) reduces to Eq. (3). The other case is that of an elastic foundation, where $\eta \approx \infty$. In this case, Eq. (7) reduces to the form as: $P_m = [A(N+1)\dot{P}]^{1/(N+1)}$, which is Eq. (7) of Ref. [1].

Since $N \approx 18$ for glass [1], we can fit the experimental data of Lee et al. [1] to Eq. (7). The results of the fit are shown in Fig. 5. All the material properties are taken from our measurements. The only fitting parameter is A , whose value is $352.8\ \text{N}^{18/19} \cdot \text{s}^{1/19}$ for the glass/epoxy/polycarbonate structure. The solid line with solid square dots corresponds to the results from the creep-assisted slow crack growth model. The solid dots in Fig. 5 correspond to the experimental data

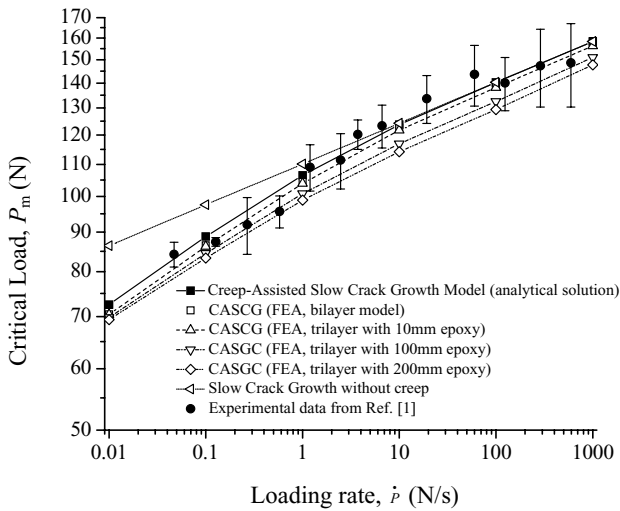


Fig. 5 Critical load as a function of loading rate in the multiplayer system containing glass top layer

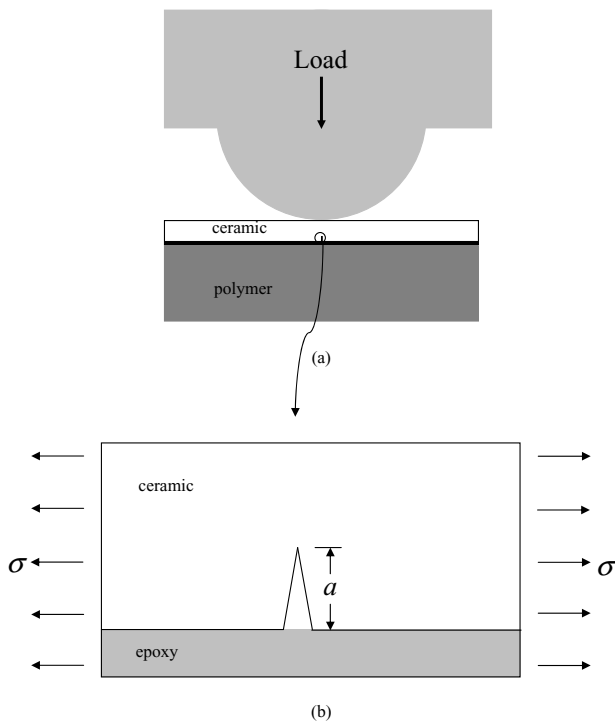


Fig. 6 Schematic to show the flaw in the ceramic (a) Fullview image showing 3-layer structure under Hertzian contact load; (b) Enlarged image of local area around middle of the subsurface of glass layer and, therefore the local stress state is tension

obtained from prior work by Lee et al. [1]. In general, the predictions obtained from the creep-assisted slow crack growth model are in good agreement with the experimental data at both low and high loading rates. In contrast, the predictions obtained from the slow crack growth model were not in good agreement with the experimental data obtained at

slow loading rates where creep effects are more important. The loading rate effects of tri-layer structures with different thickness are also obtained by substituting finite element results into Eq. (4). As expected, the epoxy layer does not play important role when its thickness is less than 100 μm . Our results suggest that the modeling of creep effects is important in the prediction of critical conditions for radial cracking in the top glass layers in glass/epoxy/polycarbonate multi-layers.

5 Conclusions

Epoxy and foundation creep play an important role in determining the critical loads of different loading rates under Hertzian indentation. The loading rate effects on the critical loads of silicon top layer can be explained by the foundation creep, and the creep-assisted slow crack growth can be used to explain the loading rate effects on the critical loads of glass top layer. A similar model can be developed to study the creep effects on the fatigue of multilayered structures under different loading frequencies.

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References

1. C.-S. LEE, D. K. KIM, J. SÁNCHEZ, P. MIRANDA, A. PAJARES and B. R. LAWN, *J. Am. Ceram. Soc.* **85**(8) (2002) 2019.
2. M. HUANG, X. NIU, P. SHROTRIYA, V. P. THOMPSON, E. D. REKOW and W. O. SOBOYEJO, "Contact damage of dental multilayers: viscous deformation and fatigue mechanisms." *Trans. ASME, J. Eng. Mater. Tech.* **127** (2005) 33.
3. B. R. LAWN, D. B. MARSHALL and P. CHANTIKUL, *J. Mater. Sci.* **16**(7) (1981) 1769.
4. B. R. LAWN, K. S. LEE, H. CHAI, A. PAJARES, D. K. KIM, S. WUTTIPHAN, I. M. PETERSON and X. HU, *Adv. Eng. Mater.* **2**(11) (2000) 745.
5. S. TIMOSHENKO and S. WOINOWSKY-KRIEGER, in "Theory of Plates and Shells," 2nd edn, (McGraw-Hill, New York, 1959) p. 275.
6. C. ZENER, in "Elasticity and Anelasticity of Metals" (Chicago Press, 1948).
7. S. M. WIEDERHORN and L. H. BOLZ, *J. Am. Ceram. Soc.* **53**(10) (1970) 543.
8. T. P. DABBS, B. R. LAWN and P. L. KELLY, *Phys. Chem. Glasses* **23**(2) (1982) 58.
9. S. M. WIEDERHORN, "Subcritical crack growth in ceramics," in "Fracture Mechanics of Ceramics", edited by R. C. Bradt, F. F. Lange and D. P. H. Hasselman, vol. 2 (Plenum, New York, 1974).