



# An investigation into strain rate dependent constitutive properties of a sandwiched epoxy interface



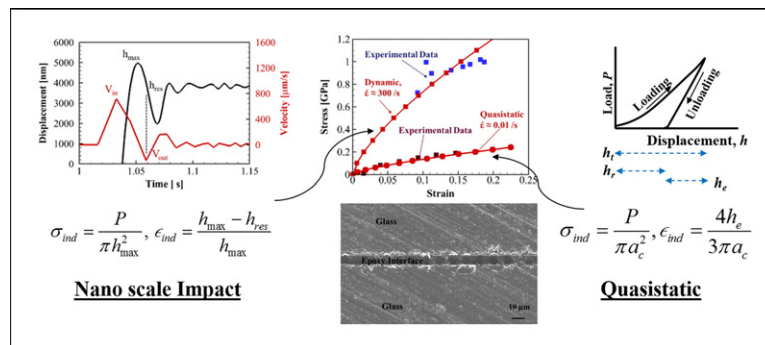
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## HIGHLIGHTS

- The stress-strain behavior of 2 μm to 10 μm-thick epoxy interfaces between glass plates is examined.
- Proposed model captures well the lateral stress (confinement) and strain rate effects during indentation of glass-epoxy interfaces.
- An analytical model is also proposed to predict the lateral stress during indentation experiments.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

### Article history:

Received 28 May 2016

Received in revised form 15 September 2016

Accepted 17 September 2016

Available online 27 September 2016

### Keywords:

Interface strength

High strain rate

Dynamic loading

Confinement effect

Viscoplastic deformation

## ABSTRACT

A composite material fracture strength can significantly depend on the constitutive description of interfaces. A computational model of composite deformation should, therefore, incorporate interface constitutive behavior. However, separating main phase constitutive behavior from interface constitutive behavior in mechanical property measurement experiments is an arduous task. In this work, an epoxy interface is analyzed under quasistatic and dynamic loading conditions to obtain a description of interfacial constitutive response at strain rates from  $10^{-2}$  to  $10^3 \text{ s}^{-1}$ . The approach relies on describing interfaces as a confined material phase between two unconfined phases. Dynamic microscale impact tests are used to obtain stress-strain response as a function of strain rate for the analyzed interface. The rate dependent stress-strain response is fitted to the Johnson-Cook constitutive model. Based on the analyses of confinement effects, a power law constitutive model is proposed to predict the interface deformation behavior with a dependence on both strain rate and interface thickness.

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## 1. Introduction

It is well established that a material exhibits changes in its constitutive response as a function of changes in temperature, length scale of analyses, and strain rates [1–4]. The material damage under different impact scenarios such as due to sand particles, various projectiles etc.

occurs via different deformation mechanisms. An understanding of such mechanisms is required to develop new constitutive laws. While a number of constitutive laws for polycrystalline materials are available in literature [5], recently the focus has shifted to understand the influence of interfaces [5]. The current article aims at investigating interface failure under micro scale dynamic impacts. There are several material constitutive models available to understand the strain rate effect on a material's dynamic strength and failure [6–9] and the development of such material models requires precise experiments because of smaller

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temporal and spatial resolution. One such example is the split Hopkinson bar experiment for intermediate strain rate experiments in tensile, torsion, and compression modes [10]. The experimental procedure involves impacting bulk material samples and measuring average stress-strain response for the whole sample. The development of nanoindentation techniques in the last few decades has enabled measurement of nanoscale to microscale material site-specific behavior such as an interface sandwiched between two materials [11]. The present work focuses on using a small-scale impact test based on nanoindentation at high strain rates to measure strain rate dependent interface constitutive behavior.

In the last several decades, significant efforts have been devoted to understand the interface behavior in materials. The interface stress consists of two parts, both arising from the distorted atomic structure near the interface: the first part, independent of the deformation of solids, is the interface residual stress, and the second part which contributes to the stress field related to the deformation is related to the interface elasticity. Interface plastic deformation, particularly the initial yield point is sensitive to the local strain (or local stress) of a heterogeneous material, which includes both the local surface or interface residual stress and local stress-strain relationship [12]. The interface mechanics studies are broadly divided in two parts, (1) studies that consider the interface as zero thickness entity [13–18] and (2) studies that consider interfaces to have a finite thickness interface as a finite thickness [19–28]. For interface of zero thickness as given by Gibbs [18], surface stress formulations were developed using continuum mechanics by Shuttleworth [17] relating the interfacial excess energy to surface stress [17]. The formulation was further modified by Gurtin and coworkers to find relations between the surface and body stresses [16]. Such relations have been widely used and modified in the recent years by Sharma et al. [29], Yang et al. [30], Cammarata et al. [15] etc. Dingerville and Qu [31] related surface stress at interface with interface in-plane strain and in-plane stresses based on a modification of Shuttleworth and Herring model. Interface strength properties of finite thickness interfaces have been studied for metallic interface layers [19,20,26,32], interfaces at the glass/epoxy in composites [22,23,27], interface as thermal barriers [21] and molecular interfaces [33,34]. Interfaces are subjected to the confinement lateral stresses from the adjacent phases. Mechanical strength of materials in confined spaces has been shown to be strongly affected by lateral stresses [35]. Various analysis techniques have been adopted to model the confinement effect on crack formation process with pre and post peak behavior in confinement [36], stress strain model for compressive fracture [37], the fracture mode changes in armor materials [38], and the lateral expansion associated with the compression [39]. All such studies are applicable on a specific material system investigated by researchers whether it is composite, metallic or ceramic material. There has not been a general model provided that relates the deformation mechanisms of the interface to its properties.

In the present paper, the response of interfaces under dynamic and quasistatic loading is modeled by developing a dynamic response constitutive model. We have used a combination of Raman spectroscopy and nanoindentation experiments to analyze the deformation of glass/epoxy interfaces at different strain rates. The interface stresses were measured by developing an analytical solution based on the work of Boussinesq, Sneddon, and Flamant [40–42] to provide the in-plane stress-strain components. The combination of these results was used to develop a model coupling both the strain rate and confinement effect to predict interface thickness dependent constitutive behavior.

## 2. Materials and method

During the nanoscale impact experiments, impact points were precisely located at the interfaces using an objective with a resolution of one micrometer. The impactor tip impacted analyzed interfaces in precisely predetermined locations confirming that the impact energy was imparted only in the interfacial region. Strain rate during impact was

calculated to correlate the damage during impact with impact energy and interface damage in both quasistatic and dynamic loading. The interface deformation behavior was modeled using a modification of the Johnson-Cook multiaxial plasticity model which couples the effect of both strain-rate and effect of confinement.

### 2.1. Material interface sample preparation for testing

Single interface samples of glass and epoxy were prepared with an epoxy interface sandwiched between two glass plates. The samples were prepared using two part industrial epoxy procured from Composite Polymer Design (South St. Paul, MN, USA). The resin, CPD4505A, and hardener, CPD 4507B, were thoroughly mixed in recommended proportions of 100A:28B by weight. The epoxy layer thickness was controlled by putting tabs of appropriate thickness in between the glass plates. The interface thickness was kept at 10  $\mu\text{m}$ , achieved by placing the epoxy between glass plates with 10  $\mu\text{m}$  tabs on the sides. The samples were cured at a prescribed temperature of 250 °F for one day. The thickness of the interface was measured with a microscope to make sure that it was in the error margin of  $10 \pm 0.5 \mu\text{m}$ . The sample surfaces as shown in Fig. 1 were polished to remove scratches that could interfere with the data measurement during experiments.

### 2.2. Experimental procedure

#### 2.2.1. Quasistatic indentation

Nanoindentation method was used to perform elastic-plastic property measurements at nano and micro-scales in the current experiments. The experiments are performed by indenting the interfaces at predefined peak load ( $P_{\text{max}}$ ) by stepwise increase in the load or peak depth ( $h_{\text{max}}$ ) by increasing the depth in small steps. A spherical indenter was used for performing the indentations and the unloading portion of the curve was used for calculating the mechanical properties from contact mechanics framework. The maximum indentation load  $P_{\text{max}}$  and the corresponding area of indentation  $A$  was measured during experiments. The analyses procedures are according to Oliver and Pharr method [43]. More details on these procedures can be found in the author's earlier articles [44,45]. The load-displacement data was further analyzed to find the stress-strain behavior. The contact depth was calculated from the indentation depth using Eq. (1)

$$h_c = h - 0.5 \left[ \frac{3P}{4E^*} \right]^{2/3} \left[ \frac{1}{R} \right]^{1/3}. \quad (1)$$

Here,  $P$  is the applied load,  $h$  is the indentation depth,  $E^*$  is the modulus of material from the experiments, and  $R$  is the radius of the indenter.

The contact radius  $a_c$  was measured by Eq. (2) given as

$$a_c = \sqrt{2h_c R - h_c^2}. \quad (2)$$

The indentation stress and strain were then defined by Eq. (3) as

$$\sigma_{\text{ind}} = \frac{P}{\pi a_c^2}, \quad \epsilon_{\text{ind}} = \frac{4h_e}{3\pi a_c}. \quad (3)$$

Here,  $h_e = h - h_c$  is the elastic depth of indentation. These stresses and strains were also corrected for the zero load and zero displacement. Reader can find more details on the procedures in the article by Kalidindi [46]. The experimental set up is a multi-module mechanical testing instrument from NanoTest, Micro Materials Ltd., platform 2, as shown in Fig. 2. The experimental set up consists of a 3D stage that allows it to move in x, y and z directions with nanometer precision. The instrument has a vertical pendulum with an indenter. The pendulum hangs on frictionless springs which indents during experiments in the

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