Conference Proceedings of the Society for Experimental Mechanics Series

Jay Carroll · Shuman Xia · Alison M. Beese
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Fracture, Fatigue, Failure and Damage Evolution, Volume 7

Proceedings of the 2017 Annual Conference on Experimental and Applied Mechanics

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ISSN 2191-5644 ISSN 2191-5652 (electronic)
Conference Proceedings of the Society for Experimental Mechanics Series
ISBN 978-3-319-62830-1 ISBN 978-3-319-62831-8 (eBook)
DOI 10.1007/978-3-319-62831-8

Library of Congress Control Number: 2015951232

Printed on acid-free paper

This Springer imprint is published by Springer Nature
The registered company is Springer International Publishing AG
The registered company address is: Gewerbestrasse 11, 6330 Cham, Switzerland

Preface

Fracture, Fatigue, Failure and Damage Evolution represents one of nine volumes of technical papers presented at the 2017 SEM Annual Conference and Exposition on Experimental and Applied Mechanics organized by the Society for Experimental Mechanics and held in Indianapolis, IN, June 12–15, 2017. The complete proceedings also includes volumes on *Dynamic Behavior of Materials; Challenges in Mechanics of Time-Dependent Materials; Advancement of Optical Methods in Experimental Mechanics; Mechanics of Biological Systems, Materials and Other Topics in Experimental and Applied Mechanics; Micro- and Nanomechanics; Mechanics of Composite, Hybrid and Multifunctional Materials; Residual Stress, Thermomechanics and Infrared Imaging, Hybrid Techniques and Inverse Problems; and Mechanics of Additive and Advanced Manufacturing.*

Each collection presents early findings from experimental and computational investigations on an important area within experimental mechanics, fracture and fatigue being one of these areas.

Fatigue and fracture are two of the most critical considerations in engineering design. Understanding and characterizing fatigue and fracture has remained as one of the primary focus areas of experimental mechanics for several decades. Advances in experimental techniques, such as digital image correlation, acoustic emissions, and electron microscopy, have allowed for deeper study of phenomena related to fatigue and fracture. This volume contains the results of investigations of several aspects of fatigue and fracture such as microstructural effects, the behavior of interfaces, the behavior of different and/or complex materials such as composites, and environmental and loading effects. The collection of experimental mechanics research included here represents another step toward solving the long-term challenges associated with fatigue and fracture.

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Chapter 1
Interface Mechanical Strength and Elastic Constants Calculations via Nano Impact and Nanomechanical Raman Spectroscopy

Devendra Verma and Vikas Tomar

Abstract Interfaces are ubiquitous in important natural and manmade materials. Research evidence has shown that interface chemistry, structure, and thickness together strongly influence material microstructure and mechanical properties. The focus of the present work is on presenting an experiment based theoretic advancement to predict thickness dependent elastic properties of materials interfaces by treating the interfaces and the area around them in a material as an elastic continuum. The experiments are based on the nanomechanical Raman spectroscopy (NMRS) developed by authors earlier with a capability to simultaneously measure stress components in orthogonal directions during an in-situ nanomechanical loading. An analytical model is developed based on boundary conditions of interface to predict thickness dependent interface elastic constants. The interface elastic constants are compared with the relations provided in literature.

Keywords Nanomechanical Raman spectroscopy • In-situ interface deformation • In-situ nanomechanical measurements • Interface thickness • Interface elastic constants

The first ever mention of interfaces is in the work of Gibbs [1] where he formulated thermodynamic foundations of interface excess energy. Gibbs definition of interfaces was a zero thickness mathematical entity. The focus of the present work is different from interface thermodynamics, interface chemistry, and interface structure characterization work available in literature. The interface in this study is considered to be of finite thickness with a non-zero volume. Emphasis is on deducing the influence of interfaces on mechanical deformation using a classical approach that incorporates interface multi-axial properties. The present work uses a nanomechanical Raman spectroscopy (NMRS) [2–7] based experimental framework to measure direct in-situ interface deformation properties.

In the classical work by Dingreville and Qu [8–10] the interfacial mismatch stress was related to the in-plane strain and applied stress in the case of a zero thickness interface. These formulations provide a way to calculate interfacial elastic properties based on the contribution of interfacial coherent surface stress, incoherent surface stress and transverse excess strain. The role of transverse direction properties of interfaces is accounted for mathematically. In an another formation, the interface is explicitly considered as a finite thickness entity to calculate the interface elastic constants in the case of heterogeneous thin interfaces by Ustinov et al. [11] showing the interface stiffness dependence on their thickness. The present work focuses on using NMRS based direct observations of multiaxial interface deformation to develop a theoretical framework for predicting interface elastic constants as a function of interface thickness.

Interfaces in composite materials are considered as a material phase confined between two separate grains or phases. In this experimental work, the interface elastic constants are measured in the case of an idealized epoxy interface between two glass plates. Single interface samples of glass and epoxy were prepared with an epoxy interface sandwiched between two glass plates. The thickness of the interface in samples was measured with a microscope to make sure that it was in the error margin of 10 ± 0.5 μm.

Considering different stiffness for the interface and the bulk phases, this boundary condition leads to a jump in the stresses. It has continuous strain distribution for the component in indentation direction while the stresses perpendicular to the interface must fulfill the condition of equilibrium. To solve this problem, a fictitious homogeneous isotropic elastic half space is assumed which for a given applied load through the indenter exhibits the same normal surface displacement as the material with interface. Figure 1.1 shows the schematic of solution procedure for this case. The key assumption of the analytical approach presented here is that the classical strain solution and the solution for the layered half space are similar for the applied loads. The assumptions for these components are therefore:

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J. Carroll et al. (eds.), *Fracture, Fatigue, Failure and Damage Evolution, Volume 7*, Conference Proceedings of the Society for Experimental Mechanics Series, DOI 10.1007/978-3-319-62831-8_1

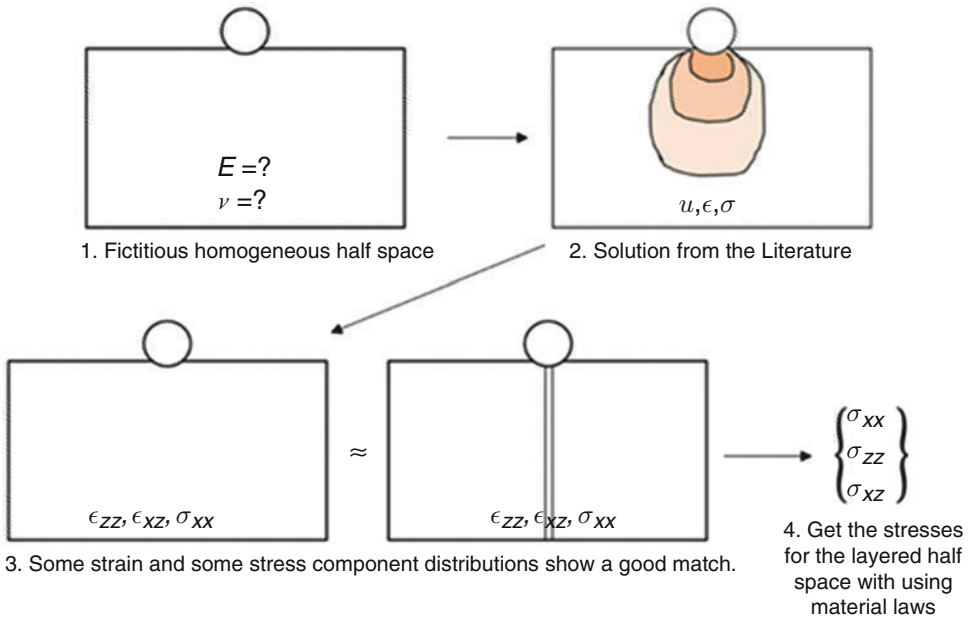

Fig. 1.1 Schematic illustration of the solution procedure

$$\begin{aligned} \sigma_{xx,fict} &\approx \sigma_{xx,lay}, \\ \sigma_{xz,fict} &\approx \sigma_{xz,lay}, \\ \sigma_{xy,fict} &\approx \sigma_{xy,lay}. \end{aligned} \tag{1.1}$$

The remaining strain components in interface plane direction, and must fulfill the conditions of compatibility and strain jumps cannot occur.

$$\begin{aligned} \epsilon_{yy,fict} &\approx \epsilon_{yy,lay}, \\ \epsilon_{yz,fict} &\approx \epsilon_{yz,lay}. \end{aligned} \tag{1.2}$$

Due to limitations of the stress measuring technique, it is not possible to obtain a full stress tensor for the interface in the current indentation setup. Nevertheless, an equivalent stress can be obtained, therefore the calculated stress tensors are transferred to an equivalent stress by:

$$\sigma_v = \sqrt{\sigma_{xx}^2 + \sigma_{yy}^2 + \sigma_{zz}^2 - \left(\sigma_{xx}\sigma_{yy} + \sigma_{yy}\sigma_{zz} + \sigma_{xx}\sigma_{zz}\right) + 3\left(\sigma_{xy}^2 + \sigma_{yz}^2 + \sigma_{xz}^2\right)}. \tag{1.3}$$

Here, σ_v is the von Mises equivalent stress. The flat punch corrections were then applied in the model. A flat ended and a spherical indenter produce different load distributions on the surface of the sample.

To validate the assumptions of the model, an interface FE model and a homogeneous isotropic half space model was simulated with 10 μm thick interface in the middle. Plane strain loading boundary conditions were applied and the loading was given in the displacement boundary condition. The strains were measured at the maximum displacement of 500 nm that was equivalent to the indentation depth in the actual experiments. The strains and stresses were compared for both models. The stresses showed the similar profile validating the stress assumptions given in Eq. (1.1).

Raman spectroscopy is an excellent tool to measure properties such as the crystalline structure, chemical signature without a necessity of sample preparation. The Raman spectroscopy has been used for other material systems such as epoxy in recent years to measure the curing state as well as the residual stresses in the sample44. We have used the Raman spectroscopy developed in our lab by Gan and Tomar to measure the stresses in the interface at different applied loads during nanomechanical loading to compare the stress distribution [3, 4]. The mechanical load was applied using the nanoindentation platform manufactured by Micro Materials Inc., UK, [12, 13] with load range from 0.1 to 500 mN, with the accuracy of 0.01 mN. The nanomechanical loading was performed using a flat punch indenter attached to the pendulum.

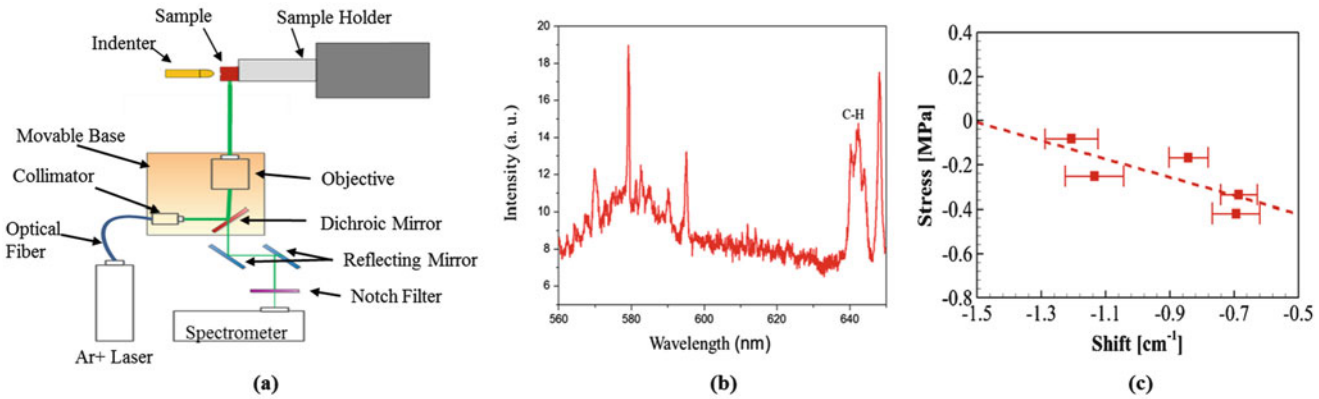

Fig. 1.2 (**a**) Setup of the nanomechanical Raman spectroscopy experiments. (**b**) Raman spectrum collected from epoxy showing the peak corresponding to C–H bond. (**c**) Raman shift versus stress calibration curve for epoxy

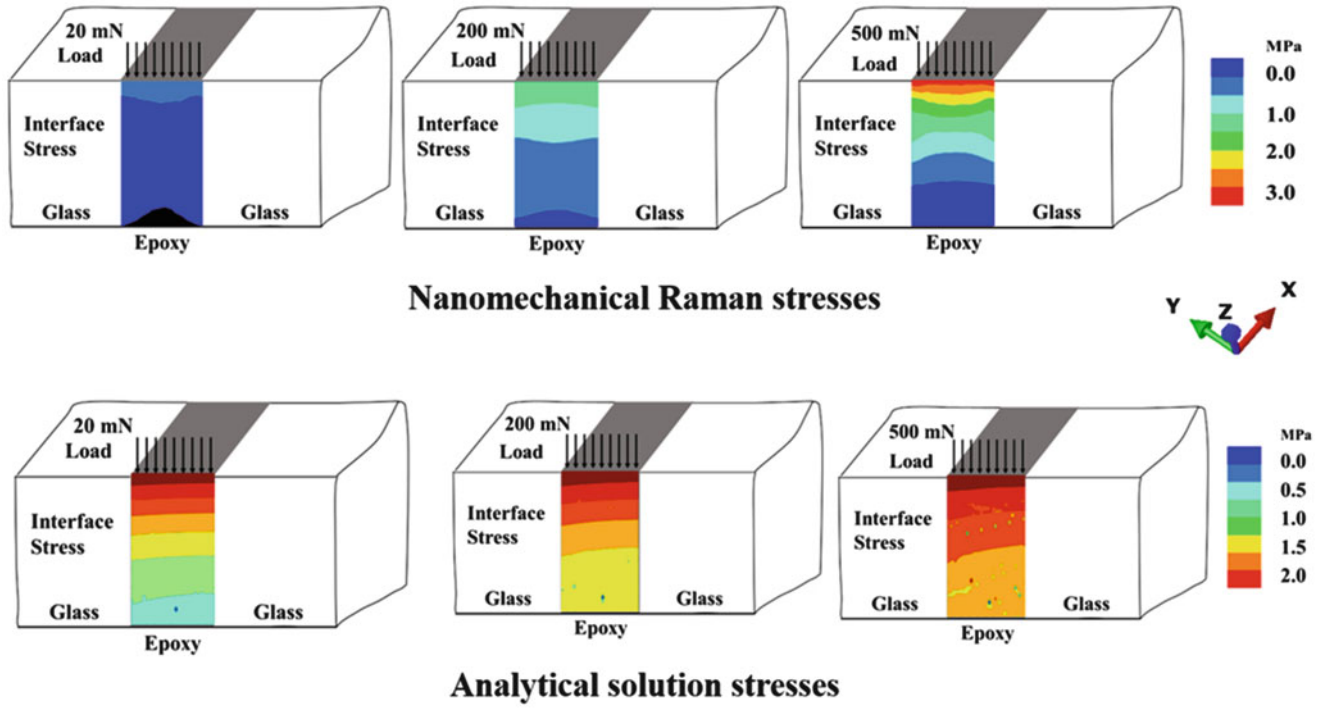

Fig. 1.3 Distribution of the equivalent stress for the indentation with a flat indenter for 20, 200, and 500 mN. The height in each map is 80 μm and width is 10 μm

The epoxy samples show Raman peaks in a wide range from 560 to 645 nm. The Raman signal at each wavelength depends on the mass of the atoms involved and the strength of the bonds between them. In the current system we measured the strongest signal around 641.1 nm as shown in Fig. 1.2b. The change in shift was obtained by subtracting the shift at the applied load for the shift at zero load. The calibration curve for shift versus load for epoxy is given in Fig. 1.2d. The Raman shift versus stress calibration curve was used to calculate the stress maps on the interfaces. The Raman maps were measured at 0, 6, 60, and 150 MPa. The measurements were performed while holding the load constant. The stress distribution across interfaces is shown in Fig. 1.3.

The Raman spectroscopy only provides the average stress at the interface but stress tensors in different directions are needed to fully understand the behavior of interfaces. Even in the experiments, it is difficult to measure the lateral stresses. An analytical solution is therefore developed to calculate the lateral stresses during indentation of interfaces. In the present analyses, the indentations are quasistatic and fully elastic which gives small indentation depths compared to the indenter

Table 1.1 Elastic constants *for glass/epoxy* interface

	A11 (Pa-m)	A22 (Pa-m)	A33 (Pa-m)	A13 (Pa)
This work	34,029	6821	2.03×10^{14}	2.80×10^{9}
Theoretical approximations [11]	53,571	53,571	5.35×10^{14}	3,571,428,571

radius. This condition allows for the simplifications of the loaded region. An analytical method to calculate the stresses in interfaces with vanishing thickness is presented by Ustinov et al. [11].

In this work, we replace the intermediate layer in the internal energy equation given in [11] by a layer of thickness *h,* which is less than the thickness of two original layers. A new two-layered system with additional interface elastic constants is obtained with its elastic energy as a function of the thickness of the intermediate layer. While the longitudinal strains stay the same as for the initial system, a new relation for the transverse strains are presented, by claiming that the surface displacements should coincide with the initial system. By substituting equation of elastic energy of the modified system expressed in terms of the strains of the initial system and solving the resulting equation system which results by equating after performing the limit transition the elastic properties of the interface with vanishing thickness can be obtained [14].

The abovementioned model was programmed in a MATLAB code to calculate the interface stresses for different scenarios, [14]. The stresses were then calculated for the quasistatic case for the same applied load as in the experiments. The values from the Nanomechanical Raman spectroscopy measurement and the analytical solution are of the same order showing the validity of the model to measure the stress components in the given case, Fig. 1.3. The stress and strains were then calculated in all direction using the analytical model to calculate the interface elastic constants.

The elastic constants were then calculated from these stress-strain data using the linear fit. These formulas were further used to calculate the interface elastic constants of the epoxy interfaces analyzed in the current study. The interface elastic constants for epoxy interface measured from the indentation experiments after conversion to surface constant is listed in the same Table 1.1 with the values obtained from theoretical approximation for comparison.

A new formulation based on the NMRS is presented to calculate the interface elastic constants using an analytical model. The measured and calculated elastic constants are compared with the strain energy frameworks provided in the literature. A comparison between the current and literature methods shows the dependence of the interface elastic constants on the interface thickness. The elastic constants calculated from the stress-strain data match the literature values after the thickness effect correction.

References

1. Gibbs, J.W.: The Collected Works of J. Willard Gibbs, Volume I: Thermodynamics. Yale University Press, New Haven (1928)
2. Gan, M., Samvedi, V., Tomar, V.: Raman spectroscopy-based investigation of thermal conductivity of stressed silicon Microcantilevers. J. Thermophys. Heat Transf. **29**(4), 845–857 (2014)
3. Gan, M., Tomar, V.: An in situ platform for the investigation of Raman shift in micro-scale silicon structures as a function of mechanical stress and temperature increase. Rev. Sci. Instrum. **85**(1), 013902 (2014)
4. Gan, M., Tomar, V.: Surface stress variation as a function of applied compressive stress and temperature in microscale silicon. J. Appl. Phys. **116**(7), 073502 (2014)
5. Zhang, Y., Gan, M., Tomar, V.: In-Situ Combined Sensing of Uniaxial Nanomechanical and Micromechanical Stress with Simultaneous Measurement of Surface Temperature Profiles by Raman Shift in Nanoscale and Microscale Structures. Purdue Research Foundation, West Lafayette (2016)
6. Zhang, Y., Gan, M., Tomar, V.: Small scale thermomechanics in Si with an account of surface stress measurements. In: Ralph, C., et al. (eds.) Mechanics of Composite and Multi-Functional Materials, Volume 7: Proceedings of the 2015 Annual Conference on Experimental and Applied Mechanics, pp. 247–250. Springer International Publishing, Cham (2016)
7. Zhang, Y., Gan, M., Tomar, V.: Raman thermometry based thermal conductivity measurement of bovine cortical bone as a function of compressive stress. J. Nanotechnol. Eng. Med. **5**(2), 021003 (2014)
8. Dingreville, R., Hallil, A., Berbenni, S.: From coherent to incoherent mismatched interfaces: a generalized continuum formulation of surface stresses. J. Mech. Phys. Solids. **72**(0), 40–60 (2014)
9. Dingreville, R., Qu, J.: Interfacial excess energy, excess stress and excess strain in elastic solids: planar interfaces. J. Mech. Phys. Solids. **56**(5), 1944–1954 (2008)
10. Dingreville, R., Qu, J., Mohammed, C.: Surface free energy and its effect on the elastic behavior of nano-sized particles, wires and films. J. Mech. Phys. Solids. **53**(8), 1827–1854 (2005)
11. Ustinov, K.B., Goldstein, R.V., Gorodtsov, V.A.: On the modeling of surface and interface elastic effects in case of eigenstrains. In: Altenbach, H., Morozov, F.N. (eds.) Surface Effects in Solid Mechanics: Models, Simulations and Applications, pp. 167–180. Springer Berlin Heidelberg, Berlin (2013)

12. Verma, D., Tomar, V.: Strain rate dependent failure of metallic interfaces at nano- microscale via nanoimpact experiments. In: 20th International Conference on Composite Materials, Copenhagen (2015)
13. Verma, D., Tomar, V.: An investigation into environment dependent nanomechanical properties of shallow water shrimp (Pandalus platyceros) exoskeleton. Mater. Sci. Eng. C Mater. Biol. Appl. **44**, 371–379 (2014)
14. Verma, D., et al.: A Combined Theoretic and Experimental Advancement to Measure Interface Stress-Strain Relations and Interface Elastic Constants using Nanomechanical Raman Spectroscopy. Manuscript in preparation (2017)

Chapter 2
Effect of Strain Rate and Interface Chemistry on Failure in Energetic Materials

Chandra Prakash, I. Emre Gunduz, and Vikas Tomar

Abstract We study the failure at interfaces between Hydroxyl-terminated polybutadiene (HTPB)-Ammonium Perchlorate (AP) based energetic material. In this work, interface mechanical strength of a set of HTPB-AP interfaces is characterized using nano-scale impact experiments at strain rates up to 100 s^{-1}. A power law viscoplastic constitutive model was fitted to experimental stress-strain-strain rate data in order to obtain constitutive behavior of interfaces, particle, and matrix. A mechanical Raman spectroscopy is used to analyze the effect of binding agent at different temperature. A tensile fracture experiment combined with In-situ Mechanical Raman Spectroscopy was used to obtain fracture properties. Stress maps are obtained near the interface using In-situ Mechanical Raman Spectroscopy to analyze the changes in the stress distribution around interfaces for different loads till failure. Cohesive zone model parameters were obtained from the consideration of local stress during failure and the cohesive energy required for delamination of AP from HTPB matrix. Effect of binding agent on the interface strength is found to be quite significant. The cohesive zone parameters and the viscoplastic model obtained from the experiment were then used in the cohesive finite element method to simulate the dynamic crack propagation as well as the delamination. Results show that interfacial properties are affected by the rate of loading and are also dependent upon the binding agent.

Keywords Energetic material • Stress/strain relationship • HTPB • AP • NRS

Energetic compounds are employed in a large number of applications, such as, explosive, propellant, and pyrotechnic formulations. An example of a heterogeneous solid propellant used in rocket industry is a crystalline oxidizer (e.g., ammonium perchlorate-AP) embedded in a polymeric binder (e.g., Hydroxyl-Terminated Polybutadiene or HTPB). Aluminum (Al) particles are sometimes added to enhance the propellant performance. A typical industrial solid propellant consists of 70% AP, 10% HTPB and around 20% Al by weight, [1]. Three main failure mechanisms in the composite material are identified as particle fracture, interfacial failure and the cavitation in the binder [2]. Palmer [2] investigated a number of polymer bonded explosives (PBXs) under tensile loading and observed finding a wide range of responses. They found that the crack propagation was mostly confined to binder and that the interface debonding was the dominant failure mode. Interface strength depends on the constituent material, i.e., particle, matrix and/or binding agents [3, 4].

Several experiments [5–7] have suggested a particle size effect on the performance of energetic materials. Yeager [8] has shown the effect of interface/interphase and the microstructure on the mechanical behavior of PBXs. Interfacial structure was altered by adding a plasticizer in the composite. The plasticizer was shown to inhibit the formation of a large interface/interphase and was more likely to have film delamination than the no-plasticized composite. The difference in interfacial properties was also shown to have significant effect on the crack initiation and explosive sensitivity.

Two samples were prepared for analyzing the effect of functionalization on the interface mechanical properties. One consist of ammonium perchlorate (AP) particles embedded in hydroxyl-terminated polybutadiene (HTPB). In the second sample, a surface binding agent (Tepanol) was added at a mass ratio of 0.5 to fabricate samples with higher surface adhesion, while keeping the same index ratio.

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J. Carroll et al. (eds.), *Fracture, Fatigue, Failure and Damage Evolution, Volume 7*, Conference Proceedings of the Society for Experimental Mechanics Series, DOI 10.1007/978-3-319-62831-8_2