

REPORT DOCUMENTATION PAGE

Form Approved
OMB NO. 0704-0188

Public Reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comment regarding this burden estimates or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188,) Washington, DC 20503.

1. AGENCY USE ONLY (Leave Blank)		2. REPORT DATE 10 Dec 2004	3. REPORT TYPE AND DATES COVERED Final; 1 May 2001-31 Dec 2004	
4. TITLE AND SUBTITLE Fracture Toughness Improvement of Composites Reinforced with Optimally Shaped Short Ductile Fibers			5. FUNDING NUMBERS DAAD19-01-1-0529	
6. AUTHOR(S) Robert C. Wetherhold, Abani K. Patra				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Research Foundation of State University of New York, State University Plaza, Broadway and State Street, Albany, NY 12246-0001			8. PERFORMING ORGANIZATION REPORT NUMBER N/A	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) U. S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211			10. SPONSORING / MONITORING AGENCY REPORT NUMBER 42188.1-MS	
11. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.				
12 a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited.			12 b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) The fracture toughness of brittle matrix composites reinforced with ductile fibers has been greatly improved by shaping the fibers so that they fully contribute their plastic work to the fracture process. This has been accomplished by anchoring the fiber ends so that the largest possible fiber volume contributes to the toughness during pullout. A combined experimental and analytical program has shown that it is possible to organize fibers into shape families and to optimize the toughness with respect to geometric parameters within a given shape family. The interface conditions during pullout have been modeled using a new cohesive zone/Coulomb friction model with three parameters. Furthermore, adaptive methods in space and time were developed to reduce computational time. New indicators for error in spatial and temporal discretizations were derived and implemented. Once these parameters were inferred from one embedded length from the single fiber pullout test (SFP), predicted pullout toughness values at other embedded depths matched well with experimental results. In addition, fracture values predicted from SFP tests matched well with experimental composite fracture values.				
14. SUBJECT TERMS composite toughness, shaped fibers, ductile fibers, adaptive finite elements, cohesive zone model			15. NUMBER OF PAGES	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OR REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION ON THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL	

FINAL PROGRESS REPORT

Fracture Toughness Improvement of Composites Reinforced with Optimally Shaped
Short Ductile Fibers
DAAD19-01-1-0529

Statement of the Problem Studied

Short fiber reinforced composites have a number of advantages over continuous fiber composites, including low-cost manufacturing methods and flexibility of properties based on controlling the flow of materials. Conventionally shaped short (CSS) fibers are usually round in cross-section (other shapes with higher surface area/volume are possible) and prismatic—i.e. their cross-section remains constant along their length. The fibers offer stiffness and strength to the matrix through complimentary, but in some regimes competing, mechanisms. In general, the *better the bond* between fiber and matrix, the more efficient the load transfer to the fiber and the *stiffer the composite*. (If there is a debond or a poor bond, a high interfacial shear stress is desirable for high stiffness.)

The mechanism of strength and toughness increase provided by the fibers is somewhat more complicated. In general, fibers can bridge the fracture surface, providing a decrease in the net stress intensity factor/increase in energy dissipation through bridging, often with fiber pullout, fiber fracture or plasticity, etc. The role of fiber strength is direct: the higher the strength, the better. It is the interfacial properties that play a more involved role for bridging fibers. The interface strength or shear stress should be as high as possible (more energy dissipation via pullout) *right up to the point where brittle fibers start breaking*. This is the trade-off that limits the improvement in properties possible in CSS fiber composites. The fundamental question to be addressed, then, is if an adjustment in fiber shape and surface treatment would permit this inherent trade-off to be removed. This requires an exploration of the fiber-shape and surface design space using a combination of experiments (see “Task 1”) and modeling (see “Task 2”). The inspiration for this solution comes from biomimetics, as exemplified by bamboo fibrils, which have a dog bone shape and achieve good anchoring and excellent pullout behavior of the fibers.

Summary of Most Important Results

This research is focused on improving the fracture and impact toughness of brittle thermoset matrix-copper fiber composites, without decreasing their strength or stiffness. This is accomplished by using short shaped ductile fibers which utilize the plastic work potential of the fiber volume by anchoring the fibers into the matrix and by controlling the friction at the interface.

Task 1: The goal was to identify how the bonding and fiber pullout behavior in selected fiber-matrix combinations affects the composite fracture toughness increment and from this identify what constitutes “desirable” behavior. The matrices tested were a brittle low shrinkage (LS) epoxy, a more ductile high shrinkage (HS) epoxy, and polyester. The fiber morphologies tested were: straight, flat end-impacted, end-oxidized, rippled, and acid roughened. Single fiber pullout (SFP) experiments were conducted to determine the adhesion quality, debonding behavior, and subsequent matrix fracture behavior for numerous fiber-matrix combinations. The pullout experiments were used with a theoretical model that predicts the fiber contribution to the fracture toughness increment “ ΔG ” and the fiber shapes that perform better than a straight fiber were determined from these values. Based on the SFP results, fiber shapes that perform better than straight fibers were placed into HS Epoxy and tested in four-point bending to determine the accuracy of the predicted fracture toughness increment equation. Selected shaped fibers were then placed into Izod impact specimens to determine the effect the selected fiber morphologies have on the composite impact toughness. Lastly, EMI shielding effectiveness tests were performed to determine the EMI shielding capability of selected fiber morphologies in a composite. Highlights of the research were:

- Four-point bending test results agreed well with the theoretical predictions of the fracture toughness increment ‘ ΔG ’ of a short metal fiber-brittle thermoset matrix composite based on SFP tests [6]. This close agreement demonstrates that SFP testing, along with the theoretical model, can be used as an effective fiber shape screening tool for ductile fibers before full scale composite testing. Further, four-point bending results illustrate that shaped copper fibers improve both the flexural

- strength and modulus of the composite, indicating that shaped ductile fibers provide improved stress transfer by anchoring the fibers into the matrix.
- Results indicated that the most important factors for determining toughness are the matrix fracture behavior and the interface bond behavior [1, 3, 4]. These behaviors determine the fiber debonding and pullout behavior, the fiber shapes which give increased toughness, and the length where fibers fail rather than pull out of the matrix. When the matrix fails around the fiber, the toughness is significantly less compared to matrices with the same fiber that have minor matrix fracture during SFP testing, four point-bending testing, or impact testing.
 - The fiber shape is also very important because when a fiber debonds from the matrix, it must be able to pull out to improve toughness. Because of the high effectiveness of shaped fibers, composites with such fibers, many of which fail during fracture and impact, can still perform better than composites with straight fibers, all of which pull out during fracture or impact.
 - Lastly, results indicate that besides improving the fracture and impact toughness, short shaped copper fibers also improve the EMI shielding effectiveness of the composite through increased internal reflections and improved conductivity [submitted manuscript 1; also M.S. thesis “EMI Shielding Improvements using short Shaped Copper fibers in a Thermoset Matrix”, J.M. McManaman, University at Buffalo, SUNY 8/2004]. In conclusion, results from SFP, four-point bending, and impact tests indicate that the greatest improvement in toughness occurs with a fiber-matrix combination that has moderate adhesive bond strength with an adhesive bond failure, moderate post debonding friction, and minor matrix damage during fiber pull out.

Task 2:

The quasi-static analysis of elastoplastic fiber pullout process is simulated by the *hp*-adaptive finite element method based on an updated Lagrangian formulation. The highlights of the research were:

- To approximate the nonlinear behavior of the fiber-matrix interface, we propose a new mixed cohesion-friction model [5]. The use of such a model enables us to simulate the entire pullout process.
- A combination of von Mises yield criterion and associated flow rule is utilized as the constitutive model to describe the plasticity in the initial annealed copper fiber.
- Through comparison with the experimental results from straight fiber pullout experiments, key parameters for describing the interface model are determined and the model is validated.
- The model is then applied to simulate the pullout of the “nail-head” shaped fiber family. Simulation results are used to design an optimal shape of the head to maximize the pullout work [2, 5].
- An explicit *a posteriori* estimator based on the patch recovery technique and element residuals method gradient is applied to measure and enhance the quality of finite element computations of nonlinear material and interface structure. Then, we propose an optimal *hp*-adaptive mesh that limits the iterations and minimize the computation cost.
- Finally, we develop a scheme to automatically adjust the load step based on an indicator of error in the constitutive modeling. This greatly reduces the iterations required to reach convergence.

Publication and Technical Reports

A) Papers in peer-reviewed journals

1. R.M. Bagwell, R.C. Wetherhold, “Improvement in Fracture Toughness of an Epoxy/Copper Composite through the use of Various End-Shaped Fibers,” *Mat Sci Eng A* 361 (2003) p294- 301.

2. J-H Tsai, A.K. Patra, R.C. Wetherhold, "Numerical Simulations of Fracture-Toughness Improvement Using Short Shaped-Head Ductile Fibers," *Compos A*, 34 (2003) p.1255-1264. .
3. R.M. Bagwell, R.C. Wetherhold, "Debond Behavior of Copper Fibers in Thermoset Matrices and their Effect on Fracture Toughening," *J Adhesion Sci Techn*, 17 (2003) p. 2223-2242.
4. R.M. Bagwell, R.C. Wetherhold, "Fiber Pullout Behavior and Impact Toughness of Short End-Modified Copper Fibers in Thermoset Matrices," *J Compos A*, in press.
5. J. Tsai, A. Patra, R. Wetherhold, "Finite Element Simulation of Pullout of Shaped Ductile Fibers using a Cohesive Zone Type Model," *Compos A*, in press.
6. R.M. Bagwell, R.C. Wetherhold, "End-Shaped Copper fibers in an Epoxy Matrix—Predicted versus Actual Fracture Toughening," *Theo Appl Fract Mech*, in press.

B) Papers in non peer-reviewed journals or conference proceedings

1. R.M Bagley, R.C. Wetherhold, "Volume Optimization for a Given End-Shape Family: Using Short End-Shaped Ductile Fibers to Improve the Fracture Toughness of a Brittle Composite", *Intl Conf Compos Matls (ICCM) 14*, San Diego, CA (July 2003); oral presentation & technical paper SME TP03PUB395.
2. R. Wetherhold, R. Bagwell, "Improving Fracture Toughness of Brittle Matrix Composites using End-shaped Ductile Fibers: the Effects of Adhesion and Matrix Shrinkage," *ASME IMECE*, Washington, DC (Nov 2003); oral presentation & technical paper IMECE2003-42967.
3. R.C. Wetherhold, R.M. Bagwell, "Short Shaped Copper Fibers in an Epoxy Matrix: Their Multifunctional Use—Fracture and Impact Toughening and EMI Shielding," *ASME IMECE*, Anaheim, CA (Nov 2004); oral presentation & technical paper IMECE2004-60405.

C) Papers presented but not published

1. R.C. Wetherhold, F.K.Lee, "Shaped fibers to Improve Toughness of Composite

- Materials,” Finno-Ugric Int Conf of Mechanics, Budapest (May 2001).
2. R.C. Wetherhold, R. Bagwell, “Shaping Fiber Ends to Improve Fracture Toughness of Brittle Matrix Composites,” at Institut für Verbundwerkstoffe, Kaiserslautern, Germany (June, 2002).
 3. R.C. Wetherhold, R. Bagwell, "Improved Fracture Toughness of Brittle Composites Through the use of Shaped Ductile Fibers", ASME IMECE, New Orleans, LA (Nov 20, 2002).
 4. J. Tsai, R. Bagwell, A. Patra, R.C. Wetherhold, “Fracture Toughness Improvement Using Shaped Short Ductile Fibers,” Fourteenth U.S. National Congress of Theoretical and Applied Mechanics, Blacksburg, VA (June 23-28, 2002).
 5. R.C. Wetherhold, R Bagwell, “Fiber Pullout Behavior of Ductile Fibers in Various Brittle Matrices and their Effects on Fracture Toughening”, **invited lecture**, 1st International Workshop on Polymers and Composites at IVW Kaiserslautern, Germany (May 2003).
 6. J. Tsai, A. Patra and R. Wetherhold, Simulation of Ductile Fiber Pullout Based on a Mixed Cohesion and Friction Model, VII US National Congress on Computational Mechanics, Albuquerque, NM, (July 28-31, 2003).
 7. R.C. Wetherhold, R Bagwell, “End-modified Copper Fibers and Their Role in a Multifunctional Brittle Matrix Composite: Fracture & Impact Toughening and EMI Shielding, **plenary (invited) talk** at Iberomet (VIII Iberoamerican Congress in Metallurgy and Materials), Quito, Ecuador (May 2004).

D) Manuscripts submitted but not published

1. R.C. Wetherhold, J.M. McManaman, “EMI Shielding Effectiveness of Cu/Epoxy Composites,” Polym Polym Compos, in review.
2. R.M. Bagwell, R.C. Wetherhold, “Short Shaped Copper Fibers in an Epoxy Matrix: Their Role in a Multifunctional Composite,” Compos Sci Tech, in review.
3. J. Tsai, A. Patra, “Finite Element Simulation Of Shaped Ductile Fiber Pullout Using A Mixed Cohesive Zone/Friction Interface Model Adaptive Space and Time Discretizations

of Updated Lagrangian Formulations – Application to Shaped Elastoplastic Fiber Pullout Using a Hybrid Interface Model”, in review Int. J. Num. Meth. Eng.

E) Technical reports submitted to ARO—N/A

List of personnel

Robert C. Wetherhold, PI

Abani K. Patra, Co-PI

Renee Bagwell, M.S and Ph.D. earned.

Jeng-Hsiu Tsai, Ph.D. earned.

Joseph. M. McManaman, M.S. earned.

Jennifer Parks, M.S. earned (degree conferral Feb 2005).

Report of Inventions N/A

Bibliography

1. R.M. Bagwell, R.C. Wetherhold, “Improvement in Fracture Toughness of an Epoxy/Copper Composite through the use of Various End-Shaped Fibers,” Mat Sci Eng A 361 (2003) p294- 301.
2. J-H Tsai, A.K. Patra, R.C. Wetherhold, “Numerical Simulations of Fracture-Toughness Improvement Using Short Shaped-Head Ductile Fibers,” Compos A, 34 (2003) p.1255-1264.
3. R.M. Bagwell, R.C. Wetherhold, “Debond Behavior of Copper Fibers in Thermoset Matrices and their Effect on Fracture Toughening,” J Adhesion Sci Techn, 17 (2003) p. 2223-2242.
4. R.M. Bagwell, R.C. Wetherhold, “Fiber Pullout Behavior and Impact Toughness of Short End-Modified Copper Fibers in Thermoset Matrices,” J Compos A, in press.
5. J. Tsai, A. Patra, R. Wetherhold, “Finite Element Simulation of Pullout of Shaped Ductile Fibers using a Cohesive Zone Type Model,” Compos A, in press.

6. R.M. Bagwell, R.C. Wetherhold, “End-Shaped Copper fibers in an Epoxy Matrix— Predicted versus Actual Fracture Toughening,” *Theo Appl Fract Mech*, in press.

Appendices N/A