

ARMY RESEARCH LABORATORY



**Resin/Dye Infusion Technique of Composite
Integral Armor Targets for Post-Impact Study**

**by Paul Moy, James Pritts, James Harris, Daniel Deschepper,
and Chad Ulven**



ARL-TN-204

July 2003

Approved for public release; distribution is unlimited.

20030806 113

NOTICES

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Citation of manufacturer's or trade names does not constitute an official endorsement or approval of the use thereof.

Destroy this report when it is no longer needed. Do not return it to the originator.

Army Research Laboratory

Aberdeen Proving Ground, MD 21005-5069

ARL-TN-204

July 2003

Resin/Dye Infusion Technique of Composite Integral Armor Targets for Post-Impact Study

**Paul Moy, James Pritts, James Harris,
Daniel Deschepper, and Chad Ulven
Weapons and Materials Research Directorate, ARL**

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 information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.					
1. REPORT DATE (DD-MM-YYYY) July 2003		2. REPORT TYPE Final		3. DATES COVERED (From - To) June 2002–September 2002	
4. TITLE AND SUBTITLE Resin/Dye Infusion Technique of Composite Integral Armor Targets for Post-Impact Study			5a. CONTRACT NUMBER		
			5b. GRANT NUMBER		
			5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S) Paul Moy, James Pritts, James Harris, Daniel Deschepper, and Chad Ulven			5d. PROJECT NUMBER AH84		
			5e. TASK NUMBER		
			5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Research Laboratory ATTN: AMSRL-WM-MA Aberdeen Proving Ground, MD 21005-5069			8. PERFORMING ORGANIZATION REPORT NUMBER ARL-TN-204		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)			10. SPONSOR/MONITOR'S ACRONYM(S)		
			11. SPONSOR/MONITOR'S REPORT NUMBER(S)		
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT A technique has been developed to study the interior fracture surfaces on alumina oxide ceramic tiles as well as the delamination zones of S-2 Glass laminates within ballistic-impacted composite integral armor (CIA) targets. The process is an alternative to other inspection methods used such as ultrasonic (C-scan) or x-ray (CAT-scan) for post-analysis of these impacted armor structures. A low viscosity resin mixed with a fluorescing dye was injected into the targets by means of the vacuum assisted resin transfer molding (VARTM) method. The fractured surfaces of the failed ceramic tiles and cavities created by the composite delamination and tile debonding were filled with the resin/dye mixture. The resin/dye mixture, which is trapped in between the layers of composites and damaged tiles, fluoresces naturally when exposed to a "black light" or ultraviolet light source, as a result highlights the resin against the target components. Digital images of the cross-sections are compared under normal and black-lighting conditions.					
15. SUBJECT TERMS composite, armor, CIA, infusion, VARTM, fluorescing dye					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UL	18. NUMBER OF PAGES 21	19a. NAME OF RESPONSIBLE PERSON Paul Moy
a. REPORT UNCLASSIFIED	b. ABSTRACT UNCLASSIFIED	c. THIS PAGE UNCLASSIFIED			19b. TELEPHONE NUMBER (Include area code) 410-306-0604

Contents

List of Figures	iv
List of Tables	iv
Acknowledgments	v
1. Introduction	1
2. CIA Target Material	2
3. Experimental Procedure	2
3.1 Target Preparation	2
3.1.1 Creating Resin Flow Channels	2
3.1.2 Isolating Sides	3
3.1.3 Bagging the Target Top	4
3.1.4 Bagging the Target Bottom (If Necessary)	5
3.2 Target Infusion	5
3.2.1 Vacuum Assisted Resin Transfer Molding (VARTM) Preparation	5
3.2.2 Resin/Fluorescing Dye	7
4. Results and Discussion	7
5. Conclusion	10
6. References	11
Appendix. Key Materials List	13

List of Figures

Figure 1. Schematic of a cross-section for CIA panels.....	2
Figure 2. Drill pattern on the back surface of a ballistic-impacted CIA target.....	3
Figure 3. (a) Corner of target with an opened weave and (b) epoxy-sealed corner of target's sides.....	4
Figure 4. Top view of bagged target.....	5
Figure 5. Sealed backside of target.....	5
Figure 6. Vacuum distribution ports (a) top view (b) bottom view.....	6
Figure 7. Target ready for resin transfer.....	7
Figure 8. Top view of infused target.....	8
Figure 9. Bottom view of infused target.....	8
Figure 10. Cross-sectional view of target without EPDM elastomer layer under (a) normal and (b) UVA lighting conditions.....	9
Figure 11. Cross-sectional view of target with EPDM elastomer layer under (a) normal and (b) UVA lighting conditions.....	9
Figure 12. Close-up of Al ₂ O ₃ tile under (a) normal lighting condition and (b) black light exposure.....	10

List of Tables

Table 1. Fluorescein data.....	1
Table 2. Typical Properties for Vantico Resinfection 8601R/8602H.....	2

Acknowledgments

The authors wish to extend their gratitude to Dr. Travis Bogetti for providing references and technical input on the subject matter, Dr. Matthew Bratcher for his expertise on the selection and availability of the fluorescing dyes used in this project, and to Mr. John Brown and Mr. James Wolbert for their assistance in obtaining supplies and facility usage.

INTENTIONALLY LEFT BLANK.

1. Introduction

Researchers at the U.S. Army Research Laboratory (ARL), Aberdeen Proving Ground, MD have developed a technique for a post-analysis study of ballistic tested composite integral armor (CIA) targets. This technique is similar to that of Gama et. al. (1, 2) on the repair of composites. The effort by Gama et al. primarily focused on the repair of a ballistic-impacted target and used a colored resin to observe the effectiveness of its infusion procedures on the backing plate only. The concept of infusing a resin was taken one step further by introducing a fluorescing dye into the resin. This process involves the use of a fluorescing powder dye mixed with a low-viscosity resin system and later infused into the entire damaged target including the cover plate and tiles. The fluorescing dye creates very high contrast for the fractures/cracks in the Al₂O₃ ceramic tiles as well as delamination within the layers of the S-2 Glass* composites.

Several commercially available dyes were considered and fluorescein was selected based on performance, availability, and cost. The dye itself is a fine powder form of reddish-orange color. The common use of this vegetable dye is primary as a stain or marker for human tissues, particularly in the area of ophthalmology where Fluorescein is injected into the blood vessels in the arm during angiograms (3, 4). Table 1 lists specifications of this dye obtained from the Material Safety Data Sheet.

Table 1. Fluorescein data.

Fluorescein Dye	
Cas No.	2321-07-5
Molecular Formula	C ₂₀ H ₁₂ O ₅
Molecular Weight	332.081
Appearance	Orange-red crystalline powder
Physical State	Solid
Freezing/Melting Point	290 °C
Solubility	Insoluble in water
Chemical Stability	Stable under normal temperatures and pressures

Initially, vinyl ester resin was used, but its viscosity was found to be too high for this application. A lower viscosity resin with an extended gel time was necessary to obtain a successfully infused part. A second issue with the vinyl ester was that the gel time was decreased when the Fluorescein dye was added. Various vendors were contacted and Vantico's Resininfusion[†] 8601R/8602H epoxy was chosen. This resin is a two-part epoxy and hardener system with a 4:1 mixing ratio, respectively. Table 2 lists the typical physical and mechanical properties for the Resininfusion.

* S-2 Glass is a registered trademark of Owens-Corning Fiberglass Technology, Inc.

† Resininfusion is a registered trademark of Vantico, Inc.

Table 2. Typical Properties for Vantico Resininfusion 8601R/8602H.

Typical Properties		
Color		Transparent
Mixed Viscosity, cP at 77 °F (25 °C) at 125 °F (51 °C)	ASTM D-2393 (5)	175
Gel Tim, 14 fl oz, min	ASTM D-2471 (6)	70
Recommended Cure Schedule (minimum before use)		3 days at 77 °F (25 °C)
Typical Cured Properties*		
Hardness, Shore D	ASTM D-2240 (7)	82
Ultimate Flexural Strength, psi	ASTM D-790 (8)	11,013
Ultimate Tensile Strength, psi	ASTM D-638 (9)	7,871
T _g by DMA, °F (°C)	ASTM D-4065 (10)	164 (73)
Elongation, %	ASTM D-638	6

* Tested at 77 °F (25 °C), unless otherwise noted, on neat product form.

2. CIA Target Material

A CIA target is a multifunctional layered hybrid structural composite comprised of fiberglass, alumina ceramic tiles, and often a thin layer of elastomer. A thorough descriptive lay-up of the CIA and the purpose and functionality of each layer are given by Fink (11). Depending on the actual target lay-up construction, each target is ~12 × 14 in with a thickness from 1 1/2 to 2 in (Figure 1).

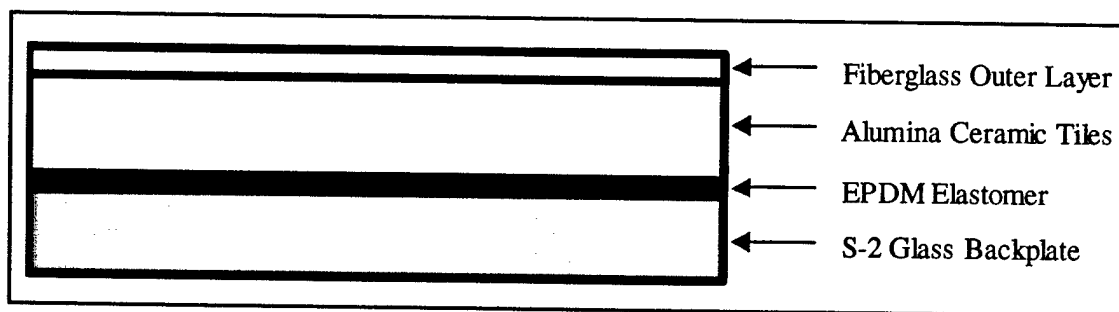


Figure 1. Schematic of a cross-section for CIA panels.

3. Experimental Procedure

3.1 Target Preparation

3.1.1 Creating Resin Flow Channels

In preparation for the infusion process, each target was cleared of loose debris of fractured ceramic tiles and damaged fiberglass from the impacted surface area. An industrial shop vacuum

with a flexible hose was effective in removing the majority of the loose debris. In some cases, these targets had a punctured opening with fiberglass protruding out of the front and back faces. A Dremel[†] hand tool with a circular saw blade was used to cut off the protruding fiberglass around the impacted zone at both the front and back (if applicable). A pattern of holes was drilled from the composite backplate of the CIA target. These holes created channels for a vacuum pump to draw the resin between the layers of the delaminated composites, during the infusion process. A 1/8-in-diameter drill bit was used. Holes were located in the proximity to the impact zone, and hole depths reached to the interior surface of the tiles.

The actual number of vacuum holes varied from 10 to as many as 48, and was determined by the extent of damage and size of the target. Panels with greater damage required fewer holes. Targets that were not completely penetrated needed additional holes drilled, as they typically had less delamination of the composite. See Figure 2 for the drill pattern for a typical ballistic-impacted target.

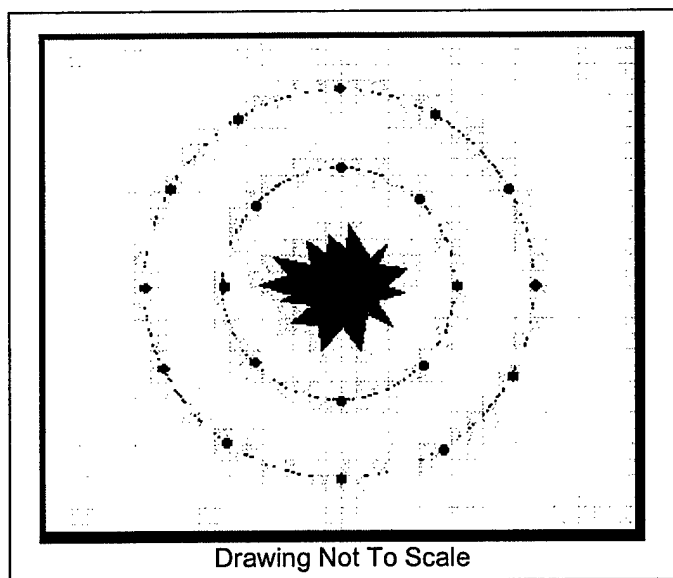


Figure 2. Drill pattern on the back surface of a ballistic-impacted CIA target.

3.1.2 Isolating Sides

After the holes were drilled, the target needed to be sealed for proper through-thickness infusion. To accomplish this, the target was first visually inspected for such defects as openings in the fiberglass composite cover layer. Coating the sides with liberal amounts of a fast-curing epoxy sealed these openings (Figure 3). This procedure was necessary to block the fluorescing resin from discharging out the sides, thus forcing the resin to flow through the thickness, resulting in a quality target infusion.

[†] Dremel is a registered trademark of the Dremel Corporation.

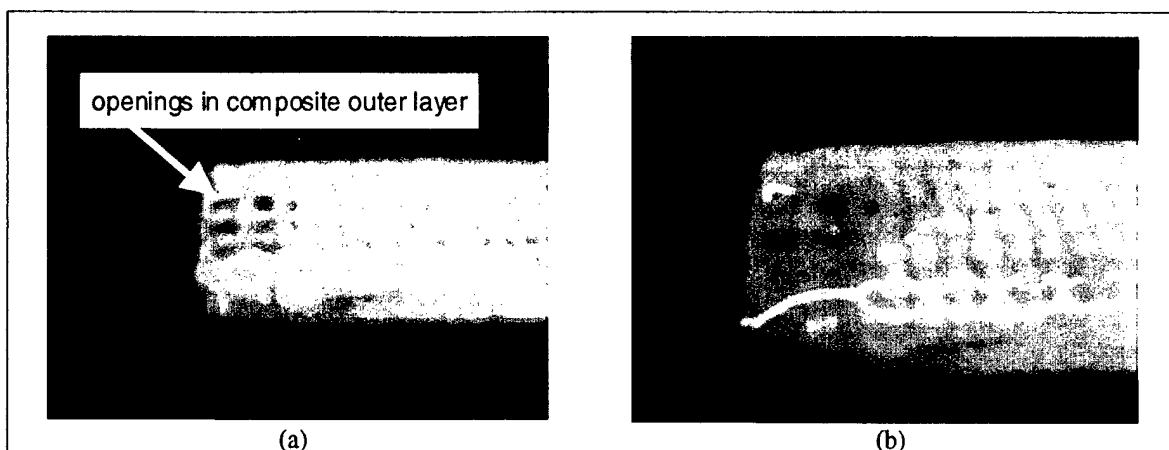


Figure 3. (a) Corner of target with an opened weave and (b) epoxy-sealed corner of target's sides.

3.1.3 Bagging the Target Top

Once the sides were sealed, the target front strike face had to be completely isolated from the rest of the target (sides and back), and this was done using sealant tape (Appendix) and a layer of nylon bagging film (Appendix). The impact hole on top of the target is the point of entry for the fluorescing resin mixture. Because the target is laid on its back, this report will occasionally refer to the front and back as top and bottom, respectively.

A piece of polyamide release fabric (Appendix) is cut ~1 to 2 in less than the target's top dimension and placed on the target's top surface. A resin distribution material (Appendix), smaller in size, is placed on top of the release fabric and both sheets are held by tape. Trimming the resin distribution material slightly less in size than the release fabric allows for easier removal of the bagging material after the system has been fully cured.

A section of 3/8-in ID spiral polyethylene tubing (Appendix) commonly used as a harness for electrical wiring was cut ~20 in in length. A spray adhesive (Appendix) was applied on the tubing and then rolled-on with the same resin distribution material of the same length. This is the resin transfer medium for the infusion process. A doughnut shape was created with the tubing and joined by a polyethylene T-connector; and from this connector, a solid polyethylene tubing (Appendix) of ~36 in was attached. The tubing ring was positioned with the impacted area at the center and held in place by small strips of the sealant tape.

Finally, the sealant tape is applied by finger pressure along the top perimeter of the target. The nylon bagging film is then carefully laid over the entire target surface area. Figure 4 shows a top view of the bagged target.

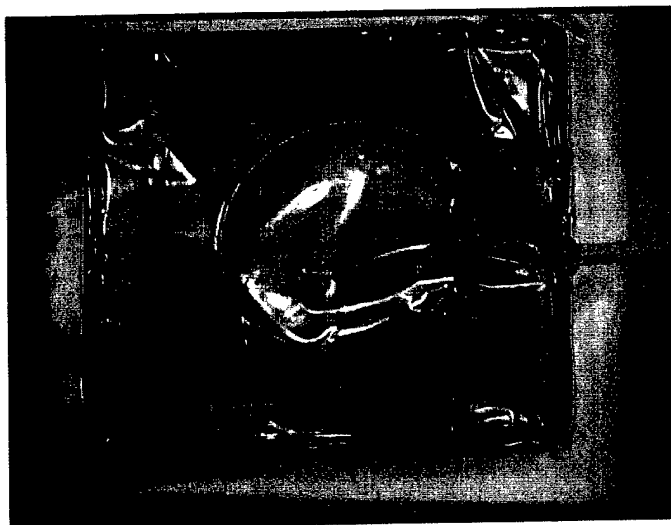


Figure 4. Top view of bagged target.

3.1.4 Bagging the Target Bottom (If Necessary)

In some cases, targets had penetration through the backside, requiring the exit hole to be sealed. Good infusion cannot be accomplished if the resin is not properly isolated and thus, not forcing the resin/dye to exit the laminate through the drilled holes. The exit hole can be easily patched with sealant tape and the nylon bagging material as shown in Figure 5.

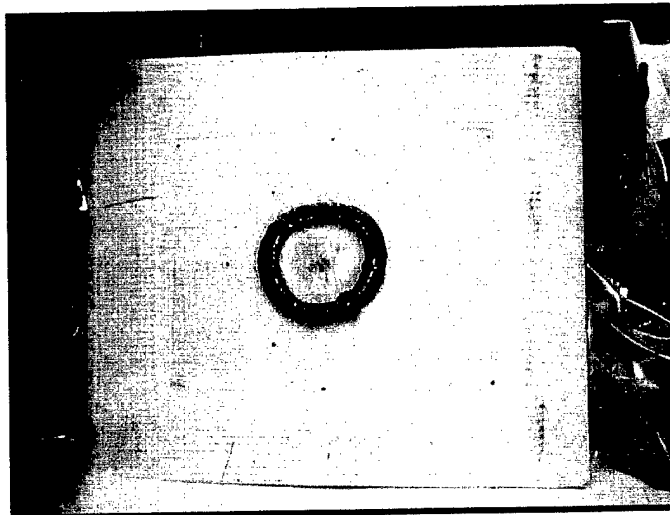


Figure 5. Sealed backside of target.

3.2 Target Infusion

3.2.1 Vacuum Assisted Resin Transfer Molding (VARTM) Preparation

A large transparent polycarbonate table with an 8- × 4-ft, 1/2-in-thick work surface was used as the tool plate for the infusion process. The polycarbonate tabletop was drilled with several holes

for vacuum distribution lines (Figure 6). Notice in Figure 6b the center port is not used and was sealed off. This port was unnecessary, as the vacuum was to be drawn away from the center of the target.

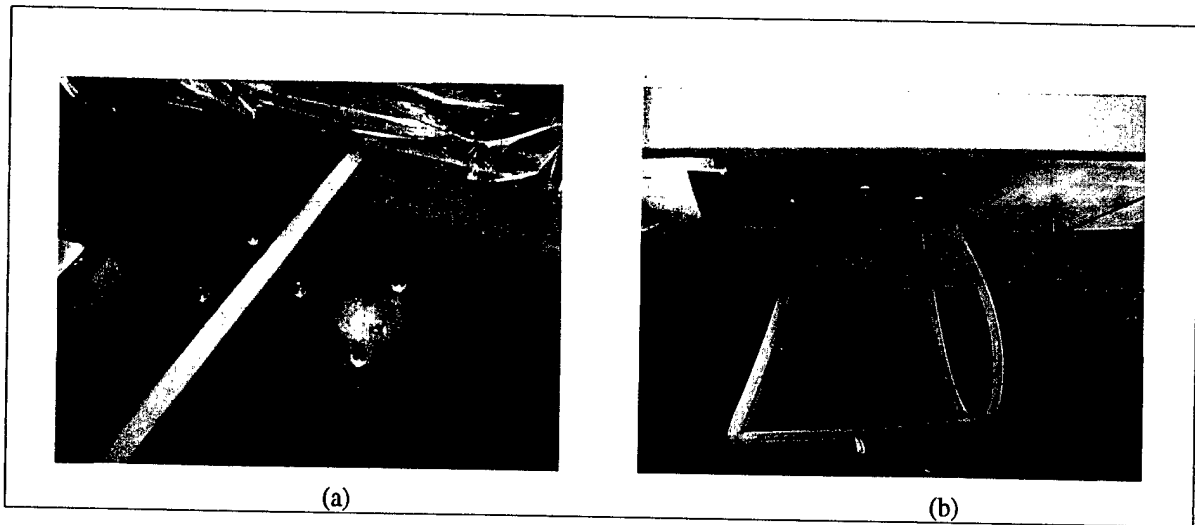


Figure 6. Vacuum distribution ports (a) top view (b) bottom view.

An area of 2×2 ft of the table surface was treated with a liquid release agent (Appendix). After the mold release agent dried, polyester felt breather (Appendix) material was cut $\sim 2-3$ in larger than the target's length and width and placed on center over the vacuum ports.

The breather material allowed air to continuously flow under the target during the evacuation cycle and also acted to absorb any excess resin that may flow out from the target. The same release fabric (used in the prior section) of approximate dimension was placed on top of the polyester breather. The target was then laid centrally on top of the breather and release fabric.

A section of the table surface was marked off from the center with sealant tape, a perimeter of $\sim 28 \times 48$ in. Relief loops/strips of sealant tape were placed in-line with the corner edge of the target at all sides. The strips are approximately the same height as the target and prevent the bagging film from forming a bridging between the part and tool surface, which can possibly tear the film during the vacuum draw, by adding additional slack in the bagging material. The nylon film is carefully adhered to the tacky sealant tape one side at a time, leaving the side where the tubing extends from the target to be finished last. By doing so, the excess bagging material that may accumulate is taken up by the tubing and/or extra sealant tape. Finally, the tubing is then clamped with a nonscaring pinch-off style Vise-Grip[§], and a vacuum is then pulled to check for possible air leaks. Figure 7 shows the target under vacuum and ready for the infusion process.

[§] Vise-Grip is a registered trademark of the American Tools Company.

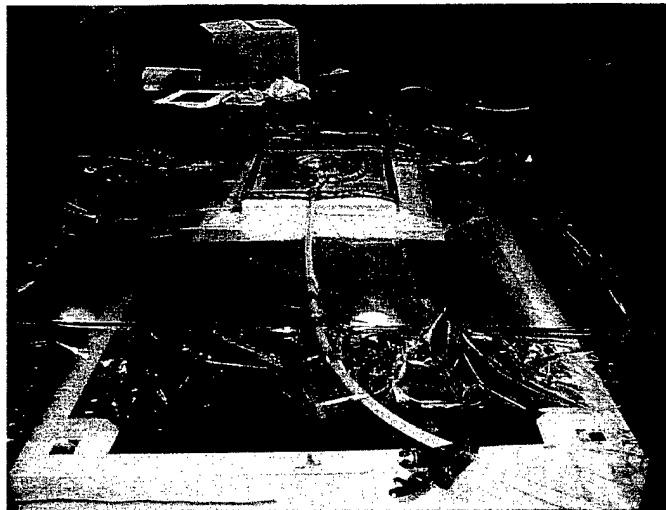


Figure 7. Target ready for resin transfer.

3.2.2 Resin/Fluorescing Dye

Depending on the size of the target and extent of the impact damage to the target, the amount of resin mix varied. On the average, a total of ~500 g was prepared for a target geometry of $14 \times 12 \times 2$ in. An appropriate amount of resin was poured into a 1 L container and weighed for accuracy. Approximately 1 g (0.2% of total weight to resin/hardener) of the fluorescein powder was added to the resin and thoroughly hand-mixed until the resin and powder dye became uniform in color. Next, the hardener was added (4:1 mix ratio w/w) and again thoroughly mixed.

After a final check for leaks of the evacuated bag, the resin/dye mixture was infused into the part through the polyethylene tubing after releasing the Vise-Grip. When the resin flow from the container to the target decreased, the tubing was again clamped off. Figure 8 is a view of the top with the resin/dye drawn into the target. The vacuum pump was left on continuously for about 2–3 hr. The target itself was monitored about every 1/2 hr to look for any leakage or resin/dye discharging from the sides. Observation from underneath the polycarbonate tabletop can reveal whether the infusion is penetrating through the target thickness by an indication of minute absorption onto the breather material. After the pump is powered off, the infused target was left to cure for about 12–15 hr before the bagging material was removed.

4. Results and Discussion

Figure 9 shows the bottom of an infused target with good resin penetration. Notice the resin flow out from the holes, and none is specifically concentrated at the center. This particular target had the backside sealed to block and divert the resin outward from the center. The number of holes with the resin bleed is an indication of extensive delamination in the composite back plate for this target. Targets are next typically cut into quarters by either a water jet or industrial diamond saw. Sections are intentionally cut through the center of the impact location.

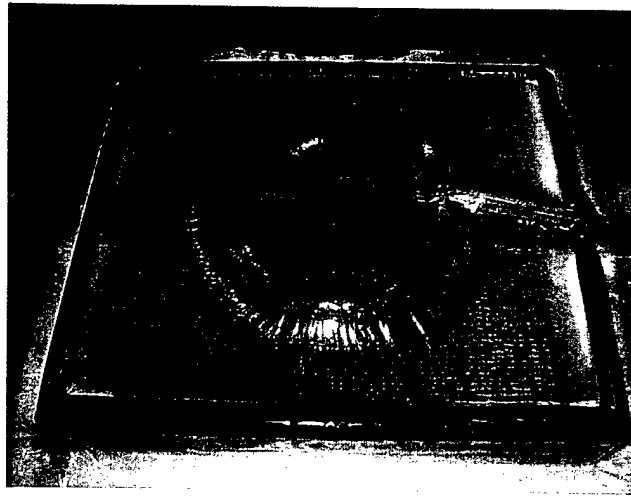


Figure 8. Top view of infused target.

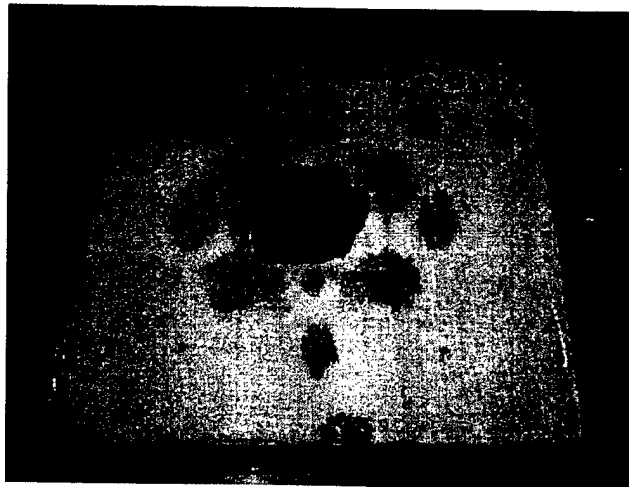


Figure 9. Bottom view of infused target.

By visual observation of the target cross-sections, the orange-red and green color of resin/dye can be seen clearly where it has filled in adequately, the impact location, between the fiberglass laminates and cracks within the tile, and tile debonding from the ethylene-propylene diene (EPDM) elastomer. Figures 10–12 shows the digital images taken of the cross-sections of a few CIA targets under normal lighting conditions and exposure to a black light source. A thick layer of the resin/dye creates a dark greenish hue effect as shown in Figure 10. Using a handheld “black light” or ultraviolet A (UVA) light source, the contrast of the resin/dye is further enhanced and can easily be distinguished among the resin and the target materials. Even 0.2 weight-percent of the powder dye was enough to brilliantly highlight the resin.

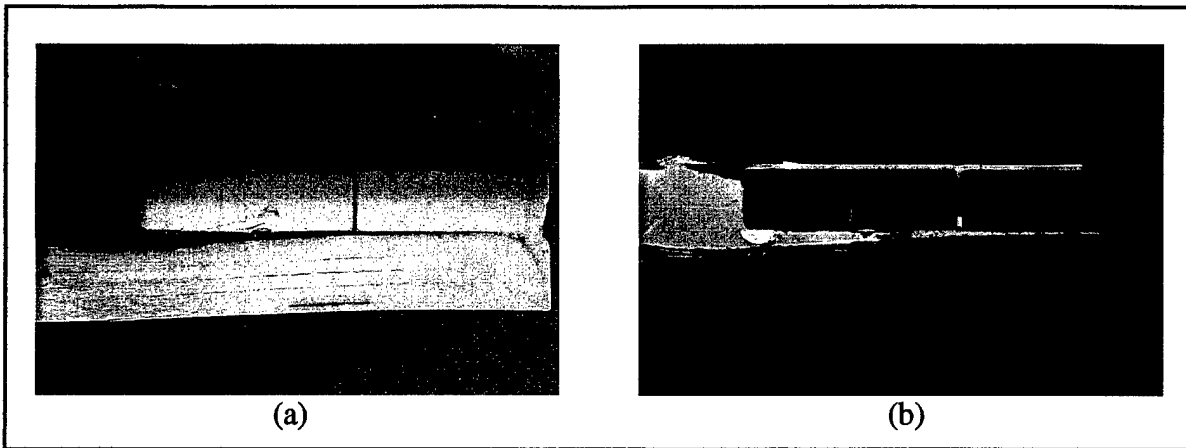


Figure 10. Cross-sectional view of target without EPDM elastomer layer under (a) normal and (b) UVA lighting conditions.

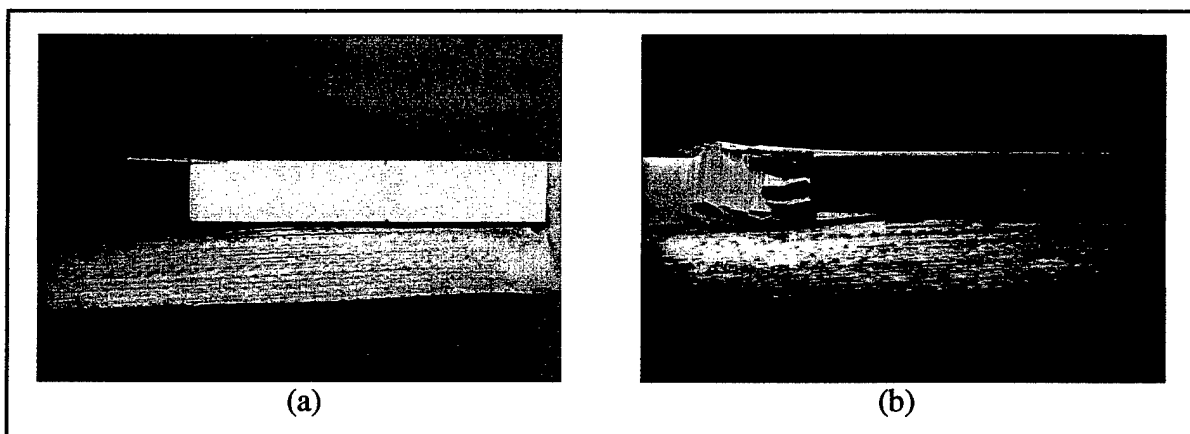


Figure 11. Cross-sectional view of target with EPDM elastomer layer under (a) normal and (b) UVA lighting conditions.

From the images, the resin/dye seeped into all the possible cavities available thus retaining the shape and clearly defining damage of the target. Figures 10a and 10b are photos of the same target. This target, which does not have the EPDM elastomer layer, shows a significant amount of debonding that occurred in comparison to the target with the EPDM shown in Figure 11. The tile debonding from the elastomer layer is well depicted, going out from the center of the target toward the edge. The fracture patterns of the ceramic tiles are also frozen in the resin for this particular target (Figure 11b). However, more delamination is widespread in the target with the additional elastomer layer.

Figure 12 shows extremely fine macrocracks in the tile made visible with the aid of the black light and not prevalent under normal room lighting. The details in the outline of the cracks are exceptionally well preserved. Even a small amount of the resin/dye has penetrated between the tiles.

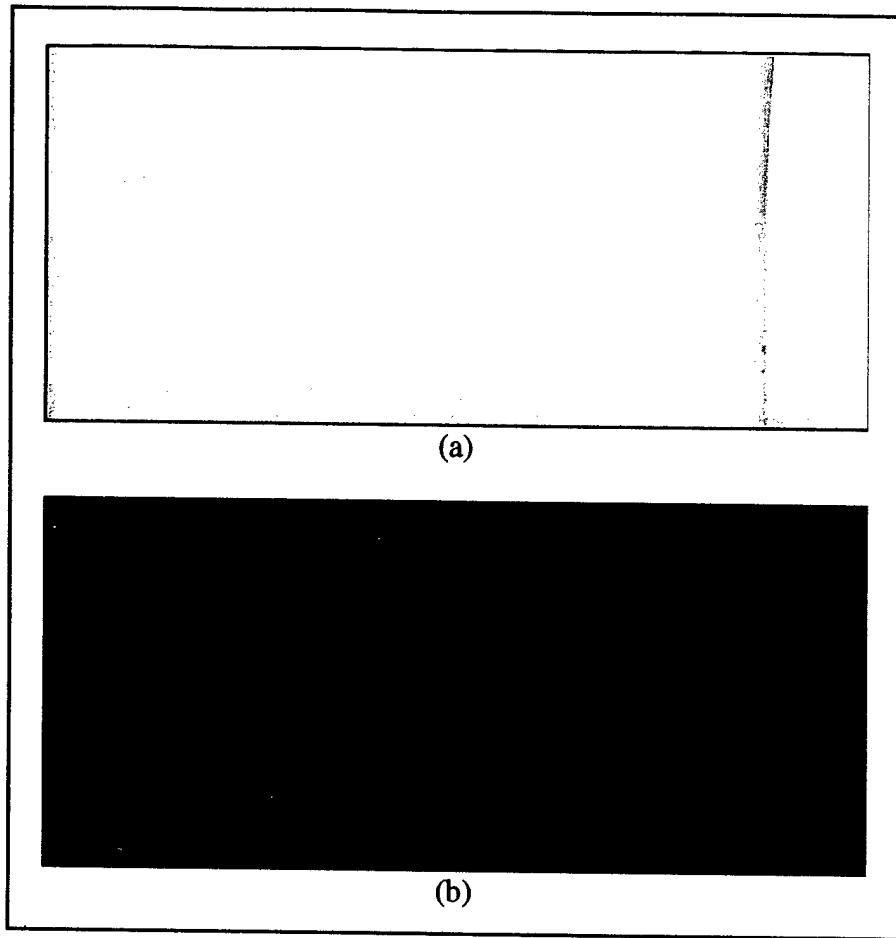


Figure 12. Close-up of Al_2O_3 tile under (a) normal lighting condition and (b) black light exposure.

5. Conclusion

This technique provides a new approach for the investigation of the delamination zones in a thick-section composite and fractures in the alumina oxide tiles of a ballistic-impacted CIA target. In comparison to nondestructive testing, the effort involved for the target resin/dye infusion is much more time consuming and requires additional materials on a per-target basis. However, this method allows a hands-on, visual aid for all the components in the targets. The fractures and delamination as well as debonding in the target materials are well preserved and offer a history synopsis of the damage initiated during the ballistic impact test itself. Overall, this method cannot only be applied to CIA targets but to other areas for post-analysis of composite structures as well. Additional work in this area may investigate the use of other fluorescing dyes to infuse a target with one specific colored dye prior to a ballistic test and infusing again with another color dye after impactation. This will possibly demonstrate the effects in the types of processes during target lay-ups as well as defects that may occur during fabrication of the target.

6. References

1. Gama, B. A.; Bogetti, T. A.; Fink, B. K.; Gillespie, J. W., Jr. Processing, Ballistic Testing and Repair of Composite Integral Armor. *Proceedings of the 32nd International SAMPE Technical Conference*. Boston, MA, November 5–9, 2000.
2. Gama, B. A.; Yarlagadda, S.; Bogetti, T. A.; Fink, B. K.; Gillespie, J. W., Jr. Repair of Composite Integral Armor. *Proceedings of the 46th International SAMPE Symposium*. Long Beach, CA, May 6–10, 2001; Vol. 46.
3. Desmettre, T.; Devoisselle, J. M.; Mordon, S. Fluorescence Properties and Metabolic Features of Indocyanine Green (ICG) as Related to Angiography. *Survey of Ophthalmology*, **2000**, *45* (1), 15–27.
4. van Norel, J.; van den Biesen, P. R.; Groen, G. J.; van Norren, D. Hold Up of Dye in the Arm During Fluorescein Angiography: A Quantitative Demonstration. *American Journal of Ophthalmology*, **2000**, *129* (4), 551–552.
5. ASTM D 2393. Standard Test Method of Viscosity of Epoxy Resins and Related Components, *Annu. Book ASTM Stand.* 1986.
6. ASTM D 2471. Standard Test Method for Gel Time and Peak Exothermic Temperature of Reacting Thermosetting Resins, *Annu. Book ASTM Stand.* 1994.
7. ASTM D 2240. Standard Test Method for Rubber Property—Durometer Hardness E1-1999, *Annu. Book ASTM Stand.* 1997.
8. ASTM D 790. Standard Test Methods for Flexural Properties of Unreinforced Plastics and Electrical Insulating Materials, *Annu. Book ASTM Stand.* 1998.
9. ASTM D 638. Standard Test Method for Tensile Properties of Plastics, *Annu. Book ASTM Stand.* 1998.
10. ASTM D 4065. Standard Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics, *Annu. Book ASTM Stand.* 1995.
11. Fink, K. B. *Performance Metrics for Composite Integral Armor*. ARL-RP-8; U.S. Army Research Laboratory: Aberdeen Proving Ground, MD, 2000.

INTENTIONALLY LEFT BLANK.

Appendix. Key Materials List

Frekote 700NC, Mold Release Agent
Loctite Distributor, Mahogany Co.; Mays Landing, NJ

3M Super 77 Multi-Purpose Spray Adhesive
local Grainger Industrial Supply

RC-3000-10, Stretchable Polyester Felt Breather
Richmond Aircraft Products; Norwalk, CA

A-8888, Open Weave Polyamide Release Fabric
Richmond Aircraft Products; Norwalk, CA

SM 5126 Sealant Tape
Schnee-Morehead Distributor, Northern Fiber Glass Sales; Hampton, NH

40% CCF Resin Distribution Material
Roxford Fordell; Greenville, SC

3/8-in ID Spiral Wrap Polyethylene Tubing
Panduit Distributor, Graybar Electric; New Castle, DE

3/8-in ID Polyethylene Solid Tubing
Panduit Distributor, Graybar Electric; New Castle, DE

Capran 512 Nylon Bagging Film
Richmond Aircraft Products; Norwalk, CA

INTENTIONALLY LEFT BLANK.