

NSG-1217

IMPROVEMENT OF COMPOSITE FRACTURE TOUGHNESS

BY FUSIBLE FIBERS AND COATINGS

by

James H. Williams, Jr.

Composite Materials and  
Nondestructive Evaluation Laboratory

December 1976

Massachusetts Institute of Technology  
Cambridge, Massachusetts 02139



(NASA-CR-149310) IMPROVEMENT OF COMPOSITE  
FRACTURE TOUGHNESS BY FUSIBLE FIBERS AND  
COATINGS Final Report, Aug. 1975 - Dec.  
1976 (Massachusetts Inst. of Tech.) 20 p

N77-71276

Unclas

00/98 58219

REPRODUCED BY  
**NATIONAL TECHNICAL  
INFORMATION SERVICE**  
U. S. DEPARTMENT OF COMMERCE  
SPRINGFIELD, VA. 22161

N77-71276

IMPROVEMENT OF COMPOSITE FRACTURE  
TOUGHNESS BY FUSIBLE FIBERS AND COATINGS

Final Report, August 1975-December 1976

(NASA Grant No. NSG-1217)

Submitted to the  
Materials Division of the Langley  
Research Center  
(Grant Monitor: Dr. Wilbur B. Fichter)

by

James H. Williams, Jr.  
Associate Professor of Mechanical Engineering  
Massachusetts Institute of Technology  
Cambridge, Massachusetts 02139

December 1976

REPRODUCED BY  
**NATIONAL TECHNICAL  
INFORMATION SERVICE**  
U. S. DEPARTMENT OF COMMERCE  
SPRINGFIELD, VA. 22161

## ABSTRACT

The results of a program to improve the fracture toughness of graphite fiber epoxy composites by thermoplastic fibers and fiber coatings are summarized. The concept can provide the simultaneous improvement of both composite strength and toughness, in particular, for laminates containing fibers coated with polysulfone. Very limited tests indicate that fatigue behavior may be improved by polysulfone coatings whereas polyvinylalcohol coatings may degrade the fatigue behavior. The low velocity impact toughness is increased by polyvinylalcohol coatings more than the increase achieved by polysulfone coatings.

The fracture modes of the composites are affected substantially by the choice of coating and stacking sequence. Thus, in instances where a particular mode of fracture or crack propagation direction is desired, the choice of coating, if any is used, is very important.

## ACKNOWLEDGMENTS

This report represents the first general summary of results relating to a concept which the author originated during 1973 when he was holder of the Edgerton Young Faculty Professorship. Work on discontinuous reinforcing fibers was initiated at the Union Carbide R & D Center during the summer of 1974 when the author held an NSF Industry Participation Grant. That work was continued under this Grant (NSG-1217), resulting in two publications and other preliminary analyses. The work on the fiber coatings has been totally supported by this Grant which also has subsidized a theoretical analysis of interlaminar stresses. (Refer to Cumulated Bibliography of Issued Publications.) The support of the Materials Division and Dr. W.B. Fichter the grant monitor, of the NASA Langley Research Center is greatly appreciated.

TABLE OF CONTENTS

ABSTRACT .....	1
ACKNOWLEDGMENTS .....	2
TABLE OF CONTENTS .....	3
INTRODUCTION .....	4
MATERIALS .....	5
STATIC STRENGTH AND FRACTURE TOUGHNESS .....	6
FATIGUE AND IMPACT TESTS .....	9
Fatigue .....	9
Impact .....	10
INTERLAMINAR STRESSES .....	13
CONCLUSIONS .....	14
REFERENCES .....	15
CUMULATED BIBLIOGRAPHY OF ISSUED PUBLICATIONS .....	16
FIGURES .....	17

## INTRODUCTION

One of the most common structural trade-offs in the utilization of graphite fiber composites is strength versus toughness. The goal of this research program was to improve the toughness characteristics of continuous graphite fiber composites [1]. The attempt to achieve the toughness improvements was by means of thermoplastic fibers and fiber coatings. The characteristics of the coatings enable them to (1) control fiber-matrix debonding; (2) contain individual fiber fractures, thus decreasing fiber-fiber flaw communication; and (3) control fiber pull-out. A model of the composite structure is shown in Fig. 1.

The following theoretical and experimental aspects of the proposed research effort have been undertaken (See the "Cumulated Bibliography of Issued Publications" section of this report.):

- 1) theoretical analyses of fiber intersections and the orientation efficiency factor relating to the thermoplastic reinforcing fibers;
- 2) theoretical and experimental analyses of the static strength and the fracture toughness of coated graphite fiber composites;
- 3) fatigue and impact experiments on coated graphite fiber composites;
- 4) a theoretical analysis of the unnotched interlaminar stresses for arbitrary lamina stacking sequences.

Only items (2), (3) and (4) will be reviewed briefly here as item (1) is already a part of the open technical literature.

## MATERIALS

The specimens for all the tests were cut from nine (9) graphite fiber epoxy panels (23cm x 23cm x 0.24cm) which were fabricated by Fiber Materials Incorporated. The fibers were Celanese GY-70. In some of the panels, the fibers were coated with either polyvinylalcohol (PVOH) or polysulfone (PPS). In all tests the specimens were grouped according to the lamination construction (A:  $[0^\circ, \pm 45^\circ]_s$  ; B:  $[0^\circ, \pm 45^\circ, 0^\circ]_s$  ; C: Unidirectional) and further classified by the fiber coatings (I: PVOH ; II: PPS ; III: No Coating). Extensive details concerning the constituents, panels and fabrication procedures are given in [2].

## STATIC STRENGTH AND FRACTURE TOUGHNESS

The static strength and notched fracture toughness characteristics are detailed in [2]. These results are summarized in Table I below where for each material the maximum value of the tensile strength ( $\sigma_{fr}$ ) for three unnotched specimens and the maximum value of the fracture toughness for two notched specimens are reported. Note that the tensile strength is normalized with respect to the fiber volume fraction. It can be seen that the fiber coatings provide significant increases in both  $\sigma_{fr}$  and  $K_Q$ .

In Table II the typical fracture characteristics of the various types of composites are summarized. Although these descriptions are more prominent in the notched specimens, they are typical for unnotched specimens as well. Photographs of fractured specimens and additional discussions are given in [2].

TABLE I

Maximum Values of Unnotched Static Strength  
and Notched Fracture Toughness

		Fiber Coating			
		PVOH	PPS	No Coating	
Laminate	$[0^\circ, \pm 45^\circ]_s$	328 17.0 0.60:0.11	452 13.9 0.64:0.14	375 8.5 0.52:0	$\sigma_{fr}/V_f$ $K_Q$ $V_f:V_c$
	$[0^\circ, \pm 45^\circ, 0^\circ]_s$	624 21.2 0.58:0.11	540 17.2 0.70:0.13	396 8.7 0.46:0	$\sigma_{fr}/V_f$ $K_Q$ $V_f:V_c$
	$[0^\circ]$	418 21.3 0.65:0.12	587 27.1 0.67:0.14	664 6.8 0.42:0	$\sigma_{fr}/V_f$ $K_Q$ $V_f:V_c$

Unnotched Fracture Stress:  $\sigma_{fr}$  (MN/m<sup>2</sup>)

Approximate Fiber Volume Fraction:  $V_f$

Approximate Coating Volume Fraction:  $V_c$

Mode I Fracture Toughness:  $K_Q$  (MN/m<sup>3/2</sup>)  $\equiv \sigma_c \sqrt{\pi c}$

$\sigma_c$  is the notched fracture stress referred to the unnotched cross-sectional area of the specimen.

$c$  is the notch length having 0° flank angle.

All notched specimens except the PPS[0°] had a 3.68 cm width and a 0.92 cm notch length; the corresponding dimensions for the

PPS[0°] notched specimens were 2.54 cm and 0.64 cm, respectively.

Matrix Elastic Modulus: 3930 MN/m<sup>2</sup>; Matrix Fracture Stress: 75 MN/m<sup>2</sup>.

All specimens had a total thickness of 0.24 cm.

TABLE II

Typical Fracture Characteristics of Composite Specimens

		Fiber Coating		
		PVOH	PPS	No Coating
Lamin ate	$[0^\circ, \pm 45^\circ]_s$	Large amounts of delamination. $0^\circ$ laminae shatter extensively. Major cracks propagate along interfaces* in the $+45^\circ$ and $-45^\circ$ directions of the respective laminae.	A single crack propagates along either a $+45^\circ$ or $-45^\circ$ interface with small amounts of delamination. When delamination occurs, major cracks propagate along interfaces in both $+45^\circ$ and $-45^\circ$ laminae simultaneously.	A single crack propagates horizontally (orthogonal to load axis) accompanied by small amounts of delamination in vicinity of crack path.
	$[0^\circ, \pm 45^\circ, 0^\circ]_s$	Major cracks propagate along interfaces of the $+45^\circ$ and $-45^\circ$ laminae, respectively, accompanied by moderate delamination. $0^\circ$ laminae fracture same as adjacent $+45^\circ$ or $-45^\circ$ laminae. Later delamination ends, single saw-tooth major crack propagates horizontally.	Moderate delamination accompanied by simultaneous major cracks along interfaces of respective $+45^\circ$ and $-45^\circ$ laminae. $0^\circ$ laminae fracture along $+45^\circ$ or $-45^\circ$ direction in accordance with adjacent $+45^\circ$ or $-45^\circ$ laminae.	A single crack propagates horizontally (orthogonal to load axis) accompanied by small amounts of delamination in vicinity of crack path.
	$[0^\circ]$	A single crack propagates vertically (parallel to load axis) along interfaces all the way to the specimen tabs, resulting in a complete separation of the specimen and tabs.	A single crack propagates vertically (parallel to load axis) toward tabs along interfaces, then horizontally, then vertically in the opposite direction of initial motion. (Occasionally, specimen behaves same as $[0^\circ]$ - No Coating specimen.)	A single crack propagates horizontally (orthogonal to load axis). Smooth cleavage-type surfaces are created.

\* Interfaces refer to the intralaminar fiber-matrix, fiber-coating and coating-matrix surfaces along the fiber direction.

## FATIGUE AND IMPACT TESTS

Due to materials limitations, all the composites could not be impact tested and only very abbreviated fatigue tests were conducted. These are summarized below.

### Fatigue

All specimens were sinusoidally fatigue loaded at 30 Hz in a cantilevered bending mode with zero mean stress. Emphasis was placed on testing two types of specimens, namely BIII ( $[0^\circ, \pm 45^\circ, 0^\circ]_s$  and no coating) and CI (Unidirectional and PVOH coating). In both cases the  $0^\circ$  laminae were parallel to the longitudinal axis of the specimen. Sufficient data to establish S-N curves were not generated in either case as only ten (10) specimens of each material were available.

The BIII material was tested without fracture to  $5 \times 10^6$  cycles when the oscillating stress in the extreme fibers was 0.75 of the uniaxial static fracture stress ( $\sigma_{fr}$ ). At a fatigue stress of  $0.80 \sigma_{fr}$ , the specimen fractured at  $1.7 \times 10^6$  cycles, and at a fatigue stress of  $0.90 \sigma_{fr}$ , the specimen fractured at about  $2 \times 10^4$  cycles. In both the fractured and unfractured specimens, no delamination was observed.

Fatigue data for the CI material was  $1.2 \times 10^6$  cycles at  $0.80 \sigma_{fr}$  without rupture;  $7.4 \times 10^5$  cycles at  $0.88 \sigma_{fr}$  without rupture; but almost immediate rupture when the fatigue stress exceeded  $0.90 \sigma_{fr}$ . Despite the fact that the specimens did not rupture in a number of the tests, significant fiber/coating/matrix debonding occurred. Also, in the ruptured specimens, fiber pullout was evident.

Finally, in addition to the above tests, two (2) C II specimens (unidirectional and PPS coating) were fatigued to  $3 \times 10^5$  cycles at  $0.9 \sigma_{fr}$  without rupture and with no visible debonding damage.

### Impact

A pendulum impact tester located at the U.S. Army Materials & Mechanics Research Center (Watertown, Mass.) was used. The rated capacity of the tester was 22.4 J with an impact head weight of 3.6 kg. The impact velocity was 3.4 m/sec. The specimen geometry and loading are shown in Fig. 2.

Because the impact tester was not in a state of calibration, the data obtained must be interpreted officially in a qualitative sense\*. Despite this, significant trends are indicated by the data obtained. The average normalized† impact energies for a minimum of three of each type of specimen are given in Table III.

Unnotched specimens were tested to determine the notch sensitivity. The results indicate that the unnotched specimens had normalized impact energies that were approximately 60% to 70% higher than those of their notched counterparts.

---

\* This statement is made in accordance with an agreement dated May 24, 1976 and signed by the author.

† The impact energies are normalized with respect to the net cross-sectional area at the notch. These values have not been normalized with respect to the fiber volume fractions.

TABLE III

Approximate Notched Impact Energies (kJ/m<sup>2</sup>)

		Fiber Coating		
		PVOH	PPS	No Coating
Laminate	[0°, ± 45°] <sub>s</sub>	No Data	34 kJ/m <sup>2</sup>	6 kJ/m <sup>2</sup>
	[0°, ± 45°, 0°] <sub>s</sub>	49 kJ/m <sup>2</sup>	21 kJ/m <sup>2</sup>	5 kJ/m <sup>2</sup>
	[0°]	57 kJ/m <sup>2</sup>	30 kJ/m <sup>2</sup>	No Data

The impact fracture behavior of the various specimens is quite consistent with the descriptions in Table II. PVOH coating resulted in significant intra-ply shattering and delamination; PPS coating produced no intra-ply shattering but some delamination along the fracture path; and the uncoated fiber composites displayed a single cleavage-type fracture plane with no visible damage away from the fracture surface.

## INTERLAMINAR STRESSES

A theoretical formulation of the interlaminar stresses in a fiber composite laminate having an arbitrary stacking sequence and subjected to uniform axial extension has been developed [3]. Assuming a set of three-dimensional displacement functionals, a variational principle was used to derive the governing equations which were solved in closed-form. A set of examples remains to be computed.

## CONCLUSIONS

The results of a program to improve the fracture toughness of graphite fiber epoxy composites by thermoplastic fibers and fiber coatings have been reviewed. The effects of polyvinylalcohol and polysulfone fiber coatings were investigated. Polyvinylalcohol coatings provide the greater impact toughness improvement although it appears that it degrades the fatigue strength. Polysulfone coatings enhance strength, notched fracture toughness and low-velocity impact toughness. Although the effect of polysulfone coatings on fatigue behavior is not clear, it appears to be favorable.

The various aspects of the program which have been considered are represented by the cumulated bibliography. Further development of the concept appears to be attractive although its effectiveness must be compared with those of competing concepts.

## REFERENCES

1. Williams, J.H., Jr., "Improvement of Composite Fracture Toughness by Fusible Fibers and Coatings", Proposal submitted to the Materials Division of the NASA Langley Research Center, March 1975.
2. Kousiounelos, P.N., "Fracture of Graphite Fiber Composites", S.B. and S.M. Thesis, Massachusetts Institute of Technology, May 1976.
3. Bin Ahmad, Z. and Williams, J.H., Jr., "Theoretical Formulation of Interlaminar Stresses in Arbitrarily Laminated Composites", Composite Materials and Nondestructive Evaluation Laboratory, Department of Mechanical Engineering, Massachusetts Institute of Technology, December 1976.

CUMULATED BIBLIOGRAPHY OF ISSUED PUBLICATIONS

The documents listed below were issued during the conduct of the research program and were supported either wholly or partially by funds from the subject grant.

1. Williams, J.H., Jr., Lee, S.S., and Wasserman, C.S., "Fibre Intersections in a Planar Randomly Oriented Fibre Composite", (To appear in Fibre Science and Technology).
2. Williams, J.H. Jr. and Bosy, B.J., "Fibre Orientation Efficiency Factor for Planar Randomly Oriented Discontinuous Fibre Composites", (To appear in Fibre Science and Technology).
3. Kousiounelos, P.N., "Fracture of Graphite Fiber Composites", S.B. and S.M. Thesis, Department of Mechanical Engineering, Massachusetts Institute of Technology, May 1976.
4. Williams, J.H., Jr. and Kousiounelos, P.N., "Thermoplastic Fiber Coatings Enhance Composite Strength and Toughness", (Submitted for publication).
5. Kousiounelos, P.N. and Williams, J.H., Jr., "Heterogeneous Anisotropic Model for Notched Fiber Composites", (Submitted for publication).
6. Bin Ahmad, Z. and Williams, J.H., Jr., "Theoretical Formulation of Interlaminar Stresses in Arbitrarily Laminated Composites", Composite Materials and Nondestructive Evaluation Laboratory, Department of Mechanical Engineering, Massachusetts Institute of Technology, December 1976.

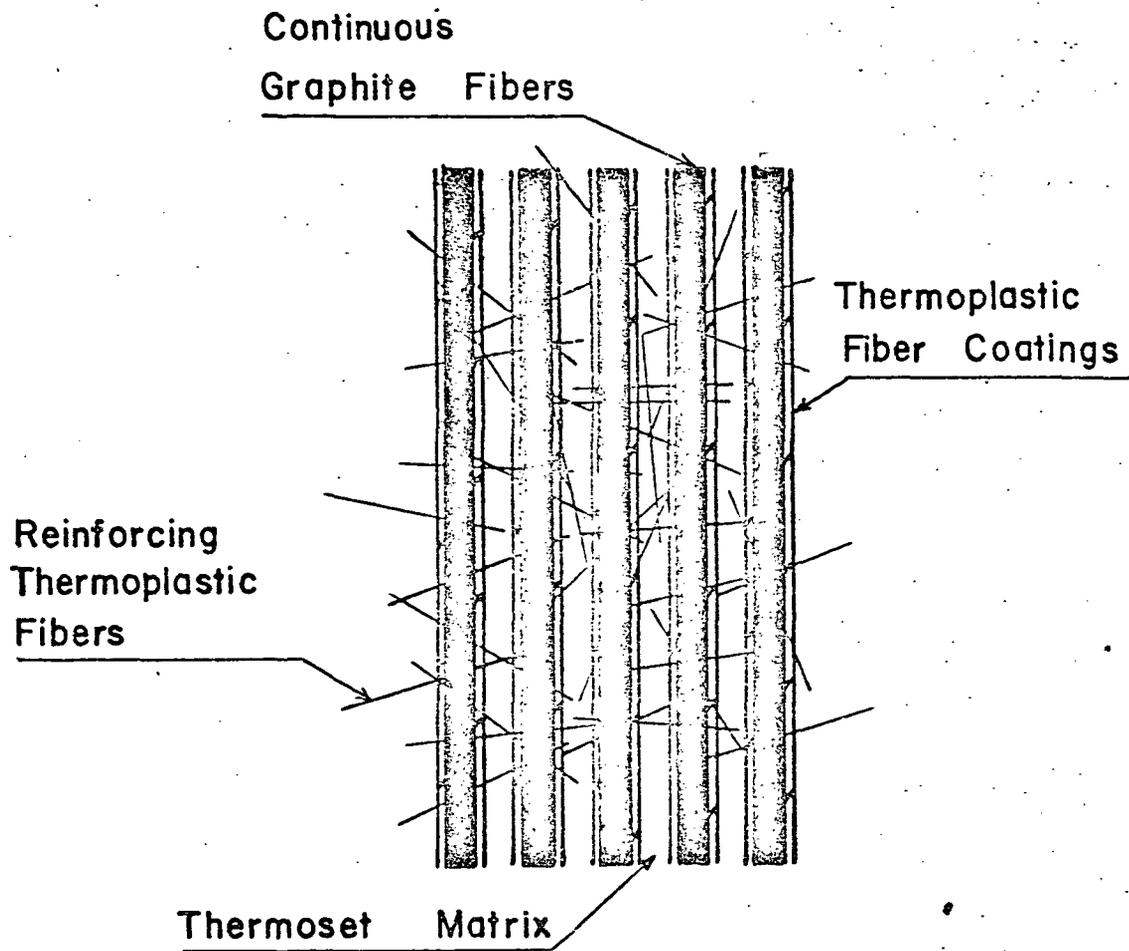


Fig. 1 Model of unidirectional graphite fiber composite showing thermoplastic fiber coatings and thermoplastic reinforcing fibers.

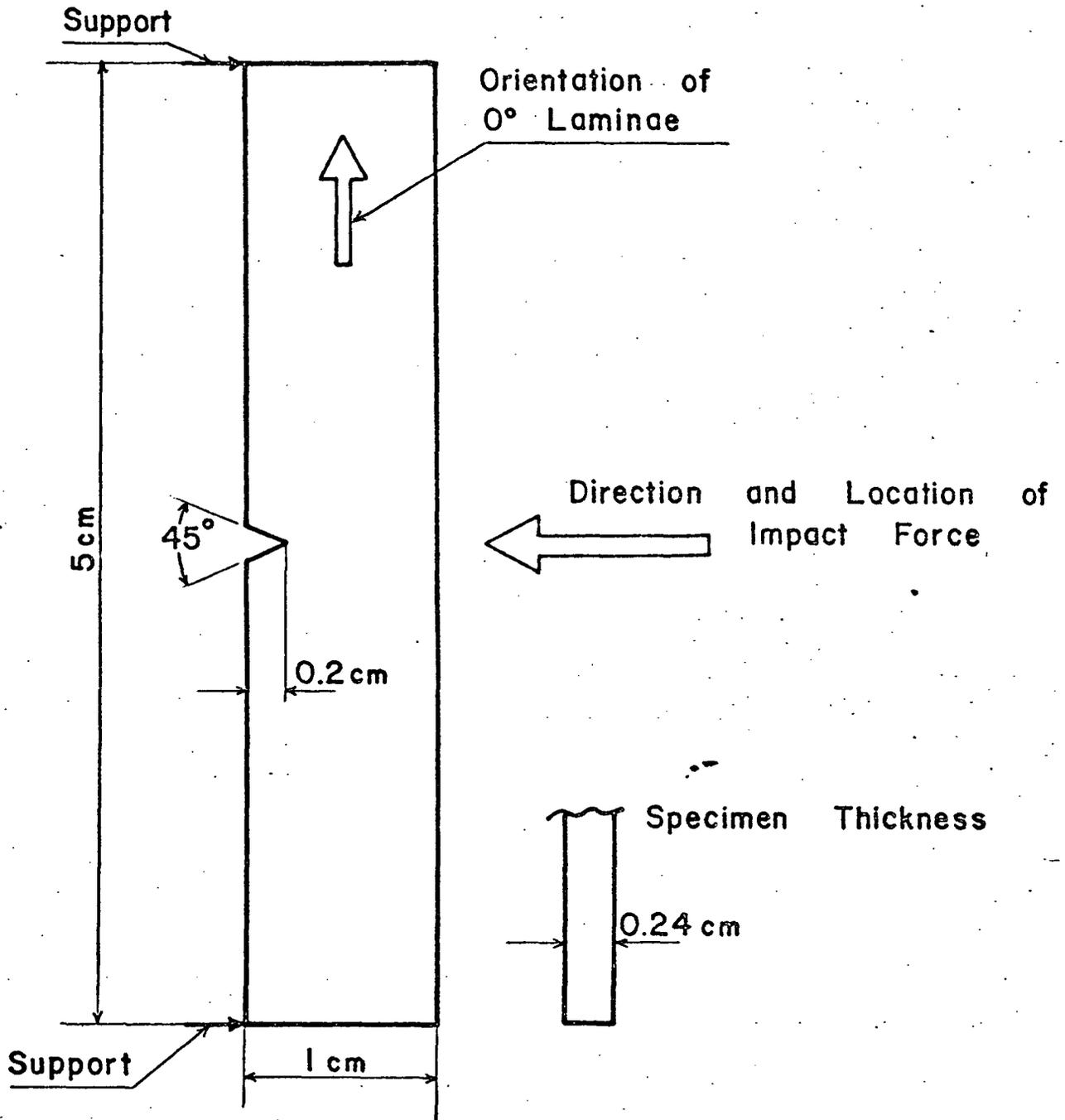


Fig. 2 Sketch of impact specimen showing geometry, orientation, loading and support.