

N

NOLTR 66-2

AN AUTORADIOGRAPHIC TECHNIQUE FOR
STUDYING CRACK GROWTH IN PLASTICS
COMPOSITE MATERIALS

DISTRIBUTION STATEMENT A

Approved for public release;
Distribution Unlimited

19960419 078

1 APRIL 1966

NOL

UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

NOLTR 66-2

Distribution of this document is unlimited.

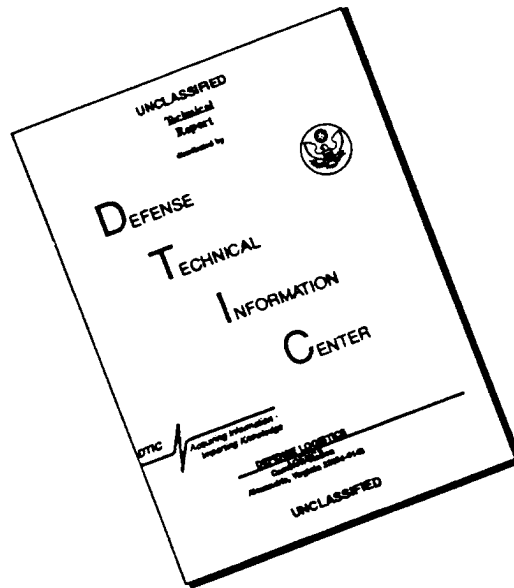
DTIC QUALITY INSPECTED 1

DEPARTMENT OF DEFENSE
PLASTICS TECHNICAL EVALUATION CENTER
PICATINNY ARSENAL, DOVER, N. J.



PLASTEC 8/19
6/19

DISCLAIMER NOTICE



THIS DOCUMENT IS BEST QUALITY AVAILABLE. THE COPY FURNISHED TO DTIC CONTAINED A SIGNIFICANT NUMBER OF PAGES WHICH DO NOT REPRODUCE LEGIBLY.

AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING
CRACK GROWTH IN PLASTICS COMPOSITE MATERIALS

Prepared by:
Marlin A. Kinna

ABSTRACT: Preliminary work has been conducted on segments of NOL Rings to study crack propagation characteristics in fiber reinforced plastics composite materials. Samples were submitted to a variety of fatigue loading conditions. They were then immersed in a tritiated water-ethylene glycol solution and after suitable processing, autoradiograms were prepared for analysis. Radiation emanating from the solution retained by the samples indicated that minute cracks and fissures were present in all of the specimens and that the radiation intensity varied in direct proportion to the severity of the fatigue loading conditions. When photomicroscopy was used to analyze similar specimens, cracks could be detected only for the most severe test conditions.)

APPROVED BY:

F. Robert Barnet
Non-Metallic Materials Division
CHEMISTRY RESEARCH DEPARTMENT
U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, SILVER SPRING, MARYLAND

1 April 1966


AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING CRACK GROWTH IN PLASTICS
COMPOSITE MATERIALS

This report presents information pertaining to the development of a technique for studying crack growth in reinforced plastics composite materials. The technique involves immersion of specimens in a radioactive tracer solution and then processing them to produce autoradiograms of the surface being analyzed. Photographs of the autoradiograms are prepared for analysis, and provide permanent records of the results.

The information presented in this report is of an exploratory nature. The tests were conducted on a single series of ring specimens which had been subjected to multiaxial fatigue loads, and the technique has not been applied to samples submitted to other types of mechanical testing. Furthermore, analytical procedures associated with the application of radioactive materials, handling of exposed specimens, and documentation of results are still in a state of flux, and the techniques described are not to be considered final. The preliminary work reported herein has been presented as papers at the 1966 Annual Technical Conference of the Society of Plastics Industry and at the first symposium on "Radioisotope Applications in Aerospace," jointly sponsored by the Wright Patterson Air Force Flight Dynamics Laboratory and the U. S. Atomic Energy Commission (February, 1966).

The technique for applying the radioactive solution and preparing the autoradiograms was developed under contract by Philco Scientific Laboratories, Blue Bell, Pennsylvania. The work was supported by Task No. RREN-ST-204/212-5/0000 and was carried out over the period from July 1964 to July 1965.

J. A. DARE
Captain, USN
Commander


ALBERT LIGHTBODY
By direction

CONTENTS

INTRODUCTION	1
BACKGROUND	2
EXPERIMENTAL WORK	3
RESULTS AND DISCUSSION	3
CONCLUSIONS	6
RECOMMENDATIONS.....	6
ACKNOWLEDGMENT.....	6

ILLUSTRATIONS

Figure	Title
1	NOL Multiaxial Fatigue Test Device
2	Schematic Representation of NOL Ring Test Specimen
3	Procedure for Radioactive Tracer Analysis of NOL Ring Samples
4	Autoradiogram of NOL Ring Segment (No Fatigue Test Conditioning) (35 X)
5	Autoradiogram of an NOL Ring Segment After 500 Cycles of Fatigue Testing at 10 CPM (35 X)
6	Autoradiogram of NOL Ring Segment After 500 Cycles of Fatigue Testing at 100 CPM (35 X)
7	Autoradiogram of NOL Ring Segment After 5000 Cycles of Fatigue Testing at 100 CPM (35 X)
8	Autoradiogram of NOL Ring Segment After 5000 Cycles of Fatigue Testing at 100 CPM (Ordinary Water) (35 X)
9	Autoradiogram of NOL Ring Fabricated with Preimpregnated Roving (35 X)
10	Mirror-Image Autoradiogram of NOL Ring Fabricated with Pre-Impregnated Roving (35 X)
11	Photomicrograph of Autoradiogrammed NOL Ring Cross-Section (400 X)
12	Photomicrograph of Autoradiogrammed NOL Ring Cross-Section (1800 X)
13	Photomicrograph of NOL Ring Segment After 500 Cycles of Fatigue Testing at 100 CPM (35 X)

REFERENCES

- (a) Erickson, P. W., A. A. Volpe and J. J. Frankenthaler, "Chemical and Physical Effects of Glass Surfaces Upon Laminating Resins," Sec. 21-A, 19th Annual Tech. Conf. of S.P.I. Reinforced Plastics Div., Feb. 1964.
- (b) Outwater, J. O. and M. J. Yendryeski, "On the Strengthening of the Roving by Modification of the Finish," Sec. 19-B, 20th Annual Tech. Conf. of S.P.I. Reinforced Plastics Div., Feb. 1965.
- (c) Bascom, W. D., "Some Surface Chemistry Aspects of Glass-Resin Composite: Part 1. Wetting Behavior of Epoxy Resins on Glass Filaments," Sec. 15-B, 20th Annual Tech. Conf. of S.P.I. Reinforced Plastics Div., Feb. 1965.
- (d) Kinna, M. A., "NOL Ring Test Methods," NOLTR 64-156, Sep. 1964.
- (e) Prosen, S. P., "A Method of Test for Determining the Compressive Properties of Filament Wound NOL Rings," NOLTR 64-80, July 1964.
- (f) "Tensile Strength Apparent of Parallel Reinforced Plastics by Split Disc Method," American Society for Testing and Materials ASTM 2290-64T.
- (g) "Apparent Horizontal Shear Strength of Reinforced Plastics by Short Beam Method," ASTM 2344-65T, American Society for Testing and Materials.
- (h) "Tensile Properties of Glass Fibers, Glass Fiber Strands, Yarns and Rovings Used in Reinforced Plastics," ASTM 2343-65T, American Society for Testing and Materials.
- (i) Bradford, John R., "Radioisotopes in Industry," Reinhold Publishing Corp., New York, N.Y., 1953.
- (j) Overman, R. T. and H. M. Clark, "Radioisotope Techniques," McGraw-Hill Book Company, Inc., 1960.

INTRODUCTION

Much work has been done in the field of fiber reinforced plastics composites to gain an understanding of the relationships between fabrication parameters, inherent mechanical properties, and crack formation and propagation. The interaction between glass fibers and resin matrices has been studied by several investigators. Erickson, et al (ref. (a)) working in this field, have found indications that factors exist at the glass-resin interface which could lead to changes in the resin-hardener ratio. The role played by glass surface finishing materials has also been widely studied. Outwater, et al (ref. (b)) for example, have shown that the surface shear strength of certain finishing materials is enhanced by boiling the finished roving for short periods of time. Working in the same area of research, Bascom (ref. (c)) found that epoxy monomers and resin liquids exhibit high contact angles on E-glass filament coated with siloxane type finishing materials. Subsequently, he showed that the number of microvoids in composite structures could be significantly decreased by reducing the contact angle to zero. In order to effectively study the relationships between fabrication parameters and the physical properties of composite structures, test methods were developed (refs. (d), (e), (f), (g), (h)) which enabled the investigator to obtain ultimate strength properties on carefully prepared samples. Although much information has been acquired as a result of these and other studies, conflicting evidence has also arisen and the actual formation and growth of minute cracks within the plastics material, when it is placed under stress, are still not clearly defined.

When plastics materials undergo mechanical failure, the failure seldom, if ever, occurs instantaneously. The time of the initial failure and the extent of the progressive degradation, however, are of prime importance. The development and progression of minute cracks are of particular interest when the structure is exposed to water or water vapor, since water is adsorbed on and interacts with the glass fibers exposed in these microscopic fissures. When the composite structure is placed under stress, unpredictable changes, influenced by the volume of absorbed water and the length of time it has been present, occur in the mechanical properties of the material. An analytical procedure is thus required which can be used to define crack initiation and propagation characteristics in reinforced plastics composite materials.

The Naval Ordnance Laboratory is studying a technique for evaluating crack initiation and propagation characteristics in plastics composite structures. A radioactive isotope is used to define the microscopic fissures which are formed under load or due to aging. The isotope penetrates the fissures and the radiation emitted can be observed by use of autoradiographic techniques. The location and concentration of the tracer material present in the structure can thus be determined, and the dimensions of the fissures can be estimated. Results for a series of NOL ring samples studied by using the autoradiographic technique are presented in this report.

BACKGROUND

Radioactive isotopes are widely used today in scientific, medical and industrial research (ref. (i)). One of the important applications is in their use as tracers. Since radioactive isotopes are chemically identical to stable isotopes of the same element, they can be used without interference to the process being carried out. Radioactive isotopes, when present in the amounts of 10^{-11} to 10^{-14} grams, can be detected using standard counting techniques, and when autoradiographic methods are used, even greater sensitivity may be obtained. Because of such high sensitivity of detection, radioisotopes have proven useful in numerous analytical procedures.

Not all radioisotopes are suitable for a given application, and selection of the correct tracer involves a number of considerations (ref. (j)). First, an isotope must have decay characteristics suitable for study of the problem at hand. Second, the experiment, when performed, should produce data of statistically sound significance within the required limits of interpretation. Third, due regard must be given to the necessary safety precautions and health hazards which exist in the handling of all radioactive materials. Consideration must also be given to other factors, such as the necessary equipment required and the ease of preparation or availability of the isotope.

Radiation is usually measured in one of two ways: (1) counting techniques or (2) autoradiography. Counting techniques involve the use of equipment suitable for recording the number of events of ionization or scintillations which are produced when radiation interacts with matter. Autoradiography makes use of a photographic emulsion to record radiation events such as gamma rays, X-rays, beta particles or alpha particles. In many applications, the emulsion is applied directly to the surface of the sample and intercepts and records radiation, incident to the emulsion, which is emitted by the radioactive isotope present in or on the specimen. The particular emulsion selected for use is determined by the type of radiation to be recorded, the level of radiation, and the degree of resolution required.

Since a solution of ethylene glycol and tritiated water of known specific activity was already available, tritium was selected as the tracer material to be used in the preliminary study of crack initiation and propagation in fiber reinforced plastics composite materials. Tritium is suitable since it radiates a beta particle which can be detected by autoradiographic techniques. In addition, when combined in proper proportions with water or other organic liquids, tritium replaces the hydrogen atom without changing the chemical characteristics of the liquid. The role played by water in the formation and propagation of internal cracks in fiber reinforced composite structures can thus be studied.

EXPERIMENTAL WORK

A series of NOL rings were fabricated and cured under identical conditions. After machining, they were submitted to a variety of multiaxial fatigue loads in the NOL Multiaxial Fatigue Tester shown in Figure 1. On completion of the fatigue tests, the portion of each ring which had received maximum stress (see Fig. 2) was used for the radioisotope analysis. A solution consisting of three parts ethylene glycol, two parts tritiated water and one part oxalic acid was used as the tracer material. The specific activity of the solution was one curie per milliliter.

The analytical procedure was carried out by placing NOL ring segments in an air-tight chamber and evacuating. The radioactive solution was then admitted to the chamber and the specimen was completely immersed in the liquid for a period of 72 hours or more. The sample was then removed from the chamber and the excess tracer material on the surface was quickly washed off. After mounting in a casting resin for easy handling, a section having a nominal thickness of 0.062 cm (0.025 inches) (thicknesses of 0.025 cm can be cut when required) was cut using liquid nitrogen as the coolant. The specimen was then stored in liquid nitrogen to prevent evaporation of the radioactive solution until coated with the photographic emulsion. Following application of the emulsion, the specimens were stored in a light tight container at 4°C for a predetermined period of time. The emulsion was then processed photographically to obtain autoradiograms, and photographs of the autoradiograms were then prepared for study. The procedure is summarized in Figure 3.

Control samples for the radioisotope analysis technique were prepared by exposing specimens in the manner described to ordinary water. Samples were prepared for each of the fatigue test conditions used.

In addition to the samples analyzed after multiaxial fatigue testing, segments of an NOL ring prepared with preimpregnated roving were analyzed. The samples were 0.5 cm long, and after exposure to the radioactive solution autoradiograms were prepared and photographed.

RESULTS AND DISCUSSION

Figure 4 shows the cross-section of an NOL ring segment which received no mechanical testing prior to immersion in the tritiated water solution. Radioactive emission is indicated by the grey and black areas present in the structure. Each such area represents a void or fissure end which is capable of retaining radioactive liquid. The largest discontinuity is approximately 6.5×10^{-3} square millimeters in area. Many are visible, however, which are only 1.5×10^{-3} square millimeters or less in area. The diagonal lines on this and other autoradiograms are machining marks.

Autoradiograms of NOL ring sections which were exposed to the radioactive solution after more extensive test conditioning are shown in Figures 5 and 6. The tracer solution has penetrated at least one lateral crack in the surface shown in Figure 5. Crack formation is also indicated by the presence of tracer material at the upper left side of the specimen. As in Figure 4, void areas which have filled with the tracer are sharply defined. Structural failure, sufficient to retain appreciable quantities of radioactive liquid, is readily indicated on the specimen surface shown in Figure 6. The shading and general configuration of many of the tracer-containing areas indicate that failure has progressed, under the test conditions imposed, in a radial manner from void areas which were present in the specimen prior to test. The radioactive tracer also indicates the presence of hairline cracks, probably just forming, in the central area of the specimen.

Figure 7 shows large quantities of tracer material which have been retained in the separations produced by severe test conditions. The large cracks are clearly defined, and smaller vertical and lateral fissures are also made apparent by the radiation. A sample taken from a position adjacent to the specimen in Figure 7 is shown in Figure 8. The autoradiogram was prepared after the sample had been processed with ordinary water. Examination of the autoradiogram led to the conclusion that the extensive damage indicated by the tritiated water solution could not be detected when ordinary water was used. Similar analysis of samples exposed to each of the other test conditions resulted in the same conclusion.

Autoradiograms of a specimen from a ring fabricated with pre-impregnated roving are shown in Figures 9 and 10. After immersion in the tritiated water solution, both ends were coated with photographic emulsion. Examination of the resulting mirror-image autoradiograms led to the conclusion that many of the voids present on the surface were indeed fissures which progressed longitudinally through the specimen for its entire length. In addition, several of the lateral cracks indicated in Figure 9 by the presence of tracer material are continuous for the length of the test specimen and appear in the mirror image presented in Figure 10. The potential value of the radioisotope technique is thus clearly demonstrated. By exposing a composite sample to a tracer material and taking sections in an appropriate manner, the length, diameter and location of internal fissures and discontinuities can be studied. The materials characteristics thus described may then be correlated with mechanical test data and ultimately may be expected to be useful in forecasting the lifetime of plastics materials.

Figures 11 and 12 are autoradiograms which have been enlarged 400 and 1800 times respectively. Fiber ends can be observed in the void areas which are completely unattached to resinous material, and excellent definition of void configuration is obtained with the use of the radioactive tracer solution, in spite of the high magnifications. Enlargements

of the area around the fissure ends may provide detailed information, in subsequent studies, on the nature and origin of crack initiation.

Five specimens were taken from each sample after immersion in the tritiated water solution and were examined to evaluate the reproducibility of the test method. Comparison of the resulting autoradiograms indicated that for each of the test levels used, a distinct failure pattern was obtained. Approximately the same number of tracer-filled fissures (or bubble-like voids) appeared in all the specimens at each test level, while distinct differences, such as lateral cracks and fissures of substantial size, could be noted for the samples tested at different conditions. All observations indicated that tracer analysis offered a reliable technique for the study of internal degradation in fiber reinforced plastics materials.

Three samples which had been subjected to multiaxial fatigue loads were studied using photomicroscopy techniques. Specimens were cut, mounted and polished, and a microscopic examination of the cross-sectional surface was made. The sample which had received 5000 cycles of test at 100 cycles per minute showed a large crack across the width dimension near the center of the cross section (Fig. 13). Several smaller cracks were also forming at other locations within the structure. The remaining two samples, which had been tested at less severe conditions, showed no indication of fissures. Autoradiographic techniques, however, showed that minute cracks were indeed present in samples after fatigue testing at the less severe conditions.

The experimental techniques employed in this work are preliminary, and it is expected that more definitive data will be obtained with refinement of the method described herein. Use of tritium limited this work to an investigation of surface phenomena; other isotopes may be used in the future, however, which will permit the investigator to study internal phenomena as well. Materials which should be considered for future work are the radioactive isotopes of sodium, phosphorus, silver, etc. Since these isotopes radiate at different levels of energy, specific analytical procedures will be required for each. The variety of available materials, however, is expected to be more than sufficient to provide complete information on crack propagation characteristics in plastics composites.

The potential value of radioactive isotopes for the study of crack growth and propagation cannot be overestimated. The methods and materials used in the work discussed herein provided good resolution and clarity in the autoradiograms obtained. Special photographic emulsions are now available, however, which offer even greater resolution, and it is expected that dimensions in the order of 100 Angstroms or less may be detected. Examination of tracerized materials with photomicroscopy techniques also offers promise. Careful analysis of highly magnified surfaces generally requires the services of skilled microscopists. By photographing autoradiograms, on the other hand, surface discontinuities can be easily identified by the technician even at high magnifications. The value of isotopes will be realized to the fullest extent, however, when techniques have been developed to study the interior of plastics composite materials. By using

NOLTR 66-2

isotopes which radiate at sufficient levels of energy, crack density profiles may be obtained which will indicate the length, size and location of discontinuities in specimens after controlled mechanical and environmental conditioning. Ultimately, techniques can be devised which will be essentially of a nondestructive nature, permitting samples to be alternately conditioned and evaluated. Valuable information on the progressive nature of failure in plastics composite materials can thus be documented.

CONCLUSIONS

The information reported herein demonstrates the potential value of radioactive tracer techniques in the study of fiber reinforced plastics composite materials. It has been shown that tracers are capable of indicating the presence of small internally situated cracks and fissures, and that the growth of such discontinuities may be followed and related to the fabrication and/or test conditions imposed upon the structure. Studies carried out on replicate samples have further indicated that the analytical procedure used herein is sufficiently reliable to produce statistically significant data.

RECOMMENDATIONS

It is recommended that additional studies be carried out leading to the development of a reliable technique for investigation of crack growth in plastics composite materials. The use of tracer isotopes other than tritium should be considered, as should the use of organic carrier materials which can be polymerized after sample exposure. On completion of these phases of study, use of the finalized procedure is recommended in all areas where basic information is required on the nature of internal changes in composite materials as a function of load and/or storage conditions.

ACKNOWLEDGMENT

The author wishes to thank Dr. Soo San Choi of the Philco Scientific Laboratories, Blue Bell, Pennsylvania, for his work leading to the development and application of autoradiographic techniques suitable for use in this study. Thanks are also extended to Dr. R. H. Cornish of the IIT Research Institute for his cooperation in the performance of the microscopic studies discussed in this report.

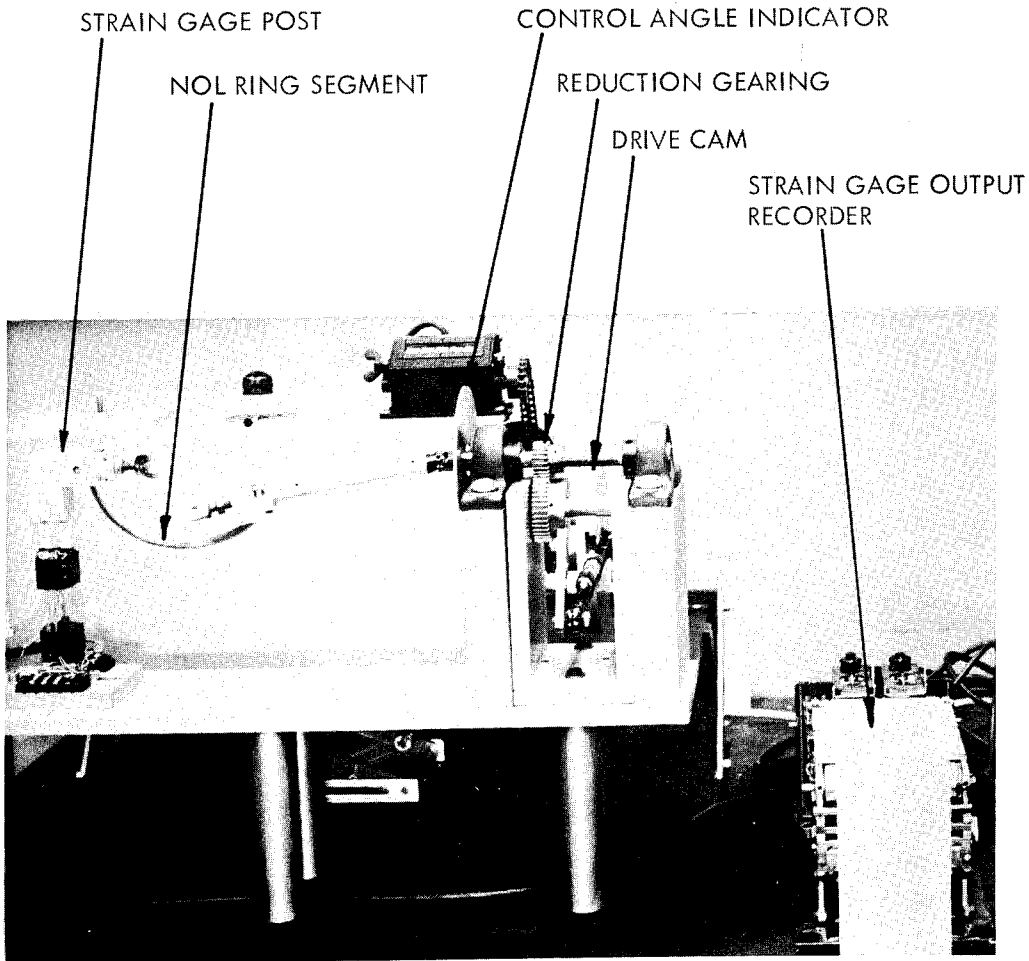


FIG. 1 NOL MULTIAXIAL FATIGUE TEST DEVICE

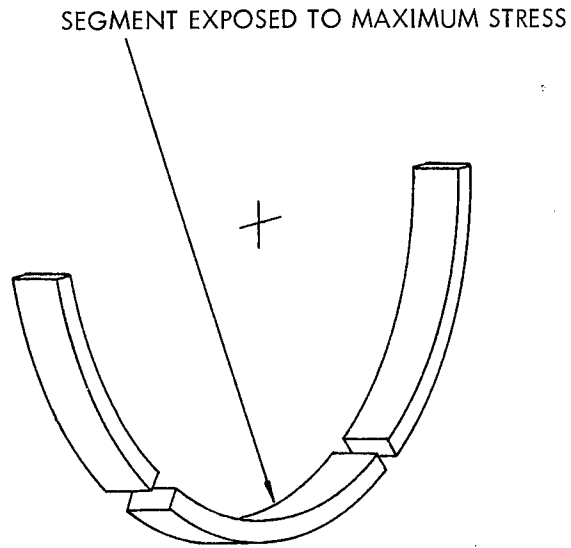
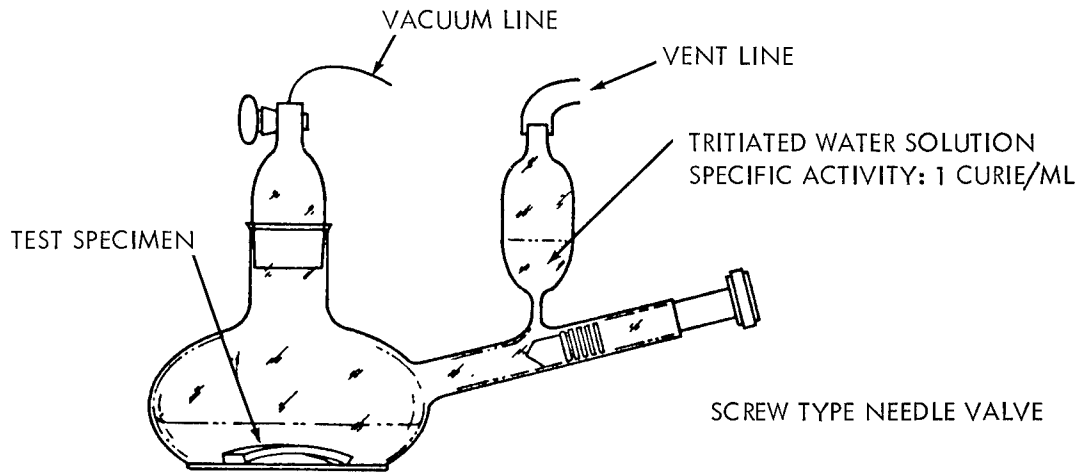


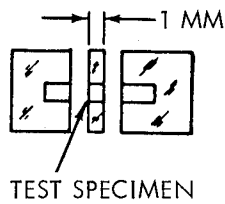
FIG. 2 SCHEMATIC REPRESENTATION OF NOL RING TEST SPECIMEN

STEP 1. IMMERSE SECTION OF FATIGUE TEST SPECIMEN IN RADIOACTIVE TRACER SOLUTION

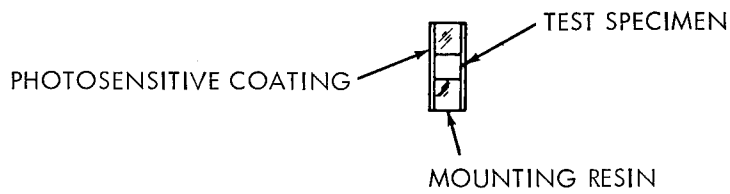


STEP 2. REMOVE AND WASH SPECIMEN

STEP 3. MOUNT SPECIMEN IN CASTING RESIN AND CUT SPECIMEN FOR ANALYSIS



STEP 4. APPLY PHOTSENSITIVE COATING AND STORE FOR EXPOSURE



STEP 5. PHOTOGRAPH AUTORADIOGRAM FOR PERMANENT RECORD

FIG. 3 PROCEDURE FOR RADIOACTIVE TRACER ANALYSIS OF NOL RING SAMPLE

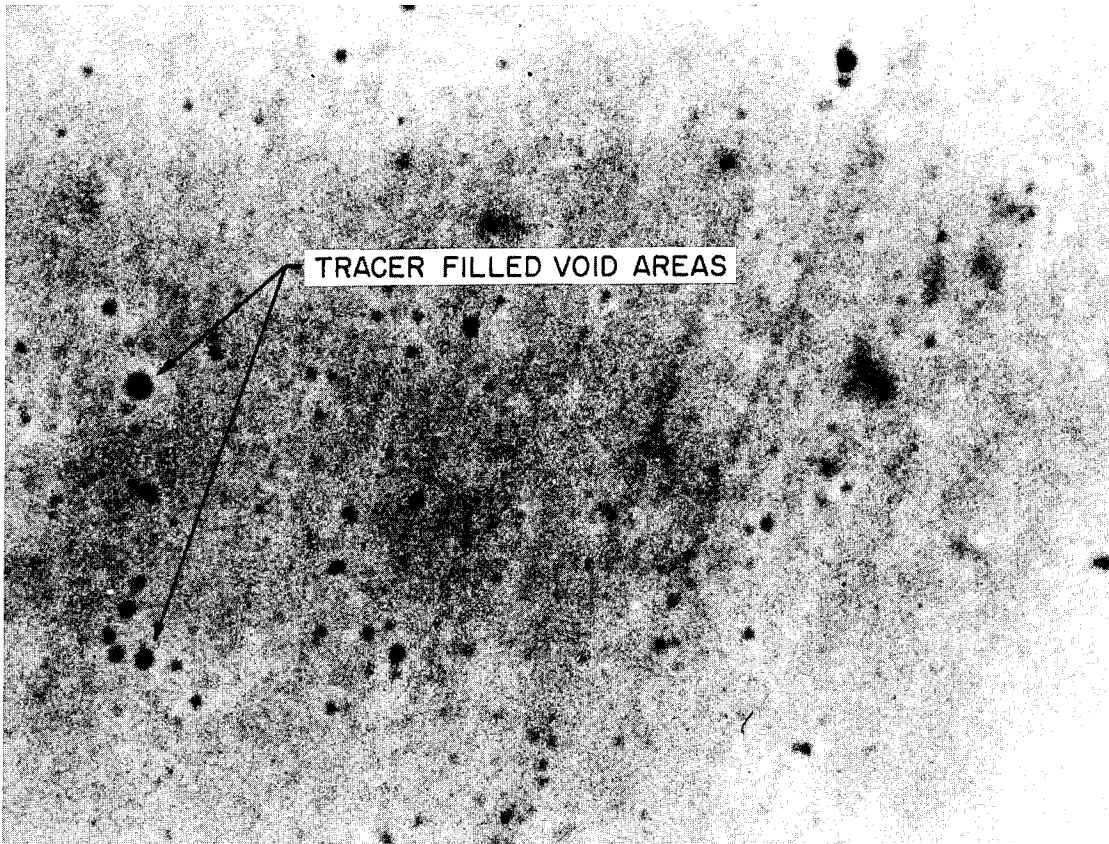


FIG. 4 AUTORADIOGRAM OF NOL RING SEGMENT (NO FATIGUE TEST CONDITIONING)
(35 X)

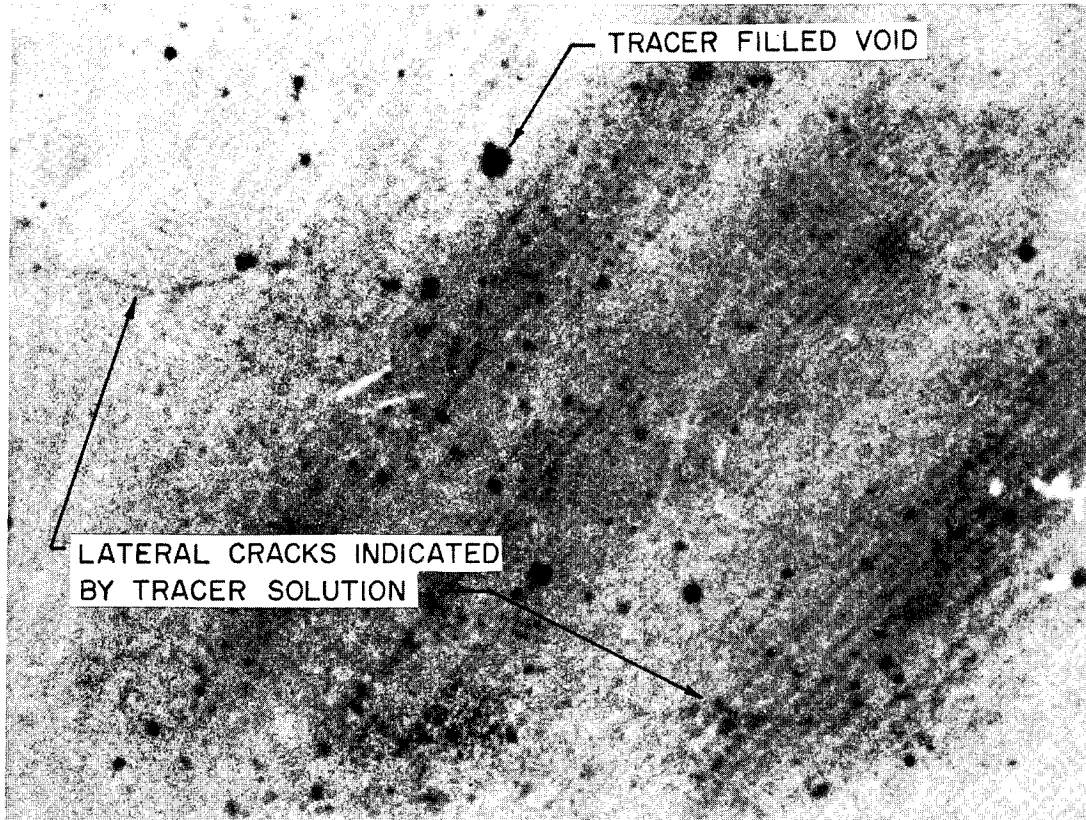


FIG. 5 AUTORADIOGRAM OF AN NOL RING SEGMENT AFTER 500 CYCLES OF FATIGUE TESTING AT 10CPM (35X)

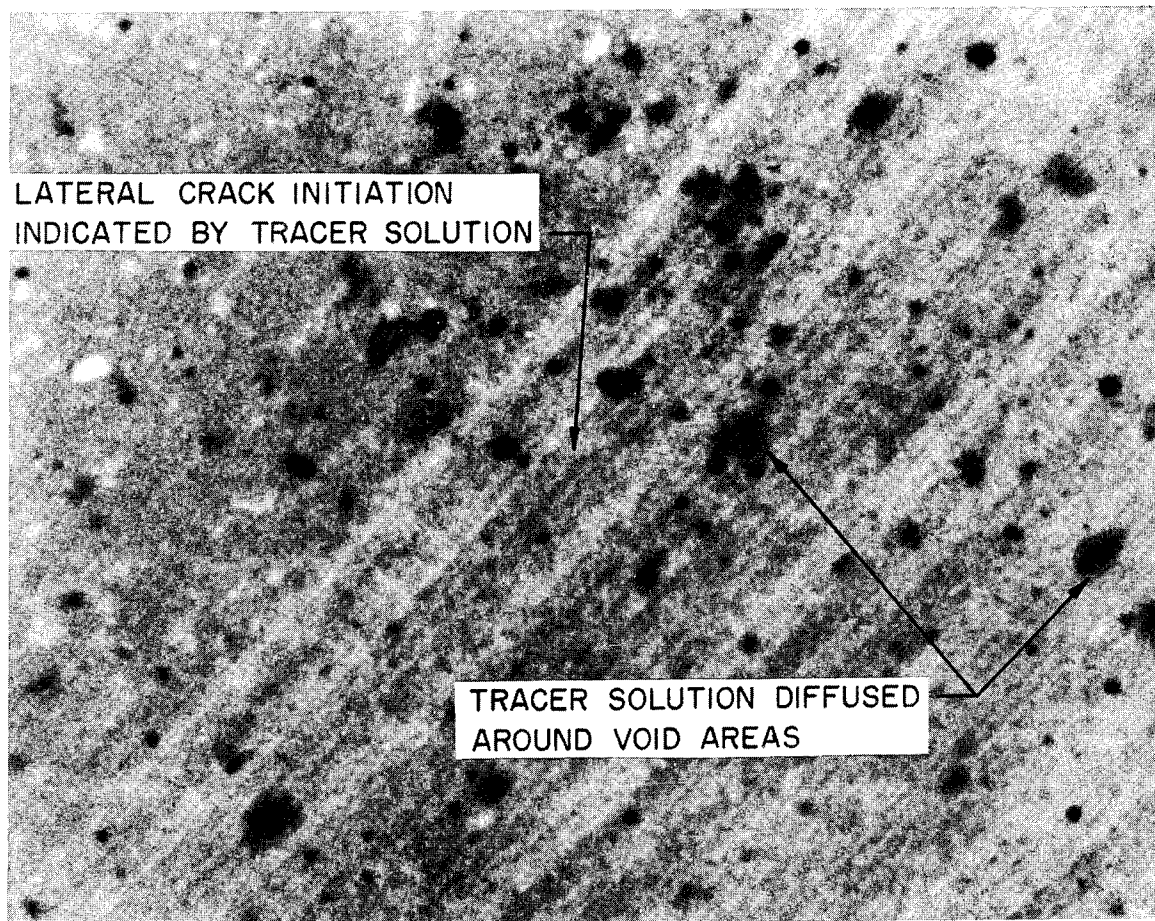


FIG. 6 AUTORADIOGRAM OF NOL RING SEGMENT AFTER 500 CYCLES OF FATIGUE TESTING
AT 100 CPM (35 X)

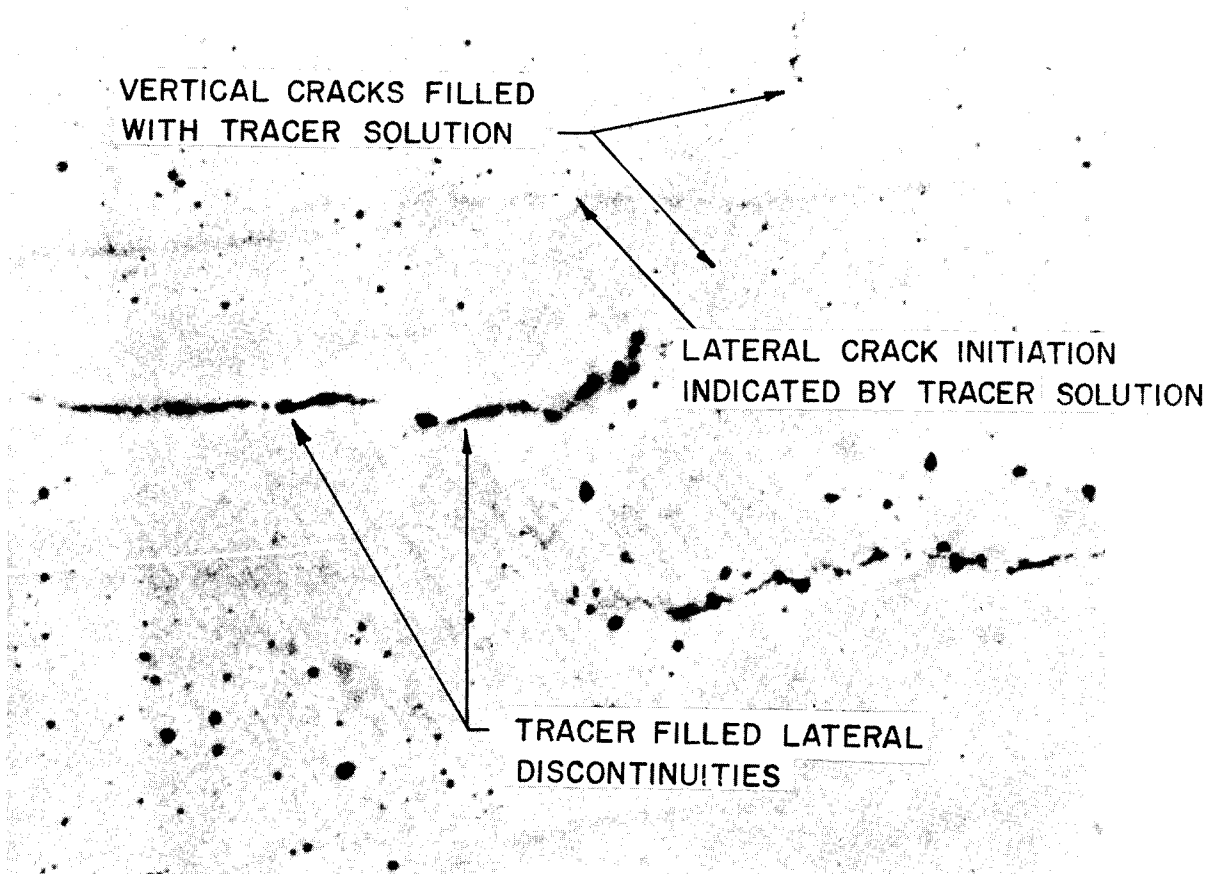


FIG. 7 AUTORADIOGRAM OF NOL RING SEGMENT AFTER 5000 CYCLES OF FATIGUE TESTING AT 100 CPM (35X)

NOLTR 66-2

BACKGROUND RADIATION

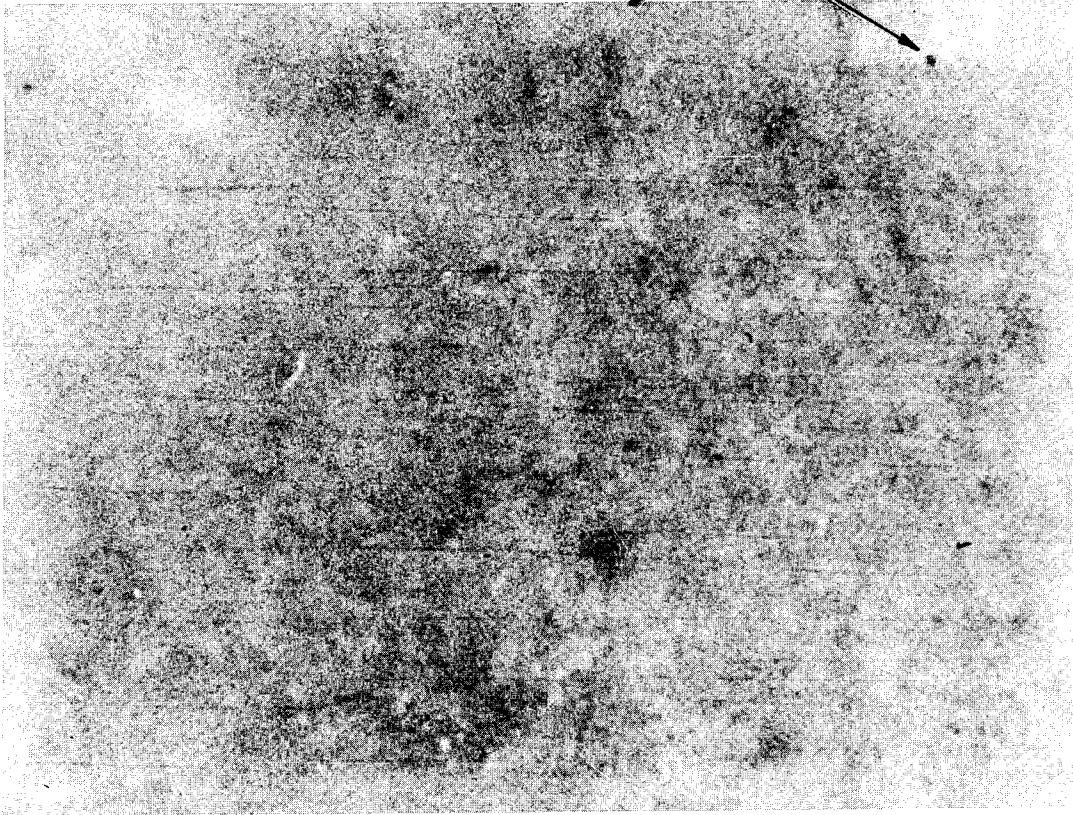


FIG. 8 AUTORADIOGRAM OF NOL RING SEGMENT AFTER 5000 CYCLES OF FATIGUE TESTING AT 100 CPM (ORDINARY WATER)(35 X)

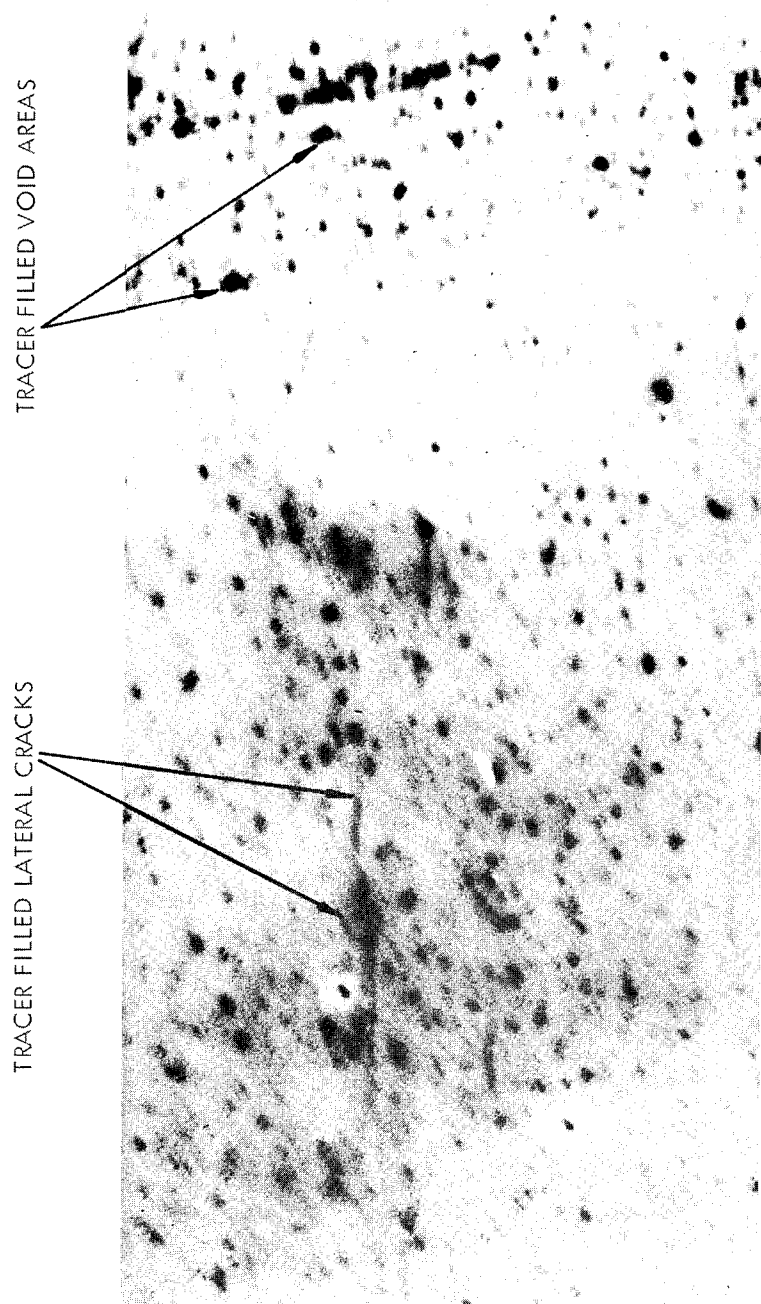


FIG. 9 AUTORADIOGRAM OF NOL RING FABRICATED WITH PREIMPREGNATED ROVING (35 X)

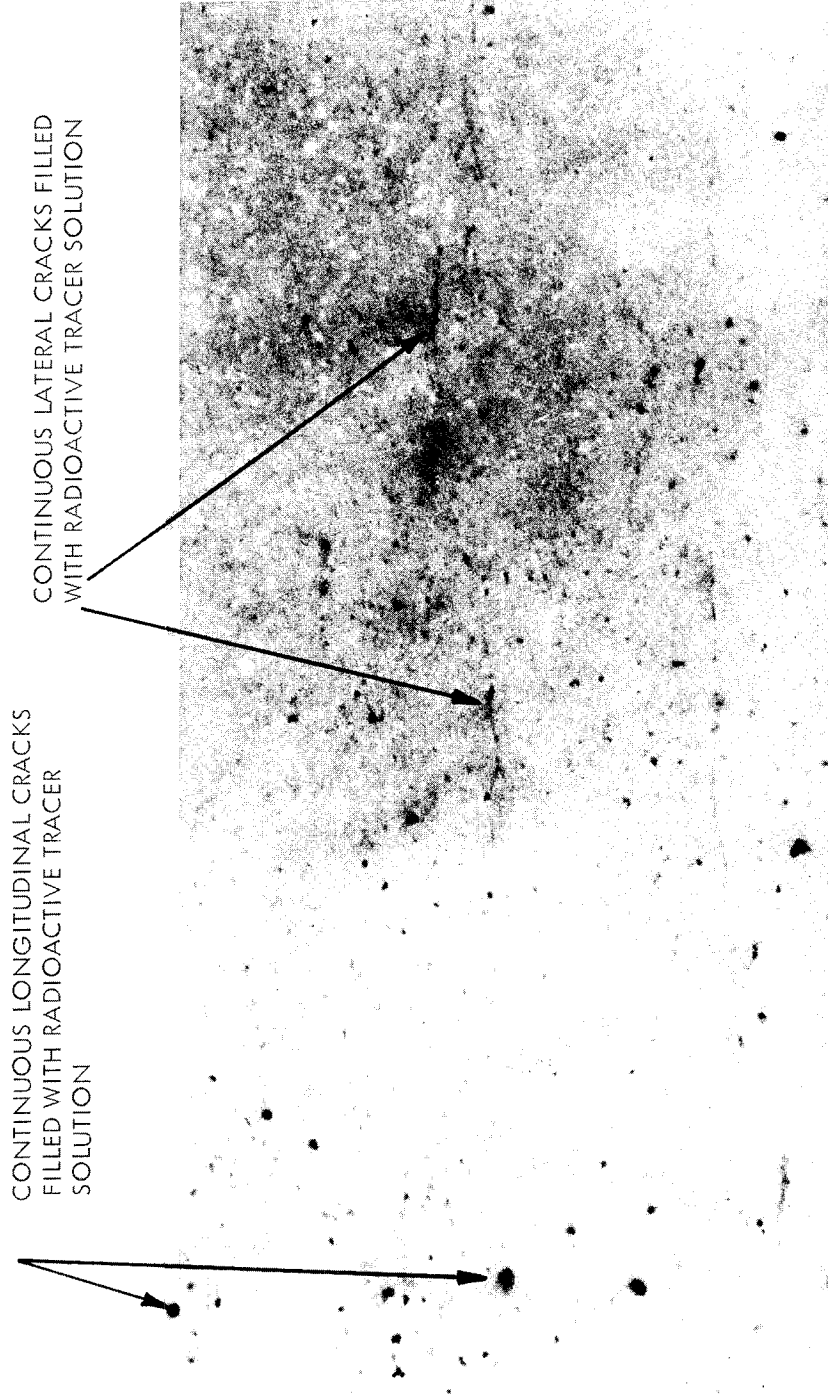


FIG. 10 MIRROR-IMAGE AUTORADIOGRAM OF NOL RING FABRICATED WITH PRE-IMPREGNATED ROVING (35 X)

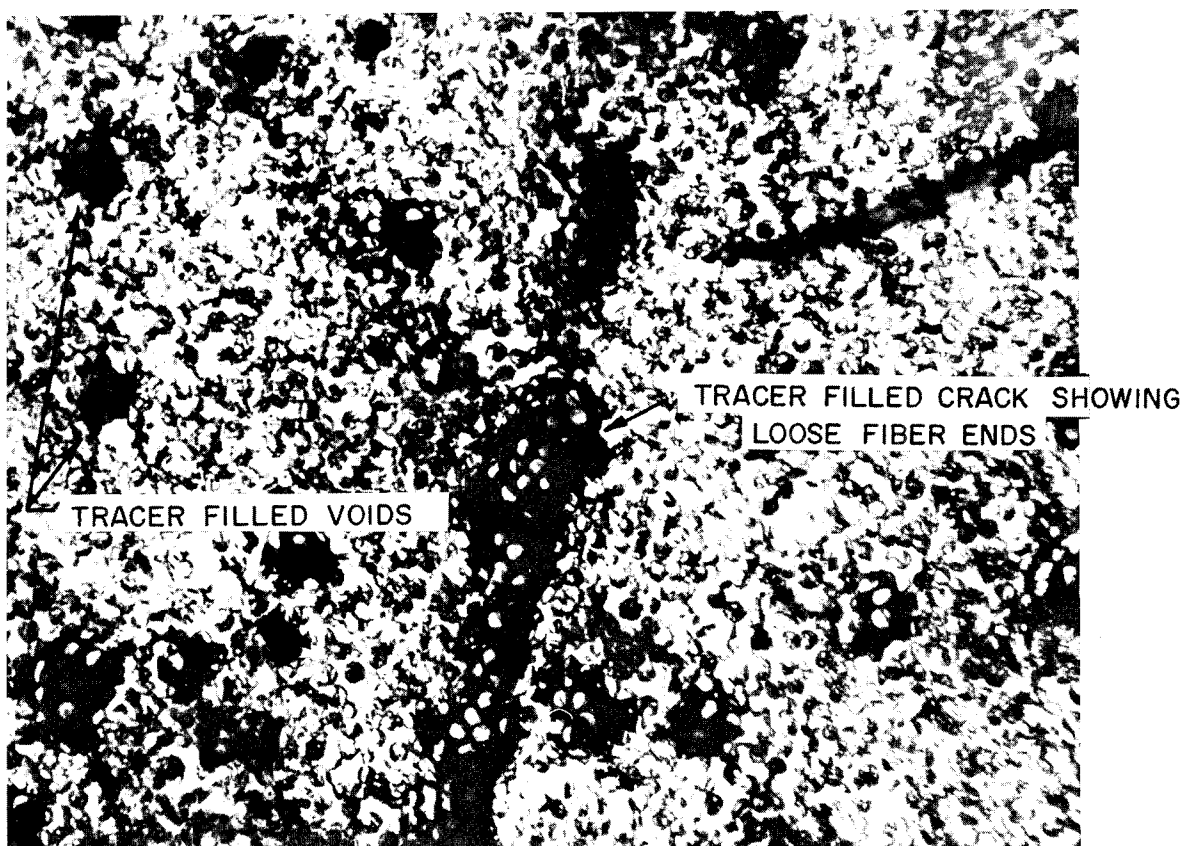


FIG. 11 PHOTOMICROGRAPH OF AUTORADIOGRAMMED NOL RING
CROSS-SECTION (400 X)

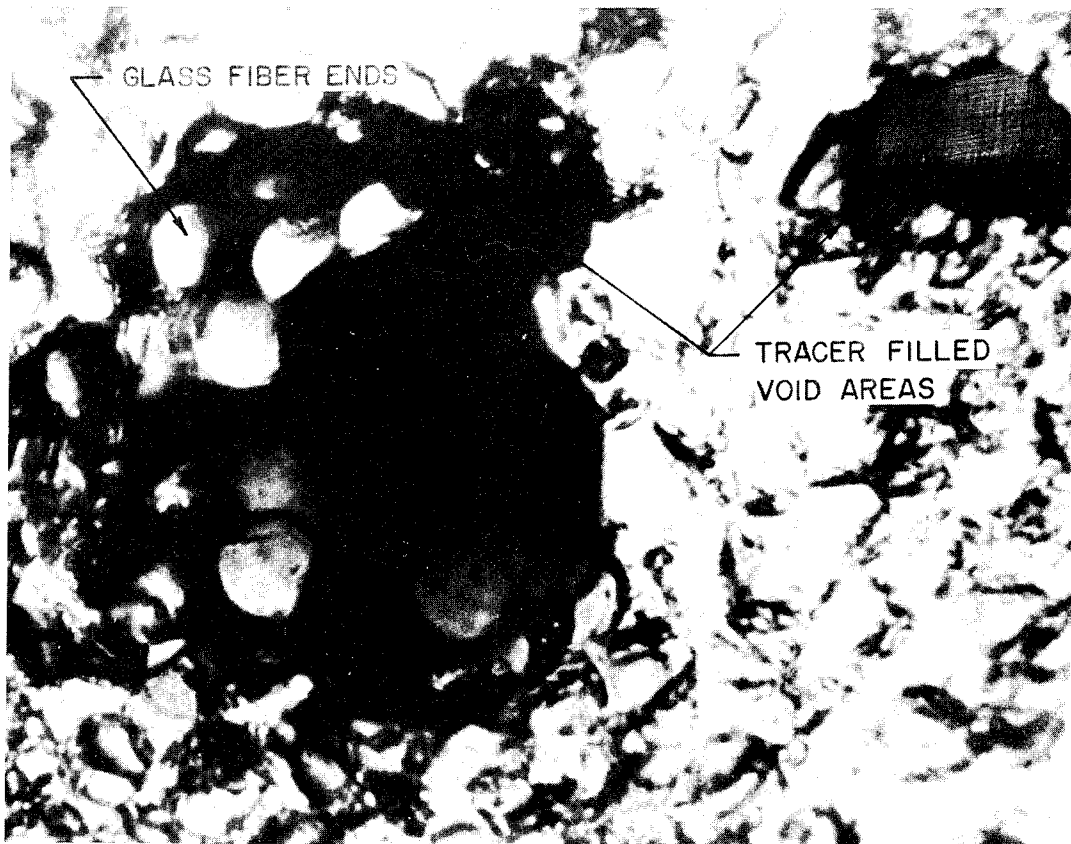


FIG. 12 PHOTOMICROGRAPH OF AUTORADIOGRAMMED NOL RING
CROSS-SECTION (1800 X)

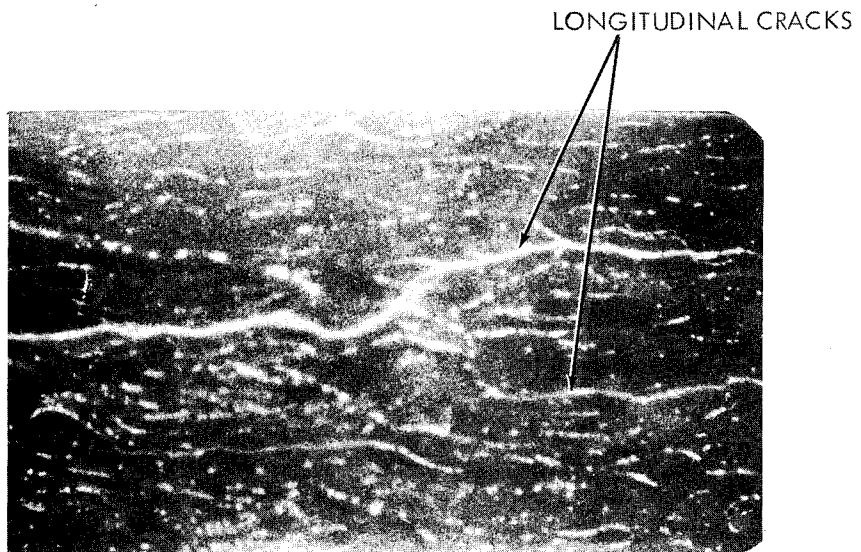


FIG. 13 PHOTOMICROGRAPH OF NOL RING SEGMENT AFTER 5000 CYCLES OF FATIGUE TESTING AT 100 CPM (35 X)

DISTRIBUTION

Copies

Chief, Bureau of Naval Weapons

RMMP-11 (I. Silver)
RMMP-23 (S. Matesky)
RRMA (N. E. Promisel)
RRMA-10 (P. Goodwin)
RRMA-3 (P. Stone)
RRMA-32 (M. Stander)
RRMA-34 (C. Bersch)
RRMA-35 (J. Gurtowski)
DIS-3 (Library)

4

Chief, Bureau of Ships (Code 210L)

Code 634-C (W. Graner)
(J. Alfors)

Director, Special Projects Office

SP-001 (Dr. J. P. Craven)
SP-2711 (H. Bernstein)
SP-2731 (R. Gott)

Chief of Naval Research

Navy Department

Washington, D. C. 20360

Attn: Dr. J. Shenk
Dr. H. Liebowitz

Director, U. S. Naval Research Laboratory

Washington, D. C. 20390

Attn: J. Kies
Dr. I. Wolock, Code 6213
J. Cowling

Commanding Officer and Director

David W. Taylor Model Basin

Washington 7, D.C.

Attn: K. Hom, Code 732
A. Stavovy, Code 725

Chief, Bureau of Yards and Docks (Code 41.236A)

Navy Department

Washington, D.C. 20360

U. S. Navy Applied Science Laboratory (Code 9350)

New York Naval Shipyard

Naval Base

Brooklyn 1, New York

NOLTR 66-2

DISTRIBUTION

Commander, Mare Island Naval Shipyard
Vallejo, California

Commander, Philadelphia Naval Shipyard (Code 254)
Naval Base
Philadelphia, Pennsylvania

U. S. Naval Electronics Laboratory
San Diego, California

U. S. Naval Marine Engineering Laboratory
Annapolis, Maryland

U. S. Naval Underwater Sound Laboratory
New London, Connecticut

Commander, U. S. Naval Ordnance Test Station
China Lake, California
Attn: S. Herzog - 5557

Commander, U. S. Naval Air Development Center
Johnsville, Pennsylvania

Director, Air Force Materials Laboratory
Wright-Patterson AFB, Ohio 45433
Attn: MAG
MANC (Plastics and Composites Branch)
MANC (S. Litvak)
ASRCE-22 (T. Reinhart)

Director, Air Force Office of Scientific Research
T-D Building
Washington, D. C. 20333
Attn: SRIL

Office of the Chief of Ordnance
Department of the Army
Pentagon Building
Washington, D. C. 20350
Attn: ORDTA
ORDTE
ORDTB

Commanding Officer, Picatinny Arsenal
Dover, New Jersey 07801
Attn: Plastics Laboratory

DISTRIBUTION

	<u>Copies</u>
U. S. Army Engineer Research and Development Laboratories Ft. Belvoir, Virginia Attn: STINFO Branch	2
Director, Plastics Technical Evaluation Center Picatinny Arsenal Dover, New Jersey 07801	2
Director, National Aeronautics and Space Administration 1512 H Street, N. W. Washington 25, D. C. Attn: Bernard Achhammer	
Director, Forest Products Laboratory Madison, Wisconsin 53705	
Narmco Research and Development Division Whittaker Corporation 3540 Aero Court San Diego, California Attn: B. Duft	
IIT Research Institute Technology Center 10 West 35th Street Chicago, Illinois 60616 Attn: R. Cornish	
H. I. Thompson Fiberglass 1600 W. 135th Street Gardena, California Attn: N. Meyers	
Owens-Corning Fiberglas Corporation 806 Connecticut Avenue, N.W. Washington, D.C. Attn: R. J. Weaver	
Shell Chemical Company Plastics and Resin Division 110 W. 51st Street New York 20, N. Y. Attn: Barton C. Brown	

DISTRIBUTION

Goodyear Aerospace Corporation
1210 Massillon Road
Akron 15, Ohio
Attn: R. A. Burkeley, Dept. 481

University of Illinois
Department of T and AM
Urbana, Illinois
Attn: Professor H. T. Corten

Minnesota Mining and Manufacturing Co.
Reinforced Plastics Division
900 Fauquier Avenue
St. Paul, Minnesota 55104
Attn: J. William Davis

Monsanto Research Corporation
800 North Lindberg Boulevard
St. Louis, Missouri 63166
Attn: Dr. Lucius Gilman

Ferro Corporation
4150 E. 56th Street
Cleveland 5, Ohio
Attn: R. W. Pelz

Mellon Institute
4400 Fifth Avenue
Pittsburgh 13, Pennsylvania
Attn: T. E. Doyle

Thiokol Chemical Company
Plant Site, Mail Stop 620
Development Laboratory
Brigham City, Utah 84302
Attn: Mike Wright

U. S. Polymeric Chemicals, Inc.
700 E. Dyer Road
Santa Ana, California
Attn: J. A. Martenson

Department of Aerospace Engineering
Polytechnic Institute of Brooklyn
333 Jay Street
Brooklyn 1, New York
Attn: Dr. J. Kempner

DISTRIBUTION

University of Dayton
Research Institute
Dayton 9, Ohio
Attn: Research Librarian

Johns-Manville Fiber Glass Division
Waterville, Ohio
Attn: J. Gilbert Mohr

Materials Research
P. O. Box 428
Warsaw, Indiana
Attn: R. H. Bradt, Director of Research

Materials Research Laboratory, Inc.
22333 Governors Highway
Richton Park, Illinois
Attn: Dr. E. J. Ripling

Jones-Dabney Company
15th and Lytle Streets
Louisville, Kentucky
Attn: David Shimp

B. F. Goodrich Company
1112 19th Street, N. W.
Washington, D. C.
Attn: Charles R. Collins

General Technology Corporation
708 N. West Street
Alexandria 14, Virginia
Attn: Lawrence Davies

Pittsburgh Plate Glass Company
One Gateway Center
Pittsburgh 22, Pennsylvania
Attn: J. F. Corey

National Aeronautics and Space Administration
Langley Station
Hampton, Virginia
Attn: R. A. Pride, Mail Stop 188

NOLTR 66-2

DISTRIBUTION

Copies

Air Force Flight Development Center (FDCL)
Wright-Patterson Air Force Base
Dayton, Ohio 45433
Attn: H. W. Basham

U. S. Atomic Energy Commission
Washington, D. C. 20545
Attn: N. Bassin

Martin Company
Baltimore Division
Mail No. 836
Baltimore, Maryland 21203
Attn: N. E. Campbell

Ohio State University
OSU Nuclear Reactor Laboratory
1298 Kinneau Road
Columbus, Ohio 43212
Attn: W. E. Carey

National Bureau of Standards
Washington, D. C. 20234
Attn: J. A. Coleman
L. Costrell

Bureau of Naval Weapons
Washington, D.C.
Attn: S. Englander

Goddard Space Flight Center
Greenbelt, Maryland 20771
Attn: Joseph Epstein

NASA Langley Research Center
Langley Station
Hampton, Virginia 23365
Attn: J. J. Olivero

Defense Documentation Center
Cameron Station
Alexandria, Virginia

20

NOLTR 66-2

DISTRIBUTION

Copies

Chief, Systems Engineering Development Section
Radiation Application Branch
Division of Isotopes Development
Washington, D.C. 20545
Attn: Dr. G. J. Rotariu

2

Atlantic Research Corporation
Shirley Highway at Edsall Road
Alexandria, Virginia
Attn: Mr. Joseph Kivel, Supr.
Nuclear Laboratory

Air Force Flight Dynamics Laboratory
Wright Patterson Air Force Base
Dayton, Ohio 45433
Attn: Dr. Paul Polishuk

Division of Isotopes Development
U. S. Atomic Energy Commission
Washington, D. C. 20545
Attn: Mr. G. C. Dempsey

Oak Ridge National Laboratory
P. O. Box X
Oak Ridge, Tennessee
Attn: P. S. Baker

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R&D		
<i>(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)</i>		
1. ORIGINATING ACTIVITY (Corporate author) U. S. Naval Ordnance Laboratory White Oak, Silver Spring, Maryland		2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED
		2b. GROUP
3. REPORT TITLE An Autoradiographic Technique for Studying Crack Growth in Plastics Composite Materials		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Interim		
5. AUTHOR(S) (Last name, first name, initial) Kinna, Marlin A.		
6. REPORT DATE 1 April 1966	7a. TOTAL NO. OF PAGES 20	7b. NO. OF REFS 10
8a. CONTRACT OR GRANT NO. RREN-ST-204/212-5/0000	9a. ORIGINATOR'S REPORT NUMBER(S) NOLTR 66-2	
b. PROJECT NO.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) -	
c.		
d.		
10. AVAILABILITY/LIMITATION NOTICES Distribution of this document is unlimited.		
11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Bureau of Naval Weapons	
13. ABSTRACT Preliminary work has been conducted on segments of NOL Rings to study crack propagation characteristics in fiber reinforced plastics composite materials. Samples were submitted to a variety of fatigue loading conditions. They were then immersed in a tritiated water-ethylene glycol solution and after suitable processing, autoradiograms were prepared for analysis. Radiation emanating from the solution retained by the samples indicated that minute cracks and fissures were present in all of the specimens and that the radiation intensity varied in direct proportion to the severity of the fatigue loading conditions. When photomicroscopy was used to analyze similar specimens, cracks could be detected only for the most severe test conditions.		

DD FORM 1473
1 JAN 64

Unclassified

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
plastics - reinforced plastics - fabrication composite plastics plastics testing NOL Rings density control filament wound autoradiography tracer isotopes tritium						

INSTRUCTIONS

1. **ORIGINATING ACTIVITY:** Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization (*corporate author*) issuing the report.
- 2a. **REPORT SECURITY CLASSIFICATION:** Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.
- 2b. **GROUP:** Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.
3. **REPORT TITLE:** Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.
4. **DESCRIPTIVE NOTES:** If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.
5. **AUTHOR(S):** Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.
6. **REPORT DATE:** Enter the date of the report as day, month, year, or month, year. If more than one date appears on the report, use date of publication.
- 7a. **TOTAL NUMBER OF PAGES:** The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.
- 7b. **NUMBER OF REFERENCES:** Enter the total number of references cited in the report.
- 8a. **CONTRACT OR GRANT NUMBER:** If appropriate, enter the applicable number of the contract or grant under which the report was written.
- 8b, 8c, & 8d. **PROJECT NUMBER:** Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.
- 9a. **ORIGINATOR'S REPORT NUMBER(S):** Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.
- 9b. **OTHER REPORT NUMBER(S):** If the report has been assigned any other report numbers (*either by the originator or by the sponsor*), also enter this number(s).
10. **AVAILABILITY/LIMITATION NOTICES:** Enter any limitations on further dissemination of the report, other than those

imposed by security classification, using standard statements such as:

- (1) "Qualified requesters may obtain copies of this report from DDC."
- (2) "Foreign announcement and dissemination of this report by DDC is not authorized."
- (3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through _____."
- (4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through _____."
- (5) "All distribution of this report is controlled. Qualified DDC users shall request through _____."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. **SUPPLEMENTARY NOTES:** Use for additional explanatory notes.
12. **SPONSORING MILITARY ACTIVITY:** Enter the name of the departmental project office or laboratory sponsoring (*paying for*) the research and development. Include address.
13. **ABSTRACT:** Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as (TS), (S), (C), or (U).

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. **KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.

1. Naval Ordnance Laboratory, White Oak, Md.
(NOL technical report 66-2).
AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING
CRACK GROWTH IN PLASTICS COMPOSITE MATERIALS,
by Marlin A. Kinna. 1 April 1966. 6p. illus.
BuWeaps task RREN-ST-204/212-5/0000.

UNCLASSIFIED
Preliminary work has been conducted on segments of NOL rings to study crack propagation characteristics in fiber reinforced plastics composite materials. Samples were immersed in a tritiated water-ethylene glycol solution and autoradiograms were prepared for analysis. Radiation emanating from the solution retained by the samples indicated minute cracks and fissures were present in all of the specimens, and that the radiation intensity varied in direct proportion to the severity of fatigue loading conditions.

Abstract card is unclassified.

1. Plastics,
Reinforced
2. NOL ring
test
I. Title
II. Kinna,
Marlin A.
Project
III.

2. Naval Ordnance Laboratory, White Oak, Md.
(NOL technical report 66-2).
AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING
CRACK GROWTH IN PLASTICS COMPOSITE MATERIALS,
by Marlin A. Kinna. 1 April 1966. 6p. illus.
BuWeaps task RREN-ST-204/212-5/0000.

UNCLASSIFIED
Preliminary work has been conducted on segments of NOL rings to study crack propagation characteristics in fiber reinforced plastics composite materials. Samples were immersed in a tritiated water-ethylene glycol solution and autoradiograms were prepared for analysis. Radiation emanating from the solution retained by the samples indicated minute cracks and fissures were present in all of the specimens, and that the radiation intensity varied in direct proportion to the severity of fatigue loading conditions.

Abstract card is unclassified.

1. Plastics,
Reinforced
2. NOL ring
test
I. Title
II. Kinna,
Marlin A.
Project
III.

1. Naval Ordnance Laboratory, White Oak, Md.
(NOL technical report 66-2).
AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING
CRACK GROWTH IN PLASTICS COMPOSITE MATERIALS,
by Marlin A. Kinna. 1 April 1966. 6p. illus.
BuWeaps task RREN-ST-204/212-5/0000.

UNCLASSIFIED
Preliminary work has been conducted on segments of NOL rings to study crack propagation characteristics in fiber reinforced plastics composite materials. Samples were immersed in a tritiated water-ethylene glycol solution and autoradiograms were prepared for analysis. Radiation emanating from the solution retained by the samples indicated minute cracks and fissures were present in all of the specimens, and that the radiation intensity varied in direct proportion to the severity of fatigue loading conditions.

Abstract card is unclassified.

1. Plastics,
Reinforced
2. NOL ring
test
I. Title
II. Kinna,
Marlin A.
Project
III.

2. Naval Ordnance Laboratory, White Oak, Md.
(NOL technical report 66-2).
AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING
CRACK GROWTH IN PLASTICS COMPOSITE MATERIALS,
by Marlin A. Kinna. 1 April 1966. 6p. illus.
BuWeaps task RREN-ST-204/212-5/0000.

UNCLASSIFIED
Preliminary work has been conducted on segments of NOL rings to study crack propagation characteristics in fiber reinforced plastics composite materials. Samples were immersed in a tritiated water-ethylene glycol solution and autoradiograms were prepared for analysis. Radiation emanating from the solution retained by the samples indicated minute cracks and fissures were present in all of the specimens, and that the radiation intensity varied in direct proportion to the severity of fatigue loading conditions.

Abstract card is unclassified.

1. Plastics,
Reinforced
2. NOL ring
test
I. Title
II. Kinna,
Marlin A.
Project
III.