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FRACTOGRAPHY OF POLYTETRAFLUOROETHYLENE (PTFE)

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ABSTRACT

This study is an attempt to apply the science of fractography of metallic materials to a polymeric material. The material used in this study was polytetrafluoroethylene (PTFE). The approach to the study was to plan an investigation where controlled laboratory failures of PTFE would serve as standards for comparison to the unknown of a failure. These standards were planned for ambient and liquid nitrogen temperatures. The results demonstrated that the science of fractography is applicable to PTFE; further, that the failure environment of PTFE can be determined by using fractography.

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By

B. H. Nerren

MATERIALS ANALYSIS SECTION
APPLIED TECHNOLOGY BRANCH
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ACKNOWLEDGEMENT

A special thanks goes to Earl Hasemeyer, Harvey Siler Jr., and Fred Owens for their help in preparing samples used in this study.

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FRACTOGRAPHY OF

POLYTETRAFLUOROETHYLENE (PTFE)

SUMMARY

Polytetrafluoroethylene (PTFE) has been in use for some time with only limited information available on its microstructure and fracture characteristics.

The need for information of this type was realized when a PTFE seal fractured in a Parker lox fill and drain valve resulting in a leak in the valve. This valve was installed in a Saturn flight vehicle at the time of failure.

To answer some of the unknowns an investigation was planned where controlled laboratory failures of PTFE served as standards for comparison to the unknown failure. These standards were planned for ambient as well as liquid nitrogen temperatures.

The results of this investigation indicate that the failure mode of PTFE can be determined using fractography; the failure environment may also be determined.

SECTION I. INTRODUCTION

Thorough failure analysis and subsequent timely preventive redesign or selection of alternate materials is the keynote to successful prevention of more failures, schedule slippage, excessive funds expenditures, etc. Most all equipment and structures failures, functional or physical, are evidenced by materials failure. The need for thorough failure analysis of materials is most evident in the aerospace industry where so much is dependent on successful performance throughout a complex graduated sequence of fabrication, assembly, and testing operations.

The techniques for analyzing failures in metals are well established and a wealth of historical data on failure modes is available. This is not necessarily the case in non-metallic materials. Many of these materials are relatively new and are constantly being applied in areas where no background data are available. This particular void in our knowledge was recently pointed out when a Teflon® (polytetrafluoroethylene) seal failed in a lox fill and drain valve. In an effort to fill this void, this study was undertaken. The purpose of the study being to determine if fractography, a commonly used tool in metallurgical failure analysis, could be applied to failure studies on PTFE.

SECTION II. EXPERIMENTAL DETAILS

In fractography, the fracture face of a failed material is studied with the aid of an electron microscope. The minute topographical features revealed by this instrument are then compared with standards which were fractured under controlled conditions. Similarities between service failures and the standards indicate the failure mode.

This study was designed to produce a set of standards that could be used to study failures in PTFE. The conditions selected for standards are given in Table I. These conditions approximate the stress and temperature environments to which various space systems hardware such as the fill and drain valves may be exposed.

Table I. Test Parameters

Sample	Test Mode	Room Temp.	LN_2 Temp.
Notched	Tensile	72°F	-319°F
Unnotched	Tensile	72°F	-319°F
Notched	Fatigue	72°F	-319°F
Unnotched	Fatigue	72°F	

Unnotched samples were not tested in the fatigue mode at LN_2 temperature, since it was evident from the results of ambient temperature testing that such tests were inadequately contributive.

Standard specimens were prepared from PTFE sheet stock, 1/4-inch thick, according to the drawings in Figure 1. Tensile tests were performed on an Instron Tester and fatigue tests were performed on a Materials Testing System (MTS) low cycle fatigue machine. Replicas of the fractured surfaces were prepared by the commonly used two stage acetate replica technique. Initial work was attempted using the direct carbon replica, but difficulty experienced in removing these replicas precluded the use of this technique.

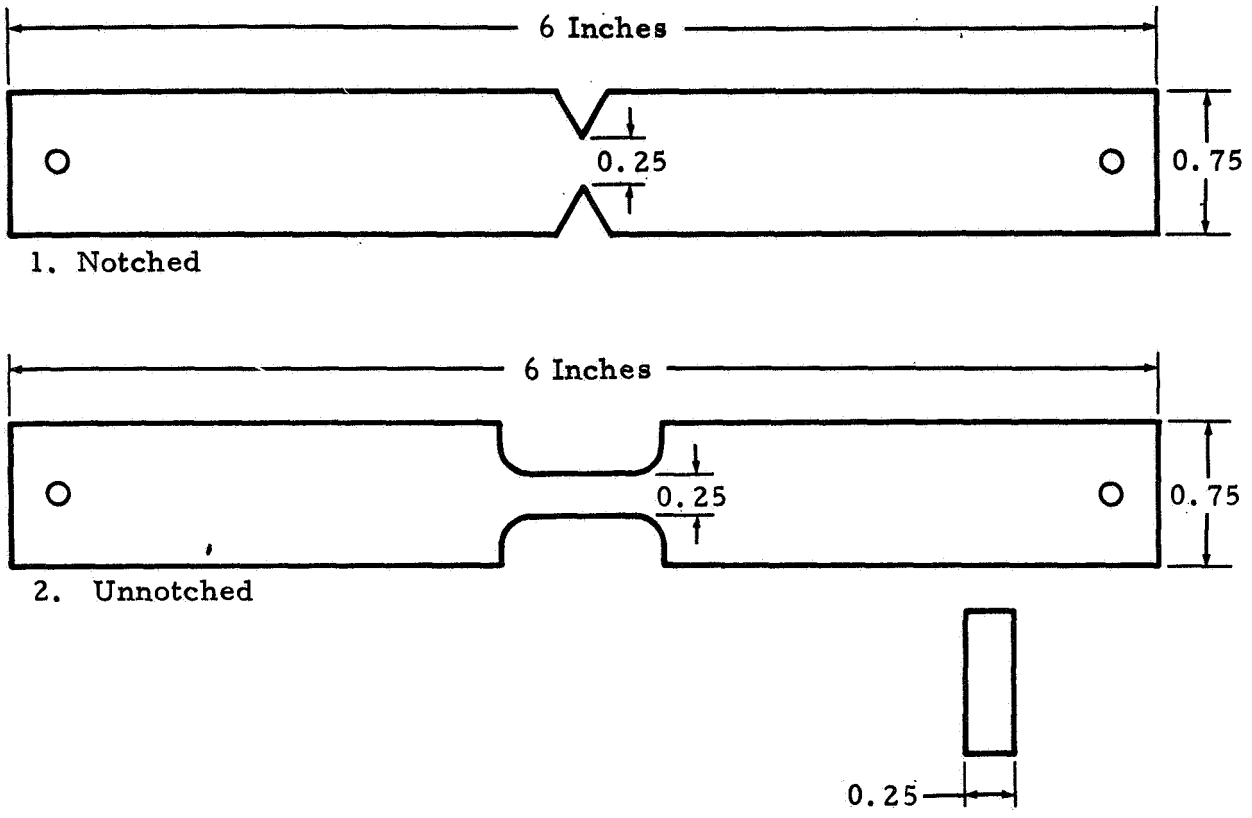


Figure 1. PTFE Sample Design

SECTION III. DISCUSSION

All fractures were studies in two magnification ranges: The macro-range, from 1 to 10X, and the micro-range, from 3500 to 15,000X. The various topographical features noted on the fracture faces were defined in terms of some observable characteristic, i.e., chevrons, dimples, striations, and tabulated for all samples. These tabular data are given in Table II.

Table II. Fractography of PTFE

Type Loading	Sample Condition	72°F Rm Temp	Fig.	Appx Page	Sample Code	-319°F Cryogenic	Fig.	Appx Page	Sample Code
Macro (1 - 10X)									
Tensile	Notched	Tear Ridges, Convex	2	A-2	NTA	Flat Fracture	6	A-10	$NTLN_2$
	Unnotched	Chevrons, Flat Fracture	4	A-4	UTA	Flat Fracture	6	A-12	$UTLN_2$
Fatigue	Notched	Tear Ridges, Concave	3	A-6	NFR	Flat Fracture	6	A-14	$NFLN_2$
	Unnotched	Chevrons, Flat Fracture	5	A-8	UFR	-	-	-	-
Micro (3500 - 15,000X)									
Tensile	Notched	Tear Ridges, Dimple	7, 8	A-3	NTA	Flat Fracture, Dimple, Crystals	7, 9	A-11	$NTLN_2$
	Unnotched	Tear Ridges, Dimple	7, 8	A-5	UTA	Flat Fracture, Dimple, Crystals	7, 9	A-13	$UTLN_2$
Fatigue	Notched	Striations, Dimple	10	A-7	NFR	Flat Fracture, Crystals, Striations, Dimple	10	A-15	$NFLN_2$
	Unnotched	Striations, Dimple	10	A-9	UFR	-	-	-	-

Basic observations revealed that the configuration of samples tested, either notched or unnotched, was the primary factor effecting the macrotopography of specimens fractured at ambient temperatures. In the case of notched sample, Figures 2 and 3, the area at the root of the notch and the area at the center of the specimen are distinguishable. It is felt that the areas at the root of the notch are zones where slow crack growth occurred prior to failure. The center area was formed during final, catastrophic failure of the specimen.

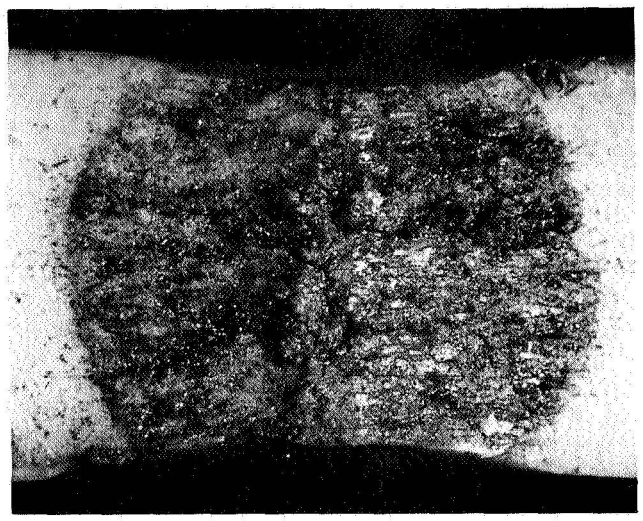


Figure 2. Macrograph of Notched, Ambient, Tensile Fracture

Figure 3. Macrograph of Notched, Ambient, Fatigue Fracture

In the case of the unnotched specimens, the macroappearance of the fracture was the same at ambient temperatures (Figures 4 and 5). Under both tensile and cyclic loading the fracture was fairly flat with chevrons pointing to the origin of failure.

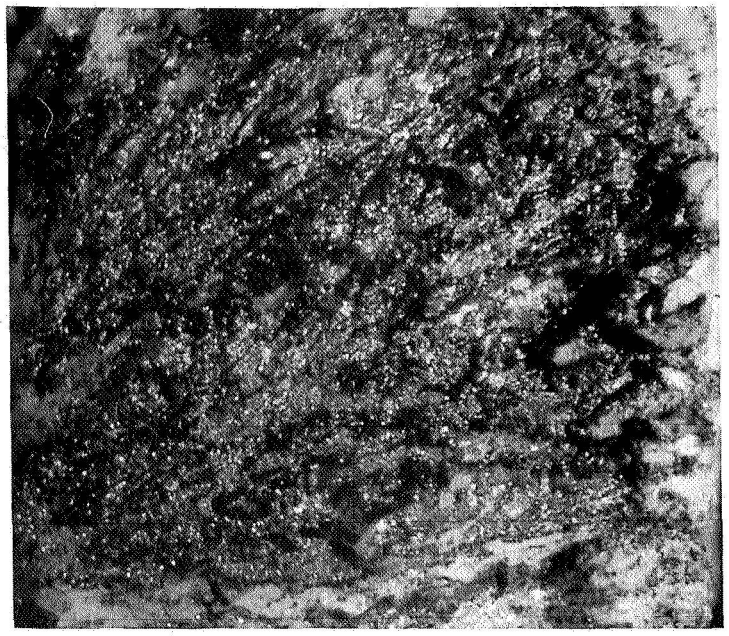


Figure 4. Macrograph of Unnotched, Ambient, Tensile Fracture

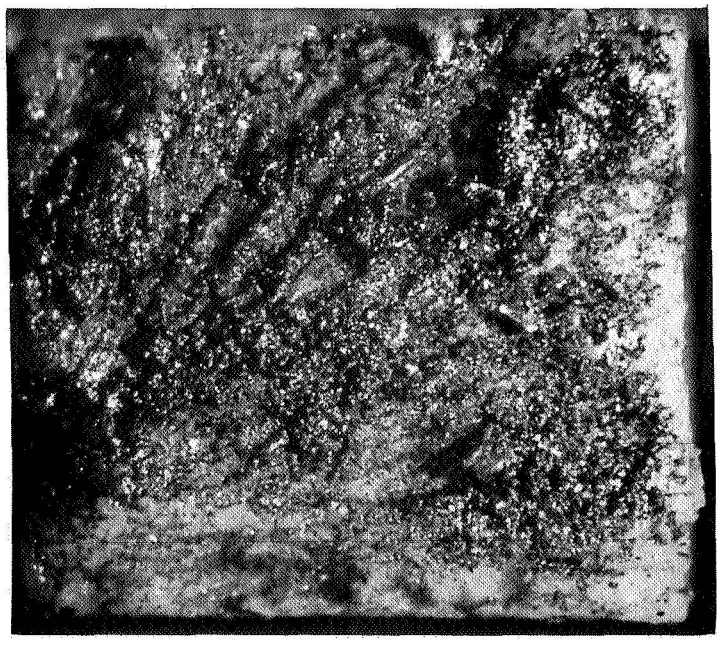


Figure 5. Macrograph of Unnotched, Ambient, Fatigue Fracture

The macrotopography of samples fractured at cryogenic temperature appeared to be unaffected by sample type or stress environment. All samples fractured at cryogenic temperatures exhibited the flat, fine grained texture shown in Figure 6.

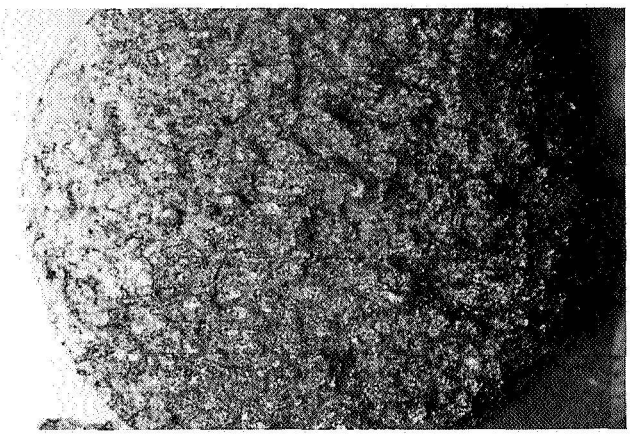


Figure 6. Macrotopography of Fracture at Cryogenics

Electron microscopy studies indicated that the microtopographical features were influenced primarily by the stress and temperature environments and were essentially independent of sample configuration. Notched and unnotched tensile failure exhibited dimple structure (Figure 7) on both ambient and cryogenic specimens.

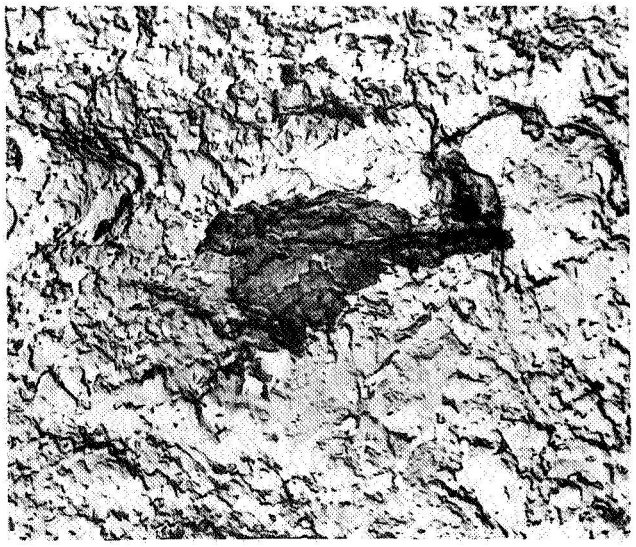


Figure 7. Electron Micrograph of Tensile Failure Exhibiting Dimples

The samples fractured under ambient condition also exhibited tear ridges (Figure 8) on the fracture face. These tear ridges indicate considerable local plastic deformation prior to fracture. The fact that these ridges are not found in the cryogenic tested samples indicates that a significant decrease in ductility occurs as the testing temperature is reduced.

Crystals were also noted in samples fractured under cryogenic conditions (Figure 9).

Figure 8. Electron Micrograph Illustrating Tear Ridges

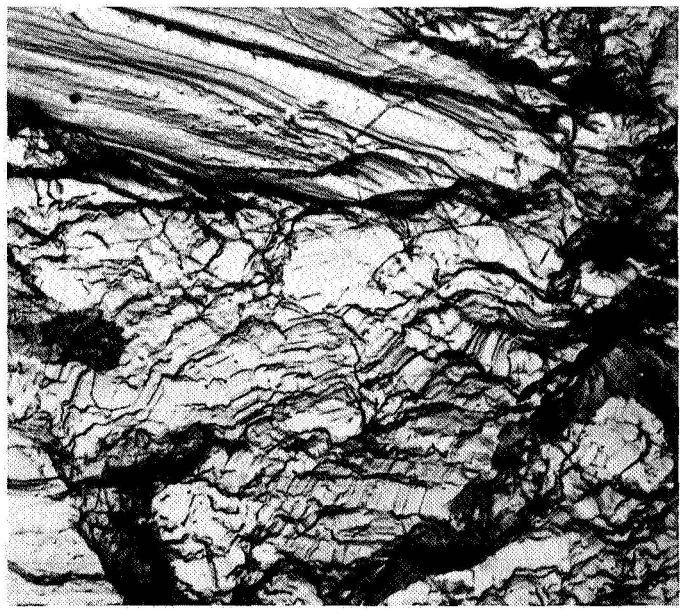


Figure 9. Electron Micrograph of Crystals

All samples which were fractured under cyclical loading exhibited striation (Figure 10). These striations are very similar to fatigue striations found on metal surfaces fractured under cyclical loading. These are probably crack arrest lines where crack propagation was momentarily stopped during stress reversal. Crystals were also noted in fatigue samples fractured at cryogenic temperatures.

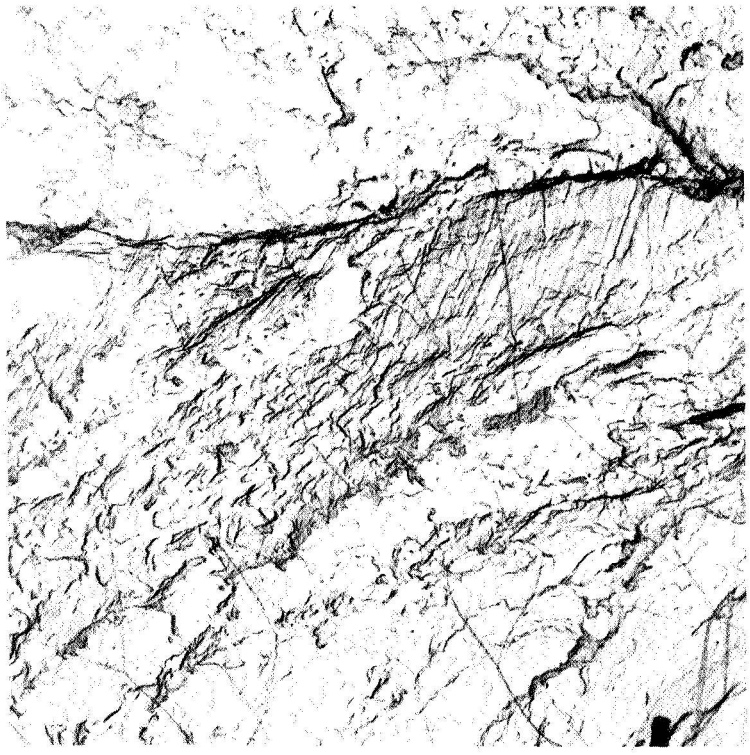


Figure 10. Electron Micrograph of Fatigued PTFE (Note Striations)

SECTION IV. CONCLUSIONS

This study indicated that fractography can be used to study fractured surfaces of Teflon to gain information concerning failure modes. This was demonstrated by the reproducibility of the topographical patterns seen during this study. In an effort to aid future failure studies, the data from this study has been arranged in a handbook format and presented in the Appendix for easy reference.

Two interesting observations were made during the course of this study: the extreme notch sensitivity of teflon and the appearance of crystals on surfaces fractured at cryogenic temperatures. Additional studies should be performed in these areas to yield a better understanding of these phenomena.

APPENDIX

PAGES APPROPRIATE FOR HANDBOOK ACCUMULATION AND LABORATORY REFERENCE

Index of Handbook

CODE	CONDITION	TEMP	ENVIRONMENT
NTA	Notched - Tension	72°F	Air
UTA	Unnotched - Tension	72°F	Air
NFR	Notched - Fatigue	72°F	Air
UFR	Unnotched - Fatigue	72°F	Air
$NTLN_2$	Notched - Tension	-319°F	Liquid Nitrogen
$UTLN_2$	Unnotched - Tension	-319°F	Liquid Nitrogen
$NFLN_2$	Notched - Fatigue	-319°F	Liquid Nitrogen

CODE NO: NTA

MATERIAL AND FORM: PTFE 1/4-Inch Sheet

LOAD AT FAILURE: 106 lbs.

CONDITION OF TEST: Notched - Tension

TEST TEMPERATURE: Ambient (72^{o}F)

TEST ENVIRONMENT: Air

TEST RESULTS:

APPEARANCE OF FRACTURE SURFACE

The fracture had its origin at the root of the notch on either edge and then propagated to the center of the test specimen forming a peak with river-like patterns pointing to the origin.

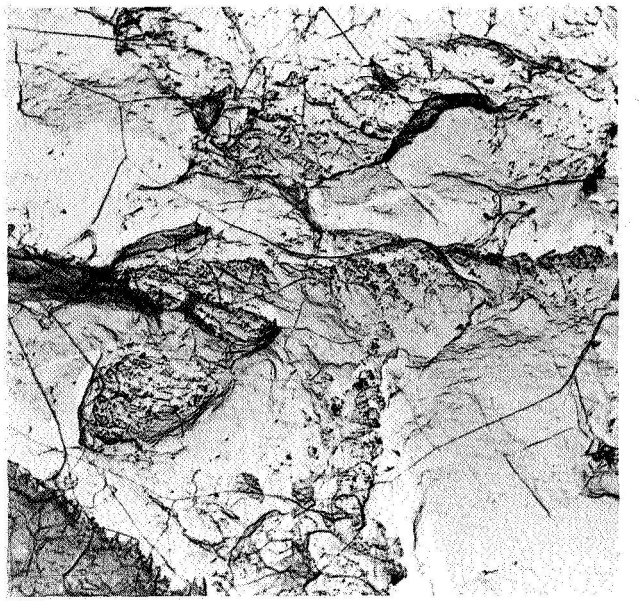
FRACTOGRAPHIC ANALYSIS

The fracture was characterized by dimples and tear ridges perpendicular to the notches.

7X

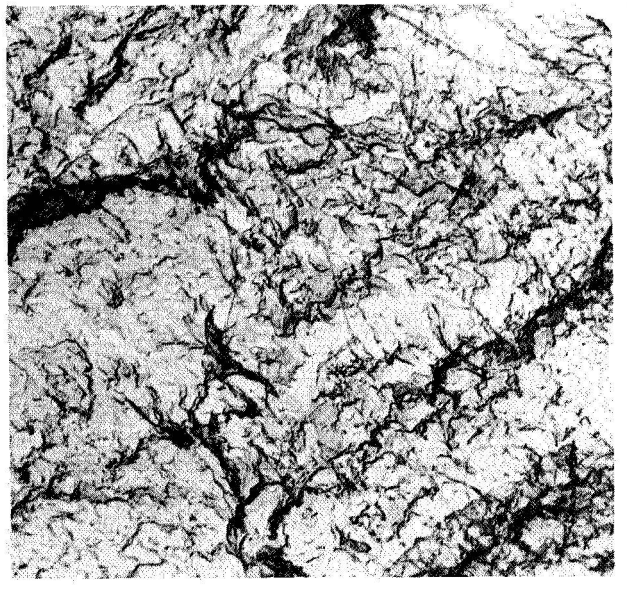
Micrographs of Fractured Surface of PTFE
at Ambient Temperature (Notched Samples) (Sheet 1 of 2)

Area A



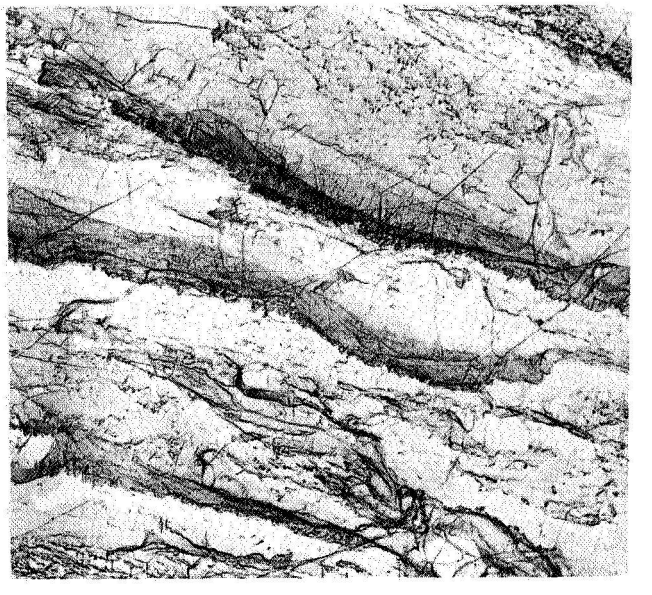
#340 4000X

Area A



#338 4000X

Area B



#341 4000X

Electron Micrographs of Fractured Surface of PTFE
at Ambient Temperature (Notched Samples) (Sheet 2 of 2)

CODE NO: UTA

MATERIAL AND FORM PTFE 1/4-Inch Sheet

LOAD TO FAILURE: 167 lbs.

CONDITION OF TEST: Unnotched - Tension

TEST TEMPERATURE: Ambient (72°F)

TEST ENVIRONMENT: Air

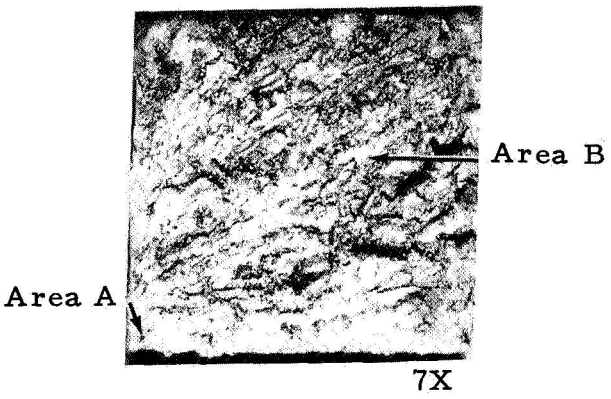
TEST RESULTS:

APPEARANCE OF FRACTURE SURFACE

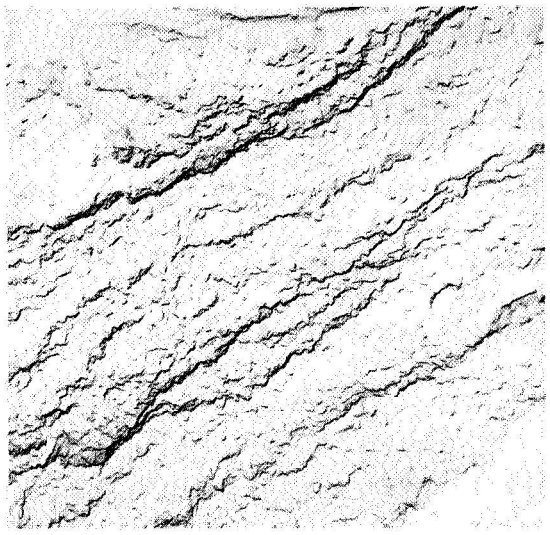
The fracture exhibited separation lines pointing to the origin of failure, similar to chevron patterns in metal failures.

FRACTOGRAPHIC ANALYSIS

The fracture exhibited tear ridges and typical PTFE tension separation patterns similar to dimples in metallic fractures.

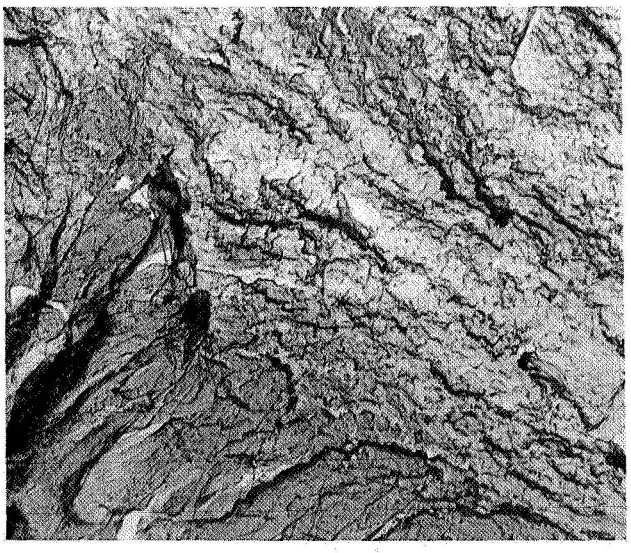


Micrographs of Fractured Surface of PTFE at Ambient Temperature (Unnotched Samples) (Sheet 1 of 2)



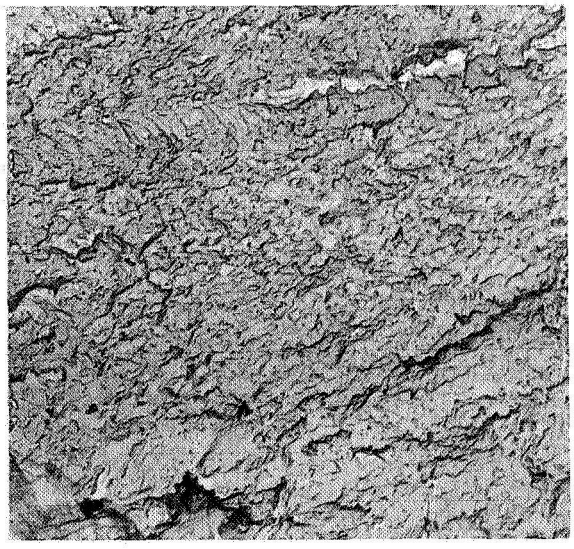
#278 4000X

Area B



#355 4000X

Area B



#357 4000X

Electron Micrographs of Fractured Surface of PTFE
at Ambient Temperature (Unnotched Samples) (Sheet 2 of 2)

CODE NO: NFR

MATERIAL AND FORM: PTFE 1/4-Inch Sheet

MAXIMUM DYNAMIC LOAD: 850 lbs.

CONDITION OF TEST: Fatigue - Low Cycle - Notched

TEST TEMPERATURE: Ambient (72^{o}F)

TEST ENVIRONMENT: Air

TEST RESULTS:

DYNAMIC PROPERTIES

Maximum Stress: 850 lbs.
Cyclic: 22.5 lbs.
Test Frequency: 3 cps
Cycles to Failure: 96,959

APPEARANCE OF FRACTURE SURFACE

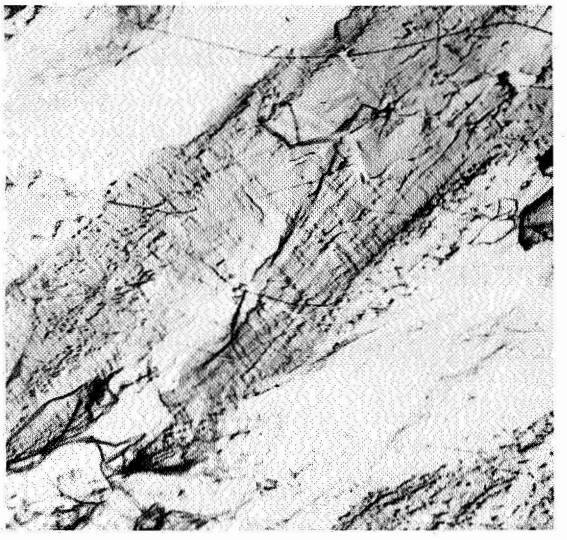
The fatigue area consisted of a relatively flat zone. Rapid fracture was by tension that formed a valley in the center.

FRACTOGRAPHIC ANALYSIS

The fatigue striations were well formed but scattered. Rapid fracture was by dimple-like rupture.

Micrographs of Fractured Surface of PTFE at Ambient Temperature, Low Cycle (Notched Sample) (Sheet 1 of 2)

Area A



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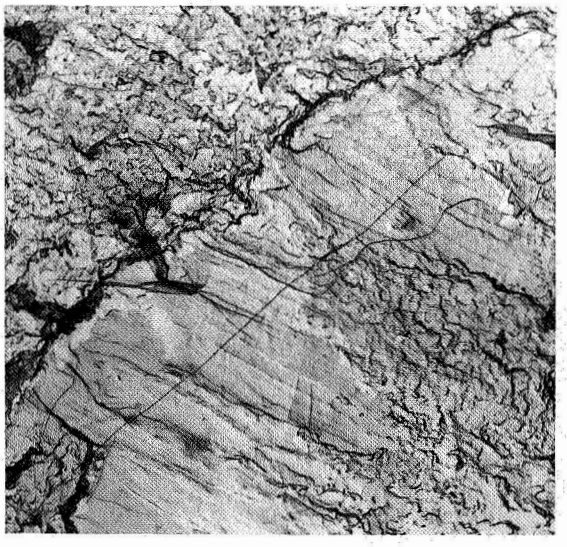
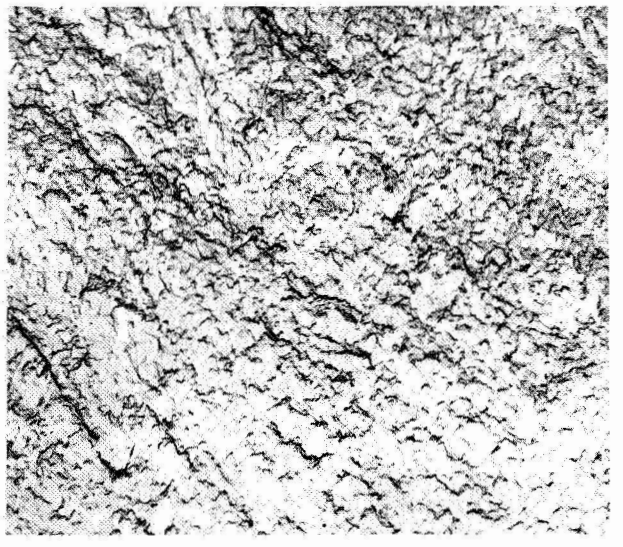
Area A



#399 4000X

Area B



#239 4000X

Electron Micrographs of Fractured Surface of PTFE
at Ambient Temperature, Low Cycle (Notched Sample) (Sheet 2 of 2)

CODE NO: UFR

MATERIAL AND FORM: PTFE 1/4-Inch Sheet

MAXIMUM DYNAMIC LOAD: 210 lbs.

CONDITION OF TEST: Fatigue - Low Cycle - Unnotched

TEST TEMPERATURE: Ambient (72^{o}F)

TEST ENVIRONMENT: Air

TEST RESULTS:

DYNAMIC PROPERTIES

Maximum Stress: 210 lbs.
Test Frequency: 3 cps
Cycles to Failure: 3218

APPEARANCE OF FRACTURE SURFACE

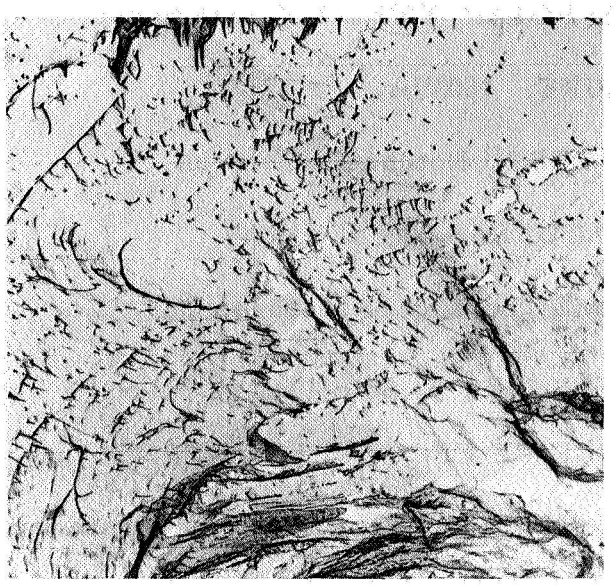
The fracture exhibited separation lines pointing to the origin of fracture very similar to the unnotched tension failure.

FRACTOGRAPHIC ANALYSIS

The fracture exhibited tear ridges with faint striation near origin of failure, then a twisted dimple-like fracture near fast rupture.

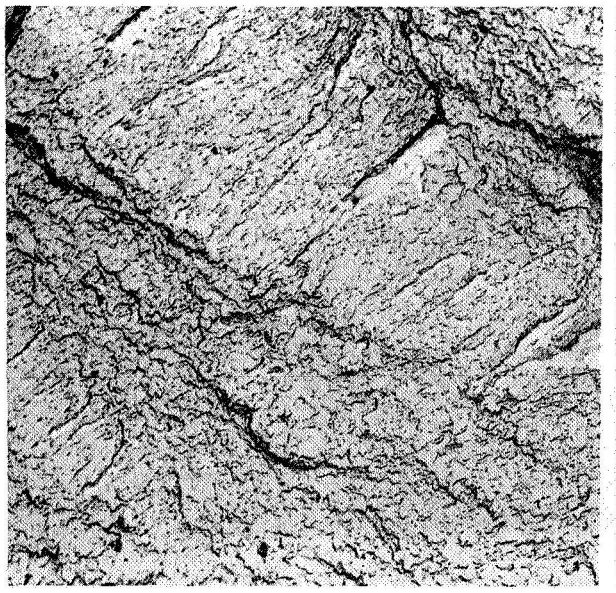
Micrographs of Fractured Surface of PTFE at Ambient Temperature, Low Cycle (Unnotched Sample) (Sheet 1 of 2)

Area A



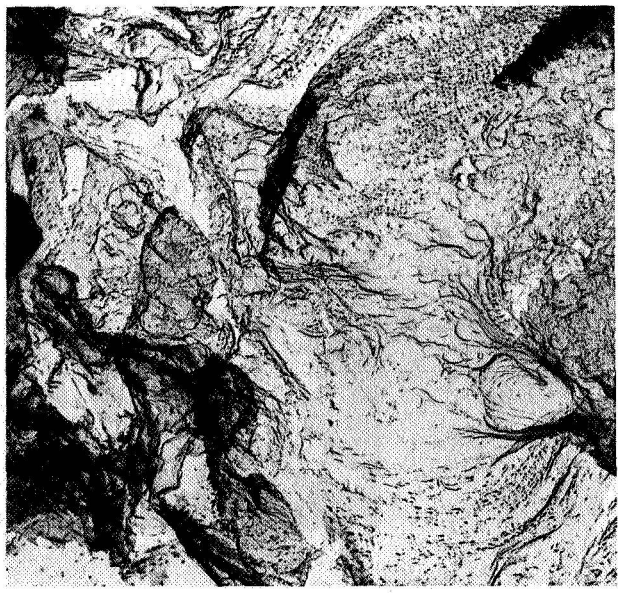
#326 4000X

Area B



#325 4000X

Area C



#328 4000X

Electron Micrographs of Fractured Surface of PTFE
at Ambient Temperature, Low Cycle (Unnotched Sample) (Sheet 2 of 2)

CODE NO: $NTLN_2$

MATERIAL AND FORM: PTFE 1/4-Inch Sheet

LOAD TO FAILURE: 460 lbs.

CONDITION OF TEST: Notched - Tension

TEST TEMPERATURE: $-319^{o}F$

TEST ENVIRONMENT: Liquid Nitrogen

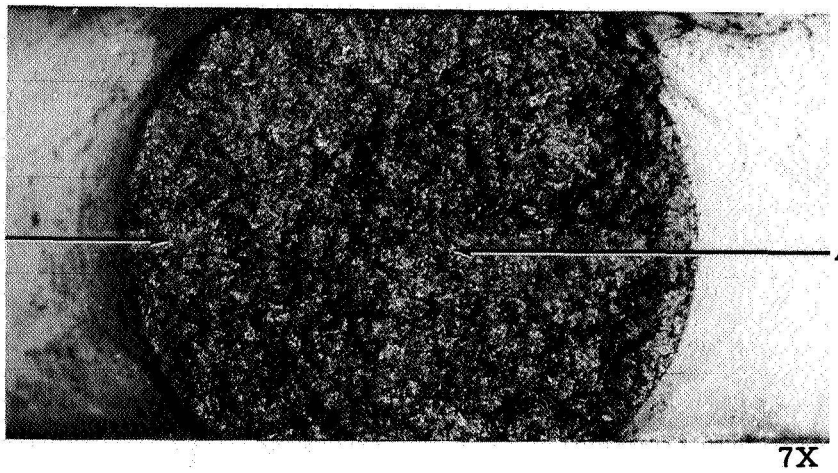
TEST RESULTS:

APPEARANCE OF FRACTURE SURFACE

The fracture exhibited a flat, fine-grain texture.

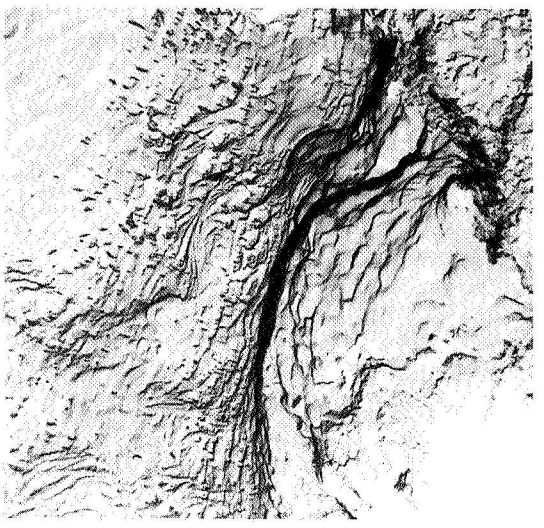
FRACTOGRAPHIC ANALYSIS

The fracture exhibited small and large dimple-like structure. An unusual texture was noted but unexplained.



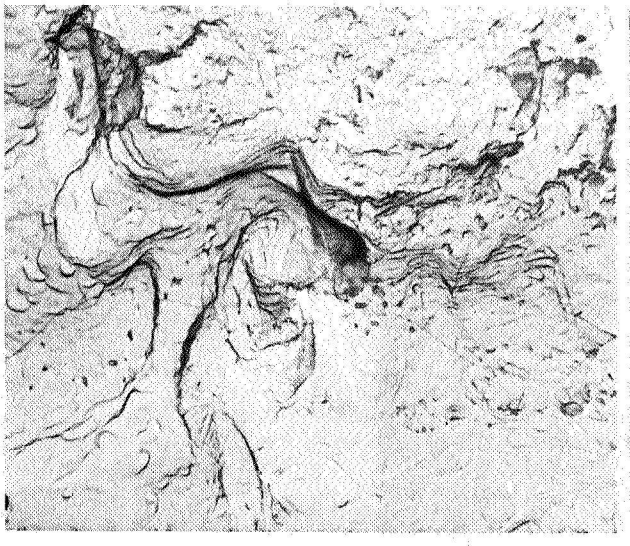
Micrographs of Fractured Surface of PTFE at LN_2 Temperature (Notched Sample, Tensile Failure) (Sheet 1 of 2)

Area A



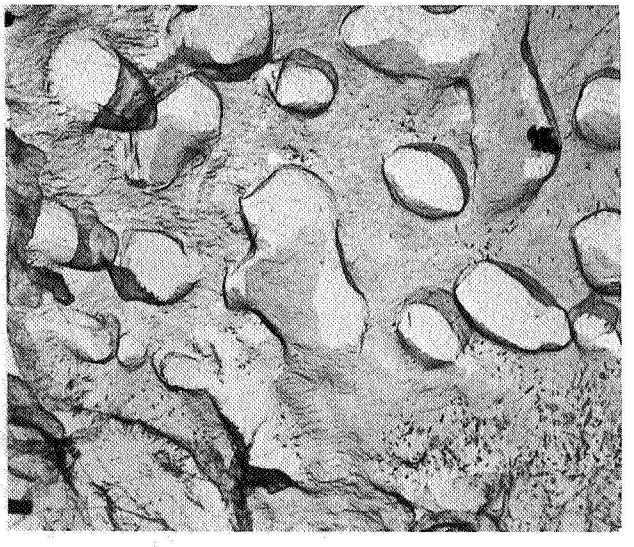
#376 4000X

Area A



#425 4000X

Area B



#420 4000X

Electron Micrographs of Fractured Surface of PTFE
at LN_2 Temperature (Notched Sample, Tensile Failure) (Sheet 2 of 2)

CODE NO: $UTLN_2$

MATERIAL AND FORM: PTFE 1/4-Inch Sheet

LOAD AT FAILURE: Not Recorded

CONDITION OF TEST: Unnotched - Tension

TEST TEMPERATURE: $-319^{\circ}F$

TEST ENVIRONMENT: Liquid Nitrogen

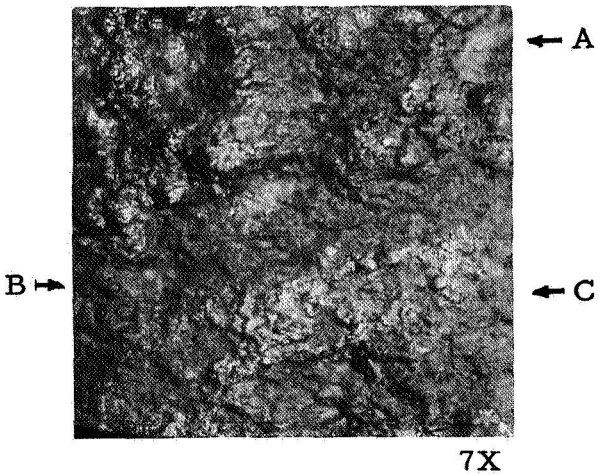
TEST RESULTS:

APPEARANCE OF FRACTURE SURFACE

The fracture face exhibited a flat, smooth topography texture.

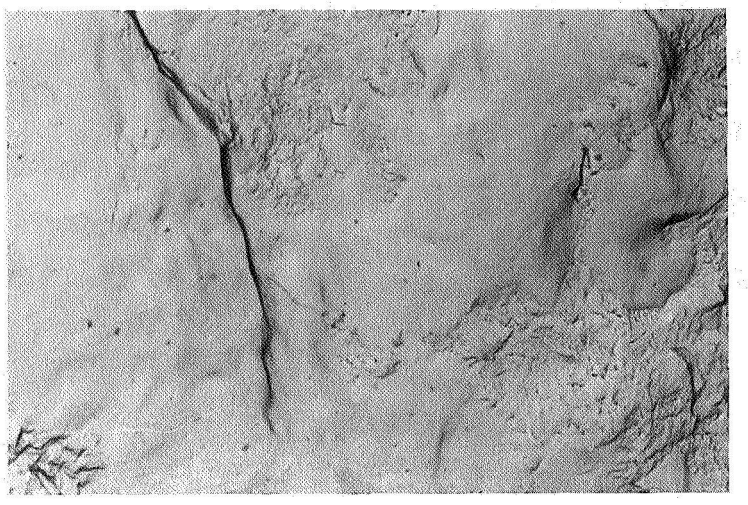
FRACTOGRAPHIC ANALYSIS

The fracture exhibited a flat waxy texture with scattered areas of dimples.



Micrograph of Fractured Surface of PTFE at LN_2 Temperature (Unnotched Sample) (Sheet 1 of 2)

Area A

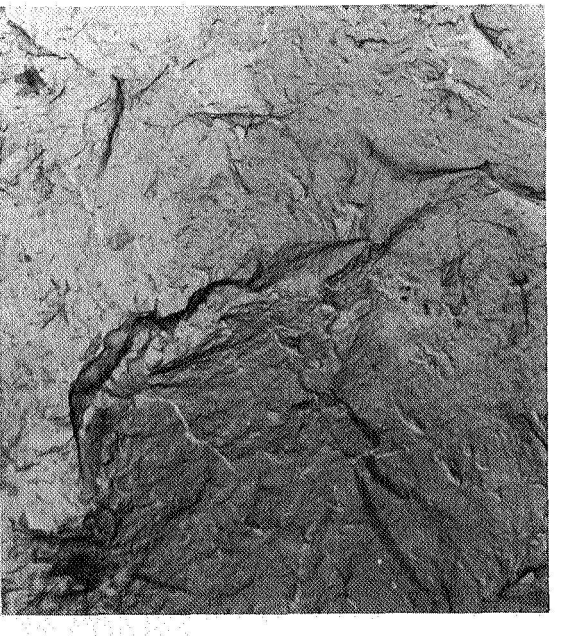


#244 4000X

Area B

#252 4000X

Area C



#247 4000X

Electron Micrograph of Fractured Surface of PTFE at LN_2 Temperature. (Unnotched Sample) (Sheet 2 of 2)

CODE NO: $NFLN_2$

MATERIAL AND FORM: PTFE 1/4-Inch Sheet

MAXIMUM DYNAMIC LOAD: 385 lbs.

CONDITION OF TEST: Notched - Fatigue

TEST TEMPERATURE: $-319^{\circ}F$

TEST ENVIRONMENT: Liquid Nitrogen

TEST RESULTS:

DYNAMIC PROPERTIES

Maximum Stress: 385 lbs.
Mean Stress: 365 lbs.
Minimum Stress: 345 lbs.
Test Frequency: 5.5 cps
Cycles to Failure: 297,120

APPEARANCE OF FRACTURE SURFACE

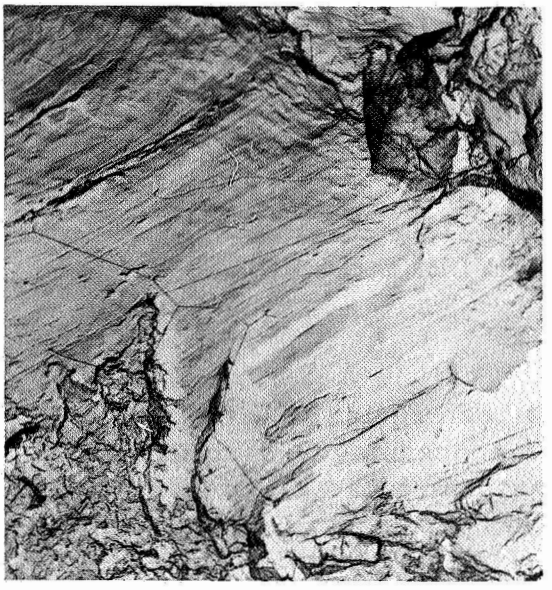
The fracture exhibited a rough flat surface.

FRACTOGRAPHIC ANALYSIS

The fracture exhibited typical fatigue striations at the root of each notch with elongated dimple-like structure in the fast fracture area.

Micrographs of Fractured Surface of PTFE at LN_2 Temperature (Notched Sample, Fatigue Failure) (Sheet 1 of 2)

Area A



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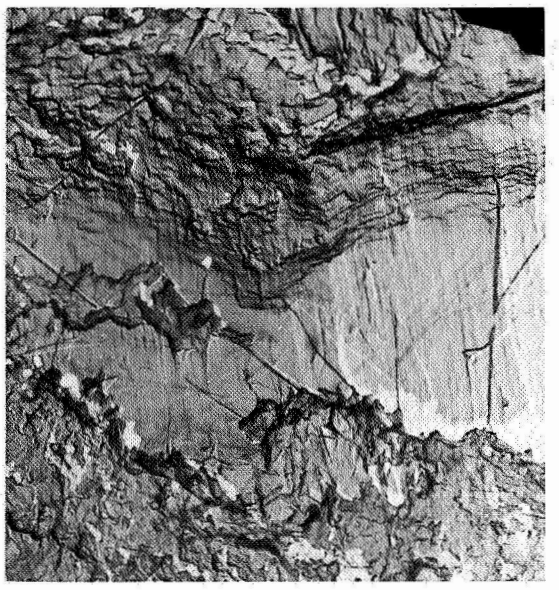

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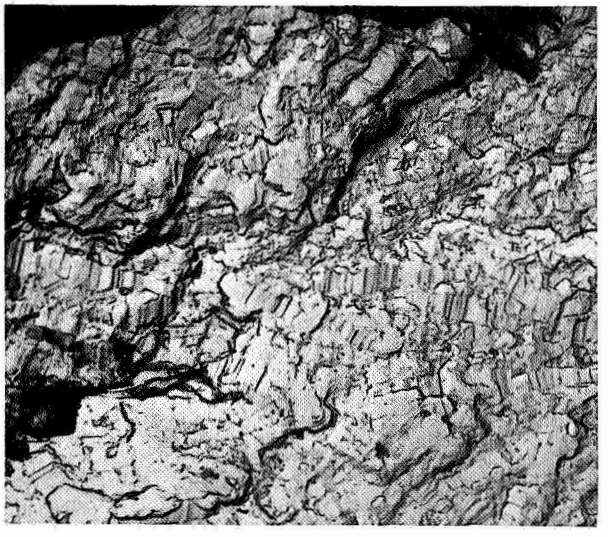
Area A



4000X

Area B

#400 4000X

Electron Micrographs of Fractured Surface of PTFE
at LN_2 Temperature (Notched Sample, Fatigue Failure) (Sheet 2 of 2)

TECHNICAL MEMORANDUM TM X-53728

APPROVAL

FRACTOGRAPHY OF POLYTETRAFLUOROETHYLENE (PTFE)

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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