NASA
Technical
Paper
3179

December 1991

p.12

A Statistical Comparison of Two Carbon Fiber/Epoxy Fabrication Techniques

A. J. Hodge

NASA

NASA Technical Paper 3179

1991

A Statistical Comparison of Two Carbon Fiber/Epoxy Fabrication Techniques

A. J. Hodge
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama

NASA

National Aeronautics and Space Administration

Office of Management

Scientific and Technical Information Program

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

TECHNICAL PAPER

A STATISTICAL COMPARISON OF TWO CARBON FIBER/EPOXY FABRICATION TECHNIQUES

I. INTRODUCTION

Carbon fiber/epoxy composite materials can be fabricated in several different manners. Two techniques for fabrication are autoclave and press curing. Press curing is not used for large components because the size of the press limits the size and shape of the composite. There can be a greater degree in the complexity of the shape of autoclaved composites, and larger components can be cured in an autoclave. An autoclave is normally used in industry, while the platen press is used in the laboratory. In support of ongoing research and materials testing and evaluation at Marshall Space Flight Center (MSFC), a statistical comparison of the compression strengths was performed on materials fabricated in both an autoclave and a platen press. This project will relate composite test specimens manufactured by both methods at MSFC and enable them to be compared.

A comparison of these two fabrication techniques was made by compression testing samples from both techniques of fabrication to determine how closely their strengths are related. There is a great deal of scatter when compression testing carbon fiber/epoxy composites due to the material's brittle mode of failure. Weibull analysis is generally used to model the strengths of brittle materials. Assuming that the weakest link theory applies to the voids, cracks, and other flaws which are the origins of failure, the compressive strength data can be modeled by the Weibull distribution. The Weibull distribution has successfully modeled the strength of unidirectional fiber-reinforced composites.[1–4]

When compression testing fiber-reinforced composites, the dominant mode of failure is local buckling of the fibers when loaded in the fiber direction. When loaded in the transverse direction, shear is the mode of failure.[5] A fiber-reinforced quasi-isotropic composite of $(0,45,90,-45)_{S2}$ configuration will fail with a combination of both modes. Specimens appear to fail by delamination and shear. The ASTM D3410 or Celanese test procedure has been shown to give correct values for testing carbon fiber/epoxy composites.[6–9]

II. MATERIALS USED AND SAMPLE PREPARATION

The material used in this project was Hercules IM6 carbon fiber in a 3501-6 epoxy resin. The prepreg was layed-up in a 16-ply $(0,45,90,-45)_{S2}$ quasi-isotropic configuration. Four panels of 650 cm^2 (100 in^2) in area were cured in a programmable platen press with a 5.4 °C/min (3 °F/min) temperature increase and a 2-h dwell time at 177 °C (350 °F). The panel was vacuum bagged, and a vacuum was drawn on the material as 550 kPa (80 psi) was applied from the press. Four more panels were cured in an autoclave using the same temperature, pressure, and ramp rate, as well as the same amounts of bleeder cloth and the same type of release film. The autoclaved panels, however, had a mean thickness of 0.292 cm (0.115 in) as opposed to the 0.257-cm (0.101-in) average thickness of the press-cured panels. This suggests that less epoxy was bled off of the autoclaved panels. This may be due simply because of the difference in the nature of the two processes.

Fiberglass tabs were then bonded to the panels leaving a 1.3-cm (0.5-in) gauge length. Cyanamid FM-300 epoxy film adhesive was used for the bonding. The tabs were bonded in a heated press at 121 °C (250 °F) at 140 kPa (20 psi) for 1 h.

After the tabs were bonded, the panels were cut into 0.65-cm (0.25-in) wide Celanese compression samples. After sanding the cut surfaces, the dimensions of the gauge area were measured and recorded. Two hundred samples of both types were labeled in preparation for compression testing.

III. TESTING

1. Compression Testing

Compression testing was performed on an Instron 1125 testing machine at a compression rate of 2.5 mm/min (0.1 in/min). A Celanese test fixture was utilized in the compression testing. Ultimate compressive strength was the only data taken for each sample. It was observed that the samples of both lots failed through delamination and buckling. The press-cured samples had an average compressive strength of 370 MPa (54.0 ksi), while the autoclave-cured samples had a greater average strength of 450 MPa (65.5 ksi).

2. Acid Digestion

Acid digestion tests were done on both lots of the samples to determine the fiber content. A square sample—approximately 100 mg—was weighed, and the volume was measured. The sample was then placed in nitric acid to dissolve the epoxy matrix. After filtering, washing, and drying the remaining fibers, they were weighed to determine the percent fiber content. The fiber weight percents for 67.1 percent for autoclaved and 77.4 percent for pressed specimens.

IV. DATA ANALYSIS

1. Weibull Analysis

Weibull analysis was performed on the compressive strength data. The 200 data points of each lot were first ranked from lowest to highest strength and assigned an F value of $F = (n-0.5)/N$, where n is the sample number and N is the total number of samples (200). When the natural log of the strength is plotted against $ln[ln(1/1-F)]$, the resulting representation becomes linear (fig. 1). The slope of this is defined as the shape parameter β in the Weibull distribution: $F(x) = 1-\exp(x/n)^{\beta}$. $F(x)$ is the probability of failure at a given load x, where the scale parameter n is the load at $F(x) = 0.632$. Once these values are found, the distribution function can be plotted (fig. 2). The beta values for the autoclaved and pressed material were determined to be 14.4 and 11.3, respectively, when ln(ksi) is used as the ordinate. The n values were 67.6 and 56.5 ksi, respectively.

The derivative of the Weibull distribution gives its density function which is as follows:

$$F(x) = \beta/n(x/n)^{\beta-1}\exp[-(x/n)^{\beta}] \ .$$

When the data are plotted using the above equation, the probability densities of the two functions can be compared (fig. 3). The area under each curve is unity. Thus, the area shared by both curves is the probability that they are of the same population. This area can be found first by setting the two equations equal to each other to find the intercept which is 60.9 ksi. Then by summing the integrals of the autoclaved curve from 0 to 60.9 and the pressed curve from 60.9 to ∞. The sum was determined to be 0.30. Thus, there is a 30-percent probability that the two types of composites have the same strength.

The Weibull means and variances were found for the two lots. The means were found using the gamma function:

$$E(x) = n\Gamma\,(1+1/\beta)\ .$$

The means of the autoclaved and pressed samples were 450 MPa (65.2 ksi) and 370 MPa (54.0 ksi), respectively. Pressed samples had an average of 17-percent lower strength value than the autoclaved samples. A student t-test suggests that the samples are not of the same population. The pressed samples had a slightly smaller variance—48.9 kPa (7.1 ksi) as opposed to 51.7 kPa (7.5 ksi)—from the gamma function:

$$V(x) = n^2[\Gamma\,(1+2/\beta)-\Gamma(1+1/\beta)^2].$$

2. Fiber Content

Acid digestion found that the fiber content of the composites was 67.1 percent for the autoclaved and 77.4 percent by weight for the pressed material. Utilizing the accepted values of the epoxy and fiber densities—1.27 and 1.76 g/cm^3, respectively—the volume percent fiber was found for both lots. Volume fraction was found with the equation:

$$V_f = \rho_m W_f/(\rho_f W_m+\rho_m W_f)\ ,$$

which gave 59.5-percent fiber for the autoclaved and 71.2-percent fiber for the pressed material. With the means of the two lots and the volume fraction, it can be shown that the compressive strength is inversely proportional to the fiber content, i.e., $\sigma_1 V_{f1} = \sigma_2 V_{f2}$.

The compressive strength is inversely proportional to the fiber content because of the failure mechanisms involved. Although the fibers carry a substantial portion of the load compared to the epoxy, the fibers need the epoxy for support and rigidity. The epoxy prevents the fibers from flexing, buckling, and shearing. Thus, up to limit, increasing the percentage of epoxy will increase the compressive strength of the composite.

V. RESULTS

It was determined that the two processing techniques at MSFC yield different, yet easily related, products. Less epoxy is bled off during autoclaving than during press curing. This results in a thicker autoclaved specimen with a smaller percentage of carbon fibers. The acid digestion tests revealed the percentage of fiber in both types of composites. The autoclaved specimens were approximately 60-percent fiber while the press cured were approximately 71-percent fiber by volume. Because of its greater percentage of epoxy, the autoclaved material was significantly stronger than the pressed material. Student *t*-tests determined the two lots were not of the same population. Weibull analysis shows that there is only a 30-percent probability that the two lots are of the same population. The Weibull mean for the compressive strength was 450 and 370 MPa for the autoclaved and pressed specimens, respectively.

VI. CONCLUSION

It was determined that of the two techniques studied for fabrication of 16-ply $(0,45,90,-45)_{S2}$ carbon fiber/epoxy composites at MSFC, autoclaving yields a greater compressive strength than pressing. This is due to the greater percentage of epoxy in the autoclaved composites, which is probably due to differences in the nature of the two processing techniques. There is no change in the thickness of the composite when the pressure or the temperature ramp rate is varied in press curing. When autoclaving, there is only the bagging material and no top plate over the specimen. This may be the cause of the difference in thickness. It was proven from the data obtained in the experiments that the compressive strength is directly proportional to the percentage of epoxy in the composite. Thus, $\sigma V_{f1} = \sigma V_{f2}$ is true for the two types of fabrication.

Fiber-reinforced composites, however, have different failure modes when tensile testing than when compression testing. The tensile strength is dependent upon the strength of the fibers. Thus, the tensile strength can be assumed to be greater for the pressed specimens because of the greater percentage of fiber. Future tests should be conducted on the two lots to determine if this assumption is true.

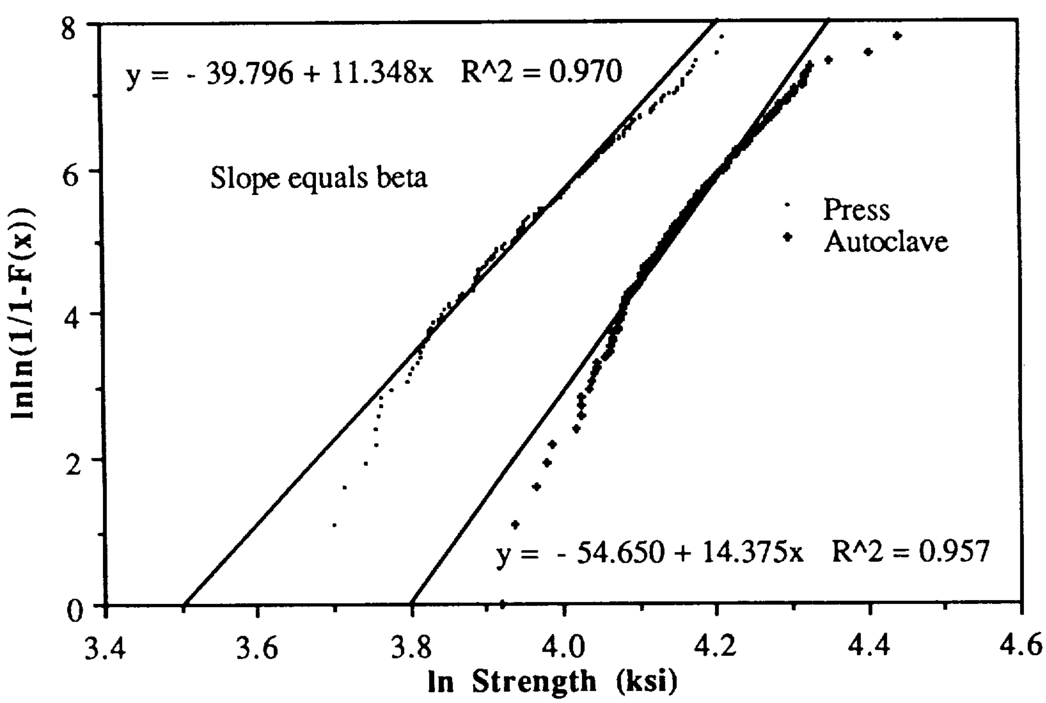


Figure 1. Linear rank regression of compression strength data.

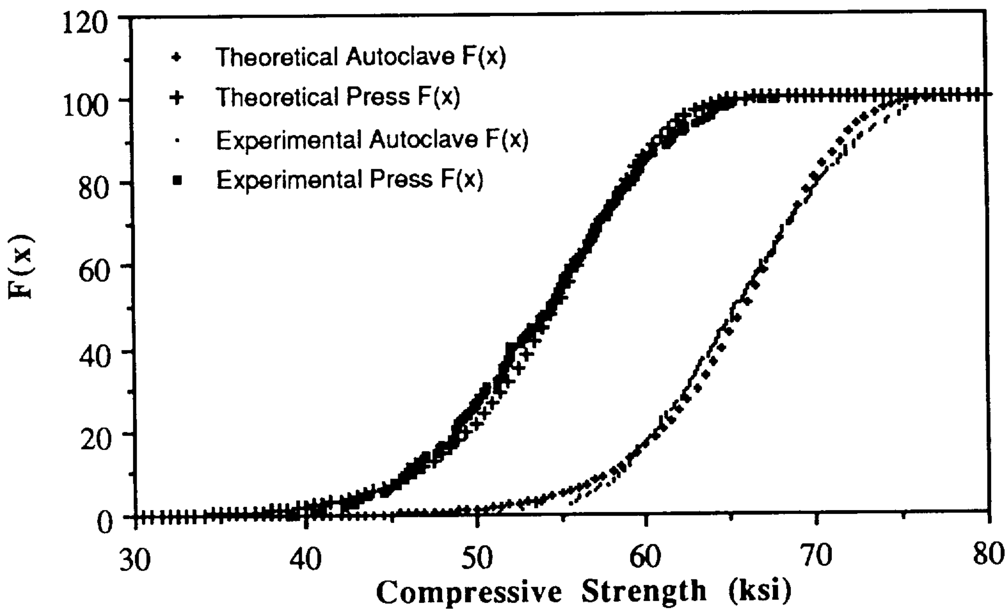


Figure 2. Experimental versus theoretical probability of failure.

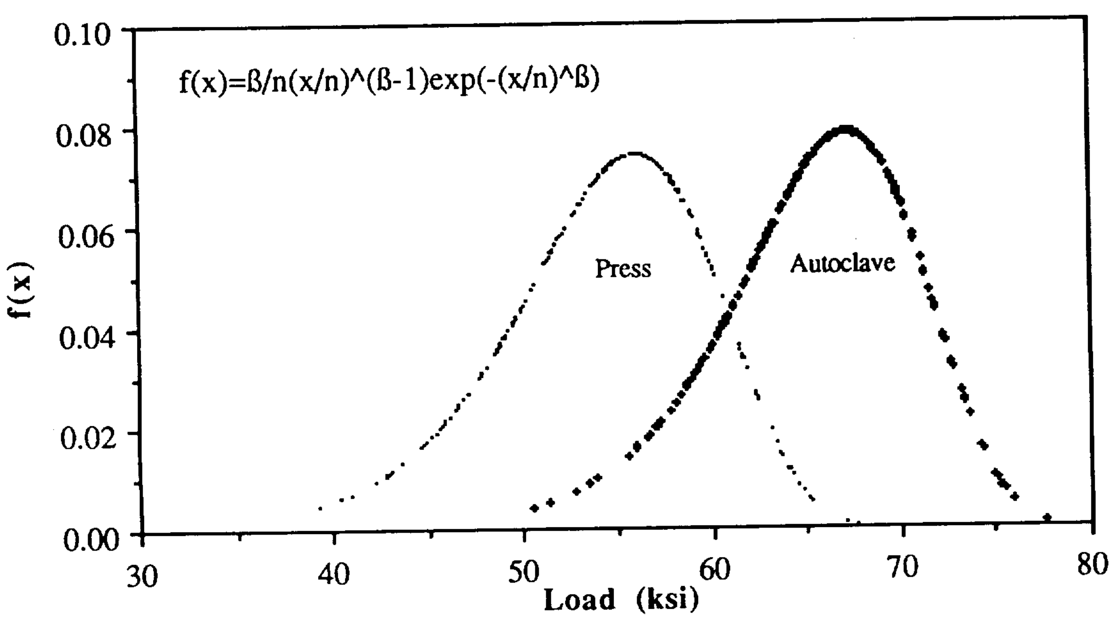
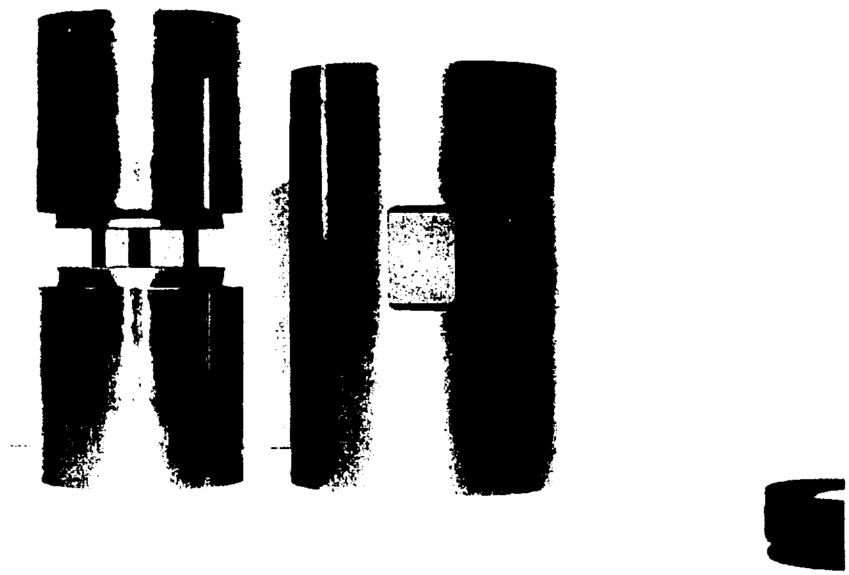
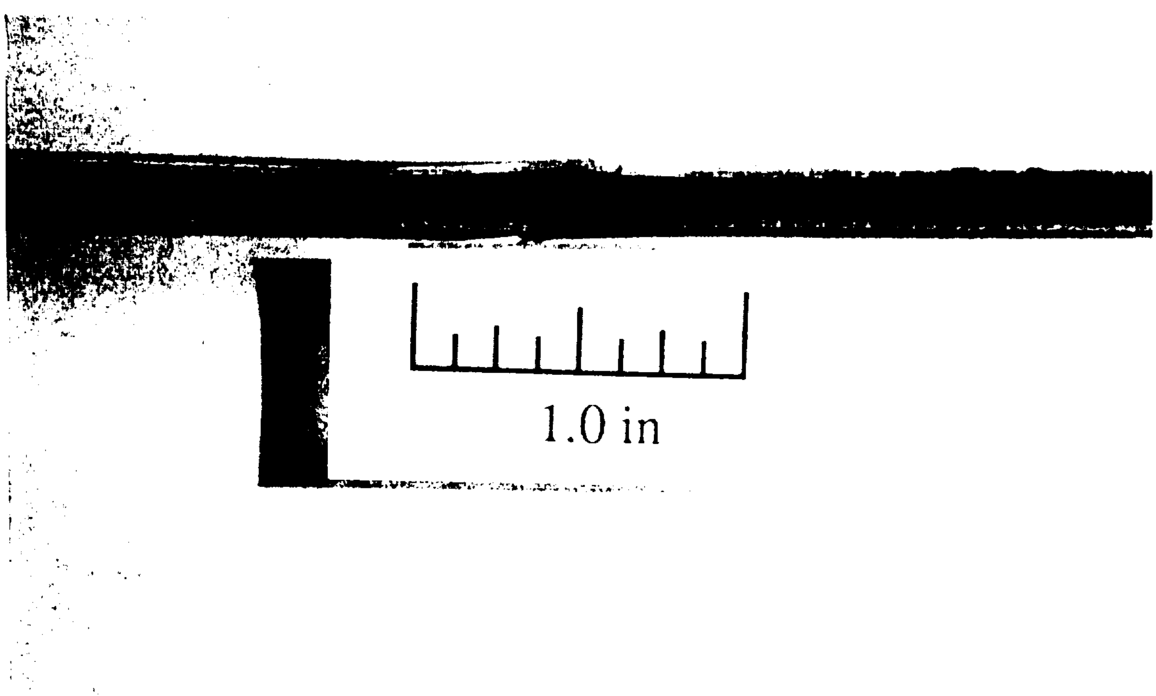


Figure 3. Probability densities of autoclave and press-cured specimens.

APPENDIX



Celanese Test Fixture



Failed ASTM D3410 Specimen

REFERENCES

1. Harlow, D.G., and Phoenix, S.L.: "Bounds on the Probability of Failure of Composite Materials." International Journal of Fracture, vol. 15, August 1979, pp. 321–335.

2. Smith, R.L.: "Probabilistic Models for Composites: Are There Flaws in the Theory?" Probabilistic Methods in the Mechanics of Solids and Structures, IUTAM Symposium, Stockholm, Sweden, S. Eggwertz and N.C. Lind Eds., Spriger-Verlag, New York, 1984, pp. 291–298.

3. Hines, W.W., and Montgomery, D.C.: "Probability and Statistics in Engineering and Management Science." John Wiley and Sons, Second Edition, New York, 1980. pp. 164–167.

4. Batdorf, S.B.: "Failure Statistics of Unidirectional Long-Fiber Composites." Probabilistic Methods in the Mechanics of Solids and Structures, IUTAM Symposium, Stockholm, Sweden, S. Eggwertz and N.C. Lind Eds., Spriger-Verlag, New York, 1984, pp. 299–304.

5. Carlsson, L.A., and Pipes, B.R.: "Experimental Characterization of Advanced Composite Materials." Prentice-Hall, Inc., New Jersey, 1987, pp. 45–52 and 69–81.

6. Adsit, N.R.: "Compression Testing of Graphite/Epoxy." Compression Testing of Homogeneous Materials and Composites, ASTM STP 808, R. Chait and Papirno, Eds., American Society of Testing Materials, 1983, pp. 175–186.

7. Lamothe, R.M., and Nunes, J.: "Evaluation of Fixturing for Compression Testing of Metal Matrix and Polymer/Epoxy Composites." Compression Testing of Homogeneous Materials and Composites, ASTM STP 808, R. Chait and Papirno, Eds., American Society for Testing Materials, 1983, pp. 241–253.

8. Hofer, K.E., Jr., and Rao, P.N.: "A New Static Compression Fixture for Advanced Composite Materials." Journal of Testing and Evaluation, JTEVA, vol. 5, No. 4, July 1977, pp. 278–283.

9. Woolstencroft, D.H., Curtis, A.R., and Haresceugh, R.I.: "A Comparison of Test Techniques Used for Evaluation of the Unidirectional Compressive Strength of Carbon Fiber-Reinforced Plastic." Composites, October 1981, pp. 275–280.

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	December 1991	Technical Paper

4. TITLE AND SUBTITLE
A Statistical Comparison of Two Carbon Fiber/Epoxy Fabrication Techniques

5. FUNDING NUMBERS

6. AUTHOR(S)
A.J. Hodge

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

8. PERFORMING ORGANIZATION REPORT NUMBER
M-673

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, DC 20546

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
NASA TP-3179

11. SUPPLEMENTARY NOTES
Prepared by Materials and Processes Laboratory, Science and Engineering Directorate.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified – Unlimited
Subject Category: 24

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

A statistical comparison of the compression strengths of specimens that were fabricated by either a platen press or an autoclave were performed on IM6/3501-6 carbon/epoxy composites of 16-ply $(0,+45,90,-45)_{s2}$ lay-up configuration. The samples were cured with the same parameters and processing materials. It was found that the autoclaved panels were thicker than the platen press-cured samples. Two hundred samples of each type of cure process were compression tested. The autoclaved samples had an average strength of 450 MPa (65.5 ksi), while the press-cured samples had an average strength of 370 MPa (54.0 ksi). A Weibull analysis of the data showed that there is only a 30-percent probability that the two types of cure systems yield specimens that can be considered from the same family.

14. SUBJECT TERMS
Composite Materials, Autoclave Cure, Compression Testing, Weibull Analysis

15. NUMBER OF PAGES
12

16. PRICE CODE
A03

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	Unlimited

National Aeronautics and
Space Administration
Code NTT-4

Washington, D.C.
20546-0001

NASA