AFRL-ML-WP-TP-2006-414

EFFECT OF PLY THICKNESS ON THE DAMAGE DEVELOPMENT IN COMPOSITE LAMINATES (PREPRINT)

Ran Y. Kim
Sangwook Sihn
Steven L. Donaldson

FEBRUARY 2006

Approved for public release; distribution is unlimited.

STINFO COPY

MATERIALS AND MANUFACTURING DIRECTORATE
AIR FORCE RESEARCH LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750

NOTICE AND SIGNATURE PAGE

AFRL-ML-WP-TP-2006-414 HAS BEEN REVIEWED AND IS APPROVED FOR PUBLICATION IN ACCORDANCE WITH ASSIGNED DISTRIBUTION STATEMENT.

//Signature//	//Signature//
STEVEN L. DONALDSON, Program Mgr. Structural Materials Branch Nonmetallic Materials Division	TIA BENSON TOLLE, Chief Structural Materials Branch Nonmetallic Materials Division

//Signature//

PERSIS A. ELWOOD, Deputy Chief
Nonmetallic Materials Division
Materials and Manufacturing Directorate

*Disseminated copies will show "//Signature//" stamped or typed above the signature blocks.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YY)*
February 2006

2. REPORT TYPE
Conference Paper Preprint

3. DATES COVERED *(From - To)*

4. TITLE AND SUBTITLE
EFFECT OF PLY THICKNESS ON THE DAMAGE DEVELOPMENT IN COMPOSITE LAMINATES (PREPRINT)

5a. CONTRACT NUMBER
FA8650-05-D-5052

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
62102F

6. AUTHOR(S)
Sangwook Sihn and Ran Y. Kim (University of Dayton Research Institute)
Steven L. Donaldson (AFRL/MLBC)

5d. PROJECT NUMBER
4347

5e. TASK NUMBER
RG

5f. WORK UNIT NUMBER
M03R1000

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

University of Dayton
Research Institute
300 College Park
Dayton, OH 45469

Structural Materials Branch (AFRL/MLBC)
Nonmetallic Materials Division
Materials and Manufacturing Directorate
Air Force Research Laboratory, Air Force Materiel Command
Wright-Patterson Air Force Base, OH 45433-7750

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Materials and Manufacturing Directorate
Air Force Research Laboratory
Air Force Materiel Command
Wright-Patterson AFB, OH 45433-7750

10. SPONSORING/MONITORING AGENCY ACRONYM(S)
AFRL-ML-WP

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)
AFRL-ML-WP-TP-2006-414

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

This work, resulting in whole or in part from Department of the Air Force contract FA8650-05-D-5052, has been submitted to The Society for the Advancement of Material and Process Engineering (SAMPE) 2006 Conference Proceedings. If this work is published, SAMPE may assert copyright. The United States has for itself and others acting on its behalf an unlimited, paid-up, nonexclusive, irrevocable worldwide license to use, modify, reproduce, release, perform, display, or disclose the work by or on behalf of the Government. All other rights are reserved by the copyright owner.

This paper contains color. PAO Case Number: AFRL/WS 06-0352, 9 Feb 2006.

14. ABSTRACT

This paper describes the experimental investigation on the effect of ply thickness on the first ply failure, delamination, and the subsequent final failure of graphite/epoxy composite laminates. The laminates considered in this paper are a cross-ply and two quasi-isotropic laminates containing thin plies in the desired interface. The onset of the first ply failure and delamination, and the stress-free temperature are experimentally determined. The experimental results for ply crack and delamination development were compared with an analytical model. The results indicated that ply thickness played a significant role on delaying damage onset. The experimental results were compared with the Weibull statistical theory in conjunction with the weakest link theory and discussed on the effect of size (ply thickness) on damage initiation.

15. SUBJECT TERMS
Fiber Reinforcement, Graphite, Damage resistance, Size effect

16. SECURITY CLASSIFICATION OF:

a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**	**17. LIMITATION OF ABSTRACT:**	**18. NUMBER OF PAGES**
Unclassified	Unclassified	Unclassified	SAR	16

19a. NAME OF RESPONSIBLE PERSON (Monitor)
Steven L. Donaldson

19b. TELEPHONE NUMBER *(Include Area Code)*
N/A

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

EFFECT OF PLY THICKNESS ON THE DAMAGE DEVELOPMENT IN COMPOSITE LAMINATES

Ran Y. Kim and Sangwook Sihn
University of Dayton Research Institute, 300 College Park, Dayton, OH 45469-0168, USA

Steven L. Donaldson
Air Force Research Laboratory, AFRL/MLBC, Wright-Patterson AFB, OH 45433-7750, USA

ABSTRACT

This paper describes the experimental investigation on the effect of ply thickness on the first ply failure, delamination, and the subsequent final failure of graphite/epoxy composite laminates. The laminates considered in this paper are a cross-ply and two quasi-isotropic laminates containing thin plies in the desired interface. The onset of the first ply failure and delamination, and the stress-free temperature are experimentally determined. The experimental results for ply crack and delamination development were compared with an analytical model. The results indicated that ply thickness played a significant role on delaying damage onset. The experimental results were compared with the Weibull statistical theory in conjunction with the weakest link theory and discussed on the effect of size (ply thickness) on damage initiation.

KEY WORDS: Fiber Reinforcement – Graphite, Damage resistance, Size effect.

1 INTRODUCTION

Application of composites in aerospace structures extends recently into primary load bearing structures as well as large cryogenic fuel tanks [1-3]. Fiber reinforced composite laminates exhibit complex failure processes under static and fatigue loading because of anisotropic nature in their strength and stiffness. Instead of a predominant single crack often observed in most isotropic materials, extensive damage throughout specimen volume usually accompanies failure in composites. Fiber/matrix interfacial debonding, matrix crack, delamination, and fiber breakage are commonly observed failure modes. Among these damage modes, the matrix related damage usually occurs at first and fiber damage follows in structural composites subjected to a thermomechanical loading. Improved damage resistance of composites may result in lower structural weight, lower cost structures, and increase in design allowable levels. Consequently, the need for improvement of damage resistance is a very desirable feature for more efficient use of these composite materials.

A considerable amount of work has been reported on the damage onset and development in literatures [4-6], especially in damage related with matrix cracking. The traditional approach in prediction of stress to initial ply failure in a $[0_n/90_n]_S$ laminate is predicted to be independent of n. A number of experimental studies for cross-ply laminates have demonstrated, however, the strain (stress) in the 90º ply at the onset of transverse cracking varies inversely with the thickness of the transverse cracking ply [4,5,8,9]. Furthermore, the strength of a 90º ply in an angle-ply laminate was found to be up to two and a half times higher than the strength of the a 16-ply of transverse laminate [5]. This dependence of first-ply failure stress on the thickness of the transverse 90º ply has been attributed to the constraining effect of the adjacent 0º layers. This constraining effect has been explained by a fracture mechanics approach [10-12] and on the basis of a statistical strength distribution in the 90º layer [13-15]. All experimental works in literatures have been dealt with the laminates made of conventional prepreg with various thickness of 90º ply until very recent. There have been a few efforts to manufacture the ply thickness thinner than the conventional ply of 0.127 mm thickness. However, this process is costly and time-consuming and furthermore, the fiber is prone to subject damage. Recently, a new novel process was developed to make the thin plies by using a tow-spreading technology [16, 17]. This new process is low-cost and robust and does not damage the fibers. We were able to obtain the unidirectional thin ply dry mat with thickness of 0.042 mm which is one-third of the conventional prepreg.

The main objective of this paper is to investigate the effect of ply thickness on the development of ply cracks and delaminations in the composites under the mechanical load. Specimens were tested to determine the onset of ply crack and delamination under uniaxial tension. Ply crack and delamination were detected from acoustic emission in conjunction with incremental loading and unloading experiments, and confirmed from microscopic examination of polished specimen edges. The thermomechanical properties required for the analysis were obtained from tests on $[0]_{8T}$, $[90]_{8T}$, and $[\pm 45]_{2S}$ laminates. The residual stresses in the plies interested were analyzed using the super Mic-Mac [18]. The results of this stress analysis were applied to predict the onset of ply cracking using the maximum stress failure theory.

2 EXPERIMENTAL PROCEDURE

In this paper, the material systems used were carbon fiber-reinforced epoxy, IM6/3501-6 supplied as a unitape prepreg with 0.127 mm thick (cured), and 0.042 mm thick TS800 dry unidirectional fiber mat. The thin dry fiber mat was placed in the desired interface between two conventional plies and the panels were cured in an autoclave according to the manufacturer's recommended cure cycle. Three types of laminates, $[0/90/0/T90/0/90]_S$ and $[0/T90/45/-45/90]_S$ and $[35/-35/-35/T90]_S$, were tested under a uniaxial tension. The thin ply is designated as T in the laminate code. We were able to maintain the nominal fiber volume content of 62% by adjusting the number of bleeder plies instead of adding the resin. Specimens were cut from the cured panel using a diamond impregnated saw after bonding fiberglass/epoxy end tabs (3.2 cm). All specimens were 7.62 cm long in gage section and 2.5 cm wide. Both side edges of the specimen were ground with sandpaper and then polished with 5- and 0.5-micron polishing powder in order to facilitate microscopic examinations. The onset and growth of ply cracking and delamination, as a function of applied uniaxial tensile load, were determined by acoustic emission technique in conjunction with an optical microscope. A Physical Acoustics Logan AT acoustic emission system was used, and the total system gain and frequency band were at 80 db and 0.1-0.3 MHZ, respectively.

All specimens were tested on a MTS testing machine with hydraulic grips while recording axial strain with an extensometer. When a sudden acoustic emission event was observed, the specimen was unloaded and examined in a microscope to confirm the presence of transverse cracking. Figure 1 shows the acoustic emission record for a specimen tested at 23°C, indicating the occurrence of ply cracking.

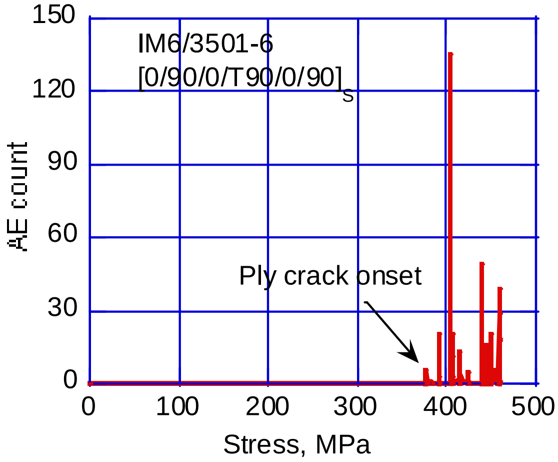

Figure 1. Acoustic emission event showing the onset of ply failure within $[0/90/0/T90/0/90]_S$.

After confirming the ply crack on a microscope, the specimen was reloaded to a stress incrementally higher than before, unloaded, and examined again. This procedure repeated until the final failure. We found after a few trials that the optical microscopic technique employed was not satisfactory to detect the small cracks in the thin ply due to crack closure upon unlading. In order to overcome this difficulty, fluorescent dye penetrant was applied into the free edge of the specimen under loading so that the penetrant could remain in the microcrack to enhance the microscopic image until it was microscopically examined after unloading. This process was repeated until the first crack was observed in the thinner plies, which is usually accomplished in a few iterations. The average value of the last two consecutive loads is then used to compute the stress required to initiate transverse cracking. With this procedure, the error in the computed stress for crack initiation is less than 5%. Figure 2 is a micrograph obtained with in situ fluorescent dye application under loading and clearly shows two ply cracks in the thin 90° ply, whereas thin ply cracks did not show under normal microscopic examination after unloading as shown in Figure. This implies that opening of such a small crack in a single thin 90° ply constrained by the 0° plies is definitely reduced to an extremely smaller size, which could not be observable with an ordinary microscopic technique.

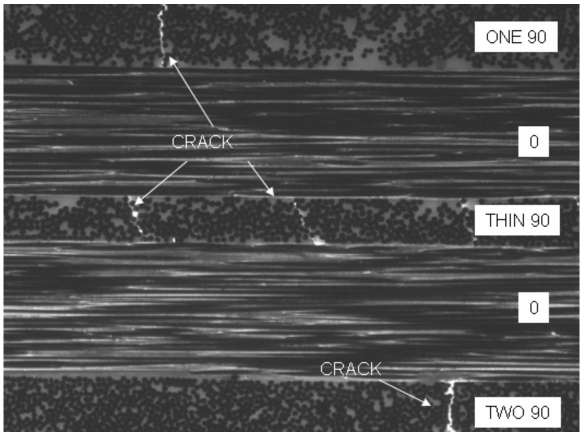

Figure 2.
Photomicrograph showing transverse ply cracks in $[0/90/0/T90/0/90]_S$ under uniaxial tensile loading. Fluorescent penetrant was applied under loading.

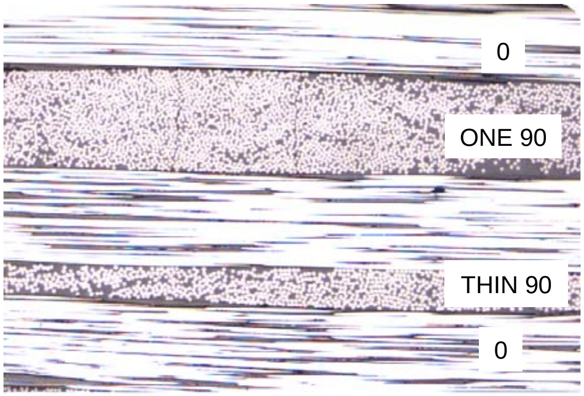

Figure 3.
Photomicrograph showing transverse ply cracks in $[0/90/0/T90/0/90]_S$ under ordinary light microscopy after unloading. Cracks in the thin 90° ply are hidden.

3 RESULTS AND DISCUSSION

3.1 Ply Stress Calculation

Stresses due to mechanical loading and curing are calculated for prediction of ply failure initiation. The constitutive equation for a ply in a symmetric multidirectional laminate is consisted of a mechanical part and a thermal (residual stress) part and can be written as [19]

$$\sigma_i = a_{ij} Q_{jk} N_k + \sigma_i^R , \tag{1}$$

where

a_{ij} = compliance matrix at the final temperature of interest,
Q_{jk} = reduced stiffness at the final temperature and
N_k = applied stress resultant.

The curing residual stresses σ_i^R is given by

$$\sigma_i^R = Q_{ij}(\varepsilon_j^N - e_j), \tag{2}$$

where

ε_j^N = laminate thermal strains measured from the stress-free state and
e_j = ply thermal strains measured from the stress-free state.

For calculation of ply stresses, the following thermoelastic properties determined by tests are utilized: E_L=175 GPa, E_T= 9.3 GPa, G_{LT}=5.5 GPa, ν_{LT} = 0.33, α_L = 0.36x10^{-6}/ºC, α_T = 26.8x10^{-6}/ºC, X=2,528 MPa and Y=58.5 MPa. E, G, and ν are Young's modulus, shear modulus and Poisson's ratio, respectively. α is the coefficient of thermal expansion and subscripts L and T denote longitudinal and transverse directions, respectively. X and Y are longitudinal and transverse, strength, respectively. The cure temperature was used as the stress-free temperature based on the previous work. The fiber volume is determined by the acid digestion method, and the average value of three samples was found to be 62%.

3.2 Results

Table 1 shows comparison of elastic constants, modulus and Poisson's ratio between analysis and experiment for the laminates tested.

Table 1. Comparison of elastic properties.

Laminate	Modulus, GPa		Poisson's ratio	
	calculation	test	calculation	test
$[0/90/0/T90/0/90]_S$	103.4	100.7	0.04	0.041
$[0/T90/45\text{-}45/90]_S$	62.7	57.2	0.26	0.27
$[35/-35/-35/T90]_S$	49.6	47.6	0.73	0.75

The laminated plate theory predicts the elastic constants accurately as expected. Figures 4-6 show the stress-strain relations for the laminates. The stress-strain curve is essentially linear up to the final failure for the first two laminates but the third laminate $[35/-35/-35/T90]_S$ shows initially linear but slightly deviates from its linearity at approximately 0.004 strain as shown in Figure 6.

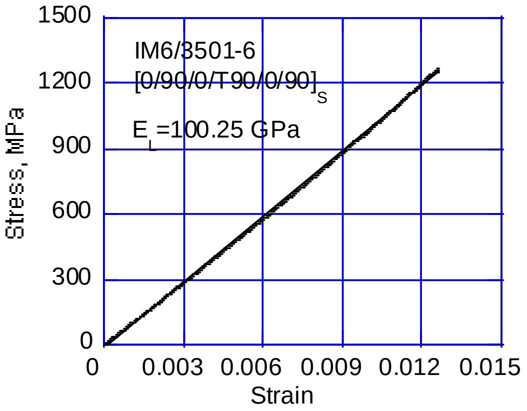

Figure 4.
Stress-strain curve for $[0/90/0/T90/0/90]_S$ under uniaxial tension.

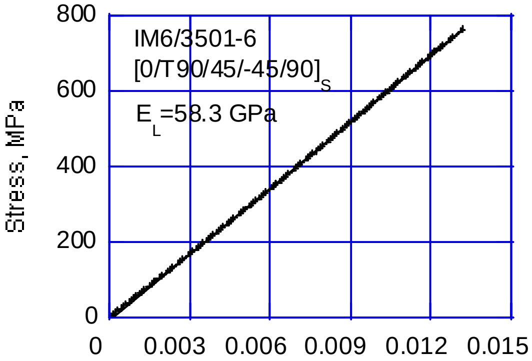

Figure 5.
Stress-strain curve for $[0/T90/45/-45/90]_S$ under uniaxial tension.

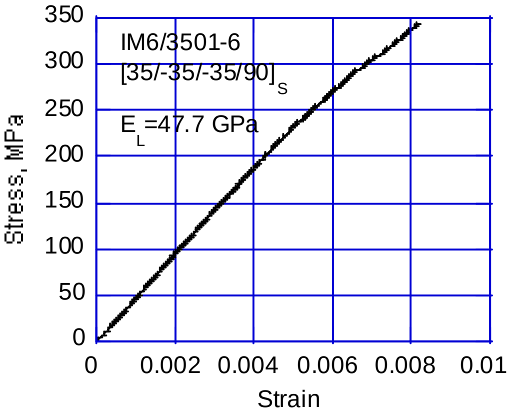

Figure 6.
Stress-strain curve for $[35/-35/-35/T90]_S$ under uniaxial tension.

Microscopic examination reveals that nearly almost all cracks were extended to the entire thickness of the ply except for those in the thin ply where some of cracks were found to be a partial crack. Crack counts on both free edges in the ordinary ply are a good agreement and aligned each other except for those cracks but there are a significant difference in crack counts, sometimes more 100 %, in the thin ply. This observation strongly suggests that the ply cracks in the thick plies are fully extended to the entire thickness as well as width whereas most of cracks in the thin ply are not.

Crack density was obtained using all the cracks counted along the entire gauge length of 10.16 cm in each 90º ply. Average crack density was used for the thin and single plies separated from each other with respect to the laminate midplane. Figure 7 shows the crack density as a function of applied stress for the cross ply laminate.

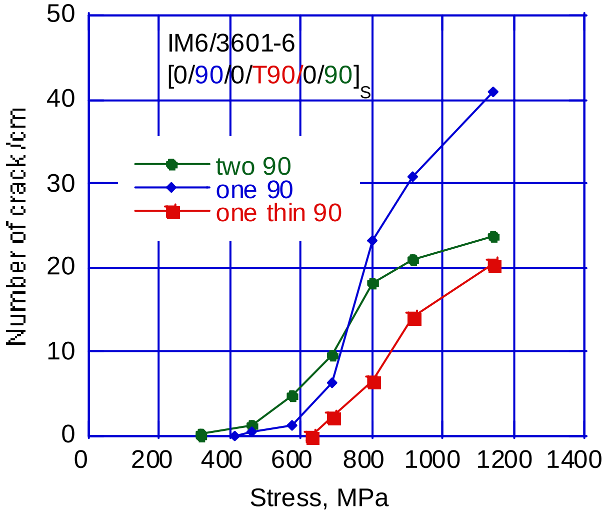

Figure 7. A typical crack density vs. applied stress for $[0/90/0/T90/0/90]_S$.

The ply crack occurred first in the thickest ply followed by the thinner plies and crack density increases as the load applied increased in all three laminates. The similar crack development was also observed in the $[0/T90/45/-45/90]_S$ laminate. Table 2 shows the comparison of ply stress at onset of ply crack and transverse tensile strength including curing residual stresses.

Table 2. Comparison of 90° ply stress at onset of ply failure.

Laminate	90° Ply thickness mm	Applied stress at FPF, MPa	Ply stress at FPF, MPa			$[90]_{8T}$ strength MPa
			curing	mechanical	total	
$[90]_{8T}$	1.12					58.5
$[0/90/0/T90/0/90]_S$	0.046	606.07	36.41	54.95	91.36	
	0.14	468.75	36.41	42.50	78.91	
	0.28	384.34	36.41	37.95	74.36	
$[0/T90/45/-45/90]_S$	0.046	248.65	34.89	42.27	77.16	
	0.28	164.59	34.89	27.98	62.87	
$[35/-35/-35/T90]_S$	0.092	165.62	35.27	35.19	70.46	

The onset of ply failure in all cases is greater than the transverse strength determined from the 8 ply thick laminate. Furthermore, the calculated stress using laminated plate theory predicts the unique value regardless of the ply thickness; whereas, the experimental results are not a unique number, but rather varies inversely with ply thickness. The experimental results were further analyzed using the Weibull statistical theory in conjunction with a weakest link theory which can be expressed as [20]

$$\frac{S_2}{S_1} = \left[\frac{V_1}{V_2}\right]^{1/\alpha_1}, \qquad (3)$$

where S and V are transverse strength and specimen volume, respectively. α_1 is Weibull shape parameter determined from the baseline strength data. Subscripts 1 and 2 refer to the baseline and other interested specimen. A total of 10 specimens of the $[90]_{8T}$ with 10-cm long in gauge section and 2.5-cm wide were tested to determine the strength and Weibull parameter. The average value of the strength is found to be 58.5 MPa. The shape parameter was estimated by the maximum likelihood method using the Newton-Raphson iterative technique and determined to be 10.2. Figure 7 shows the relationship between strength and thickness using Eq. (3) along experimental data.

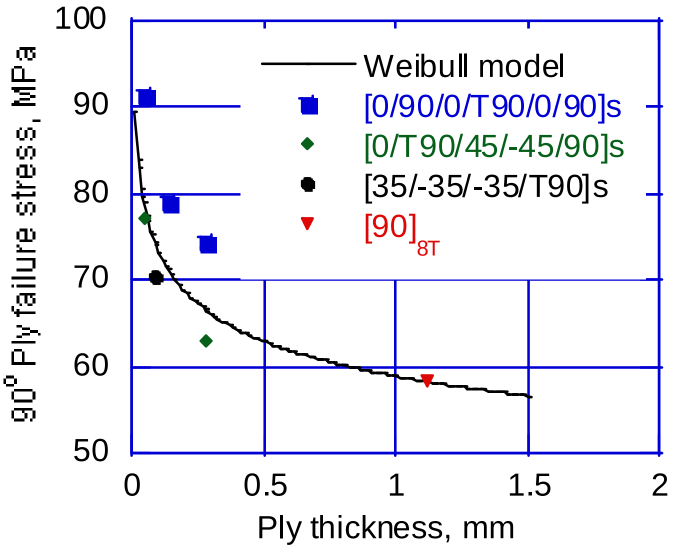

Figure 8. Average 90° ply failure in multidirectional laminates and Weibull model obtained from the $[90]_{8T}$ specimens; shape parameter 10.4.

There is a sharp decrease in the 90° tensile strength with increasing specimen thickness at the low thickness region, with a more gradual decrease thereafter. Weibull model prediction is much lower than the experimental values in all three thickness for the $[0/90/0/T90/0/90]_S$ laminate whereas it slightly overestimate for the other laminates. The degree of constrain appears to play a considerable role on the failure resistance.

4 SUMMARY

An experimental investigation was conducted to observe the onset of ply crack in a laminate using varying ply thickness in the range of 0.042 to 0.254 mm. Specimens of cross-ply and quasi-isotropic laminates were tested under uniaxial tension. The ply cracking was detected from acoustic emission and incremental loading and unloading experiments, and confirmed from microscopic examination of polished specimen edges. The curing residual stress is calculated from the thermoelastic properties of the materials and included in the ply strength prediction. The experimental data show that the transverse tensile strength in a multidirectional laminate is not a unique number but varies inversely with ply thickness. This dependence can be modeled with the Weibull failure theory which provides a closer approximation to experimental data compared to predictions which traditionally use a constant transverse strength. Thin ply used in this work which is less than one-third of the conventional ply has a great promise for the improvement of the cracking resistance in composite laminates.

ACKNOWLEDGEMENTS

This work was performed under U.S. Air Force Contract No. FA8650-05-D-5052. The authors would like to thank Ron Esterline of the University of Dayton Research Institute for sample preparation and testing. The authors would also like to thank Yasushi Kiyobayashi from Itochu Corporation, Kichiro Ishida from Mitsuya Corporation and Kazumasa Kawabe from Industrial Technology Center of Fukui Prefecture in Japan and Stephen W. Tsai from Think Composites in USA for their valuable support.

5 REFERENCES

1. R. P. Reed and M. Golda, Cryogenics, v.34, (1994).
2. H. R. Heydenreich, *Cryogenics*, v.38, (1998).
3. Y. M. Kumazawa and Y. Hayashi, ACCM 1, Kyungjoo, Korea (1998).
4. A. Pavizi, K. W. Garrett and J. E. Bailey, J. Mater. Sci., 13, 195-201 (1978).
5. D. L. Flaggs and M. H. Kural, J. Compos. Mater., 16, 103 (1982).
6. F. W. Crossman and A. S. D. Wang, ASTM STP 775. ASTM, pp 118-130, (1982).
7. A. S. D. Wang, Compos. Tech. Rev., 6, 45-62 (1984).
8. A. Crasto and R. Kim, 38th International SAMPE Symposium, pp 759-770, (1993).
9. N. J. Pagano, G. A. Schoeppner, R. Kim and F. L. Francis, Compos. Sci. and Tech., 58 1811-1825, (1998).
10. A. S. D. Wang and F. W. Crossman, J. Compos Mater., 14, 88 (1980).
11. S. Liu and J. A. Narin, J. Reinf. Plast. Compos. 11, 158-178 (Feb 1992).
12. J. Vama and L. A. Bergland, J. Reinf. Plast. Compos., 11, 708-728 (1992).
13. P. W. M. Peters and T. W. Chou, Composites, 18, 40-46 (1987).
14. H. Fukunaga, T. W. Chou, P. W. M. Peters and K. Shulte, J. Compos. Mater. 18, 339-356 (1984).
15. P. W. M. Peters, J. Compos. Mater., 18, 545-556 (1984).
16. K. Kawabe, S. Tomoda and T. Matsuo, 42nd International SAMPE Symposium, (1997).
17. Sasayama, H., K. Kawabe, S. Tomoda, I. Ohsawa, K. Kageyama and N. Ogata, 8th Japan International SAMPE Symposium & Exhibition (JISSE-8), Japan, (2003).
18. S. W. Tsai, Composites Design (1998).
19. N. J. Pagano and H. T. Hahn, ASTM-STP-617 (1977) 317.
20. R. E. Bullock, J. Compos. Mater., 8, 200-206 (1974).