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# MODELING OF FRACTURE AND DURABILITY OF PASTE-BONDED COMPOSITE JOINTS SUBJECTED TO HYGRO-THERMAL-MECHANICAL LOADING

by

#### DAVID LEE HARRIS

#### A THESIS

Presented to the Faculty of the Graduate School of the

#### MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

#### MASTER OF SCIENCE IN AEROSPACE ENGINEERING

2013

Approved by

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# PUBLICATION THESIS OPTION

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#### ABSTRACT

The objective of the research is to characterize the behavior of composite/composite joints with paste adhesive using both experimental testing and analytical modeling. In comparison with the conventional tape adhesive, joining composites using paste adhesive provides several advantages.

The carbon fiber laminate material systems employed in this study included IM7 carbon fibers and 977-3 epoxy matrix assembled in prepreg tape, and AS4 carbon fibers and 977-3 epoxy matrix as a five-harness satin weave. The adhesive employed was EA 9394 epoxy. All laminates and test specimens were fabricated and inspected by Boeing using their standard propriety procedures. Three types of test specimens were used in the program. They were bonded double-lap shear (DLS), bonded double cantilever beam (DCB) and bonded interlaminar tension (ILT) specimens. A group of specimens were conditioned at elevated temperature and humidity in an environmental chamber at Boeing's facility and their moisture absorption recorded with time. Specimens were tested at room temperature dry and elevated temperatures. DCB and DLS specimens were tested in fatigue as well as static conditions.

Two-dimensional finite element models of the three configurations were developed for determining stresses and strains using the ABAQUS finite element package code. Due to symmetry, only the one-half of the specimen needed to be considered thus reducing computational time. The effect of the test fixture is not taken into account instead equivalent distributed stresses are applied directly on the composite laminates. For each of the specimen, the distribution of Mises stress and the first strain invariant  $J_1$  are obtained to identify potential failure locations within a specimen.

#### ACKNOWLEDGEMENTS

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Center for Aerospace Manufacturing Technologies University of Missouri-Rolla Rolla, MO 65409

CAMT Task 9.2 Final Report AFRL Contract FA8650-04-C-5704

## Modeling of Fracture and Durability of Paste-Bonded Composite Joints Subjected to Hygro-Thermal-Mechanical Loading

by

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#### 1. Introduction

Bonding of carbon fiber-epoxy laminate adherends using paste adhesives is a subject of ongoing research. Generally, adhesive bonds are not currently relied on to act as unreinforced load paths in primary structure. A common means of supporting adhesive bonds is the installation of fasteners in the joint. Unfortunately, stress concentrations surrounding fastener holes are much more severe in fiber reinforced laminates than in comparable metal structures. The establishment of baseline performance data for unreinforced adhesive bonding of fiber composite laminates will be of great benefit in the design of lightweight, durable structural joints in fiber composite primary structure. This study adds to the known baseline performance data for fiber composite paste adhesive bonds.

A variety of theoretical joint configurations were subjected to finite element analysis and material testing under a range of conditions. Two-dimensional finite element models of the three configurations were developed for determining stresses and strains using the ABAQUS finite element package code. These simple joint configurations are not intended to directly simulate actual structural joints, but are intended to serve as building blocks in a broad study of a variety of relatively basic stress conditions.

The carbon fiber laminate material systems employed in this study included IM7 carbon fibers and 977-3 epoxy matrix assembled in prepreg tape, and AS4 carbon fibers and 977-3 epoxy matrix as a five-harness satin weave. The adhesive employed was EA 9394 epoxy. All laminates and test specimens were fabricated and inspected by Boeing using their standard propriety procedures. Three types of test specimens were used in the program. They were boded double-lap shear (DLS), bonded double cantilever beam

(DCB) and bonded interlaminar tension (ILT) specimens. A group of specimens were conditioned at elevated temperature and humidity in an environmental chamber and their moisture absorption recorded with time. Specimens were tested at room temperature dry and elevated temperatures. DCB and DLS specimens were tested in fatigue as well as static conditions. As part of this project, the mechanical test system at Missouri S&T was upgraded with state of the art data acquisition and controller.

Bonded Double Lap Shear (DLS) specimens were used to test bonds under primarily shear stress. The shear stress was not pure shear; the actual stress distributions were complex. The dominant failure mode of DLS specimens was laminate failure. Tape laminate specimens displayed slightly better static and fatigue performance than fabric laminate specimens. Specimens of both laminates displayed slight static and fatigue performance degradation when subjected to hygrothermal conditioning, elevated temperature test conditions, and the combination. Specimens displayed predictable, high static strength as compared to the known strengths of bulk adhesive and laminate. Consistent, predictable fatigue performance was achieved under reasonably high stresses as compared to static strengths.

Bonded Double Cantilever Beam (DCB) specimens were used to test bonds under peeling conditions. The dominant failure mode of DCB specimens was fracture at the interface of laminate and adhesive. Specimens displayed inferior static and fatigue performance as compared to the known strengths and predicted fracture qualities of bulk adhesive and laminate. Performance and failure of DCB specimens was very sensitive to the method and quality of bond surface preparation. Specimens displayed consistent, predictable fracture characteristics; such as mode I strain energy release rate, as well as consistent, predictable crack growth. Determination of fatigue performance is complicated by an unorthodox test setup and complex testing procedures and data reduction, but is still measurable and predictable.

Bonded Interlaminar Tension (ILT) specimens were used to test bonds under tensile conditions that, as opposed to peeling, may occur at locations remote from a free edge of the bond. Failure modes of ILT specimens were observed to be fracture within the laminate, within the adhesive, and at the interface between laminate and adhesive with no failure mode dominant. Specimens of both laminates displayed significant performance degradation after being subjected to hygrothermal conditioning, but elevated temperature test conditions had little effect on performance. Static strength qualities demonstrated by specimens were predictable and compared favorably to known strengths of bulk adhesive and laminate.

#### 2. Methods, Assumptions, and Procedures

#### **2.1 Materials**

The carbon fiber laminate material systems employed in this study included IM7 carbon fibers and 977-3 epoxy matrix assembled in prepreg tape, and AS4 carbon fibers and 977-3 epoxy matrix as a five-harness satin weave. The adhesive employed was EA 9394 epoxy.

#### 2.1.1 IM7/977-3 Unidirectional Tape

IM7 carbon fibers [Welsh and Wegner, 2002] and 977-3 epoxy [Cytec, 1995] matrix were arranged in a unidirectional prepreg tape (see Figure 1) at a fiber volume

fraction of 61%. This unidirectional lamina has high strength and stiffness in the longitudinal direction and low strength and stiffness in the transverse planar direction. Laminas were laid up in a general  $[(+45/-45/0/90)_x/(90/0/-45/+45)_x]$  arrangement at a thickness of 0.0052 in/ply. IM7/977-3 lamina elastic properties for layer wise analysis were obtained by using estimates provided by THICKLAM software as  $E_{11} = 23.5$  msi,  $E_{22} = E_{33} = 1.5$  (msi),  $G_{12} = G_{13} = 0.88$  (msi),  $G_{23} = 0.55$  (msi),  $v_{12} = v_{13} = 0.33$ , and  $v_{23} = 0.30$ .



Figure 1 IM7/977-3 Tape Laminate

#### 2.1.2. AS4/977-3 Fabric

AS4 carbon fibers and 977-3 epoxy matrix were woven in a five-harness satin weave fabric (see Figure 2). In the five-harness satin weave, the warp tows go over four fill tows before going under one fill tow. This fabric lamina has good strength and stiffness in the longitudinal and planar transverse directions, but lower strength and stiffness in the planar 45° direction. Laminas were laid up in a  $[+45/0/-45/0/+45/0]_s$  arrangement for some specimens and in a  $[0/+60/-60]_s$  arrangement for others. AS4/977-3 lamina elastic properties for layer wise analysis were obtained by using estimates provided by THICKLAM software as  $E_{22} = E_{11} = 10.3$  (msi),  $E_{33} = 1.5$  msi,  $G_{12} = 0.84$  (msi),  $G_{13} = G_{23} = 0.60$  (msi),  $v_{12} = 0.05$  and  $v_{13} = v_{23} = 0.30$ .

#### 2.1.3. EA 9394 Epoxy

Hysol EA 9394 epoxy is a common room temperature paste adhesive used in aerospace applications [Guess, 1995]. This adhesive could be discerned visually from either laminate system by its gray coloration (see Figure 3). It was treated as uniform and isotropic for purposes of analysis. The strength and stiffness were comparable to that of 977-3 bulk resin. Elastic properties for the adhesive are E = 0.61 msi and v = 0.37. EA 9394 over cures at 180°F, so this temperature was avoided during testing. [Loctite, 2002]



Figure 2 AS4/977-3 Fabric Laminate

#### 2.2 Specimen Configurations and Testing Arrangement

All specimen configurations addressed by this study were designed for direct comparison to analyzed models and are not intended to emulate practical bonded joints.

#### 2.2.1 Bonded Double Lap Shear (DLS) Specimens

Bonded Double Lap Shear specimens consisted of two back-to-back lap bonds (see Figure 4). The assembly was pulled in tension. DLS specimens have lower bending moments and a stress state closer to pure shear as compared to single lap shear bonds. A stiff spacer was required to minimize bending moments induced by gripping. The specimens employed in this study contained four total laminate slabs with two in the center rather than three total. Selected specimens were manufactured with artificial disbonds located in the lengthwise center of the lap bonds.

One end of DLS specimens with metal spacer is approximately 1-inch thick. None of the mechanical test machines could accommodate such a thick specimen in their grips. Therefore, special grips were designed and fabricated. DLS specimens were clamped by this custom-made wide-capacity grip and pulled in tension only (Figure 5). The measured load was divided by the total shear area of the specimen bond to obtain average shear stress, a useful comparative performance quantity.

$$t_{avg} = \frac{P}{A_{bond}} \tag{1}$$



Figure 3 EA 9394 Adhesive Layer, ILT Specimen





Adherends: 1) Unidirectional Tape: 32 Plies, IM7/977-3 Tape,  $0.0052^{\circ}$  / ply, Total thickness ~0.166 in. Layup:  $[(+45/-45/0/90)_{4}/(90/0/-45/+45)_{4}]$ 



Figure 4 Bonded Double Lap Shear Specimen

All static tests were performed under displacement control at a ramp rate of 0.04 inches per minute. Elevated temperature testing was performed at 160°F with a ten minute acclimation period prior to the ramp. Fatigue testing was performed under load control, at a frequency of 12 Hz and a load ratio of 0.25, setting peak loads at certain percentages of average static test ultimate loads.





Figure 5 Testing Arrangement and Special Grips for DLS

#### 2.2.2 Bonded Double Cantilever Beam (DCB) Specimen

Bonded Double Cantilever Beam specimens consisted of two laminate slabs bonded back-to-back (Figure 6). An artificial disbond was produced at one end to function as a crack. The crack opening was gripped by custom wire fixtures and pulled in tension only (Figure 7). When tested in displacement control, crack growth was stable, allowing for general fracture mechanics and crack growth studies. [Deobald, 2004]



Ref. ASTM D5528, ASTM D6115 Adherends: 1) Unidirectional Tape: 32 Plies, IM7/977-3 Tape, 0.0052" / ply, Total thickness ~0.166 in. Layup: [(+45/-45/0/90)\_4/(90/0/-45/+45)\_4]

> 2) Fabric Cloth: 12 Plies, AS4/977-3 Cloth, 0.014" / ply, Total thickness ~0.168 in. Layup: [+45/0/-45/0]<sub>s</sub>

Figure 6 Bonded Double Cantilever Beam (DCB) Specimen



Figure 7 Testing Arrangement for DCB Specimen

## 2.2.3 Bonded Interlaminar Tension (ILT) Specimen

Bonded interlaminar tension specimens consisted of two laminates that are already formed in to  $90^{0}$  angles bonded back-to-back (Figure 8). A set of special loading fixtures were designed and fabricated. When the ILT specimen is loaded in tension as shown in Figure 9, the adhesive is subjected to tensile stresses at the reentrant corner. [Wippich-Dienhart, undated]



Ref. CAI Test Method 4.22 in CAI Composite Test Methods Report, T6/CTM002 Adherends: 1) Unidirectional Tape: 16 Plies, IM7/977-3 Tape, 0.0052" / ply, Total thickness ~0.083 in. Layup: [(+45/-45/0/90)/(90/0/-45/+45),]

2) Fabric Cloth: 6 Plies, AS4/977-3 Cloth, 0.014" / ply, Total thickness ~0.084 in. Layup:  $[0/+60/-60]_s$ 

Figure 8 Bonded Interlaminar Tension (ILT) Specimen



Figure 9 Testing Arrangement for ILT Specimen

#### **2.2.4 Elevated Temperature Testing**

Elevated temperature testing of all specimens took place at a nominal temperature of 160°F. The test chamber containing test fixtures was warmed up from a cold start at 162°F for a minimum of two hours prior to all tests. Some tests were performed shortly after other tests, as the chamber did not require two hours to regain a stable temperature subsequent to testing. At temperature equilibrium, the chamber maintains 162°F air temperature and 157°F fixture temperature. It was not possible to achieve less than 5°F difference because the steel fixture supports conduct heat away from the chamber.

Specimens were unbagged and placed inside the chamber for the minimum amount of time that would assure a stable temperature of the specimen at the time of ramp testing. Stable temperature is considered to be  $160^{\circ}F \pm 2^{\circ}F$  at the surface of the specimen for ten minutes prior to testing for DCB and ILT specimens. This was not achievable for DLS specimens due to the large mass of the fixtures and the large area of surface contact between specimen and fixtures, so DLS specimens were considered stable at  $160^{\circ}F \pm 4^{\circ}F$  at the surface of the specimen for ten minutes prior to ramp testing. In order to minimize time at temperature, the chamber thermostat target temp was set to  $180^{\circ}F$  during specimen installation and gradually adjusted downwards as temp at the surface of the specime the passing of ten minutes from the time at which the specimen surface temperature entered the range of acceptable stable temperature.

Specimens were held at stable temperature according to the above definitions throughout ramping. Temperature at the surface of the specimen was recorded every 60 seconds during the ten minute stabilization period and during the ramp.

#### **2.3 Experimental Results**

In this section, results from the hygrothermal conditioning of selected specimens performed at Boeing are reported first. All the mechanical tests on DLC, DCB and ILT specimens were performed at Missouri S&T and these experimental results are reported next, in that order.

#### 2.3.1 Hygrothermal Behavior

Selected specimens were moisturized to an 85% RH saturation condition using a humidity chamber temperature environment of 160°F. A 2-step accelerated conditioning cycle was used. The initial step utilized a 95% RH environment at 160°F. To further accelerate conditioning, specimens were not dried first. Three specimens from each of eight groups were monitored weekly for weight increases.

Conditioning times were approximately 3-4 months at 95% RH plus 1 month at 85% RH. Specimens were considered saturated when the average cumulative weight gain (expressed as a percentage of the weight of the specimen) increased over a 7-day period by less than 0.02% of the previously recorded average cumulative weight gain. Subsequent to the conclusion of the saturation procedure, specimens were stored in airtight bags in a temperature environment of less than 32°F except during ramp testing.

Results of the hygrothermal conditioning procedure are displayed in Figure 10.



Figure 10 Moisture Content of Specimens Undergoing Hygrothermal Conditioning

#### **2.3.2 Results of DLS Tests**

Fabric and tape DLS specimens performed consistently and predictably in static and fatigue testing. Elevated temperature testing conditions and hygrothermal conditioning of specimens degraded static performance slightly. Specimens with manufactured disbonds failed at lower loads but higher average stresses than specimens without manufactured disbonds. Tape specimens as a group demonstrated slightly higher static strengths than fabric specimens. Fatigue performance data matched well against S-N curve fits. Tape specimens as a group demonstrated slightly better fatigue performance than fabric specimens. Failure within the laminate was the dominant mode.

Static results are presented in Figures 11 - 14. Results are grouped into Room Temperature-tested Dry specimens (RTD), Elevated Temperature-tested Hygrothermal conditioned specimens (ETH), Elevated Temperature Dry (ETD), and Room Temperature Hygrothermal (RTH). Static strengths are displayed as average values across each group with an indicator of scatter within each group. Where specimens failed in one lap joint before the other, results of both failures are displayed.



Figure 11 Static Performance of Fabric DLS Specimen Without Disbond



Figure 12 Static Performance of Tape DLS Specimen Without Disbond

Fatigue results are presented in Figure 15. Results are grouped into fabric specimens and tape specimens. Maximum stress levels for fabric and tape specimens

were selected as 45%, 65%, and 85% of average static failure stresses of fabric and tape specimens, respectively, without manufactured disbonds. Maximum stress levels of both fabric and tape specimens are displayed as normalized against the average static failure stress of tape specimens without disbonds,  $\sigma T = 1.84375$  ksi.



Figure 13 Static Performance of Fabric DLS Specimen With Disbond



Figure 14 Static Performance of Tape DLS Specimen With Disbond



Figure 15 Fatigue Performance of DLS RTD Specimen Without Disbond

#### 2.3.3 Results of DCB Tests

Fabric and tape DCB specimens performed consistently in static and fatigue testing. The dominant mode of failure was cracking at the interface between adherend and adhesive. This crack displayed stable growth under displacement-controlled testing. Elevated temperature testing conditions and hygrothermal conditioning of specimens appeared to have little detrimental effect on static performance. Bond surface preparation and bond curing procedures had large effects on static performance. Some tape specimen bonds were cured under a weight press; these specimens had greatly reduced static strength as compared to tape and fabric specimen bonds cured in a vacuum bag. Static results are presented in Figures 16 - 17. Results are grouped into Room Temperature-tested Dry specimens (RTD), Elevated Temperature-tested Hygrothermal conditioned specimens (ETH), Elevated Temperature Dry (ETD), and Room Temperature Hygrothermal (RTH). Static strengths are represented as the average Mode I strain energy release rate as measured over a representative period of crack propagation. This is displayed as average values across each group with an indicator of scatter within each group.



Figure 16 Static Performance of Fabric DCB Specimen



Figure 17 Static Performance of Tape DCB Specimen

Fatigue results are presented in Figure 18. Results are grouped into fabric and tape specimens. All fatigue specimens were kept at zero moisture content and tested at room temperature. Maximum stress levels for fabric and tape specimens were selected as the equivalent stresses to achieve 45%, 65%, and 85% of the critical Mode I strain energy release rate of fabric and tape specimens, respectively. Maximum stress levels of both fabric and tape specimens are converted to strain energy release rates and displayed as such in Figure 18.

DCB Cycles to Significant Crack Growth



Figure 18 Fatigue Performance of DCB Specimen

#### 2.3.3. Results of ILT Tests

Interlaminar Tensile (ILT) stresses were calculated according to Figure 19. The ILT stress  $F_{ILT}$  (psi) is equal to  $\frac{1.5 \times P \times L}{W \times T_R \times R}$ , where W = specimen width,  $T_R$  = specimen thickness at the radius, and L and R are determined by  $L = L_2 + 0.5 \times T_R$  and  $R = r + 0.5 \times T_R$  respectively.

Fabric and tape ILT specimens performed consistently and predictably in static and fatigue testing. Hygrothermal conditioning of specimens degraded static performance slightly. Specimen ILT strengths compared well with known adhesive tensile strength. Failure mode varied between specimens as well as within individual specimens. Cracking within the adhesive, at both interfaces, and between various lamina in both adherends was observed for both fabric and tape specimens. Selected failures are shown in Figure 20.



Figure 20 Selected ILT Failure Locations

Static results are presented in Figure 21. Results are grouped into Room Temperature-tested Dry specimens (RTD) and Room Temperature Hygrothermal (RTH). Static strengths are displayed for individual specimens within each group.



Figure 21 ILT Static Performance of ILT Specimen

#### 2.4 Finite Element Modeling

Two-dimensional finite element models of the three configurations were developed for determining stresses and strains using the ABAQUS finite element package code. For each of the three specimen geometries, appropriate finite element mesh was generated to get the desired accuracy. Due to symmetry, only the one-half of the specimen (DLS and ILT) is shown. Along the line of symmetry, the displacement is zero in the loading direction. The effect test fixture is not taken into account instead equivalent distributed stresses are applied directly on the composite laminates. For each of the specimen geometry and loading, the distribution of Mises stress and the first strain invariant  $J_1$  (Gosse, 2001) are obtained. Only some of the typical results are presented in Figures 22-25.



Figure 22 Misses Stresses for DLS Specimen with and without Disbond



Figure 23 Peeling Stresses for DLS Specimen with and without Disbond



Figure 24 Modeling of ILT Specimen; Loading Schematic, 2-D Symmetric Model and Details of FE Mesh for the Bent Region



Figure 25 Distribution of the First Strain Invariant J<sub>1</sub> for ILT

### **3.** Conclusions

Baseline performance data was established for a variety of basic joint configurations of paste-bonded fiber composite laminates under a range of testing conditions.

Double Lap Shear joints are subjected primarily to shear loading. These joints performed reliably and predictably in static and fatigue testing, at a range of testing temperatures, and under a variety of hygrothermal saturation conditions. Shear stresses in the joint approached the shear strength of the paste adhesive.

Double Cantilever Beam joints are subjected primarily to concentrated tensile stresses at a free edge, or peeling. These joints generally proved unable to take full advantage of the tensile strength of the structural adhesive. Preparation of the bond surfaces prior to assembly and variations in curing procedure had large effects on the void content of the bond and on performance. However, models were developed that are capable of predicting bond-peeling performance in static and fatigue loading.

Interlaminar Tension joints are subjected to moderately concentrated tensile stresses remote from a free edge. These joints generally proved able to take advantage of much more of the tensile strength of the structural adhesive than joints subjected to peeling were able to. Tensile stresses in the joint approached the tensile strength of the paste adhesive. Static performance of joints was reliable and predictable.

Strict procedures for bond surface preparation and curing are critical to obtaining a high quality bonded joint between fiber composite laminate adherends. In particular, high void content greatly degrades the strength and durability of a joint and must be minimized. The minimizing of high peeling stresses at a free edge of the bond is an important design consideration.

Two-dimensional finite element models of the test specimens showed the high strain regions that qualitatively agree with the failure locations in the experiments. So modeling of actual structural joints subjected more realistic loading could be used with some degree confidence.

#### 4. Recommendations

The objective of this project was to develop a basic understanding of pastebonded composite/composite joints by conducting a systematic study of simple joints under pure shear or peeling mode. It is highly recommended that in the future studies a more representative real life structural joints should be included.

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## 6. Publications and Inventions

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- Harris, D. L. Dharani, L. R. and T. R. Berkel, "Characterization of Paste-Bonded Composite/Composite Joints Subjected to Static and Fatigue Loading," Manuscript under preparation for a refereed journal, May 2008.

#### VITA

David Lee Harris was born in Trenton, Missouri. One year later his family moved to Cameron, Missouri, where he remained throughout his elementary and secondary education. In 2000, Mr. Harris graduated as the salutatorian of Cameron High School. He received a Bachelor of Science degree in Aerospace Engineering from the Missouri University of Science and Technology, then named University of Missouri-Rolla (UMR) in May 2004, graduating magna cum laude. Mr. Harris remained at Missouri S&T to pursue his graduate studies. He worked as a Graduate Research Assistant on an AFRL funded project and received a variety of support including a department fellowship and the Chancellor's Fellowship. As a Graduate Teaching Assistant, he taught Mechanics of Materials laboratory course for five semesters.

As a junior undergraduate, Mr. Harris was a founding member of the Miners in Space (microgravity experimental / competition and educational outreach team), held the Team Leader position for two years, flew aboard NASA's Weightless Wonder V microgravity testbed KC-135A aircraft in July 2003, and remained involved with team activities such as elementary and secondary school presentations throughout his graduate schooling.

From June 2007 to September 2012, Mr. Harris worked in Wichita, Kansas as a structural analyst and lead engineer for Spirit AeroSystems on the Boeing 747-8 and Boeing 737-NG programs. Since September 2012, he has been working in Wichita as a fatigue and damage tolerance analyst for Airbus Americas Engineering on the Airbus A350 program. Mr. Harris received his Master of Science degree from the Missouri University of Science and Technology in December 2013.