HIGH ACCURACY MEASUREMENT OF PREPREG LEVEL OF IMPREGNATION USING NON-CONTACT ULTRASOUND

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ABSTRACT

Composite prepreg is an increasingly important product in the aerospace industry, as its use continues to grow as an ingredient in primary aircraft structures. A vital material property, the level of impregnation (LOI), plays an important role in the quality and performance of prepreg. Level of impregnation has even deeper effects upon the performance of out-of-autoclave prepreg, as lower pressure curing environments cannot overcome the effects of unwanted variation in LOI, often leading to overly dry or resin-rich areas in final cured laminates. Furthermore, the ability to adequately measure prepreg LOI has been lacking as industry standard methods, such as the destructive water pickup test, have limited accuracy at approximately +/- 5%, and other methods, such as MicroCT (X-ray) are expensive and time consuming.

Advancements in non-contact ultrasound (NCU) have allowed for transmission of relatively high frequency ultrasound through materials in air, without the need for contact or liquid coupling. These advancements enable NCU to transmit ultrasonic signals through uncured prepreg, recording vital material properties instantaneously. As ultrasound is naturally sensitive to material porosity, the correlation between prepreg LOI and ultrasonic transmittance in NCU is nearly perfect ($R^2 \approx 1$) when analyzed at the appropriate frequency with suitable ultrasonic transducers and electronics, thus allowing for prepreg LOI measurement at an accuracy of 0.5% or greater. The technique of measuring prepreg LOI using NCU is applied during the manufacturing process for immediate feedback as well as offline for high-resolution imaging. Contrary to destructive methods such as water pickup, which provides a single measurement data point for a material of several square inches, NCU is non-destructive and provides localized LOI measurement with spatial resolution of $1mm^2$ or better. A study through a small business innovation research award (SBIR) with the US Air Force Research Laboratory demonstrates experimental data proving the extremely high correlation between NCU transmittance and prepreg LOI, and discusses inexpensive standardized processes which can be used to measure and certify prepreg level of impregnation industry-wide. A standard method for industry-wide prepreg measurement using NCU has been balloted with ASTM and is currently in consideration with the D30 subcommittee on composites materials.

1. INTRODUCTION

With support from an SBIR award sponsored by the U.S. Air Force Research Laboratory at Wright Patterson Air Force Base, The Ultran Group, Inc. (Ultran) and Aurora Flight Sciences Corporation (Aurora) collaborated to develop a test method capable of being standardized for measuring resin level of impregnation (LOI) in composite prepreg materials. The approach utilizes Ultran’s non-contact ultrasound (NCU) technology in through-transmission mode to establish a correlation between NCU signal amplitude and prepreg LOI. The specific prepreg studied was an out-of-autoclave (OOA) unidirectional carbon prepreg from Cytec (IM7/5320-1), provided in three different formats that correspond to different levels of impregnation: hand layup (HLU) for low LOI, automated tape layup (ATL) for medium LOI and automated fiber placement (AFP) for high LOI.

During this SBIR effort, the feasibility of measuring prepreg LOI using non-contact ultrasonic transmittance was determined. Furthermore, the accuracy of measurement was found to meet the targeted goal to be more precise than 1% under certain conditions. Initially the optimal frequency was determined by performing correlation analysis at 8 different frequencies. After the optimal range was determined (500 kHz to 1 MHz), in depth analysis was performed to identify effects of other variables which may further enhance measurement accuracy through a design of experiment (DOE) test matrix which considered the following variables: Ideal nominal frequency (500 kHz, 700 kHz and 1 MHz), transmission ultrasound, top surface reflections, bottom surface reflections, focused ultrasound, planar (non-focused) ultrasound, and prepreg LOI (high, medium, and low). 54 conditions were analyzed with 4 replicates each (216 NCU scans total).

From the multiple condition/variable analysis, we were able to achieve correlation producing R^2 values as high as 99.5% and S values (error of measure) as low as 0.5%. The simplest condition, which exhibited high correlation was 1 MHz in through transmission with planar (non-focused) transducers using a single linear correlation function produced R^2 of 98.2% and S = 1.08%. However, by combining variables together and by increasing the number of correlation functions (i.e. one function for each product type; AFP, ATL, and HLU), the correlation and accuracy of measurement can be further increased. Following this study, we are able to conclude the best combination for ease and robustness as well as accuracy of measurement. The current recommendation, 1 MHz in through transmission with either one or three linear functions, provides the basis for our proposed ASTM standard method as applicable to IM7/5320-1 prepreg.

An independent secondary test, known as guided water pickup (GWPU), was performed upon samples analyzed during this period. However, this was not the only secondary technique investigated. Further testing was conducted using MicroCT. Results from MicroCT did not produce accurate numerical data regarding prepreg LOI, but we believe there are merits in the qualitative data acquired.

While most of the research evaluated, AFP, ATL, and HLU prepreg samples with the backing paper removed, additional work was completed to determine whether or not backing paper exhibited unwanted effects upon NCU measurements. These results demonstrated that paper backing did not have negative effects upon the correlation between NCU and prepreg LOI, as the R^2 value actually increased in comparison to data gathered without backing. Towards the end of the project, in-depth analysis was conducted upon samples of HLU prepreg. This analysis was used to determine correlation and error of measurement. Although the analysis was conducted upon just one type of prepreg, the results further indicated a strong correlation with backing paper present and an error of measurement of only 1%.

1.2 Prepreg material

After discussion at the kick-off meeting, the preferred material of interest was Cytec’s OOA IM7/5320-1 prepreg material. Thus, Cytec sent Ultran and Aurora thirty (30) sheets each (24 inches wide x 12 inches long) of three different IM7/5320-1 out of autoclave (OOA) intermediate modulus carbon fiber reinforced toughened epoxy prepregs. The different materials corresponded to varying levels of impregnation, as summarized in Table 1. The types of materials consist of hand layup (HLU), automated tape layup (ATL), and automated fiber placement (AFP).

Table 1. Prepreg Materials Provided by Cytec

Material	Architecture	Type	LOI	Size of Each Sheet	Number of Sheets
IM7/5320-1	Unidirectional	HLU	Low	24" wide x 12" long	30
IM7/5320-2	Unidirectional	ATL	Medium	24" wide x 12" long	30
IM7/5320-3	Unidirectional	AFP	High	24" wide x 12" long	30

Various sheets of each material were cut in half, as shown in Figure 1, to essentially create 12" x 12" specimens for NCU scans. The sheets were cut in order to make them suitable for NCU scans on the portable setup (described in Section 3.2), as well as for repeatability. Note that the paper backing was removed prior to scanning the materials for most of the analysis presented. However, initial testing was performed upon samples with paper backing to demonstrate applicability inline during prepreg manufacturing.

Figure 1. Prepreg Sheets from Cytec Cut in Half

2. Experimentation

2.1 Non-Contact Ultrasound (NCU)

Ultran's next generation, turnkey inspection system, the U710 (Figure 2), was used to conduct NCU scans of the 6 prepreg samples. Four cylindrical supports are used to prop up the 12" x 12" samples, with the paper backing removed, to allow for through-transmission ultrasonic scanning through air. Due to the size of the transducers, the scan area was reduced to a 10" x 10" portion of each to eliminate edge effects from the generated C-scan images. The four corners of the 10" x 10" scan area were marked prior to scanning (Figure 3), such that areas of the samples could be easily detected in Ultran's SecondWave^{TM} post-processing software for subsequent analysis.

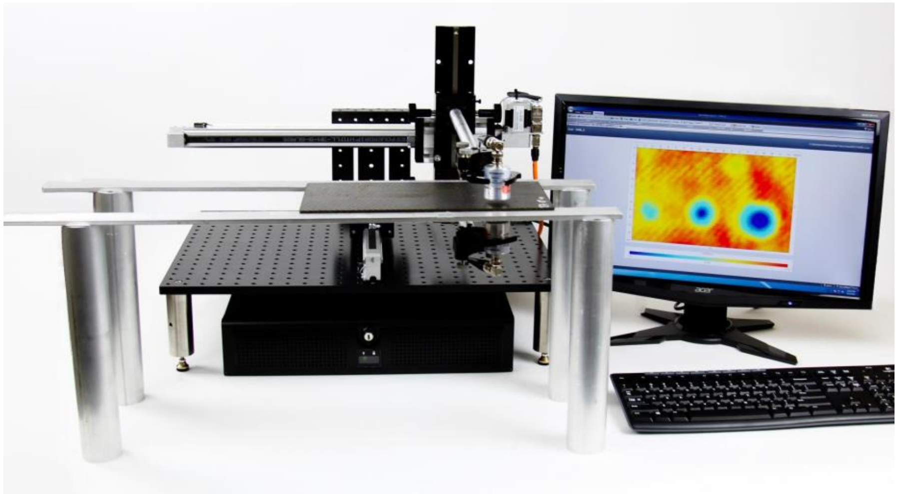


Figure 2. Ultran's U710 Ultrasonic System used for NCU Scans of Prepreg

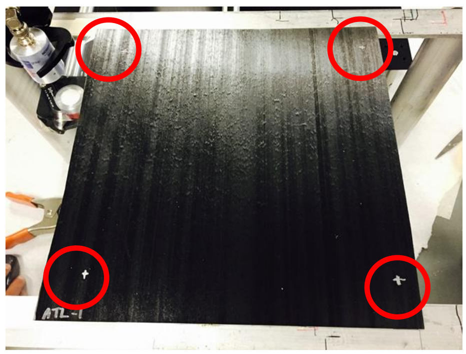


Figure 3. Four Corners of 10" x 10" Scan Area Marked on Each Sample

2.2 Guided Water Pickup Test

The standard water pickup test used to measure the level of impregnation upon prepreg specimens is expected to have accuracy of around +/-5%. Each specimen is weighed, and then clamped between two plates which are each covered with a non-porous film such as Teflon. Approximately 0.6 inches of material (aligned with the unidirectional fiber orientation) extends beyond the clamped plates, as shown in Figure 4. Approximately 0.2 inches of the material is then immersed into room-temperature water for 5 minutes, and then weighed a second time after removing any exterior water with blotting paper. The smaller the weight increase, the greater the degree of impregnation. Thus, the AFP material is expected to have less of a weight increase than the HLU material.

Figure 4. Prepreg Specimen between Teflon-covered Clamped Plates for Water Pickup Test

Nevertheless, one reason why a sample of similar impregnation could soak up more or less water is due to the fact that a well impregnated area in between the water-exposed section and the rest of the sample may obstruct the flow of water into the prepreg. Because the water pickup test attempts to measure the aggregate or average value of impregnation upon a sample, only one value of impregnation is obtained for the whole sample area. Two samples may have equal levels of impregnation across the total areas; however, if one sample has a well impregnated region which obstructs water flow into the less impregnated region, it will absorb less water overall and register a lower LOI. This non-representative sample selection may be a significant factor affecting the accuracy of the water pickup test.

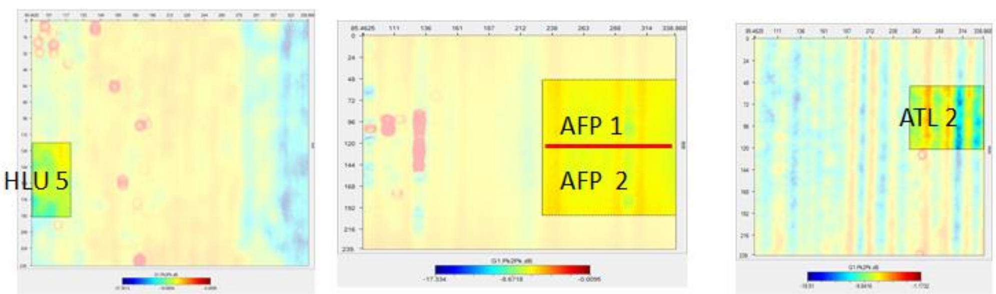


Figure 5. Selected Sections of Prepreg for Guided Water Pickup Tests

Assuming that NCU transmittance can in fact inform the level of impregnation on a localized level, we can image samples prior to the water pickup test to pre-select areas with better uniformity. Using this method we can guide our water pickup test by choosing more uniform sections, as shown in Figure 5. This method can increase the accuracy of the water pickup test and provide us with a better secondary method to validate the accuracy of NCU measurements. Acquired results validate that the guided water pickup test has significantly higher accuracy than the standard water pickup test.

2.3 Design of Experiments (DoE) test matrix

A design of experiment matrix was created to evaluate the effect of the following variables in conjunction with each other: Frequency, focused ultrasound, reflection mode, and transmission mode. The prior analysis was conducted solely in through transmission mode at a number of frequencies. In essence, it was concluded that the frequencies of 500 kHz, 700 kHz, and 1 MHz were best suited for these prepreg products and guide the DOE development. It was also hypothesized that ultrasonic reflection from the top and bottom surfaces may reveal complementary information to data acquired in through transmission.

Focused ultrasound may also provide detail around the edges of sections with stark contrast. The DoE matrix is shown below in Table 2.

Table 2. DoE Test Matrix Variables

DOE Test Matrix		
Factors	Levels	Values
Ultrasonic Frequency	3	500 kHz, 700 kHz & 1 MHz
Focus Parameter	2	Focused & Planar
Inspection Mode	3	Top reflection, bottom reflection, & through transmission
LOI	3	High, Medium, & Low

The following are descriptions of each parameter in the matrix:

- Sample count: Number of samples or replicates for each condition. 4 samples (6" x 6") were chosen; 2 to be designated for guided water pickup following NCU and 2 for MicroCT.
    - Under 18 ultrasonic conditions for 54 variable combinations (considering 3 LOI levels) with 4 replicates, this equates to 216 samples analyzed.
- Prepreg type: Prepreg at 3 levels of impregnation were provided. These are the same 3 types provided by Cytec: AFP, ATL, and HLU.
- Inspection Mode: Three inspection modes were used; through transmission, reflection from top surface, and reflection from bottom surface (see Figure 6).

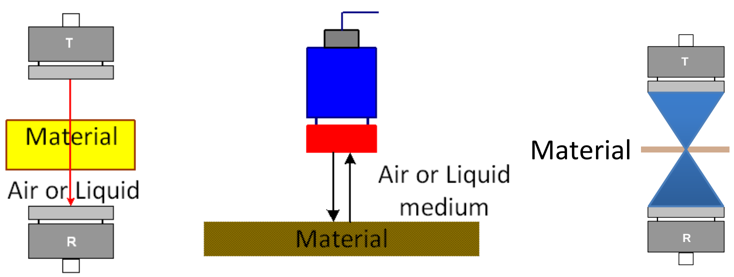


Figure 6. Modes of Analysis: Through Transmission Planar (left), Pulse-echo Reflection (center) and Through Transmission Focused (right)

- Transducer Frequency: Three frequencies were used; 500 kHz, 700 kHz and 1 MHz.
- Transducer Focus: Two types of transducers were used for analysis. These included standard planar transducers, which were used in prior analysis as well as focused transducers to create a small spot size for higher spatial resolution.

The DOE matrix, which considers the total number of combinations possible (full factorial), includes 216 scans. All of the NCU scans and secondary tests (using GWPU) have been completed. The compiled results provide the basis for our draft ASTM standard submitted for prepreg inspection (IM7/5320-1) using NCU.

3. RESULTS

This section describes the results associated with the NCU measurements and secondary tests used to determine the accuracy and correlation of the NCU analysis with prepreg LOI (%).

3.1 Initial Analysis - Frequency Determination

As described above, two samples of each of the three prepreg types (AFP, ATL, & HLU) were scanned at eight different ultrasonic frequencies for the initial analysis. Figure 7 shows examples of C-Scan images at 1 MHz frequency. Additional C-Scan images are provided in the Appendix.

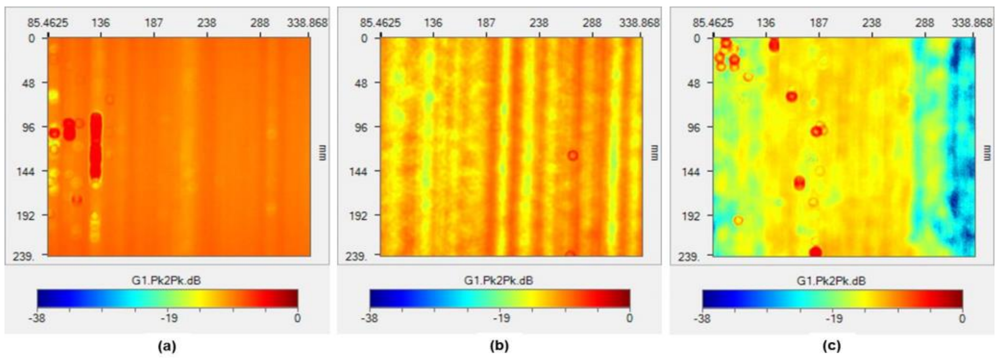


Figure 7. 1 MHz C-Scan images of (a) AFP, (b) ATL and (c) HLU prepreg

Using Ultran's SecondWave Research Studio software, four values were calculated for each scan: 1) Average Amplitude, 2) Standard Deviation, 3) Minimum Amplitude and 4) Maximum Amplitude. Figure 8 indicates a strong relationship between average ultrasonic amplitude and prepreg LOI at all frequencies but most clearly at 500 kHz, 700 kHz, and 1 MHz. The strongest correlation was found at 700 kHz and 1 MHz. A prepreg sample with high LOI produces higher average amplitude than a prepreg sample with low LOI at given transducer frequency.

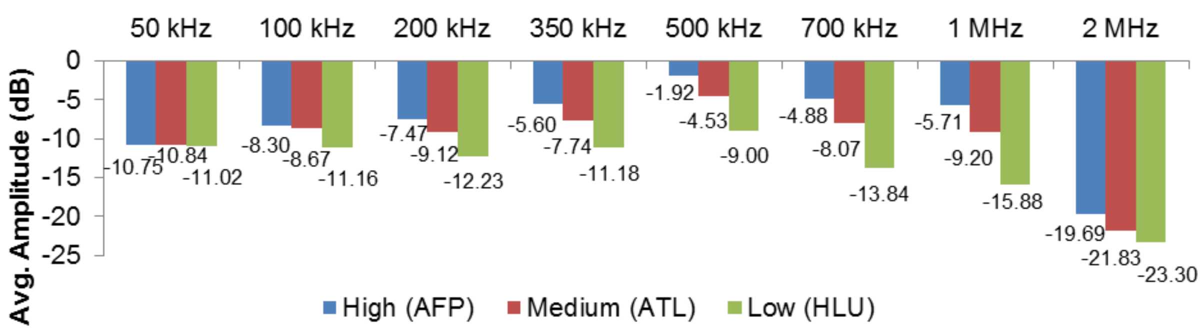


Figure 8. Average Amplitude for Each Analyzed Frequency Against LOI

3.1.1 Initial Analysis – Secondary Test and NCU Correlation

As mentioned previously, one of the key secondary tests conducted was the Guided Water Pickup (GWPU) test. When applied to the data from the initial analysis, a more accurate correlation between

NCU transmittance and prepreg LOI is achieved. Figure 9 is a graphical representation of the measured NCU transmittance values at 1 MHz vs. the LOI as measured from the guided water pickup test. From this initial analysis, the correlation between transmittance at 1 MHz and prepreg LOI as measured by guided water pickup is calculated to have a very high R^2 value of 96.30%.

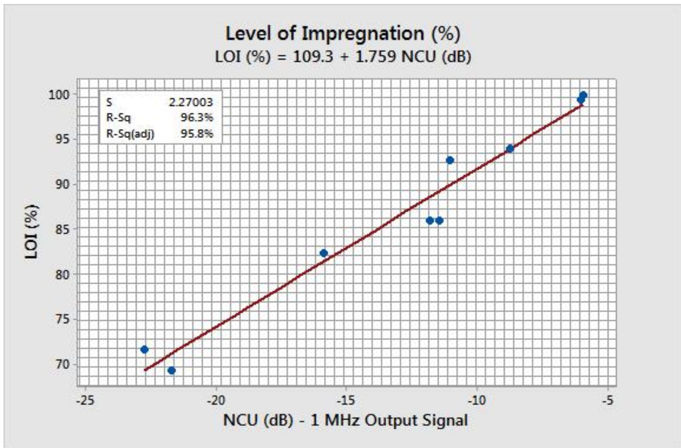


Figure 9. Graphical Representation of GWPU test and 1 MHz NCU Data

The correlation function between NCU transmittance and prepreg LOI for this material is as follows:

$LOI = 1.76 * T_x + 109.31$, where T_x is NCU transmittance.

Correspondingly, because these same samples were previously analyzed at multiple frequencies in NCU through transmission, we can compare the correlation values between frequencies.

Table 3. Correlation Comparison Between Frequencies

Freq.	**R^2 Value for Linear Correlation**	**Linear Correlation Equation**
500 kHz	89.81 %	LOI (%) = 101.92 + 2.128 * T_x (dB)
700 kHz	92.71 %	LOI (%) = 107.54 + 1.857 * T_x (dB)
1 MHz	96.30 %	LOI (%) = 109.31 + 1.759 * T_x (dB)

From Table 3, we can see that for linear correlation against the single variable of NCU transmittance, the highest correlation is achieved at 1 MHz. The R^2 value for 500 kHz and 700 kHz is still significant. By raising the correlation function to a 2nd order polynomial, the R^2 value increases but not significantly. For this reason, we plan to consider the linear function for ease of use.

3.2 Multiple Variable Analysis[1]

Following the initial analysis in the Phase I effort, which informed the ideal frequency range for analysis of the provided IM7/5320-1 material, Ultran performed analyses to determine the ideal test parameters for measurement of LOI. NCU analysis modes included transmission, reflection, and focused/planar ultrasound. In addition, multiple frequencies were considered by analyzing at 500 kHz, 700 kHz, and 1 MHz. The parameters tested are shown in Table 2.

Analyzing the provided material under a number of separate conditions also affords the opportunity to statistically process data whereby the combination of test conditions can be considered as input variables into a correlation function which calculates LOI. For this analysis, a combination of planar and focused transducers produced by The Ultran Group were used, as listed in Table 4 below.

Table 4. Transducers used for multiple variable analysis

NCU Transducer Model	Nominal Frequency	Active Dimensions	Focus
NCG500-D19	500 kHz	19 mm (diameter)	Planar
NCT500-S13-C25	500 kHz	12.5 mm x 12.5 mm	25 mm Cylindrical
NCT700-D19	700 kHz	19 mm (diameter)	Planar
NCT700-S13-C13	700 kHz	12.5 mm x 12.5 mm	12.5 mm Cylindrical
NCG750-S13-P25	750 kHz	12.5 mm x 12.5 mm	25 mm Spherical
NCT1-D13	1 MHz	13 mm (diameter)	Planar
NCT1-S13-C13	1 MHz	12.5 mm x 12.5 mm	12.5 mm Cylindrical

In addition, a combination of cylindrically (line) focused and spherical (point) focused transducers were used. The theoretical minimum spot size for cylindrically focused transducers is less than one wavelength in air when used in a cross-beam configuration (focal line of transmitter is rotated 90% relative to focal line of receiver), while the minimum theoretical spot size for point focused transducers at the focal length is two wavelengths in air. The actual achieved spot size was on the order of approximately 1mm for both cylindrically and spherically focused analyses. Focused ultrasound has certain tradeoffs; reduced spot size but lower signal to noise ratio. It was found that the focused image data was useful in a complementary fashion to the planar transducer images but not as a replacement.

Corresponding to the DOE matrix, Figure 10 lists potential variables for consideration in this analysis. For example, a detailed regression analysis can consider the degree of correlation and corresponding correlation function if transmission values from more than one frequency are input into the equation. Even larger combinations of variables, such as focused, multiple frequency, and both transmission and reflection can also be considered. However, a combination of accuracy and practicality must be balanced when considering the ideal test conditions for a proposed standard method.

[1] Method is patent pending.

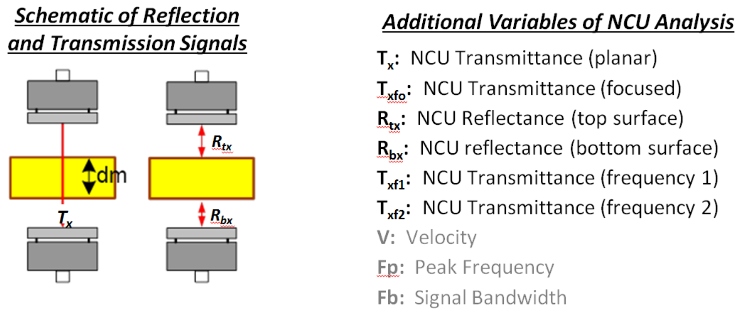


Figure 10. Representation of multiple conditions and variables for NCU analysis

As described above, 4 samples (6" x 6") of each of the three prepreg types were scanned under these 3 frequencies, 2 transducer types and 3 inspection methods which produced a total of 216 NCU scans. These NCU scans were processed using Ultran's SecondWave Research Studio software, and average amplitude was calculated. After NCU analysis, two of the four 6" x 6" samples of each type (AFP, ATL, and HLU) were cut into 4 square additional sections each to produce 8 total 1.5" x 1.5" square samples per type which would be tested using the guided water pickup test. This produced a total of 24 1.5" x 1.5" (40 mm x 40 mm) samples which were measured for actual LOI through the destructive GWPU test to correlate against the numerous NCU conditions analyzed.

Figure 11 shows captured C-scan images at 1 MHz in through transmission for all 24 sections tested under GWPU. Both the NCU transmittance value and the LOI, as measured by the guided water pickup test (GWPU), were recorded. From this data even minor variations within samples (i.e. ATL and HLU) are captured by NCU and validated from the GWPU LOI measurements. For example, the left two sections of the ATL samples (ATL #9 and ATL #11 in Figure 17) exhibit slightly higher transmittance than the right two samples. This exact phenomenon was also measured during GWPU where the LOI on the left is slightly higher than that on the right (93.41% and 93.31% versus 92.89% and 92.14%). The same behavior is apparent within the HLU sample as well.

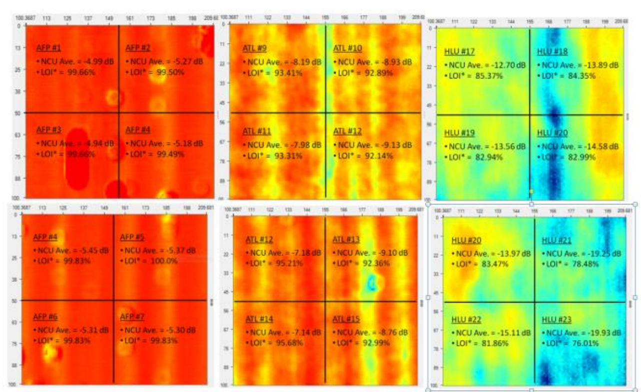
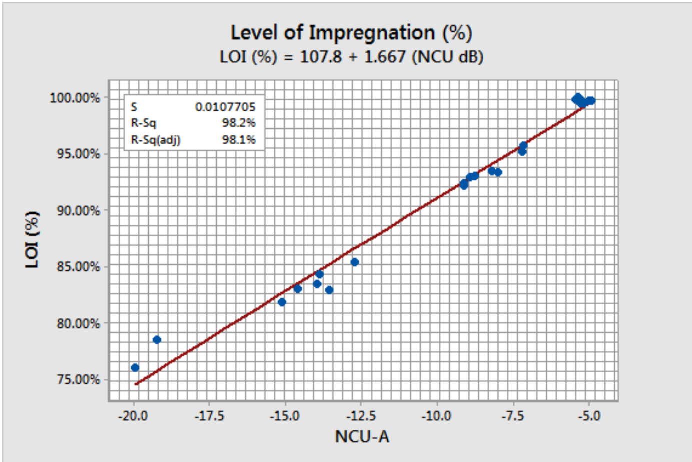


Figure 11. Images of samples analyzed during multiple variable analysis using single condition – 1 MHz through transmission.

Performing a linear regression analysis for 1 MHz through transmission upon the 24 sample sections evaluated generates a close relationship between NCU transmittance and LOI. Figure 12 graphically represents this linear relationship. As seen from the chart, the R^2 value is 98.2% and the S value (error of measurement) is 1.08%.



Prepreg Type	Sample #	NCU Tx (dB)	GWPU (%)	LOI (%)
AFP	1	**-4.99**	0.34%	**99.66%**
	2	**-5.27**	0.50%	**99.50%**
	3	**-4.94**	0.34%	**99.66%**
	4	**-5.18**	0.51%	**99.49%**
	5	**-5.45**	0.17%	**99.83%**
	6	**-5.37**	0.00%	**100.00%**
	7	**-5.31**	0.17%	**99.83%**
	8	**-5.30**	0.17%	**99.83%**
ATL	9	**-8.19**	6.59%	**93.41%**
	10	**-8.93**	7.11%	**92.89%**
	11	**-7.98**	6.69%	**93.31%**
	12	**-9.13**	7.86%	**92.14%**
	13	**-7.18**	4.79%	**95.21%**
	14	**-9.10**	7.64%	**92.36%**
	15	**-7.14**	4.32%	**95.68%**
	16	**-8.76**	7.01%	**92.99%**
HLU	17	**-12.70**	14.63%	**85.37%**
	18	**-13.89**	15.65%	**84.35%**
	19	**-13.56**	17.06%	**82.94%**
	20	**-14.58**	17.01%	**82.99%**
	21	**-13.97**	16.53%	**83.47%**
	22	**-19.25**	21.52%	**78.48%**
	23	**-15.11**	18.14%	**81.86%**
	24	**-19.93**	23.99%	**76.01%**

Figure 12. Linear regression curve for NCU transmittance at 1 MHz and prepreg LOI (left) and tabular results for all 24 samples.

The table to the right of Figure 12 lists the actual transmittance values under this single set of conditions (1 MHz transmission) and the measured LOI values from GWPU. It should be noted that this is the most simple of conditions with the lowest order regression analysis and only a single equation to fit all product types. To further increase accuracy the number of the input variables and the order of regression can be increased, while also considering 3 separate equations; one for each product type. Any one of those inclusions will increase the correlation between NCU measurements and LOI. A vast amount of data was acquired during this analysis, including images using focused ultrasound as well as reflections from top and bottom surfaces.

From the focused image of the AFP sample, we notice that the contrast in NCU transmittance between regions is much more defined and sharp as compared to the planar image in Figure 11 which demonstrates more gradual changes in transmittance. This is especially evident when observing the vertical striping pattern in this sample. The effect is somewhat easily explained by the smaller beam spot size produced using focused ultrasonic transducers. We can also see that the pin-holes are smaller as compared to the planar image but not quite as small as a single pixel. Further analysis of the pin-hole itself (and its actual size in the down-fiber direction) could help explain why they appear larger even in the focused image.

Reflection imaging was also conducted during this phase of analysis. This task was conducted by using a single transducer in pulse-echo mode, where the same transducer both sends and receives signals. Through experience, it is known that the received signal is the reflection from the top surface because back-wall reflections, which travel through the material and reflect from the bottom surface, are lost within the initial top surface reflection, as they appear slightly later in time. The results from this approach were largely unknown, and there appears to be significant variation in signal amplitude. The areas where signal amplitude is high, appear to be better reflectors of ultrasound, while the lower amplitude areas are most likely scattering the reflected signal in many directions. It is unlikely that absorption is responsible for the lower amplitude due to the acoustic mismatch between air and the composite prepreg, which causes reflection from the surface of nearly all acoustic energy. Altogether, reflectance data did not provide strong correlation to prepreg LOI. However, further studies may provide insight into the value of prepreg reflectance measurements.

In Table 5, the correlation values are compared under conditions which consider multiple input variables as well as quadratic correlation functions and three separate linear functions for each product type. While the highest achieved correlation uses a combination of 1 MHz planar and 500 kHz focused (both in transmission) with three linear functions, the most practical method which also achieves high correlation values is the use of 1 MHz planar transmission (single input variable). By using this condition, but increasing the number of linear functions to three, the R^2 value increases to 99.59% and the S value decrease to 0.53%. As advised by our associates at the Pennsylvania State University statistics department, including separate linear functions can consider factors which may vary between ranges of impregnation or product type. It is also advisable to avoid higher order regression functions (i.e. quadratic), as one can inadvertently fit outliers or unwanted outside factors.

Table 5. Correlation metrics for selected combinations of input variables and regression models

	1 MHz Planar (single Variable)	1 MHz & 500 kHz Planar	1 MHz Planar & 500 kHz Focused	1 MHz planar & 1 MHz focused
Linear Function (single equation)	**R^2 = 98.16% S = 1.08%**	**R^2 = 98.40% S = 1.03%**	**R^2 = 98.27% S = 1.07%**	**R^2 = 98.79% S = 0.89%**
Quadratic Function (single equation)	**R^2 = 99.43% S = 0.61%**	**R^2 = 99.57% S = 0.56%**	**R^2 = 99.52% S = 0.59%**	**R^2 = 99.67% S = 0.49%**
Linear Function (three equations)††	**R2 = 99.59% S = 0.53%**	**R^2 = 99.67% S = 0.49%**	**R2 = 99.77% S = 0.43%**	**R^2 = 99.59% S = 0.55%**

The above conditions have correlation functions associated with them, to allow for direct calculation of prepreg LOI from measured NCU values. For example, the equation for 1 MHz planar transmission (single input variable) with a linear function is as follows:

$LOI(\%) = 1.078 + 0.167 * T_x$, where T_x is the measured NCU transmittance value in dB.

If we consider the combination of variables, such as 1 MHz planar and 500 kHz planar (both in transmission) with a single linear function, the equation is as follows:

$LOI = 0.025 * T_{x1} - 0.011 * T_{x0.5} + 1.078$, where T_{x1} is the transmission value at 1 MHz and $T_{x0.5}$ is the transmission value at 500 kHz, both in dB.

As listed in Table 5, the accuracy of measurement can be increased by separating the correlation function into 3 equations, one for each prepreg product; AFP, ATL, and HLU. For the high accuracy recommended solution of a single variable of planar transmission at 1 MHz, the separate equations are as follows:

AFP: $LOI = 0.012 * T_x + 1.058$

ATL: $LOI = 0.012 * T_x + 1.031$

HLU: $LOI = 0.012 * T_x + 0.998$

By separating out the correlation equations by product, the R^2 value increases to 99.59% and S decreases to 0.53%. Conducting an experiment under a single condition, 1 MHz planar transmission, and separating the correlation equations by either product or range of LOI or transmittance, is relatively straightforward. A single condition test is the simplest form of measurement and algorithms in software can easily be created to apply the appropriate correlation/translation equation when measurements fall within specified ranges. Another option in software is for a user to preset the product line (in this case AFP, ATL, or HLU), and an appropriate equation will be used for all acquired data during a manufacturing run or offline test batch. For this reason, the suggested method for the initial ASTM standard method comprises using transmission at 1 MHz for IM7/5320-1 prepreg with the option to use either one or three linear correlation functions for each product type.

3.3 Paper Backing Analysis

One of the tasks of the concurrent Phase I effort is to study the effect of paper backing upon NCU and whether or not it has an adverse effect upon LOI measurement. This analysis is important as an online test standard may need to be conducted upon material while backing paper is present. Ultran will look deeper into the effects of paper backing during the final reporting period. Nevertheless, some initial analyses were conducted upon the same samples as the early frequency analysis study. From Figure 13, the values and patterns as measured by NCU appear similar whether or not the paper backing is present. Additionally, a "pin-hole" effect noticed in certain samples is less (if at all) present in samples with paper backing.

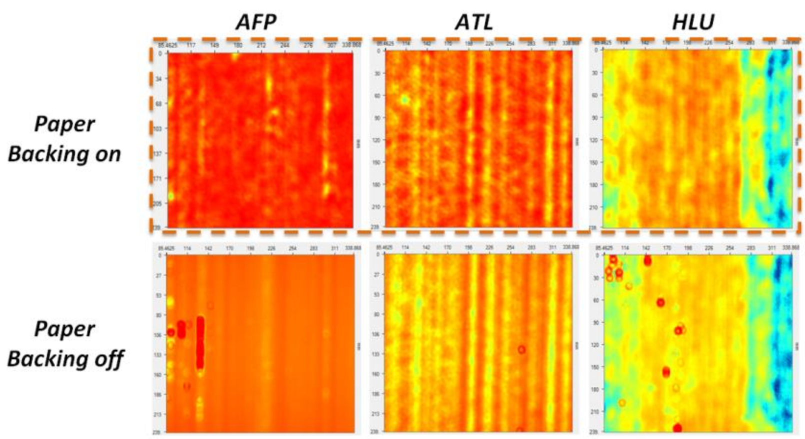


Figure 13. Images comparing NCU transmittance at 1 MHz with and without paper backing.[2]

Linear regression of data from initial analysis during Phase I measures an R^2 value of 97.0% which is actually slightly higher than the same correlation performed upon these samples with the paper backing off (R^2 = 96.3%) which was measured during the initial frequency analysis study and shown in Figure 9. The presence of paper backing seems to have positively affected the accuracy of LOI measurement. One potential reason for this is the elimination of pin-holes as noticed in some samples where backing paper is removed.

[2] Images with and without backing are included on the same color pallet and not adjusted for bias. Overall, signal amplitude was measured to be higher upon samples with paper backing. A more correct visual comparison should adjust the color pallet to correct for such bias.

During the final reporting period of Phase I, additional prepreg studies were performed to study the effect of paper backing. Because the HLU prepreg had the greatest variability in LOI during prior measurements without paper backing, it was chosen as the preferred material to investigate with paper backing in the limited remaining time of the Phase I period. Similar results and level of correlation can also be obtained for all other types of prepregs by essentially following the same analysis methods highlighted here.

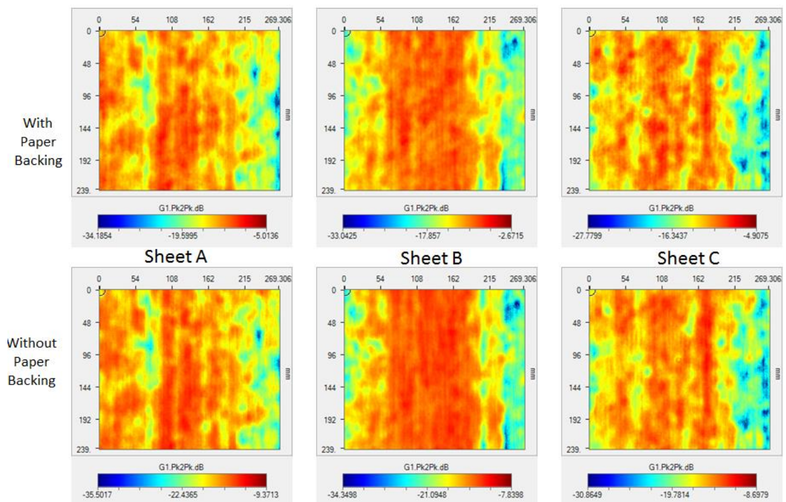


Figure 14. Qualitative comparison of HLU prepreg with and without paper backing.

First, for the purpose of qualitative comparisons, three HLU sheets that measured 254 mm x 254 mm were scanned with and without paper backing. As shown by the C-scan images in Figure 14, the transmittance data through samples with and without paper backing is remarkably similar. While the dB scale has shifted up due to the presence of the paper backing, the subsequent regression analyses (as shown in the following section) were performed such that the % LOI remains the same for the same material, regardless of whether paper backing exists. That is, an equation with some bias relative to the equation for LOI without paper backing, would be used to quantify the LOI of a material with paper backing or vice versa. While the proposed ASTM standard method for online testing will likely include transmission through prepreg with backing paper, the team may also consider proposing NCU measurement through material with paper backing for the offline test if further data demonstrates that it does not adversely affect LOI measurements.

To quantify the correlation, two additional HLU sheets were scanned with paper backing using 1MHz NCU. Following NCU analysis, each sheet was cut into 9 equal size squares of about 80 mm by 80 mm. This produced a total of 18 samples for guided water pick up test. Actual LOI (%) was measured through destructive GWPU test, and regression analysis was performed to determine the level of correlation between NCU transmittance through HLU Prepreg with paper backing and LOI (%).

3.4 ASTM Standard

The proposed method, which has been balloted to ASTM subcommittee D30 includes both an offline and online measurement techniques. The offline method is designed to support users, distributors, and service providers who conduct measurement of prepreg LOI. It can be carried out with a relatively simple 2-dimensional imaging system, such as that depicted in Figure 15. The system should have the necessary ultrasonic hardware to transmit and receive signals at the appropriate frequency and translate received into numerical values, such as amplitude (peak-to-peak).

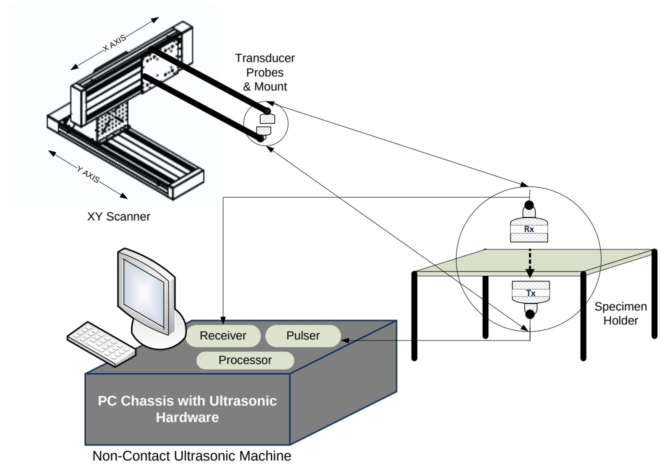


Figure 15. Offline Inspection Schematic

The second technique is designed for online inspection during manufacturing (Figure 16). This allows prepreg manufacturers to inspect their product as it is produced, for real-time mapping and feedback. The advantages of this technique will allow for cost savings and full coverage measurement. It will also allow producers the opportunity to avoid having to retest material offline following manufacturing.

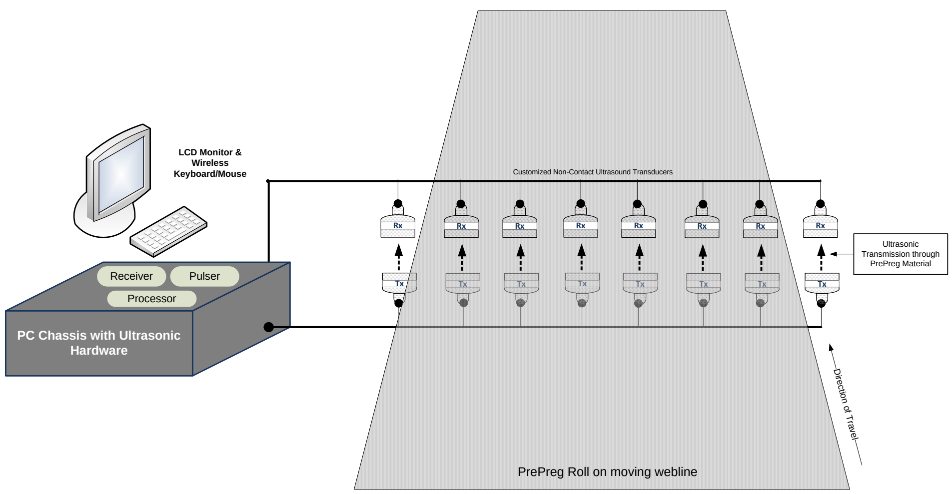


Figure 16. Online Inspection Schematic

The proposed method has been balloted to the ASTM subcommittee D30 and is currently in the process of review. Following steps include round robin testing at designated companies to validate the proposed method. It is expected that the standard will be established within the following 12-18 months.

4. CONCLUSIONS

During this effort, the Ultran Group and Aurora Flight Sciences have obtained OOA IM7/5320-1 prepreg material with three controlled levels of impregnation (low-HLU, medium-ATL, high-AFP) from Cytec, performed NCU scans of the prepreg materials to first conduct a frequency analysis test and to second perform a comprehensive multi-conditional set of tests. All NCU analysis has been validated using guided water pickup tests to develop relationships and linear correlation functions linking NCU transmittance to prepreg LOI in terms of a percentage. Qualitative analysis has also been conducted using MicroCT, performed by both Aurora and McGill University to provide qualitative insight into our findings. Furthermore, analysis was conducted to study the effects of paper backing upon NCU transmittance readings, paper backing was found to not adversely influence the measurements. This demonstrates that the proposed standard methods will be applicable for both offline and online usage, where backing paper will most likely be present. The proposed standard was balloted with ASTM subcommittee D30.03 on May 5, 2015 and is currently in process. Because this effort only explored LOI determination for one product (IM7/5320-1), additional unidirectional intermediate modulus carbon fiber reinforced toughened epoxy prepregs will be analyzed during the balloting process via round robin testing from participating prepreg suppliers and composite manufacturers.

5. ACKNOWLEDGEMENTS

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