

ULTRASOUND RESPONSES AND NON-DESTRUCTIVE TESTING FOR OPEN-MOLD GLASS REINFORCED THERMOSETS

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ABSTRACT

Ultrasound is the most common non-destructive technique used for evaluation of composite materials. It is normally used by following techniques developed for metals and is focused on time-of-flight of the ultrasonic pulse. Glass reinforced thermoset composites have inherent variation that is the result of several factors, including reinforcement content and distribution, voids, resin cure, additives, and manufacturing conditions. Some of the variations can be magnified by exposure to service conditions. In many cases, composites are qualified for use by subjecting samples to destructive tests and applying the principles of statistical process control to accept or reject a production lot. This paper discusses how ultrasonic and destructive testing information from composite samples has been evaluated to show the comparative variation of the non-destructive results compared to the destructive results. The paper concludes with a discussion of the potential for use of ultrasound in quality control during composite manufacture.

1. INTRODUCTION

It is widely accepted that glass reinforced composite materials can be used in many structural applications. In addition, the superior corrosion resistance of these materials when compared to most steels and stainless steels makes them attractive for their durability.

Just like all other materials, glass reinforced thermoset composites have inherent variation that is the result of several factors. The relevant factors for these materials include reinforcement content and distribution, voids, matrix cure, additives, and manufacturing conditions. Normally, controls are applied at the component materials preparation stage to manage these variations. After manufacture is complete, materials are tested in a number of ways, including: destructive testing of representative samples to determine aspects of the structure and non-destructive testing to identify defects.

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accept or reject a production lot. This paper discusses how ultrasonic information obtained from a range of glass reinforced composite materials has been processed to identify features.

Ultrasonic readings taken from samples were scaled and filtered to compensate for coupling conditions so that data could be extracted from the features. The resulting data was evaluated to identify variations in the composite samples. Linkage of the feature data has been made with changes in physical properties as detected from other methods, including destructive tests. The results obtained also illustrate the comparative variation of processed non-destructive ultrasonic information compared to the output of standard destructive tests. Based on the experience gained, limitations of ultrasonic evaluation will be identified.

The paper includes a discussion of some potential uses of ultrasound in quality control during composite manufacture. The potential uses discussed include early assessment of changes in the manufacturing process, identification of variability or uncertainty during composite manufacture, and assurance that the product is within required norms.

This paper addresses open mold manufacturing methods such as custom contact molded and filament winding. Many of these open mold composites are used for industrial, marine and architectural applications.

It has been shown in previous work¹ that parameters calculated from ultrasonic responses can provide quantitative information that describes changes to composite properties due to service conditions.

The purpose of this paper is to describe work that has been done to relate information that can be generated from ultrasonic readings taken from open mold composites to the properties of the composite. It is proposed that some of this information can be used to enhance process control of manufacturing and to provide

1.1 Quality Assurance of Steel

Metal alloys, in particular steel and stainless steel, are a solution of elements that is created at high temperatures then formed by casting into a billet and shaping in a rolling mill. The chemical composition and physical properties of these materials is generally included in standards that allow engineers to select a material based on known properties.

Quality control during manufacturing of steels is covered by numerous process control methods. In many cases, a small portion of the billet is removed before it enters the rolling mill. The portion removed is analyzed for chemical composition and mechanical properties. The results of this testing are then available for all products produced from the billet as a "Certified Mill Test Report" (MTR). Many construction standards and specifications require that MTR's be provided with all steel used in a project.

1.2 Current Open Mold Composite Quality Control Methods

The focus of quality control methods for industrial and civil glass reinforced composite structures has two (2) main focus areas: controlling the assembly of the constituent materials and testing the performance of the resulting or representative structures. Each of these focus areas is described below.

1.2.1 Assembly Controls

A significant portion of FRP storage and process vessels are custom made, where each vessel is designed and made independently. Each of these custom structures is usually accompanied by a custom design which details requirements for matrix, reinforcements and reinforcement assembly with layer-by-layer specification of reinforcement addition.

There are several standard specifications and practices that can be used to assist with both design and quality control^{2,3,4,5,6}. The quality assurance and inspection requirements for the structures produced to them are summarized below.

Resin Cure: This requirement is common to most standard specifications and consists of checking the resin hardness to determine if the resin has achieved adequate cure.

Physical Properties: The standards often stipulate minimum strength and elastic modulus requirements to be used for laminates. If verification is required, then destructive tests are completed using specimens that have been removed from the original structure or using specimens that have been manufactured to duplicate the actual construction⁶. Typical destructive tests that may be required are tensile strength and modulus, flexural strength and modulus and inorganic reinforcing content.

Constituent Material Certification: Many standards require that manufacturer certificates be retained for the resin and reinforcement materials used.

These inspection requirements are all in addition to verifying that the shape and dimensions of the structures comply with the design.

1.2.2 Performance Testing

In some cases, structures are produced in large enough quantity that samples can be removed for destructive testing and characterization. The purpose of these tests is to verify the performance of similar structures being manufactured by applying known loads and conditions. Several standards and specifications describe these "Performance Tests"^{6,7}.

In some cases, a sample of the composite structure is removed (such as where an opening in the structure is required) and destructive tests are conducted. If the sample is large enough, these can be tested for strength and modulus. If the sample available is too small, it may only be possible to determine the glass reinforcement fraction⁸.

Since performance testing is destructive, any samples tested cannot be returned to the population. In many cases, these tests are used to qualify a design. Once qualified, if no changes have been made to materials or manufacturing processes, re-testing is not generally required.

1.3 Damage Mechanisms and Failure Modes

A failure mode is visible to the naked eye. It is the result of changes that have occurred within the structure as a result of conditions imposed on it. In general, failure modes are only observed after failure has occurred and are therefore not useful for predicting failures.

A damage mechanism refers to changes that occur in a material that can lead to or predict failure.

For glass reinforced composites the three (3) major damage mechanisms are described below.

Damage to the resin or matrix describes damage that can occur and accumulate in the resin due to conditions imposed on the composite. This damage can take the forms of microcracking and chemical changes to the resin.

Damage to the reinforcement fibers can occur as chemical damage such as leaching elements from the glass or mechanical damage to the fiber, for example tensile fracture.

Damage to the resin-fiber interface is damage that can occur to the bond between the fibers and the resin. Examples include chemical damage from corrosion or shear fracture of the bond.

Damage mechanisms and failure modes are discussed because they are important for ongoing evaluation of the structural capacity of FRP.

2. EXPERIMENTATION

Specimens of new glass reinforced thermoset were obtained from various sources for non-destructive evaluation using ultrasonic methods. The specimens were of varying sizes and compositions.

Testing was completed on 46 specimens with an average of 39 readings per specimen. All readings were taken without overlap. Forty-one (41) of the specimens were composed only of glass fiber reinforcement in a polyester or vinyl ester matrix. Five of the specimens included various amounts of sand filler.

The readings were taken using Olympus Epoch 1000 ultrasonic flaw detector with M2008, 0.5 MHz transducer. The readings were taken following a written procedure.

2.1 Hypothesis

The hypothesis of this work is that quantitative non-destructive parameters can be obtained from new glass reinforced composites to demonstrate consistent properties.

2.2 Ultrasonic Testing Background

Ultrasonic testing is a non-destructive test method that has been in common use with composite materials for fifty (50) years. In this method, a transducer is used to apply a short, small displacement pulse to the surface of the composite. The pulse time is about 1 microsecond, corresponding to a frequency of 0.5 megahertz.

The pulse transmits the material through the thickness until it reflects from the free opposite surface and returns to the origin. Along the path, the energy of the pulse is attenuated by the material and various features within the composite. Between pulse applications, the transducer is used to receive the reflected signals. The reflected signals are then accumulated according to the time after the pulse was applied.

The output available from the ultrasonic equipment are displayed on an x-y display, with time along the horizontal axis and the averaged magnitude of the received signal displayed vertically. This output is referred to as an “A-scan”. An example is shown in Figure 1. In Figure 1, the signal reflected from the opposite surface is labelled.

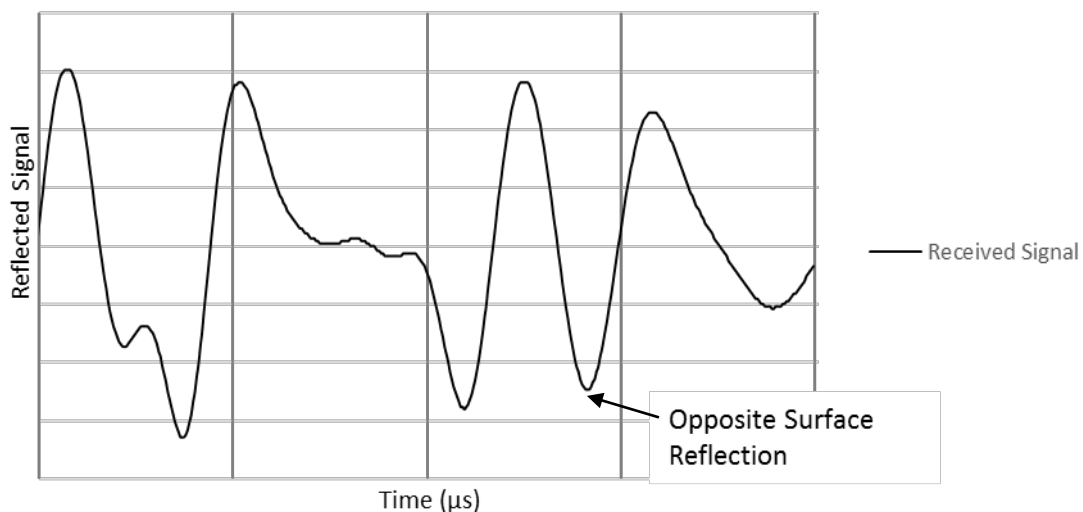


Figure 1 – Example A-scan

2.3 Sonic Velocity

A common use of ultrasound for metals is thickness measurement based on the time for transit and the sonic velocity as a characteristic of the material. The sonic velocity of the material is determined using a specimen of the material of known thickness and determining the sonic velocity. This principle is also used for composite materials.

The study that was completed was to take ultrasonic readings from one surface of the specimen, spaced adjacent to each other. The thickness of the specimens was known.

From each reading, the transit time for the applied signal to return from the opposite surface of the material was determined and the sonic velocity (V) was calculated according to equation 1.

$$V = \frac{2 \times \text{thickness}}{\text{transit time}} \quad (1)$$

2.4 Flexural Modulus Variation

Flexural modulus of a composite laminate is not usually considered as a fundamental parameter of the material. It does, however, relate to how well the layers are bonded to each other and the reinforcement is bonded to the resin. These properties are generally of importance to industrial users, especially for fluid containment and support of bending moments.

Figure 2 illustrates two feature zones within the A-Scan from Figure 1. From the Opposite Surface Feature Zone, the x-y plot data is used to calculate the overall reflected ultrasound from the full structural thickness of the composite. The x-y plot data from the Intermediate Feature Zone is processed using a proprietary algorithm and the results are incorporated by the algorithm with the opposite surface result to determine a value related to the overall attenuation of the applied signal. The value that is calculated has been given the variable name L and a functional description is given in Equation 2.

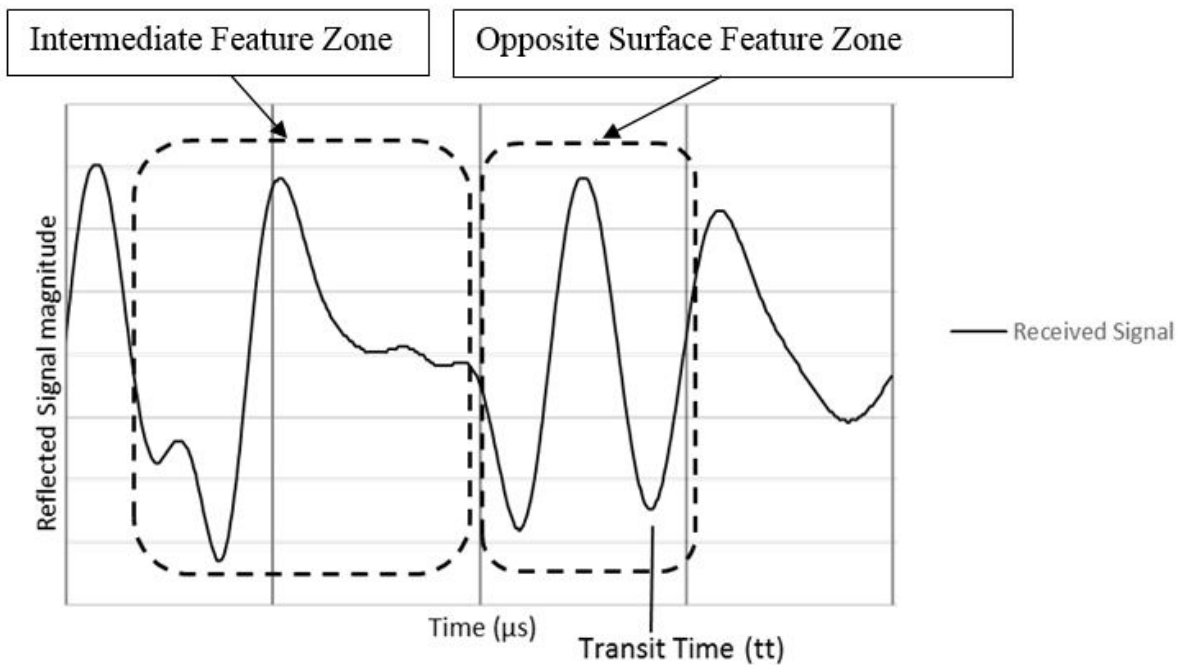


Figure 2 – Data Extraction from A-Scan

$$L = f(\text{magnitude, applied, time})_{\text{Feature Zones}} \quad (2)$$

Calculation of L is normally automated and completed by computer processing of the raw data from the ultrasonic instrument using a proprietary method. After L is determined, it is formed into the parameter Ltt , according to equation 3. Note that Ltt is determined using data that comes directly from the ultrasonic readings and does not require any additional calibrations or measurements.

$$Ltt = L \times \text{Transit Time} \quad (3)$$

This work determined Ltt from the A-Scan of each reading recorded. For each specimen, the average value was calculated and the differences between the individual values and the average were determined. The distribution of the differences was evaluated.

3. RESULTS

3.1 Sonic Velocity

The sonic velocity distribution among the samples that did not contain sand filler is shown in Figure 3. Note that for each sample, there is a fairly narrow range of velocity values calculated. There are some similarities in the data.

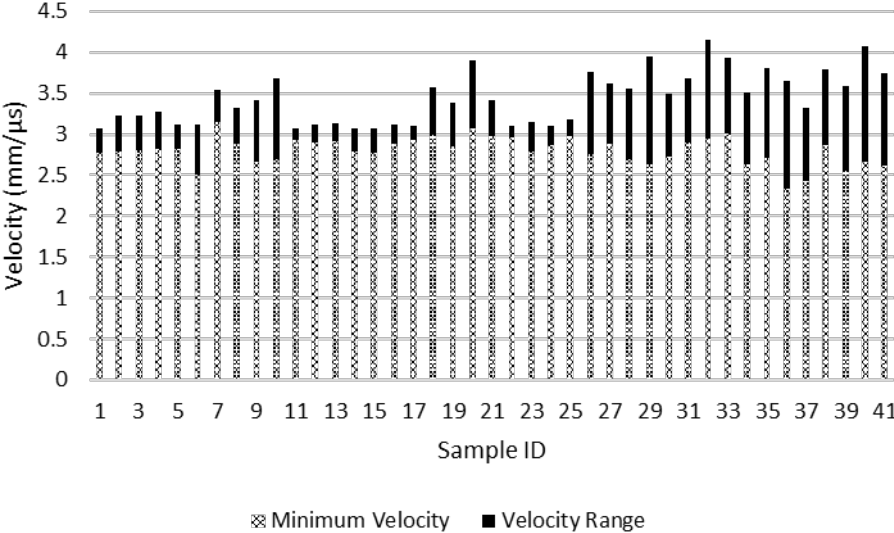


Figure 3 – Velocity for samples not containing sand filler

The sonic velocity results for the specimens with sand filler are shown in Figure 4. Note that the velocity values for this case are lower than in Figure 3. For the samples shown, the mean is 2.29 mm/μs and the standard deviation is 0.047 mm/μs.

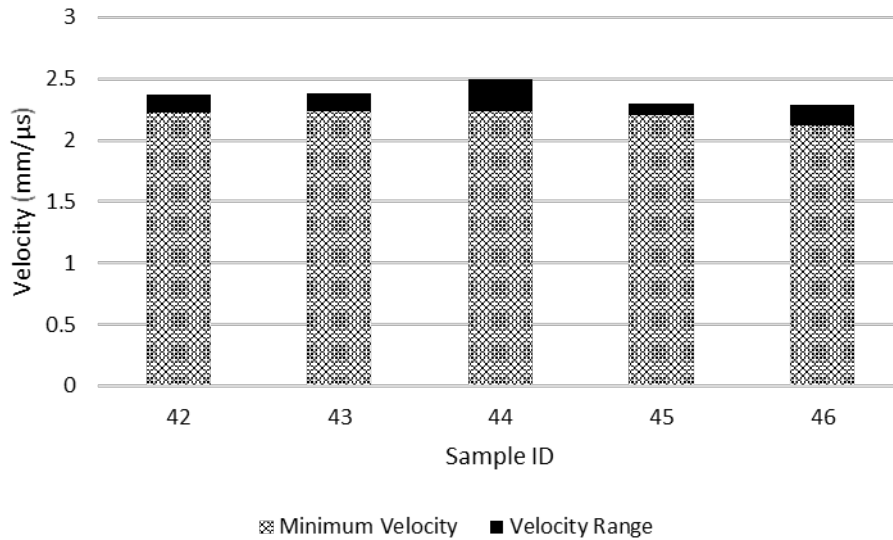


Figure 4 – Velocity for samples containing sand filler

Figure 5 shows a histogram of all sonic velocity values for the samples with no sand fill lumped together with an imposed normal curve with a mean of 3.14 mm/μs and standard deviation of 0.149 mm/μs. When the calculated probabilities are compared to the histogram fractions they are from the same population to a significance of 82%. A similar curve with different mean and standard deviation for the sand-filled specimens yields ANOVA significance of 81%.

For most new composites, the thickness is generally expected to be within a fairly narrow range, so ultrasonic readings should be expected to give a narrow range of transit time values as calculated using the velocity range shown in Figure 5. In Figure 5, the 95% confidence interval of sonic velocity corresponds to $\pm 9.3\%$ of the mean. This means that FRP with constant thickness will yield ultrasonic transit times with a corresponding confidence interval.

An appropriate quality specification for FRP would be to require that the confidence interval for transit time be within the confidence interval identified above and factored by the allowable thickness range.

It is also noteworthy that unseen fillers and porosity are expected to reduce the sonic velocity which would be directly observable from transit time results. From this, unseen defects in a FRP laminate can be detected.

Sonic velocity of FRP has been observed to decline from some service conditions¹. After the sonic velocity is determined for a new sample, this quality parameter can be reported to the buyer with the knowledge that it will provide guidance for future inspections.

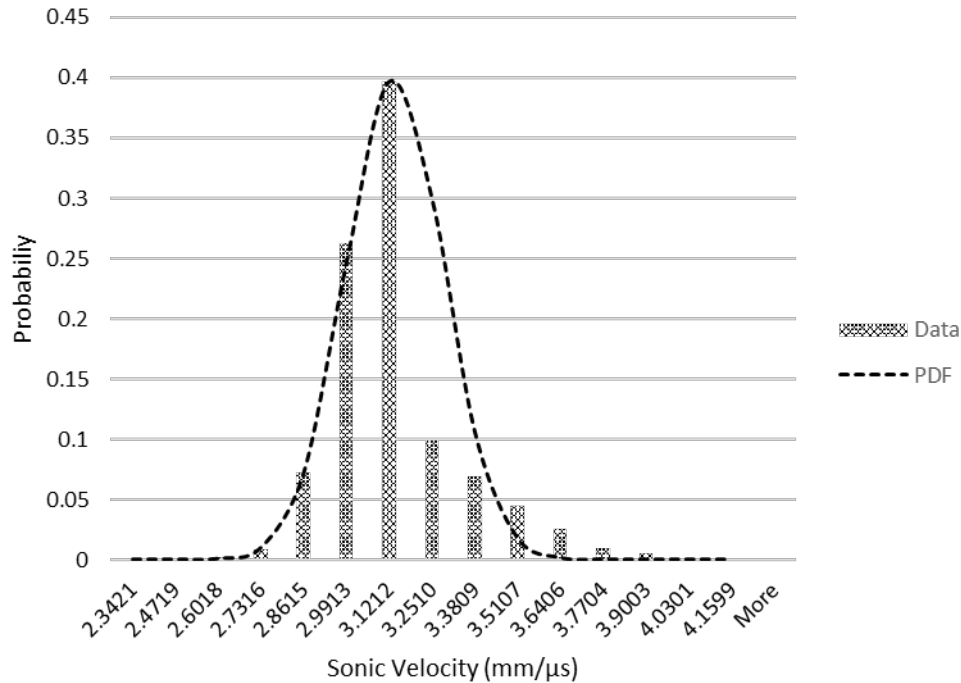


Figure 5 – Velocity Data and Normal Distribution – No Sand

3.2 Ltt Variation

As discussed above the variation of Ltt values calculated for each reading was determined. Figure 6 shows the histogram of distribution of the differences and the cumulative fraction.

It is clear that there is variation from the mean in almost all readings. Note from the results that the variation from the mean is confined to a narrow band. The actual data conforms well to a normal distribution with mean of 0 and standard deviation of 0.42, making the 95% confidence limits ± 0.81 with a nominal average Ltt of 3.50.

The average Ltt value from a new composite will also serve as a starting point to allow the progression of damage to be measured and evaluated while in service. The process for this was outlined in Reference 1, where it was shown that the value of Ltt correlates directly to the flexural modulus of FRP – thereby indicating changes in the inter-layer and fiber-matrix bonding. In related work, close correlation of Ltt to tensile modulus has also been shown.

A small value for standard deviation of Ltt is expected to relate to lower variation in the FRP and therefore more consistency in properties. An appropriate quality specification would be to require that 95% of all Ltt values for a new FRP be within the 95% confidence limits. It has been shown in a number of cases that defects in FRP will generally reduce the Ltt value to outside of the confidence limits. Thus allowing detection of defects.

Ltt of FRP has been observed to decline due to service conditions such as high stress and corrosion. After the Ltt is determined for a new sample, the corresponding structural capacity can be determined and this can be used in future inspections to determine structural changes taking place in the FRP¹.

Note that both velocity and Ltt values can be calculated from one ultrasonic reading.

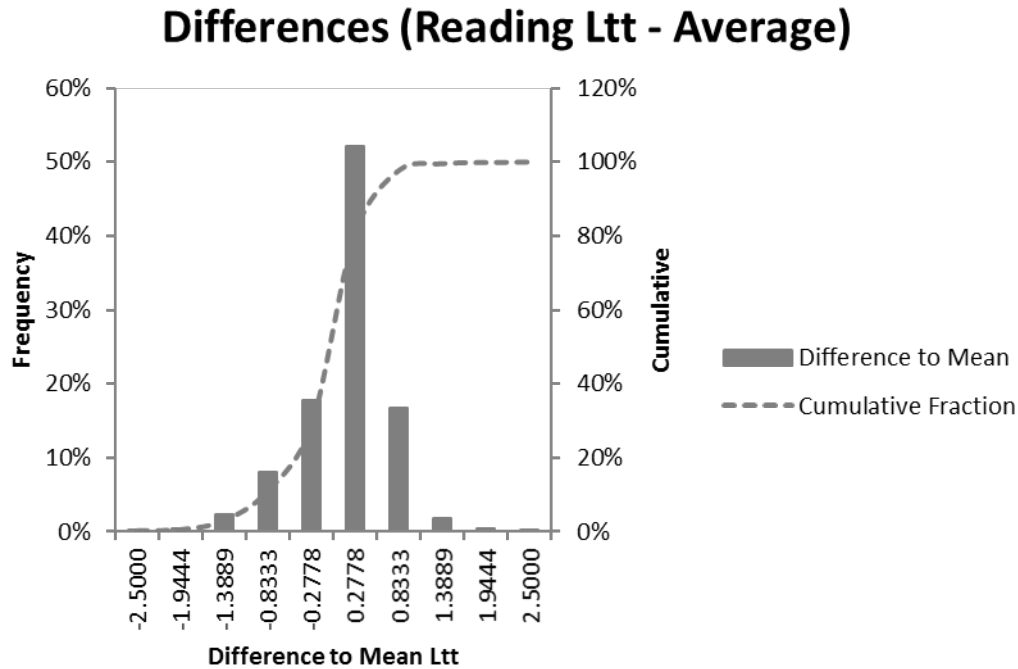


Figure 4 – Differences to Mean Ltt

3.3 Number of Readings

It can be shown that 30 ultrasonic readings from FRP that is expected to be constant thickness will produce a valid mean and standard deviation. For 30 readings, adding one reading is expected to make less than 3% difference to the standard deviation from the value determined for 29 readings. Therefore this ultrasonic quality assessment can be completed with 30 readings.

4. CONCLUSIONS

A full set of ultrasonic readings to assess FRP of the same thickness can be obtained with 30 or more ultrasonic readings taken by following the procedure used in this paper.

The sonic velocity of a composite should be expected to be within a narrow range as illustrated in Figure 5. The transit time for an ultrasonic signal through FRP is directly proportional to the thickness and sonic velocity. Based on the expected range for sonic velocity as shown above, and the thickness tolerance, the transit time for an ultrasound pulse to transit the material can be specified to be within a known range.

In addition, the existence of defects and fillers in the FRP can be detected from transit time variations that result from sonic velocity variations.

Ltt variation from the mean should be expected to be within a defined range for the composite of not more than ± 0.81 and should be expected to conform well to a normal distribution with mean of 0 and standard deviation of 0.42.

The existence of defects in the FRP can be detected from Ltt variations. The existence of significant defects will reduce the calculated Ltt value at the defect.

The values for sonic velocity and Ltt determined for new FRP can be used to determine changes taking place in service conditions.

5. REFERENCES

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