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Influence of Resin Viscosity on Physical Properties of a Composite Shell Wound on a Low-Density Material Mandrel

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ABSTRACT

This study is made to improve the structural performance of composite shells/ vessels meant for aerospace vehicles. The effect of resin viscosity on the physical properties of carbon/ epoxy composite shell wound on polyurethane (PU) foam-based mandrel is studied and presented in this paper. Cylindrical shells were manufactured through the filament winding process at different resin viscosities. The physical properties of the composite shell are found to be improved significantly with a reduction in resin viscosity. Resin pick-up in impregnated fibers is found to be lower by 4.5 %, whereas mass and thickness of the shells are recorded to be lower by 3 % and 5.4 % respectively at resin viscosity range of 600 -760 mPa.s compared to the viscosity range of 1380 – 2080 mPa.s. Fiber volume fraction and density of composite shell are found to be higher by 6.3 % and 2.8 % at the same resin viscosity range. This trend reverses/stabilizes after further heating and corresponding lowered resin viscosity. Experiment and their result indicate an optimal viscosity range of 600 – 760 mPa.s. for filament winding of efficient carbon/ epoxy composite shell.

Keywords: Filament winding; Carbon/ epoxy; Polyurethane foam; Resin viscosity; Fiber volume fraction

1. INTRODUCTION

Filament winding is the widely followed technology for manufacturing composite pressure vessels[1]. Composite is a structural material consisting of two or more constituents, where one constituent is called the reinforcement and another in which it is embedded is known as matrix[2]. Though tow-preg (pre-impregnated partially cured fiber) is a better and more consistent material for filament winding, wet winding is still preferred for manufacturing large wound composite structures[3], like pressure vessels and shells. In the wet filament winding process, fibers (reinforcement) pass through a resin bath placed on the machine itself during winding for impregnation. This impregnated fiber, under tension, gets deposited over a rotating mandrel in a certain programmed way[4-5].

Composite draws its superior property from its reinforcement, whereas matrix keeps them together in a specific shape in the overall structure[2,5]. Accordingly, in composite manufacturing, efforts are continually put into improving fractional reinforcement contribution in an overall composite. Filament winding uses continuous fiber as reinforcement, where improvement in composite properties can be tried by various processing means. Resin viscosity during winding is one such important processing parameter that demands special attention for manufacturing an efficient composite structure.

Mallick[6] discussed the essentiality of good wetting for reducing voids in filament wound parts. The author mentions resin viscosity's dependency on resin type and bath temperature. The author also discussed the lower viscosity requirement for proper impregnation, the limitation of higher resin viscosity, and shorter resin pot life at elevated temperatures. Cohen[3], *et al.* studied the effect of fiber volume fraction on filament wound carbon/ epoxy composite vessel quality. However, they varied the volume fraction by impregnating the fiber with resin by passing them through a controlled opening diameter orifice.

Cohen[7] studied the effect of process variables, such as winding tension, laminate stacking sequence, winding tension gradient, etc., on filament wound composite vessel quality and strength. Mertiny[8], *et al.* studied optimal filament winding processing parameters, where they also talked about resin dispensing and mixing through volumetric dispensing with a static mixer nozzle. However, they used glass fiber reinforced polymer (GFRP) and investigated the final part characteristics using an image analysis technique. Chamis[9], *et al.* have studied the effect of mechanical properties of matrix on graphite fiber-based composite. Researchers like Lee[10], *et al.* and Cai[11], *et al.* have briefly touched resin flow time, temperature, and viscosity in their filament winding-related work.

Sun[12], *et al.* have covered the effect of resin viscosity on composite case quality. However, case quality is only reported in terms of void and fiber volume fraction. Pandita[13], *et al.* discussed the difficulty in fiber wetting at higher resin viscosity and limited resin pot life at lower viscosity. The authors discussed a modified wet filament winding system using a special resin injection system to overcome the limitation of resin viscosity difficulty in terms of shorter pot life and non-

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uniform fiber wetting. Mertiny[14], *et al.* studied the influence of winding tension on the physical and mechanical properties of reinforced composites, where they also mentioned fiber volume fraction and effective wall thickness. They used E-glass fiber and EPON826/EPI-CURE9551 resin system, where the resin was maintained at a constant temperature of 30 ºC in the tank. However, they have not talked about resin viscosity value.

Błachut[15], *et al.* studied the effect of fiber tension on the mechanical properties of composite pressure vessels, where they used steel liners for winding. Their study concludes improvement in fiber volume fraction with increased fiber tension and, thereby, improvement in mechanical properties. Banerjee[16], *et al.* developed a model that related processing conditions like applied temperature, fiber tension to fiber volume fraction, stresses and strains in the composite cylinder. Through their model, they predicted and validated fiber volume fraction. Susan Mantell[17], *et al.* described a model simulating composite cylinders' filament winding. The models relate the processing conditions like applied temperature, pressure, and processing speed to composite temperature, viscosity, manufacturing stresses etc. Colombo[18], *et al.* studied the effect of the matrix, fiber volume fraction, and winding angle on load bearing capability of the composite pipe. However, they studied glass-based composites.

Reported studies clearly indicate the influence of resin viscosity on the properties of filament-wound composite products. However, most studies are limited to qualitative analysis or are for different material systems and processing conditions.

Composite shells/vessels for aerospace applications demand superior mechanical properties with lesser inert mass. Every unit of mass saving in structure is a gain for aerospace vehicles in terms of passenger-carrying capability or mileage. Vessels/ shells for larger vehicles are highly mass-sensitive, and accordingly, they require efficient structures. Sizable performance variation and mass inconsistency were observed in large-sized carbon/epoxy composite vessels made through wet winding technology. These variations significantly affect the performance of the vehicle. The performance and mass of composite vessels are found to be varied by altering process parameters during their manufacturing. Resin viscosity is found to be an important processing parameter that affects the health and efficiency of composite structures. Considering efficient larger aerospace structures, especially shells/ pressure vessels, and their manufacturing ease in mind, the influence of resin viscosity on the properties of a composite shell wound on a low-density material mandrel is studied.

Carbon/ epoxy composite shells are made through filament winding at different resin viscosity ranges. The physical properties of these shells are studied and presented.

2. EXPERIMENTAL STUDY

2.1 Material

2.1.1 Shell Material

Polymer-based carbon/ epoxy composite is used for this study. Polyacrylonitrile PAN-based high strength carbon fiber and Diglycidyl Ether of Bisphenol A DGEBA-based epoxy are used for composite shell manufacturing. Epoxy is a two-component resin system consisting of resin and hardener mixed in a specific proportion. Brief properties of the materials are given in Tables 1 and 2.

Table 1. Carbon fiber

Detail	Values
Tow size	12k
Filament diameter	6 – 8 microns
Density	1.7 – 1.85 g/cc

Table 2. Epoxy Resin and Hardener

Epoxy Resin	
Detail	**Values**
Specific gravity at 25 ºC	1.0 - 1.1
Viscosity at 25 ºC	120 - 200 mPa.s
Epoxy Hardener	
Specific gravity at 25 ºC	1.10 - 1.20
Viscosity at 25 ºC	14000–18000 mPa.s
Resin mix (Resin & hardener in 100:27 by weight)	
Viscosity at 25 ºC	6350 mPa.s

2.1.2 Mandrel Material

Rigid Polyurethane PU-based foam as core and Plaster of Paris (PoP) skin as the interface are selected for mandrel material. Low-density PU foam makes a mandrel multiple-fold lighter than a conventional steel mandrel. It's easy castability and machinability make it further amenable for large-sized/ contoured mandrels. The functional aspect of the material is tested and verified before use for its suitability for shell manufacturing. Foam samples and their tested properties are shown in Fig. 1 and Table 3, respectively.

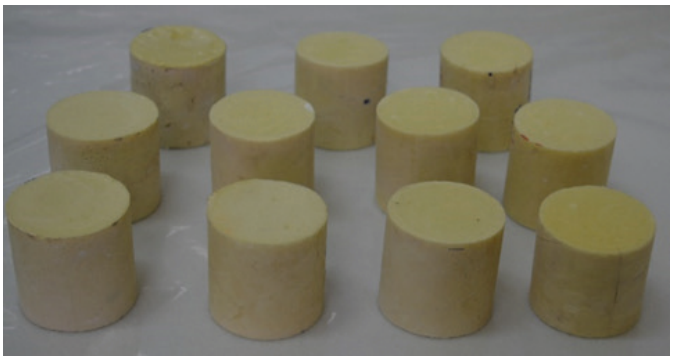


Figure 1. PU foam sample.

Table 3. PU Foam

Parameter	Measured value
Density (g/cc)	0.1 – 0.15
Compressive strength (MPa)	1.1 -1.5
Thermal resistance at 160 ºC	below 2 % volumetric change

2.2 Selection of Resin Viscosity

The resin viscosity is directly affected by temperature. Lower resin viscosity ensures proper and uniform wetting of high tow-sized fiber. Properly and uniformly wet fiber ensures improved and consistent properties of wet wound composite products. However, resin viscosity that is too low can lead to resin starvation in the composite structure. Secondly, further lower resin viscosity demands its heating at higher temperatures which causes drastically reduced pot life. Too low a resin pot life makes manufacturing large-sized/ thick products requiring a longer duration of winding difficult[4,6]. Higher resin viscosity below 36 ºC of resin temperature makes uniform wetting of high tow-sized fiber difficult, whereas, beyond 60 ºC of resin temperature, pot life becomes drastically lower. Keeping the quality of the product and processing ease in mind, the resin viscosity is selected based on temperature. Resin temperature is selected between 36 – 60 ºC. This temperature range is divided into six intervals of 4 ºC with 38 ºC as the lower base and 58 ºC as the higher base temperature. A tolerance of ±2 ºC is chosen at each base temperature, considering the practical difficulty in maintaining resin at unique temperature throughout the winding process. The resin (resin and hardener in ratio of 100:27 by weight) viscosity is measured using a Brookfield viscometer at all six base temperatures with ±2 ºC tolerance. Viscosities at each base temperature's lower and higher ranges (considering ±2 ºC tolerance) are taken as the viscosity range at each resin temperature. The average resin viscosity at each base temperature is plotted in Fig. 2.

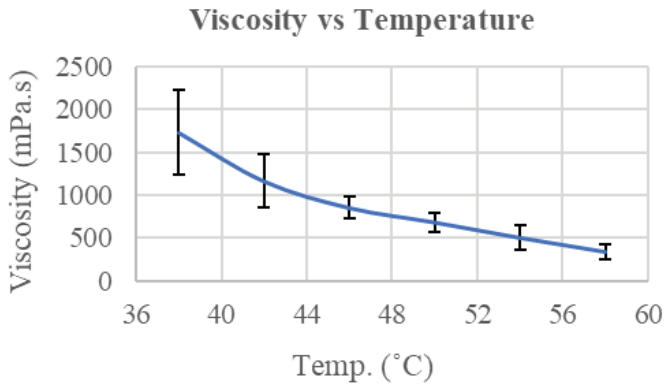


Figure 2. Viscosity plot.

It is observed that resin viscosity decreases with an increase in its temperature. However, the decrease in resin viscosity is initially higher, whereas the same slows down with further increase in temperature.

2.3 Composite Shell

A carbon/ epoxy composite shell is configured with a 550 mm internal diameter, 100 mm length, and 4 mm nominal thickness for this study.

2.3.1 Mandrel Configuration

PU foam-based mandrel was conceptualized, where the foam was built over a steel shaft. PoP skin over foam was introduced as the interface surface for a better finish and reasonable hardness to withstand fiber tension during winding. Six grooves (each 100 mm wide x 6 mm deep) were provided to manufacture composite shells at different resin viscosities. Mandrel configuration is shown in Fig. 3.

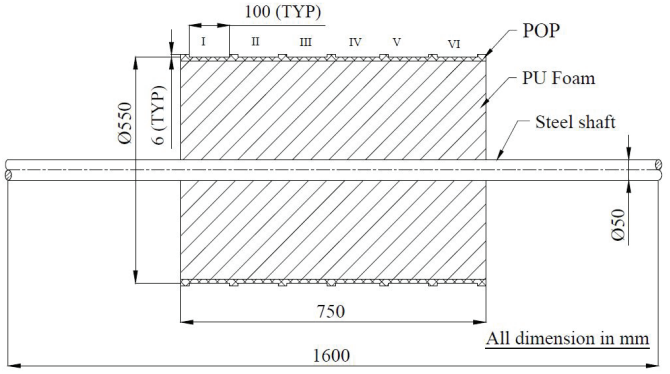
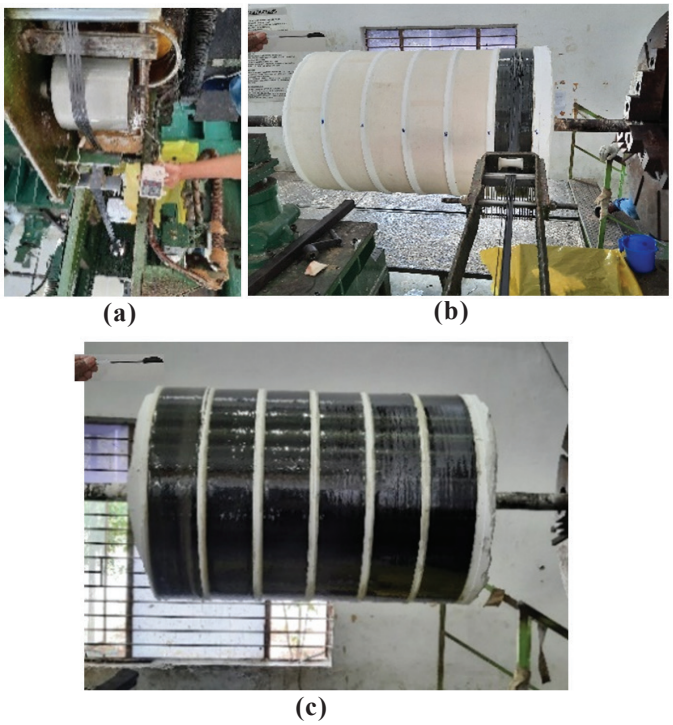


Figure 3. Mandrel configuration.

2.3.2 Shell Winding

Hoop winding was carried out in all mandrel grooves at six different identified resin viscosity ranges (Fig. 2) using carbon/ epoxy material. Resin temperature and, in turn, its viscosity were maintained through a heater placed below the resin bowl. The temperature was continuously monitored through a thermometer placed in the resin bath. Carbon fibers were wet with resin while passing over a rotating drum placed in the resin bowl. Impregnated fibers under tension were deposited over the mandrel through a Computerized Numerical Control (CNC) program. The temperature was maintained within ±2 ºC at the respective base temperatures. All other processing parameters like fiber tension, resin bath attachment setup, carriage distance from the mandrel, number of spools, etc., were kept the same during the entire winding process. All six shell windings were finished on the same day. Four hoop plies (each of ~1 mm thickness) were provided in



(a) **(b)**

(c)

Figure 4. Winding process: (a) Viscosity control; (b) Shell winding; and (c) Wound shells.

all six grooves. All six shells were wound using the same CNC program, which ensured the consumption of the same amount of fiber in all the shells. Resin viscosity controlled through its temperature was the only process variable during the different shell windings. The mandrel was rotated for 12 hours after the winding for uniform resin dispersion. The winding process with the mentioned details is shown in Fig. 4.

For resin pick-up study during the winding at different viscosities, impregnated fibers after the fiber dispensing unit and before deposition on the mandrel were cut and kept for testing. The process of cutting impregnated fiber is shown in Fig. 5, and one such cut sample is shown in Fig. 6.

Figure 5. Impregnated fiber cutting.

Figure 6. Resin pick-up sample.

2.3.3 Curing and Extraction of Composite Shell

The wound shells were cured in an oven at 160 °C for 3 hrs. After curing, composite shells were extracted by breaking/ scooping PoP and foam material. Subsequently, the shells were cleaned properly. Realized one such composite shell is shown in Fig. 7.

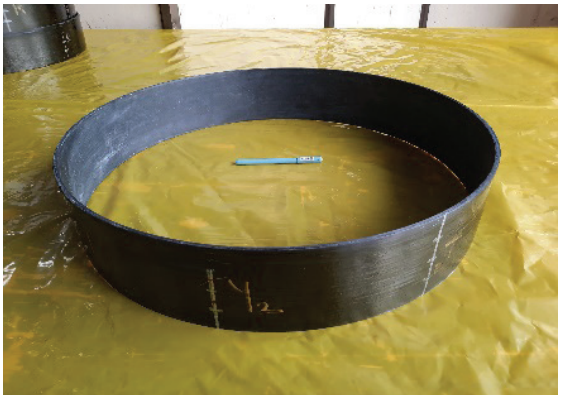


Figure 7. Composite shell.

2.4 Testing and Result

2.4.1 Resin Pick-up

Resin pick-up samples were tested through the solvent extraction method. The measured average resin and fiber mass fraction corresponding to each resin viscosity range is plotted in Fig. 8. Average resin viscosity is taken instead of its range while plotting the results.

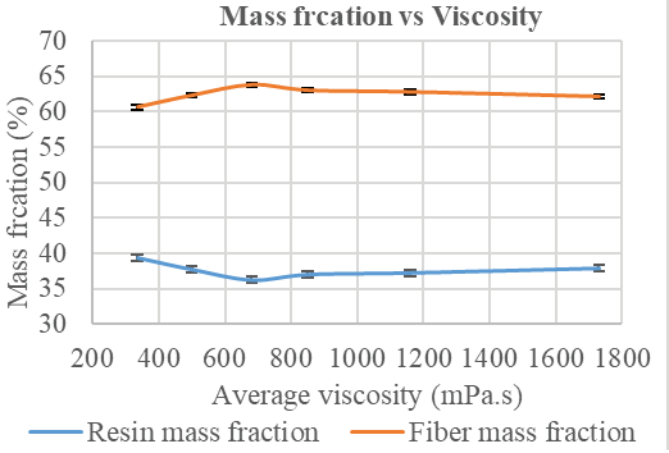


Figure 8. Mass fraction plot.

It is observed that the fiber mass fraction in impregnated fiber is maximum (63.78 %), whereas the resin mass fraction is minimum (36.22 %) at an average resin viscosity of 680 mPa.s.

2.4.2 Mass Measurement

The mass of each shell was measured using a digital weighing balance (least count - 0.5 gram). The recorded mass corresponding to each average resin viscosity is plotted in Fig. 9.

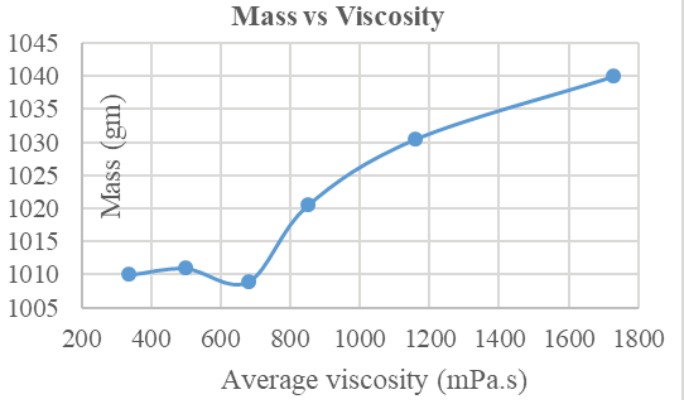


Figure 9. Mass plot.

Mass measurement result indicates the reduction in composite shell mass with the lowering of resin viscosity up to the average viscosity of 680 mPa.s.

2.4.3. Thickness Mapping

Samples were cut from the middle of the composite shell for wall thickness measurement. Four samples from equi-spaced circumferential locations were cut and taken from each shell. The thickness of each piece is measured with a digital vernier caliper. The thickness measurement is shown in Fig. 10. The measured average thickness against average resin viscosity is plotted in Fig. 11

Figure 10. Thickness measurement.

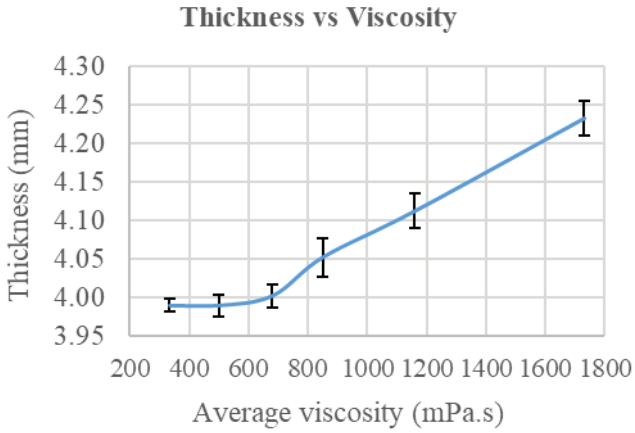


Figure 11. Thickness plot.

2.4.4 Density Measurement

Samples (15 mm x 6 mm x 4 mm) were cut from the composite thickness samples and tested for density using the water displacement method as per ASTM standard[19]. Density samples are shown in Fig. 12. A total of 4 samples taken from equispaced circumferential locations were tested from each shell. The average density corresponding to each average resin viscosity is plotted in Fig. 13.

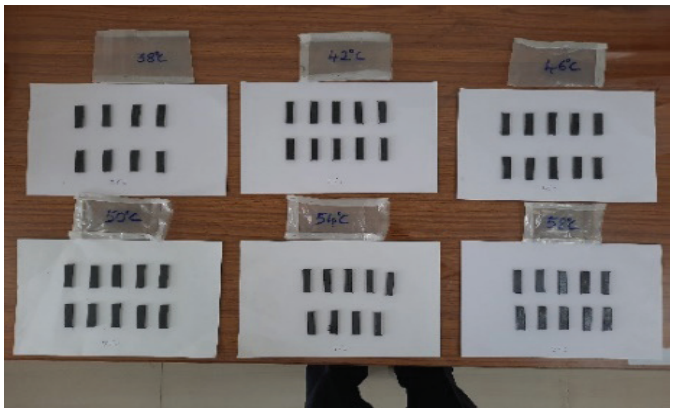


Figure 12. Density sample.

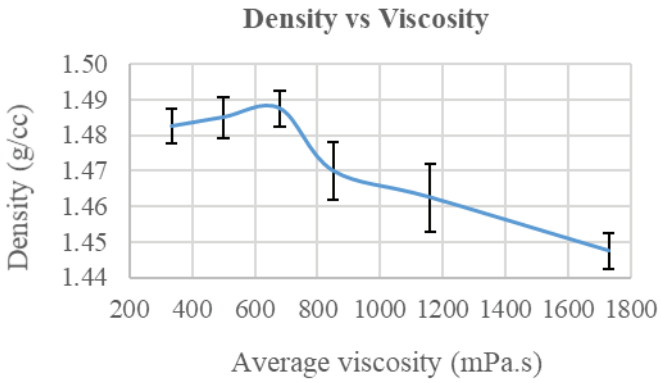


Figure 13. Density plot.

The composite shell's density is maximum (1.4875 g/cc) at an average resin viscosity of 680 mPa.s.

2.4.5 Fiber Volume Fraction

Samples (15 mm x 6 mm x 4 mm) were cut from all six shells and tested for the fiber and resin mass fraction by acid digestion method as per ASTM standard[20]. Mass fractions were converted to fiber volume fractions (V_f) using individual constituent and composite density. Four samples taken from equi-spaced circumferential locations of each shell were tested. The observed average fiber volume fraction corresponding to each average resin viscosity is plotted in Fig. 14.

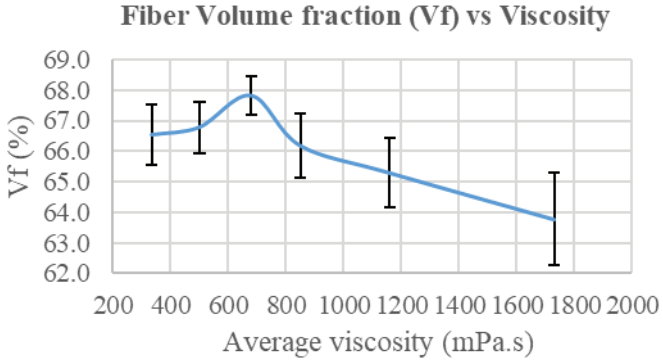


Figure 14. Fiber volume fraction plot.

It is observed that fiber volume fraction of the composite shell is maximum (67.81 %) at average resin viscosity of 680 mPa.s.

2.4.6 Non-Destructive Test (NDT)

All the carbon/ epoxy composite shells were subjected to non-destructive tests for their health examination. The

Figure 15. UT of the shell.

ultrasonic test (UT) was carried out using through transmission technique. The test was conducted as per evenly distributed grid points on each shell. A total of 24 grid points were probed for each shell. UT process of the shell is shown in Fig. 15. The average received UT signal of each shell against their processed average resin viscosity is plotted in Fig. 16.

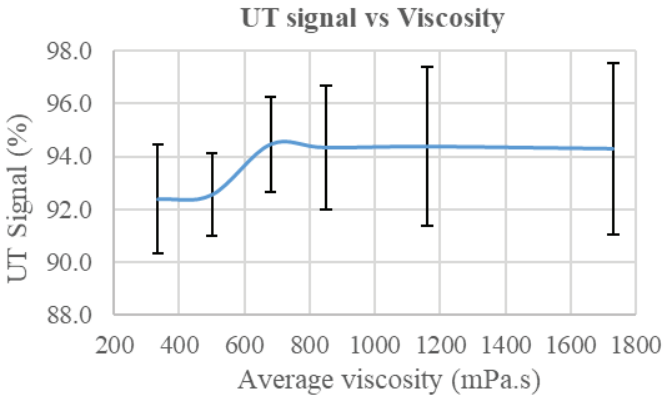


Figure 16. UT signal plot.

3. DISCUSSION

All the measured physical properties of composite shells are compared with respect to their properties at the average resin viscosity of 1730 mPa.s (viscosity range 1380 - 2080 mPa.s.)

- Resin pick-up results indicate an increase in fiber mass fraction and a decrease in resin mass fraction with a reduction in resin viscosity up to the average viscosity of 680 mPa.s. (viscosity range 600 – 760 mPa.s). As samples were taken directly after resin impregnation during winding, improvement in fiber mass fraction is due to resin viscosity alone. The trend of improvement in fiber mass fraction reverses at further lowered resin viscosity. Resin pick-up in impregnated fiber is also lowest at the same resin viscosity.
- Mass measurement result indicates the reduction in composite shell mass with the lowering of resin viscosity up to the average viscosity of 680 mPa.s. This reduction trend reverses after subsequent heating of resin. Subsequently, the shell mass is mostly stabilized as minor changes beyond 1009 grams are only within 0.2 %.

  The thickness mapping result shows a significant reduction in shell thickness up to the average resin viscosity of 680 mPa.s. The reduction in shell thickness is negligible (only 0.25 %) at further lowered resin viscosity which subsequently gets stabilized.
- The density result shows improvement in the composite shell density with reduced resin viscosity up to the average viscosity of 680 mPa.s. This trend reverses after further resin heating during the winding. As the amount of fiber employed in all six shells is the same, increased shell density is only due to reduced resin absorption.

  The fiber volume fraction plot indicates a significant increase in fiber volume up to the average resin viscosity of 680 mPa.s. This trend reverses with further resin heating during the shell winding.
- The ultrasonic test result of composite shells indicates improvement in signals up to the average resin viscosity of 680 mPa.s. However, at further lowered resin viscosity, there is a downward trend in the received UT signal, which indicates the possibility of resin starvation/ delamination in composite shells.
- Carbon/ epoxy composite shells manufactured at each resin viscosity range were of the same dimension. The PU foam-based tool was the same in all six cases. Each groove's geometric construction and dimension also remained the same. Filament winding was done on the same machine in the same condition. Winding details like creel stand, spool location, resin drum setting, pay-out eye offset setting, roving tension, winding speed, and other processing parameters were the same for all shell winding. All shells were wound using the same CNC program, ensuring the same amount of fiber consumption. Shells were cured in the same oven. Resin viscosity was the only variable employed during different shell winding.
- Lesser resin pick-up in impregnated fiber, lesser shell mass and thickness, and better fiber volume fraction at higher resin temperatures are due to the lower resin viscosity during the shell winding process. Lower resin viscosity ensures uniform and adequate wetting of higher tow-sized (12 k) fiber during the shell winding. Higher shell density at lower resin viscosity is solely related to lesser resin pick-up. Better UT signal in the composite shell at lower resin viscosity is due to better fiber-matrix interaction.

  However, the reduced properties at further lower resin viscosity are likely due to the draining of required resin during the winding. Draining of resin occurs due to too low viscosity, which leads to resin starvation/ delamination/voids in the composite structure.

4. CONCLUSION

Based on the above study, the following conclusion is made with respect to resin viscosity influence on the physical properties of carbon/epoxy composite shell wound on a PU foam-based mandrel:

- Resin pick-up in impregnated fiber is around 4.5 % lower during winding of the shell at the resin viscosity range of 600 – 760mPa.s. There is around 3.0 % and 5.4 % reduction in composite shell mass and thickness, respectively, when the shell is wound at the resin viscosity range of 600 - 760 mPa.s. Fiber volume fraction and density of the composite shell are improved by around 6.3 % and 2.8 %, respectively, at the same viscosity range. UT result also shows better laminate health when the composite shell is processed at 600 - 760 mPa.s of resin viscosity.
- It is observed that lower resin viscosity significantly improves the physical properties of the filament wound composite shell. Lower resin viscosity helps in thorough and uniform wetting of high tow-sized carbon fiber during the winding, resulting in better fiber-matrix interfacial interaction.

- It is concluded that carbon/epoxy composite shell wound on a low-density material mandrel at resin viscosity range of 600 – 760 mPa.s possesses the lowest shell mass, lowest wall thickness, and highest fiber volume fraction provided all other process parameters and winding conditions are the same.

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CONTRIBUTORS

Mr Surjeet Kumar Sinha completed his BE from B.I.T. Sindri and is presently working as a Scientist 'F' in DRDO-ASL, Hyderabad. His work domain is polymeric composite materials and processes, including pressure vessels, filament winding, and contact layup technologies.
His contributions to this work include: Conceptualization, experimental study, test results, analysis, and conclusion.

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In the current study, he has given valuable suggestions in conceptualizing and executing the work.

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