

# Quality assurance system for monitoring the online resin impregnation during Fiber-Tow-Placement: A review of the state-of-the-art approaches

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## Abstract

To raise the degree of automation for composite manufacturing, appropriate sensors for process monitoring are required. In this study an overview of the state-of-the-art approaches, their potentials and weak points as well as possible solutions for the measurement of the resin content of carbon fiber tows will be presented. Investigated technologies are weight measurement, optical cross section capturing, thermography, non-contact ultrasound, radio waves, ionizing radiation, infrared spectroscopy, nuclear magnet resonance spectroscopy, permittivity measurement and permittivity measurement by eddy current.

## 1. Introduction

The constantly growing use of composites in aeronautic and automotive applications as well as in marine, consumer products and wind energy requires highly automated, robust processes to ensure cost efficient manufacturing. The automation process itself is only half of the story. It is also necessary to have an automated evaluation.

An established technology for composite manufacturing is fiber placement, which includes sub variants for pre impregnated tows or tapes, dry fibers and various matrices (e.g. duromeric and thermoplastic). This manufacturing technology is mainly used for large parts such as wings or fuselage segments due to the high degree of automation of this technology. Currently, the associated quality assurance process for fiber placement is in most cases still a manual process, as there are no sensor systems for this application on the market.

## 2. Quality assurance for fiber placement

In this context, the parameters, which have to be monitored during a fiber placement process, should be considered. The first step is the quality assurance of the raw material (e.g. the fiber or the resin). This step is currently done manually in an offline process and should not be part of further consideration. The second step is the quality assurance of intermediate stages, e.g. the monitoring of the fiber impregnation or the laying process. The third and final step is the quality assurance of the final product. In this case a single layer or the uncured part. For the final quality assurance a few standard non-destructive testing methods are available. Therefore, the focus of the following considerations is placed on the intermediate steps.

The most important step during the fiber placement process is the fiber layup. For this application a few quality assurance systems are already available on the market. One of the market leaders is the system “Rhino” from Airbus IFSOLUTIONS [6]. The sensor captures a profile of the fiber directly behind the compaction roller. As a result the system is able to detect defects such as late add or early cut, gaps or overlaps between tows, splice, fuzballs, twists or missing tows.

For processes with integrated fiber impregnation during the layup process, it is additionally necessary to monitor the resin content of the tow or fiber. For this measurement application, there are currently no appropriate sensor systems available. Consequently, close consideration of these kinds of sensor systems is required in the following paragraphs.

### 3. Constraints

Before beginning with a technology screening, it is necessary to define the constraints and required properties for a sensor system which is able to monitor the resin content.

#### Sensor functionality

The objective of the resin content monitoring is to gather data concerning the resin content for each tow (online), ideally every few centimeters (inline). The minimum requirement is a sensor system that is able to detect dry regions respectively with resin content under or above a critical value. Ideally a sensor that delivers exact values for the resin and the fiber content would be better. A scenario for such a sensor system provides a complete cross section of the tow and therefore information about the resin distribution in the tow.

#### Required accuracy

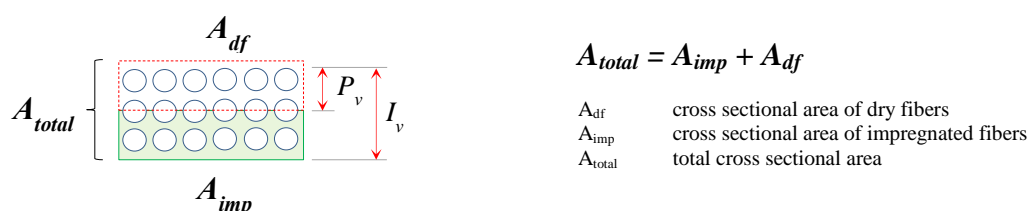
To define the required accuracy of an adequate sensor system, it is useful to consider the tolerances for the resin content of the final part. However, there are no official tolerance values such as a norm or any other specification that can be given. Normally this value is defined by the designer of a part.

In the literature values of around 60% fiber volume content with tolerances of 2-4% are often found. With consideration of further relevant tolerances, a required tolerance of +/- 1% for an appropriate sensor system is proposed.

#### Definition of impregnation

The two formulations “level of impregnation” (LOI) and “degree of impregnation“(DOI), are often found in the literature. Both terms are used synonymously to describe how well a fiber is impregnated with resin and are thereby comparable with porosity. In addition, the second parameter is the ratio between resin and fiber. This property is called resin content and is normally given as “resin volume content” or “resin content by mass”.

According to B. Thorfinnson and T. Biermann [20], the level (degree) of impregnation is defined as the ratio between the overall height of an impregnated fiber ( $I_v$ ) and the level at which the sample is filled with resin ( $I_v - P_v$ ). See Figure 1.

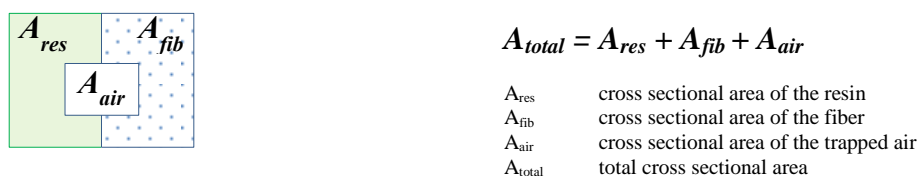


**Figure 1.** Sketch of a cross section of an impregnated fiber according to [20]

The appropriate equation (according to [20]) for the level of impregnation (LOI), which is modified for cross sectional areas instead of height, is shown in Eq 1:

$$LOI = \frac{A_{total} - A_{df}}{A_{total}} \quad (1)$$

To calculate the resin content, the three shares (resin, fiber and trapped air) must be separated intellectually as shown in Figure 2.



**Figure 2.** Schematic sketch of a cross section of an impregnated fiber with separated substances

According to H. Schürmann [19] and DIN EN 2564 [2], the resin volume content is defined as a ratio of volume shares. After canceling out the length of the fiber, an equation for cross sectional areas is attained (Eq. 3)

$$\varphi = \frac{A_{fib}}{A_{total}} = \frac{A_{fib}}{A_{fib} + A_{res} + A_{air}} \quad (2)$$

For a single tow the level of impregnation is less important than the resin content, as the amount of trapped air will be changed by the compaction roller during the lay-up process. Therefore it is better to measure the level of impregnation for a complete layer, e.g. the uncured or even the cured part. The resin content of a single fiber has a direct influence on the resin content of the final part and would not change during the lay-up process. Therefore this value should be monitored directly in the tow. Consequently, a sensor for monitoring resin content and not for monitoring the level of impregnation is required.

It can be assumed that the resin distribution in the tow is not homogeneous, especially in the peripheral areas. For broad tapes the peripheral areas can be neglected. However, for small tows or even single fibers, these areas must be taken into account. This fact leads to the requirement for an adequate measurement system that includes the whole sample including the peripheral areas.

#### 4. Current state of the art approaches

To find an adequate measurement technology to measure the resin content in a single tow, it is useful to consider the physics and the appropriate material properties of the fiber and the resin. Material properties are sought that can be measured easily and that are significantly different for the resin, the fiber and trapped air.

These requirements are fulfilled by a large number of physical quantity, beginning with measurements such as mass, density, volume (geometrical dimensions or cross sections) or parameters such as heat capacity and thermal conductivity. In addition, the material behavior in electromagnetic fields (permeability or permittivity) and the damping of mechanic or electromagnetic waves can be used to potentially measure the resin content. Mechanic waves could be acoustic, especially ultrasonic waves. Possible electromagnetic waves could be waves with a wave length in the range of microwaves, infrared or in the range of x-ray or gamma rays. To check if these approaches are applicable, more detail is required, which will be presented in the following paragraphs.

#### Measuring the weight of an impregnated tow with a scale

This is, according to the current state-of-the-art approach, the standard process to measure the resin content in an offline process. The operator cuts out a piece of tow with a defined length and measures it with a precision scale. If the weight of the fiber and the density of the resin are known, the resin content of the impregnated tow can be calculated. Trapped air has no influence on the measurement result. It is conceivable that this step is done automatically to obtain an online process. As it is necessary to cut out a piece of fiber, the measurement is just possible between two layup steps of tows. To obtain information along the whole fiber, it is necessary to measure inline. This could be done by modifying the scale and setting up a kind of belt weight. However, to gather usable results by measuring with a precision scale, it is necessary to avoid disruptions such as vibration, shock or lateral forces on the load cell. Because these disruptions cannot be avoided during the fiber laying process and have the same size than the measurement values itself, the inline approach would not be an option.

#### Measuring the cross section of an impregnated tow with optical sensors

In this approach the cross section of the impregnated tow is captured with a camera or by laser-triangulation. Both methods provide an elevation profile of the tow. With this information it is possible to derive the cross section and calculate the cross sectional area. However, the total cross sectional area does not provide enough information to calculate the resin content. According to Eq.2, there are two unknowns ( $A_{\text{air}}$ ,  $A_{\text{res}}$ ). Therefore, additional information to distinguish between trapped air and resin is needed.

#### Measuring the heat capacity or the thermal conductivity of an impregnated tow by thermography

This technology is based on the fact that different materials have different thermal conductivity and heat capacity values. During a measurement the sample is illuminated with an infrared flash light. Meanwhile the sample is captured with an infrared camera. The time lapse provides information about the material parameter in the sample.

E. Cuevas et al. [5] and M.Palardy-Sim, together with P. Hubert [17] performed some experiments with this technology. Both investigations show a general applicability of thermography to measure the level of impregnation. Certainly in both experiments the focus is on the degree of compaction and on the amount of trapped air in prepreg material and not on the resin content. Nevertheless, they show that trapped air is a good thermal resistor and hence superimpose the measurement of resin content.

#### Measuring the damping of impregnated tows by non-contact ultrasound

Non-contact ultrasound is based on the damping of acoustic waves, which is caused by reflection on the interfaces and absorption by inner friction [18]. Reflection or scattering are much more dominant than absorption, especially on interfaces between air and solids (up to 99%).

Non-contact ultrasound is a technology that is already available on the market, to measure the level of impregnation of prepreg material. A. Bhardwaj from The Ultran Group provides an excellent overview of this state-of-the-art technology in his lecture at SAMPE-CAMX conference in 2015 [4]. In his presentation at JEC Europe in 2013 [3] A. Bhardwaj mentioned furthermore that it is possible to measure the porosity as well as the resin content. However, it can be seen that trapped air causes a much higher signal damping than resin compared to carbon fiber. Therefore, it can be assumed that variations in resin content are not able to be detected with the required accuracy if the sample includes trapped air.

#### Measuring the damping of radio waves caused by an impregnated tow

This approach is based on the damping of MHz, GHz or THz waves. Because the damping of air or trapped air is negligible, Eq.2 can be solved and the resin content is calculable. Carbon fibers are electrical conductors and demonstrate shielding behavior against electromagnetic waves. Normally electromagnetic waves are polarized linearly, which means that the shielding behavior is direction dependent. For a single tow all fiber filaments are orientated in one direction. If the wave direction is parallel to the fiber orientation, the damping is almost zero. In a rectangular direction the signal damping is almost one hundred percent. The relevant experiments are shown in [13] by K. Im et al., which verify the strong directional behavior of the damping of electromagnetic waves by carbon tows. A small deviation between the fiber orientation and the wave orientation leads to a significant signal change. Thus, it becomes difficult to distinguish between the damping caused by the material and the damping caused by angular deviation. To avoid or at least reduce this direction dependent behavior seems to be possible from the theoretical point of view, if a modified experimental setup is used (other frequencies, other antenna geometry or layout). Further tests are still pending.

#### Measuring the damping of ionizing radiation (x-ray or gamma rays) caused by an impregnated tow

For technical applications two types of ionizing radiation are used: artificially created X-rays and the naturally occurring alpha-, beta- and gamma- radiation. The attenuation coefficient for roentgen radiation of carbon composites is roughly a thousand times higher than the value for dry air (according to the law of Lambert-Beer and the corresponding values for carbon and dry air [12]). Thus, it is permissible to ignore the surrounding and trapped air. Measuring the thickness of foils with this technology has been a proven method for decades. This method has also been used for quality inspection of prepreg material for the last few years.

Y.D. Huang et al. [11] performed experiments in 2002 and 2003 to detect the fiber volume content in prepreg tows with a width of 4mm by beta- radiation. His results had an accuracy between 1-2%. This investigation verifies the applicability of this technology for the given task. Although this method is proven, there are reasons against its use. Ionizing radiation requires much shielding effort and a high level of responsibility of the operator. New upcoming regulations and laws make the operation of such a device complex and expensive.

Another approach that is even able to deliver a cross section of the impregnated fiber is the computer tomography. X-ray pictures are processed to calculate a 3d model of the captured sample. Although this approach sounds interesting, it is not suitable. The main reason is the currently highest resolution of only 5µm – this value is on the same scale as the filament diameter of the fibers. Also, the fact that the fiber and the resin have almost the same density renders it impossible to calculate the resin content with the required accuracy.

#### Infrared spectroscopy

This technology is based on the fact that substances absorb certain frequency domains of electromagnetic waves. This behavior is material specific. Near Infrared (NIR) spectroscopy is a proven technology in chemistry to analyse the composition of materials. According to the literature this technology is also usable to detect the resin content of impregnated glass fibers [16], [15] or even carbon tows [14]. All three references published by the Harbin Institute of Technology show that it is possible to measure the resin content and the amount of volatile components of prepreg material. However, the results should be scrutinized. This technology works with a wavelength of near infrared. For the semitransparent resin it seems to be possible to obtain a measurement for reflection, as well as for transmission through the material. However, the black carbon fiber has a very high absorption coefficient for this wavelength which leads to a penetration depth significantly smaller than one

filament diameter. Therefore, measuring in the transmission mode is not possible. Measuring the reflection could be possible, but the results would not be representative due to the low penetration depth of the tow, just down to the first filament. Consequently, it can be assumed that this technology would simply deliver superficial information concerning the resin content of the impregnated tows. Whether this is representative information requires further discussion.

#### Nuclear magnet resonance spectroscopy

Nuclear magnet resonance (NMR) spectroscopy is a standard technology in chemistry to analyse the composition of materials. The functionality of NMR spectroscopy is explained in detail in [8].

Compared to other technologies NMR spectroscopy provides information about the amount of a substance. The detectable substances are restricted and can be selected by the measurement properties. The most common measurement setup is the H1 spectroscopy that detects hydrogen. The carbon fiber includes almost no hydrogen (except the coating and remains of non-carbonized fibers) and is thereby almost invisible. The resin consists of a large amount of hydrogen and is easily detectable with this technology. The surrounding and trapped air also includes hydrogen (water vapor), but the amount is negligibly small compared to the sample itself. Therefore it is easy to detect the resin content with this technology. G.A. Barrall et al. [1] had already conducted their first experiments in 1998 to measure the resin content of prepreg tows inline by NMR spectroscopy. They measured the resin content of different prepreg tows with different velocities and the results had an accuracy of about 1%. Because the resulting measurement signal is very noisy, it is necessary to make several repetitions. According to the experiments of G.A. Barrall, each NMR measurement requires ~1.2s and each average measurement value consists of 10 measurements. Thus, the measurement period is roughly 12 seconds.

#### Measuring the permittivity of an impregnated tow

The capacity  $C$  of capacitors depends on the dielectric constant  $\epsilon$ , which is a material specific parameter. Thus, it is possible to characterise the material composition in the capacitor by its capacity. This method is used, for example, to monitor the degree of cure of resins. The appropriate sensors are already available on the market. This method is also applicable for carbon composite characterization and is shown in [7]. In chapter 16 it is explained how the permittivity and the permeability can be measured and the extent of the influence of the carbon fiber. It is shown that the carbon fiber leads to a reduced penetration depth, but nevertheless values can generally be measured with this method.

However, there is not just one material in the capacitor, but resin, fiber and air. Therefore,  $\epsilon$  is a function of all three materials and the capacity can be approximated by a series of connections between three capacitors. Because the material thickness and composition possibly vary over the width of the tow, it is necessary to approximate the three serial connected capacitors also as a circuit of parallel connected capacitors. Thus, a capacitor matrix is created. With this approximation of a measurement capacitor, the capacity varies depending on how the material is allocated between the capacitor plates and not just by the amount of material between the plates. Therefore, it seems to be impossible to measure the resin content.

#### Measuring the permittivity of an impregnated tow by eddy current

Eddy current is a well-known method for nondestructive testing and also applicable for carbon composites. S. Gäbler et al. shows in [10] and [9] that it is also possible to measure the permittivity of isotropic materials with this technology. According to [9], the measurement of permittivity for carbon composite materials seems to be possible, but is much more difficult than measuring isotropic material. In a series of tests, S. Gäbler investigated a carbon fiber sample with local damage (thermal matrix degradation). The result shows that this damage is clearly detectable. However, the question of

whether the measured values correlate with real matrix properties was not addressed with this experiment. The relationship of the matrix properties in carbon composite material and the eddy current measurement values is still part of ongoing experiments. Trapped air is not taken into account in these publications. The permittivity measurement by eddy current measures the dielectric properties between the fiber filaments. Because the trapped air is between the fiber filaments as well, it will affect the measurement results. In addition, because resin and fiber have a dielectric constant in the same scale, it would be mathematically impossible to separate both substances in the measurement results.

## 5. Comparison

In Table 1. all technologies are listed and assessed according to the main criteria for resin content detection. The main criteria include which material shares are contained in the measured signal (column: detected shares), is it possible to separate these shares (column: signal separation) and which shares are known (column: known shares). Consequently, it is possible to discover if the resin content is mathematically determinable or not.

**Table 1.** Comparison of all technologies

Technology		Detected shares			Known shares <sup>*4</sup>			Technical evaluation			Validation		
Id	Name	Fiber	Resin	Trapped Air	Signal separation	Fiber	Resin	Air	Trapped air	Resin content is identifiable <sup>*5</sup>	Negative issues		Applicable Applicable with restrictions <sup>*6</sup>
											Exclusion criteria		
1	Weight measurement	X	X			X				X	Disruptive vibrations	X	
2	Optical cross section capturing	X	X	X		X							X
3	Thermography	X	X	X		X <sup>*3</sup>							X
4	Air coupled ultrasound	X	X	X		X <sup>*3</sup>							X
5	Radio waves	X	X	<sup>*1</sup>		X <sup>*3</sup>				X	Signal shielding	(X)	(X)
6	Ionising radiation	X	X	<sup>*1</sup>		X <sup>*3</sup>				X	Official regulations		X
7	Infra-red spectroscopy	X	X	<sup>*1</sup>	X	X <sup>*3</sup>				X	Superficial information	X	
8	Nuclear-magnet-resonance spectroscopy	<sup>*1</sup>	X	<sup>*1</sup>	X <sup>*2</sup>	X				X	Long measurement period		X
9	Capacity/ inductivity measurement	X	X	X		X <sup>*3</sup>							
10	Permittivity measurement by eddy current	<sup>*1</sup>	X	X		X							X

<sup>\*1</sup> Negligible

<sup>\*2</sup> Just for spectra not for relaxation measurements

<sup>\*3</sup> By calibration

<sup>\*4</sup> Either given or by calibration

<sup>\*5</sup> Either by separated material signals or by knowledge of the other material shares

<sup>\*6</sup> Applicable if the trapped air is negligible

After the first assessment, several technologies were discarded due to the fact that they were not able to distinguish between resin and trapped air. In the second step all approaches were evaluated from a technical point of view. Thus, further technologies are discarded because of technical issues that prevent their applicability. The issues have already been discussed in detail in the last chapter concerning the technology screening.

## 6. Conclusions

It can be concluded that the discarded approaches for an inline, resin content detection system are weight measurement, capacity or inductivity measurement and infra-red spectroscopy.

The most promising approaches are ionizing radiation and nuclear-magnet-resonance spectroscopy. Even if the administrative requirements to work with ionizing radiation are high, this technology has potential from a technical point of view. The first tests with NMR spectroscopy show potential despite the long measurement time. However, this time could be reduced by optimising the technology and the measurement procedure. To check the applicability of these two approaches, further studies should be conducted.

The radio wave technology is not able to be validated because of missing results. On the one hand, it seems to be possible to measure the resin content, as the signal damping caused by trapped air is negligible. On the other hand, the shielding behavior of carbon fibers makes it difficult to obtain reasonable results. Nevertheless the previously mentioned idea to use a modified experimental setup could potentially solve this problem.

Furthermore, it is necessary to consider closely the amount of trapped air in impregnated tows. If it is possible to prove that the amount is negligible and reproducible, other technologies (non-contact ultrasound, optical cross section capturing, thermography and eddy current) are possibly applicable. It would also be an alternative to measure not inline but online between two layup steps with the weight measurement approach and a precision scale. Apart from that, it would have to be checked if sensor combinations (e.g. optical cross section capturing combined with eddy current or thermography) provide additional information which would make it possible to distinguish between resin and trapped air.

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