SHAPE ADJUSTMENT PROCESS FOR EPOXY COMPOSITES DURING POST CURE

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ABSTRACT

There is an increasing request for high precision composite parts for aeronautic natural laminar flow and low radar cross section applications. The allowed geometrical deviations for such applications are an order of magnitude lower than todays accepted tolerances. The implementation of established measures like tool compensation for spring-in or thermal expansion prior to the manufacturing process significantly reduces the occurring geometrical tolerances. But there are also variations during the manufacturing process that cannot be compensated in advance such as deviations during ply application or varying fiber angles. This publication shows a way how to adapt the manufacturing procedure to achieve high precision composite parts despite of process deviations. The curing characteristics of epoxy resins enable a two-stage curing process. In the initial step the resin is cured to about 65 % chemical interlacing. The viscoelastic properties of partially interlaced epoxides close to their current glass transition temperature allow an adjustment of the components geometry during the following post cure. This can be achieved using a closed loop temperature control combined with a cure monitoring and a geometry measurement system. This means that process induced deviations of composite components made from thermoset matrix systems can be adjusted during the post cure process.

1. INTRODUCTION

The strong demand for lightweight components and assemblies for aeronautical applications made from fiber reinforced thermoset polymers is primarily driven by economic reasons. Decreasing fuel consumption and improving the overall aircraft performance by saving weight and reducing drag are the key requirements for new products. It makes no difference whether those new products are developed for civilian or for military aviation. Some of the technical solutions that are applied to meet those top level requirements are based on high accuracy parts and assemblies. These include natural laminar flow wings or an aerodynamic surface that provides a low radar cross section. For both applications geometrical tolerances of a few tenth of a millimeter concerning steps, gaps and the overall aerodynamic shape are critical depending on the individual component and its position on the airframe. This is at least one order of magnitude lower than the current state of the art. Regular single step manufacturing processes using temperature as a single input value do often result in composite components with poor geometrical precision. The implementation of established measures like tool compensation for spring in or thermal expansion is necessary to significantly reduce the occurring geometrical deviations [1, 2]. However, there are also process induced variations that cannot be compensated in advance. There are for example inaccurate fiber angles during ply cutting and application or the misalignment of patches and reinforcements. The resulting geometrical deviations are hard to control because they are varying from part to part. The subsequent sections show a way how to adapt the common manufacturing process for thermoset epoxies using cure monitoring and geometry control to achieve high accuracy composite components.

2. GEOMETRICAL DEVIATIONS OF EPOXY COMPOSITE PARTS

To be able to quantify the geometrical deviations and particularly the variation within a series of parts test components are manufactured. To gain realistic data, true to scale wing parts of an unmanned aerial vehicle are chosen. All test components are manufactured using the same molds, exactly the same fiber material, the same resin batch and the same curing process. The parts are made of *Huntsman LY564 / Aradur 22962* and were cured at 60 °C inside the mold. After demolding they were cured up to 150 °C in a free standing post cure process. Figure 1 shows the I-shape spar and the lower wing cover that are both made from carbon fiber reinforced plastic.

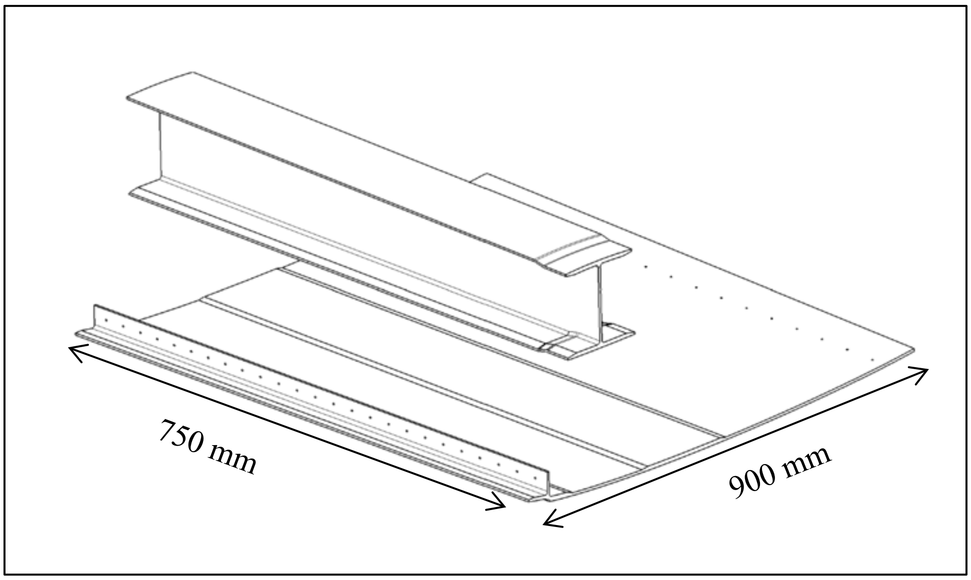

Figure 1. Main spar and lower skin of a UAV wing (courtesy of Airbus Defence and Space)

Four individual parts of each component are manufactured and subsequently measured using the optical geometry measuring system described in section 3.4. To ensure that there is no major individual distortion of a single component the global deformation of the parts of one kind is checked. The deviations are measured in predefined locations on each part. Based on this data an arithmetic average for each location concerning the series of four parts is calculated. Assuming that this resulting average distortion may be eliminated using established tool compensation methods only the variation from the mean value is investigated further. The following Table 1 shows the results depending on the measuring point.

	measuring point	mean deviation from target [mm]	sample standard deviation σ [mm]
Lower Skin	LS01	-0,173	0,039
	LS02	-0,250	0,057
	LS03	-0,103	0,090
	LS04	-0,960	0,228
	LS05	-1,105	0,241
	LS06	-0,825	0,141
	LS07	-1,113	0,377
	LS08	-0,300	0,192
	LS09	0,865	0,677
	LS10	1,390	0,564

	measuring point	mean deviation from target [mm]	sample standard deviation σ [mm]
Main Spar	MS01	-0,503	0,121
	MS02	-0,340	0,084
	MS03	-0,263	0,072
	MS04	-0,350	0,128
	MS05	-0,025	0,025
	MS06	0,233	0,067
	MS07	0,265	0,122
	MS08	0,125	0,129
	MS09	-0,545	0,177
	MS10	-0,530	0,143
	MS11	-0,538	0,135
	MS12	-0,563	0,146
	MS13	0,123	0,085
	MS14	0,213	0,149
	MS15	0,343	0,141
	MS16	0,415	0,166

Table 1. Varying deviations of four spars and four wing covers of the same make

Figure 9 which is located in the appendix of this publication shows the location of the measuring points on both components in an isometric view. Almost all of the calculated mean values exceed the allowable deformations derived from laminar flow or low radar cross section requirements. To avoid a huge number of unfeasible parts 95.4 % equal to $\mu \pm 2\sigma$ should show distortions within $\pm 0.2\ mm$. Considering the table above this is not true for almost all measuring points. This means that even after compensating major effects like spring in or thermal distortion a lot of the components manufactured this way would not be suitable for high accuracy applications. A final shape adjustment and correction process could solve this problem and increase the percentage of useable components.

3. SHAPE ADJUSTMENT DURING POST CURE

The following subsection outlines the curing behavior of epoxy based polymers which enables the shape adjustment process. Subsequently the process itself and the most relevant steps are described.

3.1 The curing of epoxy resin systems

Epoxy matrix systems consist of an epoxy resin and a curing agent that join in a polyadditive reaction. Figure 2 shows the two chemical precursors of this reaction, the epoxy resin and the diamine curing agent.

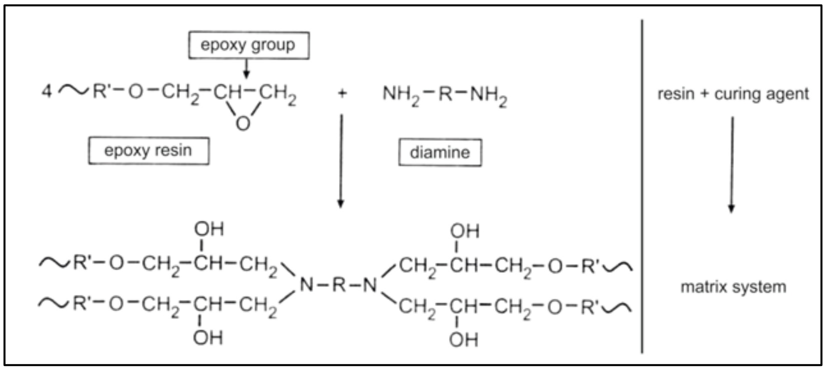

Figure 2. Polyadditive reaction between epoxy resin and a diamine curing agent [3]

The final product of this interlacing process is the epoxy matrix system. The number of crosslinks per molecule and therefore the three dimensional interlacing differs depending on the chosen resin system. In this study two systems are used. On the one hand there is *Huntsman LY564 / Aradur 22962* which is difunctional and starts curing at room temperature. On the other hand there is *Hexflow RTM6-1K* which is tetrafunctional and is usually cured at about 180 °C. The glass transition temperature is a specific property of the resin system and depends on its chemical composition. It is closely linked to the current degree of cure of the resin system. This means to achieve maximum mechanical performance and thermal resistance of the matrix it has to be cured to a degree of cure close to 100 %. Parts made from thermoset epoxy resins can usually be demolded at around 65 % degree of cure. At this point the matrix has a very low elongation at break and a low strength due to the incomplete interlacing. This makes the components extremely brittle. An important prerequisite to reduce process induced distortions of fiber reinforced epoxy components is to use a two-step curing process with a target temperature of the initial curing process that is as low as possible but sufficient to reach the necessary degree of cure of 65 %. Common post cure processes use a temperature monitoring to make sure the current curing temperature is not at or above the current glass transition temperature of the matrix. In case the curing temperature exceeds the current glass transition temperature the already established crosslinks regain rotatory degrees of freedom and the matrix system loses stiffness and starts to show a viscoelastic behavior [4]. With ongoing crosslinking and with an increased glass transition temperature stiffness is regained.

Chemical bonds break and the matrix is irreversibly damaged if the curing temperature exceeds the individual decomposition temperature of the resin system which is usually located about 20-30 °C above the maximum achievable glass transition temperature.

3.2 The shape adjustment process

The previous section explains that there is a narrow process window between 65 % and 100 % degree of cure in which a FRP (fiber-reinforced plastic) part made from thermoset epoxy can regain viscoelastic properties even when the initial cure is finished and the part is already demolded. This means that under controlled circumstances a plastic deformation is possible during post cure. This specific behavior is also explained by *W. Hufenbach et al.* to be used for processes that are commonly known from the manufacturing of thermoplastic components [5]. The following Figure 3 shows the overall view on this shape adjustment process.

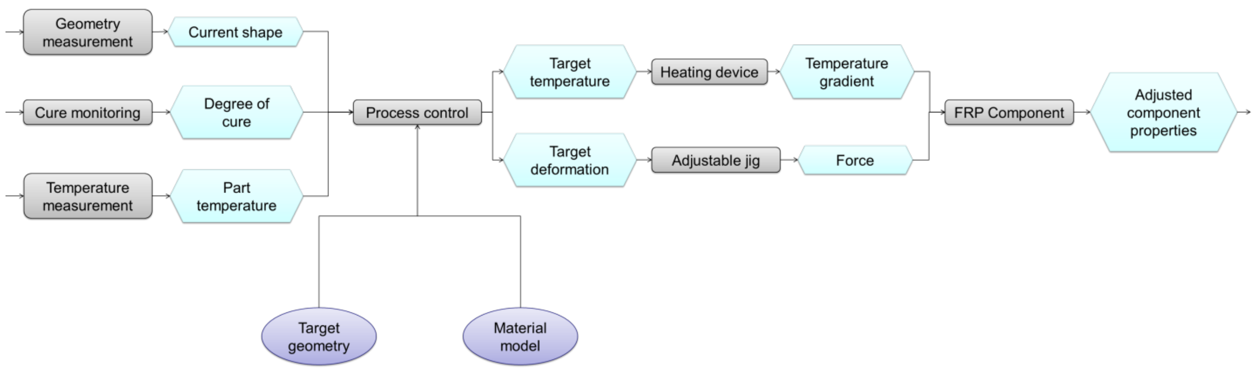

Figure 3. Diagram of the shape adjustment process during post cure

Rectangles symbolize devices or hardware. Hexagons contain properties and data that have to be acquired. The two ovals at the lower end stand for input values that are not changing during the whole process runtime. Starting from left to right in the first step the current imperfect shape of the part is measured. The second important property of the component is the current degree of cure that is acquired using a cure monitoring system. Including the current part and curing temperatures the process control unit can compute the maximum applicable strain. This happens based on the acquired sensor data and the external material model. By comparing the current measured shape and the target geometry the process control unit calculates a target deformation that does not exceed the maximum allowed strain at the current curing point. The curing temperature has to be controlled to stay within the process window that provides the viscoelastic behavior. An adjustable jig applies the necessary forces to the component based on the calculated target deformation. Both measures of interaction the curing temperature control and the deformation of the shape lead to adjusted properties of the FRP component. This means it has a new shape and a progressed degree of cure. Therefore the sensor data has to be refreshed and the significant values have to be recalculated. This leads to a closed loop adjustment process that is finished when the target deformation and a degree of cure close to 100 % is reached.

3.3 Monitoring the degree of cure

There are different cure monitoring systems for thermoset epoxy resins that reached maturity in recent past. In previous publications *S. Konstantopoulos, R.Meier and N. G. Pantelelis* give a good general view on those systems [6, 7, 8]. The most important principles are on the one hand the acoustic cure monitoring based on piezo transducers described by *N.Liebers* [9]. On the other hand there is the dielectric cure monitoring investigated and refined by *N. G. Pantelelis et al.* and *G. Gkikas et al.* [8, 10]. The main advantages of those two principles compared to spectroscopy or thermography are that they can be used without any major modification to the process or to the mold and that there is no mechanical interaction with the specimen. The usability of those cure monitoring systems has been discussed in a previous publication [11]. As a result the dielectric cure monitoring system *PDE-1* provided by *Gel Instrumente* was chosen. The main reason is that the used flat interdigital sensors can stay on or inside the component even after it was demolded. This means one can reconnect them again to monitor the crosslinking during post cure outside the mold. The measured conductivity describes the mobility of the charge carriers inside the matrix and their ability to align. Ongoing crosslinking between resin and curing agent leads to an increased viscosity and therefore to a decreased mobility of the charge carriers. As a result the conductivity declines during the curing process. At the moment the glass transition temperature exceeds the current curing temperature the matrix loses its viscoelastic behavior and dipoles and ions are not able to align anymore. This means conductivity is at the lowest level [4, 9].

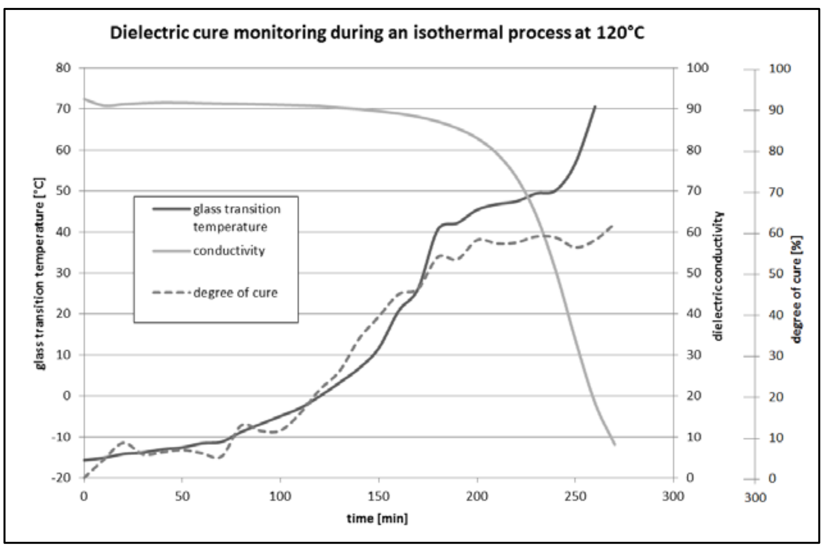

Figure 4. Cure monitoring of Hexflow RTM6-1K during an isothermal curing process at 120 °C

Figure 4s shows an exemplary dielectric cure monitoring chart acquired during an isothermal 120 °C curing process of a *Hexcel Hexflow RTM6-1K* resin sample. The glass transition temperature and the degree of cure are determined according to *ISO 11357* using a *DSC822*^{e} differential scanning calorimeter provided by *METTLER TOLEDO*. It shows that the resolution of the dielectric system at about 60 % degree of cure is sufficient to securely determine the point when the component can be demolded with the maximum process window left for the shape adjustment during post cure.

As the dielectric cure monitoring system works with the orientation and the mobility of the charge carriers it enables also a detection of the weakening during post cure. The chart in Figure 5 shows the increasing conductivity when the curing temperature exceeds the current glass transition temperature of an epoxy resin specimen. The broken line marks the beginning of this process. From this point the glass transition temperature of the specimen increases and the conductivity decreases because of the continuing crosslinking process.

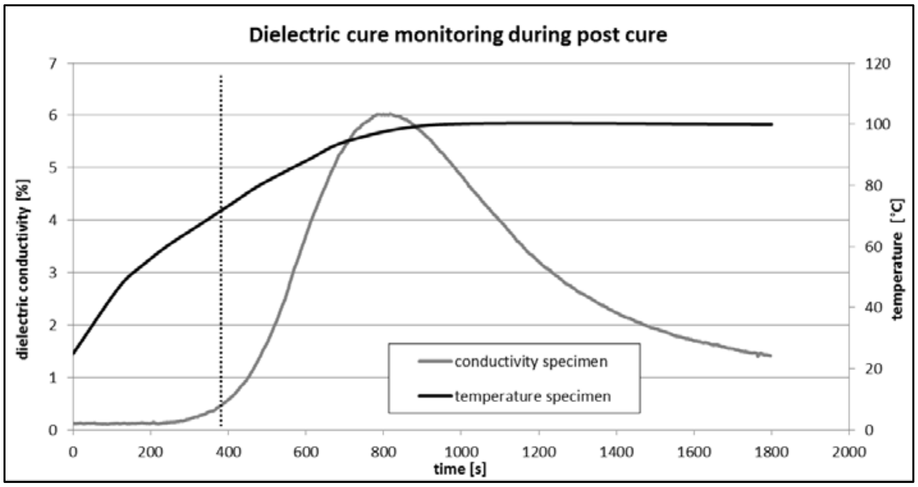

Figure 5. Detection of the weakening of the matrix during post cure

3.4 Optical geometry measurement during the curing process

In a previous publication the applicability of different geometry measurement systems has been investigated. The photogrammetry proofed to be the most suitable principle to be used for the shape adjustment process during post cure [12]. The main reason is that large components can be measured with a reasonable effort. The following Figure 6 shows the basic principle of a light stripe projection system which is capable of acquiring geometry data of the whole part surface. The output usually is a large cloud of points. A drawback of this system is its sensitivity concerning the perspective and the lighting of the specimen inside the furnace or the autoclave.

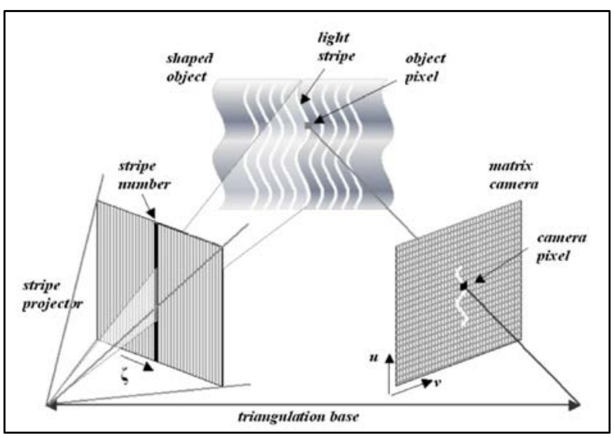

Figure 6. Photogrammetry, phase measuring projected fringe method [13]

As it is sufficient for the shape adjustment process to measure the deviation of predefined points the *GOM Pontos* dynamic measurement system has been chosen. It consists of a stereo camera system that is able to track marked measuring points with up 4 kHz depending on the number of points. The high contrast of those marks compared to the components surface enables accurate measurements also in poorly lighted environments. Figure 10 shows such a stereo camera system in front of the laboratory furnace. The technical solutions available are usually designed for optical geometry measurement at ambient pressure and room temperature. To enable shape monitoring for post cure those systems have to be adapted to process temperatures up to 200 °C. This can be achieved by either equipping the photogrammetry system with a powerful active cooling system or by using a window that enables a mounting outside the furnace or autoclave. For this study the second solution has been chosen for safety reasons as the sensitive measurement system must not be damaged by the process temperature. For this purpose an optical window made from special temperature resistant and nonreflecting borosilicate glass is installed.

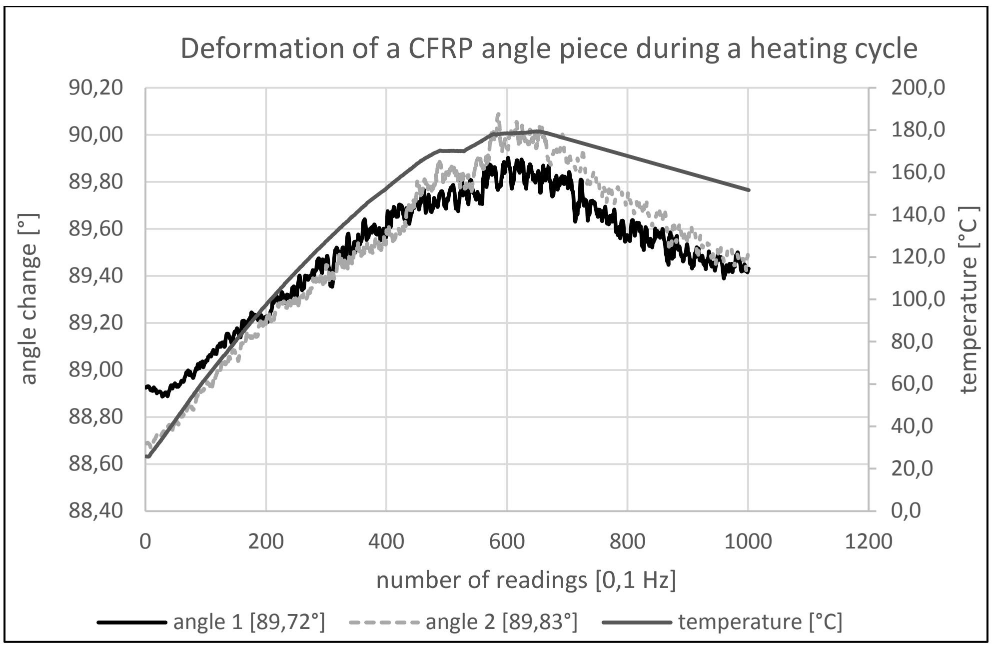

Figure 7. Photogrammetry results of CFRP angle pieces during a heating cycle

The chart in Figure 7 shows the results of an exemplary measurement of two CFRP (carbon fiber reinforced plastic) angle pieces made from *Hexcel Hexply 8552* epoxy prepreg. The parts were cured at 180 °C in a closed mold with an exact bend of 90°. The actual angles of 89,72° and 89,83° at room temperature result from process induced deformations such as chemical shrinking or the spring-in effect which are further described by *E. Kappel* and *M. Kleineberg* [1,2]. The angle pieces are heated up inside a furnace and the thermal induced deformation is monitored using an optical geometry measurement system. The graph shows that the angle pieces open up until they reach almost exactly 90° bending angle at a temperature of 180 °C. As the legs of the specimens which are shown in Figure 11 have a length of 50 mm each an angle change of 0,1 degree leads to a straight deviation of 0,088 mm at the tip. The acquired data shows that the resolution of the monitoring system is sufficient to cover this order of magnitude.

4. EXPERIMENT

To confirm the assumption that the shape of partially cured fiber reinforced epoxy components can be adjusted during post cure an experiment is performed. Two types of glass fiber fabric reinforced *Huntsman LY564 / Aradur 22962* specimens were manufactured with an initial degree of cure of 88% and a corresponding glass transition temperature of 75 °C. Both specific values were determined using a differential scanning calorimeter according to *ISO 11357*. The first type of sample was built with the fiber orientations 45°/135° the second one consists of a quasi-isotropic laminate. The wall thickness of all flat specimens is 3 mm.

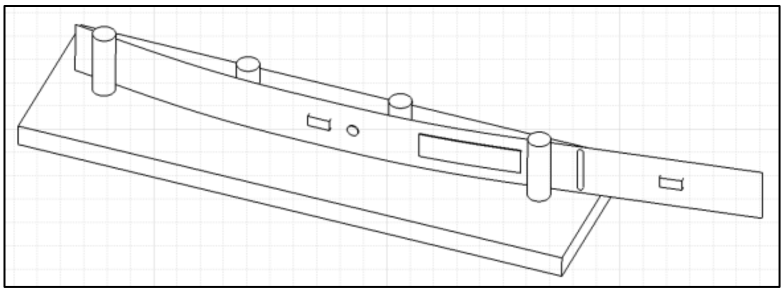

Figure 8. Static 4pt bending device with specimen

The flat samples are elastically deformed to a predefined distortion that equals a maximum applied strain of 10000 microstrain. They are fixed in a 4pt bending device as shown in Figure 8 at room temperature. The base plate of the device is made from CFRP to avoid thermal expansion during the post cure process. After that the specimens are heated up to 150 °C and cured for two hours to achieve close to 100% degree of cure. The permanent deformation after cooling down is measured using the light stripe projection system *GOM ATOS*. The resulting bending radius of the former flat 45°/135° specimens is 195 mm. The radius of the quasi-isotropic samples is 222 mm. This directly shows the influence of the chosen reinforcement on the achievable geometry adjustment during post cure. Nevertheless this test procedure proves the concept. The shape of partially cured and already demolded parts can be modified during post cure. Considering the fact that only deviations in the order of a few tenth of a millimeter should be adjusted this degree of deformation seems to be totally sufficient. Starting at a lower degree of cure than 88 % even expands the formability. A drawback is that specimens at a lower degree of cure are extremely brittle. This means they cannot bear for example 10000 microstrain at the beginning of the adjustment process. For this reason it is necessary to model the properties of the matrix under curing conditions.

5. CONCLUSION AND STEPS AHEAD

There are applications for fiber reinforced epoxy components that require extraordinary geometrical accuracy. The investigation of the process induced deviations on a series of parts shows that it is necessary to improve the common manufacturing process for thermoset epoxy resins. The curing behavior of epoxy resins is explained. The possibility to demold partially cured components and process them further in a post cure step is the basic principle of the shape adjustment process. Figure 3 shows this process including all required data and devices. Apart from the curing temperature the two most important parameters to measure are the current geometry and the current degree of cure. A dielectric cure monitoring system is investigated during initial cure and during post cure. The information it provides is sufficient to determine the moment the part can be demolded as well as the point in time the matrix regains viscoelastic properties during post cure. An optical geometry measurement system is tested too. To determine its feasibility and accuracy CFRP angle pieces are measured during a heat up cycle. This test shows that even under poor lighting conditions the system is capable to measure small deviations in real-time. A proof of concept is done using a static 4pt bending test. The flat specimen were elastically deformed and post cured in a bending device. The resulting permanent deformation turned out to be sufficient for the necessary adjustments but the influence of the chosen laminate has to be investigated further. According to the process overview those individual devices have to be connected to establish a closed loop process. A critical point will be the achievable accuracy of the combined process chain especially if an adjustable jig should be integrated. Further investigations also have to be made concerning the material properties of the matrix system after the adjustment took place. One has to make sure that there is no negative impact. A feasible approach could be a tensile test of a 45°/135° reinforced laminate that has been shape adjusted. Comparing the results to data acquired from specimen cured in a common way should show whether there is an impact or not. In addition the fiber-matrix adhesion may also be influenced by the special curing process. A fiber pushout test should be performed using the specimen from the tensile test mentioned above.

6. APPENDIX

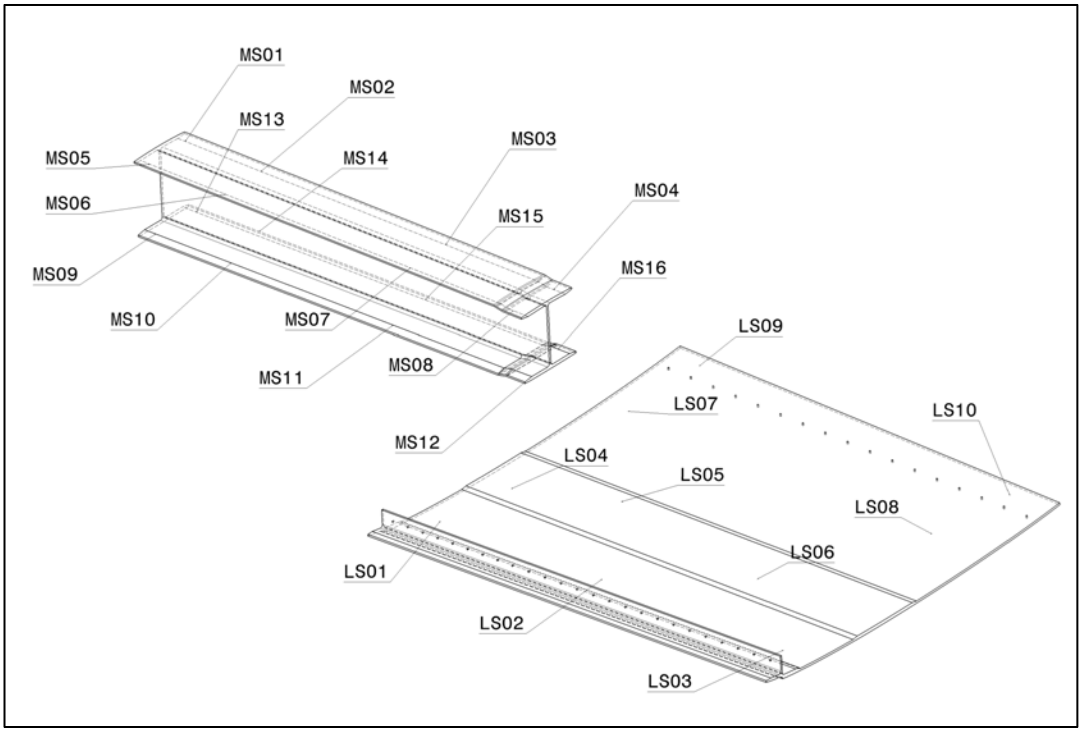

Figure 9. Measuring points on the components main spar and wing cover (courtesy of Airbus Defence and Space)

Figure 10. Stereo camera system placed in front of a furnace with a temperature-resistant and nonreflecting window.

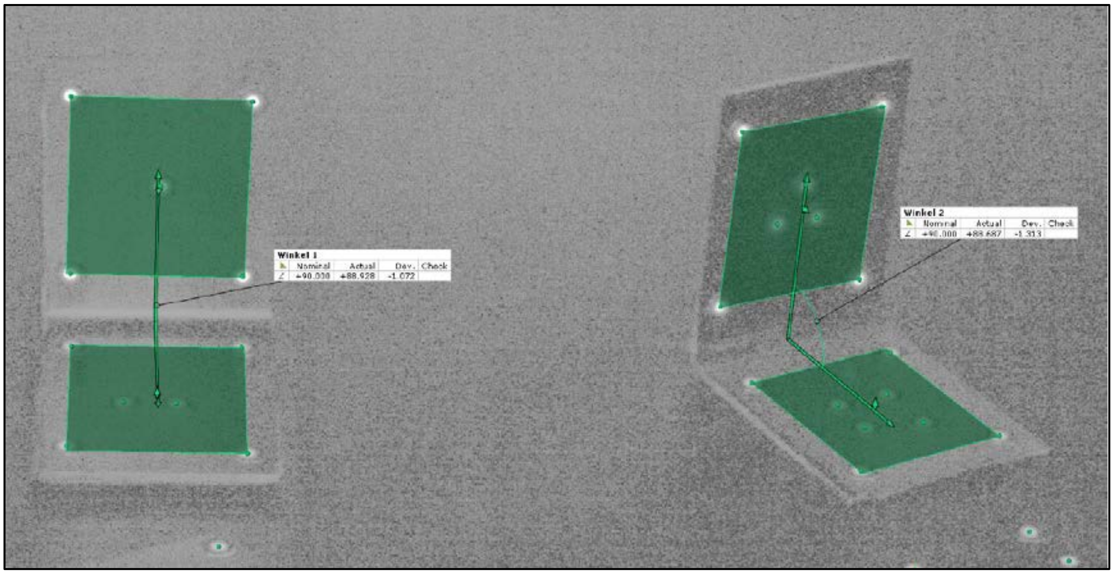

Figure 11. Camera view on both angle pieces inside the furnace

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