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Modelling of indirect laser-induced thin-film ablation of epoxy for local exposing of carbon fibers

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Abstract

Laser radiation is used as enabling technology for intrinsic joining of high-strength CFRP laminates and fiber-reinforced thermoplastic injection moulding compounds by exposure of surface-near carbon fibers. Short-pulsed NIR laser sources represent an acceptable compromise with respect to ablation performance, remote process capability by use of compact 3D scanner and the capability for closed-loop process control. However, using such a laser source means also minimizing heat-affected zones (HAZ). Based on literature research about laser ablation of thin metal films, heat flow at CFRP and thermo-mechanical behavior in FRP by pyrolysis, an analytical model was generated for thin-film ablation of cured epoxy resins at the surface of CFRP laminates by lift-off of resin chips.

A comparison between simulation and experimental results confirms the capability of the model to predict the exposure area and the HAZ with deviations below 15%. Threshold fluences for the HAZ (>1 J/cm²) and the resin ablation (>3 J/cm²) have been confirmed.

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Keywords: thin-film ablation; composites; epoxy; pyrolysis; modelling

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1. Introduction

Innovative car body structures e.g. for electric mobility are made out of carbon fiber-reinforced composites (CFRP) based on infusion technologies e.g. for BMW i3 or i8. New approaches for functionilalization of CFRP parts with thermoset matrices are investigated in a number of research projects. Bruckbauer et al. (2015) in example integrate of thermoplastic foil on CFRP surface before curing for enabling joining of onserts within short cycle times; but material combinations are limited. An alternative approach was found in laser technologies: Laser treatment is already used for micro structuring pretreatment for adhesive bonding by Buechter et al. (2011), for shafting (stepwise removal of fibers and matrix) for the preparation of FRP repair by Fischer et al. (2011), as well as for fiber exposing and following laser transmission welding of dissimilar types of FRP by Amend et. al. (2012).

Based on these exemplary high potential laser processes, a consortium of KraussMaffei Technologies GmbH, BMW AG, ARGES GmbH, Precitec GmbH & Co. KG, Sensortherm GmbH, Zeiss Optotechnik GmbH (formerly Steinbichler Optotechnik GmbH) and the Aachen Center for integrative Lightweight Production (AZL) of RWTH Aachen is developing an integrated process chain for intrinsic joining of thermoset and thermoplastic composites by laser pretreatment for the surface of thermoset CFRP in order to subsequently overmold this surface with thermoplastic compound, Emonts et al. (2015).

Fiber exposing is a 3D surface process without damaging of carbon fibers in order to locally ablate the approximately 15 µm thin epoxy layer so that the carbon fibers are exposed up to the center of the top fiber layer, see figure 1c. The fiber exposing process is a special form of surface structuring of CFRP. The resulting microstructure enables a wetting of the joining partner e.g. thermoplastic compound in overmolding process by micro-form closure and surface activation. Further specific adhesion mechanisms are assumed, for example by resulting functional groups.

Nomenclature

C_P	Specific heat capacity [J/(kg·K)]
d_{Spot}	Beam diameter [m]
E_{Puls}	Pulse energy [mJ]
F_D	Lift-off force of pyrolysis gas [N]
I	Laser intensity [W/cm²]
J_{EP}	Mass-related velocity of pyrolysis within epoxy [kg/(m^{3}s)]
K_g	Gas permeability
m_{Puls}	Mass of epoxy ablation [g]
P_u	Atmospheric pressure [Pa]
R_s	Specific gas constant [J/(kg·K)]
T_0	Room temperature [K]
T_{sub}	Sublimation temperature [K]
t_{Puls}	Puls duration [s]
V	Volume of epoxy [m³]
α_k	Absorption coefficient of carbon layer
ε_k	Emission of carbon layer
κ	Thermal diffusivity [m²/s]
λ	Thermal conductivity [W/(m·K)]
ρ	Density [kg/m³]
σ_S	Yield shear strength [N/m²]
ω	Focus radius of the laser beam [m]
φ	Volume content
Γ	Gasification coefficient

2. Material and process

A nanosecond short pulse laser was chosen, because of its potential for high exposing rates per investment costs. Further reasons for using a near infrared (NIR) laser source are the easy integration in robot-guided scanner system and supplement of sensor technology in main optical path of the scanner. Because of the interaction between laser radiation and the treated material highly depends on the used laser source, Wolynski et. al. (2011), an understanding of ablation mechanisms using a short pulsed NIR laser source was developed and will be presented in chapter 2.3.

Building up a model is done in order to minimizing the needed amount of energy per area for increasing surface processing rate and minimizing heat affected zone, laterally and analogously in depth.

2.1. Laser source

The ablation processes with nanosecond lasers were performed using a "SPI G4" of the company SPI Lasers, Southhampton, UK with 70 W, λ = 1,064 nm and 240 ns puls duration at a repetition rate of 70 kHz. A maximum pulse energy of 1 mJ can be reached. The beam quality of the laser system can be characterized by $M^2 = 1.4$. The intensity of the beam was generated out of a raw beam diameter of 16,8 mm with a rayleight length of 370 µm and a focal length of 250 mm to a focus spot of 27 µm with a $1/e^2$-intensity measured and verified with a Primes MicroSpotMonitor beam analyser for different levels relative to focus position. For beam guiding, a Lightning II Digital Scanner System "LII HI-PERF 30 mm Scanpack WTR Cooled YP" of company Cambridge Technology, CSI Group Europe GmbH, Planegg was used. All fluences are averaged values of the beams' Gaussian profile and are referring to the measured spot diameters.

2.2. Samples

The samples, used for laser process investigations and model validation, are made of carbon fibers and epoxy resin. The fully cross-linked laminates out of carbon fiber-reinforced thermoset of the company SGL, Wiesbaden, named "CFK-Platte hoch-fest" with a continuous operating temperature of 140°C and fiber content of 62% are made of Sigratex Prepreg CE 1007-310-37 with Epoxid (022).

2.3. Directly- and indirectly-induced ablation

In general, there are directly-induced ablation processes, which act at the surface of a material and indirectly-induced ablation process, where the laser penetration depth is higher and the absorption happens within the material, which leads to process concentration on deeper material interfaces if two phases with different absorption characteristics exist. In case of directly-induced ablation the optical penetration depth of laser beam is significantly smaller than the material thickness (surface absorption). This means, most energy is absorbed in the matrix material of a composite and leads to a ablation which is proportional to puls energy. For example, pulsed ultraviolet lasers with a wavelength of 200 nm up to 400 nm or pulsed CO_2 laser with a wavelength of 9,4 µm up to 10,6 µm show high absorption coefficients for thermoset resins, Kreling (2014), and can therefore be assigned to directly-induced ablation.

Using laser radiation with a wavelength of 1,064 nm leads to a completely different ablation mechanism in this case: Only a small amount of the energy is absorbed in the thin epoxy resin film, because of a low absorption coefficient of 0.005 mm^{-1}, Firbank et al. (1995). The amount of energy which reaches the carbon fibers is by roughly 90% absorbed; comparison to analysis of emissivity of thermoset prepreg by Werner et al. (2014). Based on this information it can be assumed that the major part of the radiation transmitts the thin resin layer and is absorbed at the top carbon fiber layers. This leads to high temperatures at the fiber, especially at top fiber surface. Subsequently, the resin, which is directly in contact with the hot fiber, is sublimated – in this paper assumed by pyrolysis. Because there is no way for the pyrolysis products to diffuse within the short puls duration, the pressure within the fiber-resin interface will lead to mechanical effects on the thin resin layer. This ablation can therefore be called an indirectly-induced ablation. The fluence parameter has to be chosen in such a way, the resin is ablated but fibers are unaffected as far as possible. Analysing the process zone splashy particles are visible, see figure 1a. It is assumed, that resin particels lift-off and fly through the guided beam. Based on absorption characteristics of top-surface resin layer a lift-off is assumed. In case of an indirectly-induced ablation an undesirable heat affected zone can not be avoided, Weber et al. (2011).

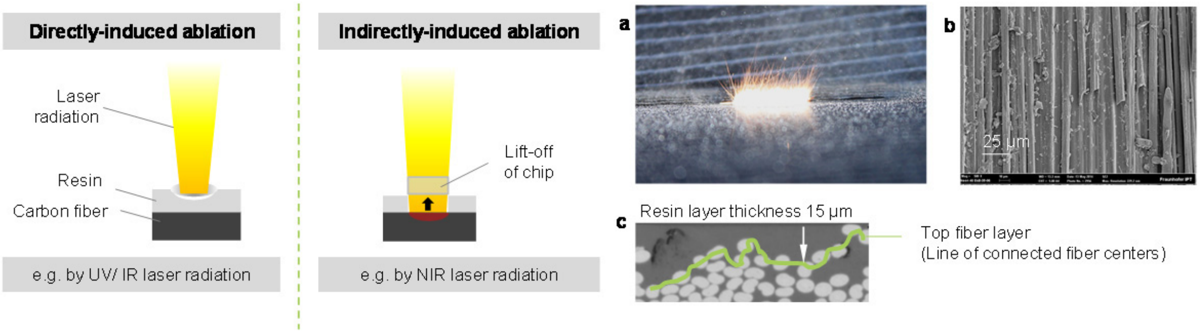


Fig. 1. Directly- and indirectly-induced ablation processing of CFRP surfaces. (a) Splashy process using NIR short-pulsed laser source. (b) Exemplary surface after ablation of resin layer by short-pulsed laser beam recorded by REM.

The theory of lift-off phenomena is derived from the ablation of thin metal films. Here, the laser lift-off process is characterized by a relative small effective penetration depth of the laser pulse compared to the thickness of the absorbing layer by having high absorption and ultrashort laser pulses. Time-resolved investigations and simulations confirm a ultrafast thermal expansion during heating and melting of the laser-matter interaction zone and mechanical bulging of the thin film by generating a shockwave with final lift-off at 20 ns, Domke et. al (2014).

3. Indirectly-induced ablation model

The basic idea of here used two layer model, see chapter 3.1, is the selection of an optimal scale of material model on the one hand. This means, focusing on the region of interface between fiber and epoxy where most reelvant mechanisms are acting. On the other hand, enables the combination of simple thermal, thermo-mechanical and mechanical calculations describing complex phenomenon.

3.1. Two layer model and procedure of model

For simplifying the process specified in chapter 2.3 a two layer model is used. Instead of round, often irregularly distributed carbon fibers, a homogeneous carbon plate is considered. This plate has isotropic thermal characteristics. The thickness of carbon fiber plate is 7 µm, which corresponds to the diameter of a carbon fiber. A further simplification is that the total energy of the Gaussian beam can pass through the epoxy resin without any absorption, due to the high transmittance of the epoxy resin and the thin epoxy film of around 15 µm.

Figure 2 demonstrates the modelling procedure. It also gives an overview about the necessary framework of equations, which are described in chapter 3.3. The green symbols define the conditions which are necessary for the validity of the model. If the condition is not fulfilled, modeling has to be stopped. A intensity of 45,000,000 W/cm² should not be exceeded. The temperature of 800 K, as well as atmospheric pressure and resin layer thickness, has to be exceeded. The output of the model are statements about the ability of ablation, dimensions of the ablation and HAZ as well as temperatures.

The equations which are necessary for calculating the model are described in the following chapter. The differential equations are solved with Matlab®. Measurements described in chapter 5 are done in order to validate the here generated model results.

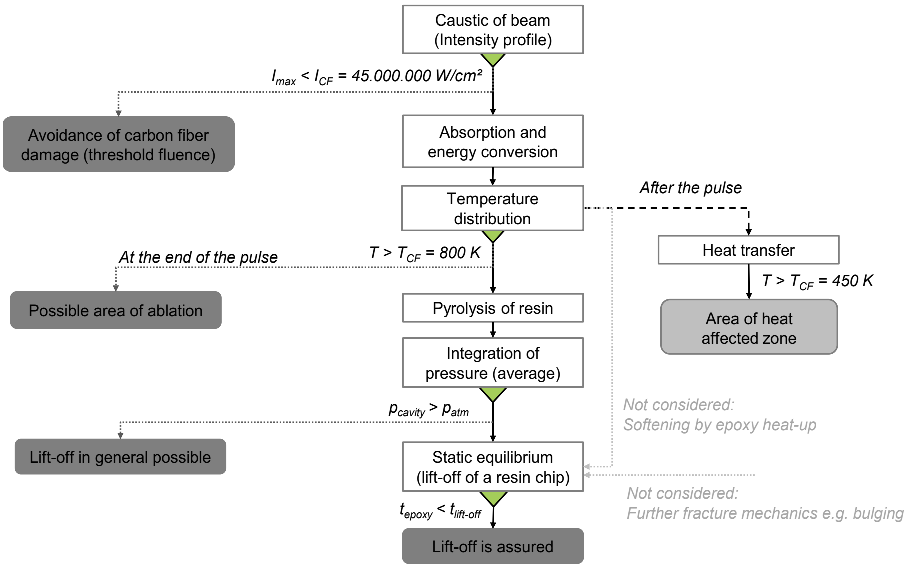


Fig. 2. Procedure for modelling of lift-off phenomena by two phase approach for processing CFRP surfaces with short pulsed NIR laser.

3.2. Material parameters

The quality of results significantly depends on the quality of the material parameters. The parameters found in literature vary. The sublimation temperature of epoxy is important for modelling. It can vary from 700 K up to 875 K. For carbon fibers this value is between 3,000 K and 4,000 K. Therefore a material parameter definition with the highest degree of coverage was done. Temperature-dependent material characteristics within constant phases were not considered.

Table 1. Defined material parameters for indirect ablation model with two phases based on literature research.

Material property	Carbon fiber	Epoxy
Density	1,850 kg/m³	1,300 kg/m³
Specific heat capacity	710 J/(kgK)	1,800 J/(kgK)
Thermal conductivity	50 W/(mK)	0.2 W/(mK)
Enthalpy of sublimation	43,000 kJ/kg	1,000 kJ/kg
Temperature of sublimation	3,900 K	800 K
Temperature of degradation	n.d.	450 K

3.3. Foundations for model

The spatial resolution of the beam caustic with Gaussian beam profil is described by equation 1. The intensity profile is characterized by puls duration t_{Puls}, puls energy E_{Puls}, puls power P_{Puls}, spot beam radius ω_0 and intensity I_{max}. The time-resolved power form is simplified by a rectangular profile.

$$I(r) = I_{\max} \cdot \exp(-\frac{2r^2}{\omega_0^2}) \quad with \quad I_{\max} = 2 \cdot \frac{P_{Puls}}{\omega_0^2 \cdot \pi} \quad and \quad P_{Puls} = \frac{E_{Puls}}{t_{Puls}} \qquad (1)$$

Pulsed laser ablation processes above a puls duration of picosecond or femtosecond base on energy conversion into heat. The energy conversion and heat transport from an ideal point source at a carbon disk surface can be described by the differential equation 2, see Weber et al. (2011). The left term corresponds to the storage capacity of the carbon disk, the first term on the right side to the thermal conduction within the disk, the second term to emission of the disk to the environment.

$$c_P \cdot \rho \cdot \frac{\partial T}{\partial t} = \lambda \cdot \left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \cdot \frac{\partial T}{\partial r} \right) - \frac{1}{h_P} \cdot \varepsilon_k \cdot \sigma \cdot (T^4 - T_0^4) \tag{2}$$

The initial and boundary conditions are defined in formula 3.

$$-\lambda \frac{\partial T_{0,t}}{\partial r} = \alpha_k \cdot I \,, \quad \frac{\partial T_{6000\mu m,t}}{\partial r} = 0 \quad and \quad T_{r,0} = 300K \tag{3}$$

The parabolic partial differential equation corresponds to a transient 2D heat transport equation, in the direction of polar coordinates with an ideal point source, Baehr et al. (2008). The Gaussian beam has been replaced by an finite number of ideal point sources. Heat conduction is neglected, due to the heat transport within 240 ns at the threshold of carbon fiber is only about 1 μm. Based on equation 2 and resulting temperatures a limiting intensity of $4.5 \cdot 10^7$ W/cm² can be identified. At this point fibers sublimate and the model is not valid anymore, because energy conversion by phase shift of carbon fibers is not implemented. Furthermore, the minimum intensity for sublimation of epoxy has to be reached. Low fluences where only the center part of the spot lies over the epoxy threshold are predestinated to validate the model. Simplifications are done by use of temperature-independent thermal and optical material parameters as well as a neglection of loss of energy by plasma.

The connection between thermal and mechanical mechanisms is integrated by pyrolysis, see equation 4. Pyrolysis is a transfer from solid to gaseous without oxygen input. This effect was already used for describing stress conditions within composites during heat up with continuous wave radiation, Dimitrienko et al. (1990), Dimitrienko (1992), Dimitrienko (1997) and Dimitrienko (2000). Based on the ideal gas law of Avogadro the gas density, determined with the differential equation 4, can be calculated together with the field of temperature and assumed volume at the beginning of bulging or lifting. The differential equation describes the mass transport of gas. The left term is the accumulated amount of pyrolysis gas. The right hand term stands for the amount of gas, which is generated by high temperatures (direct after the end of the laser puls) as well as the amount of gas which diffuses through the epoxy. Because of the minimal thermal conduction within the epoxy layer during short puls duration any time-dependent effects are minimized. The considered place of action is near to the fiber surface.

$$\frac{\partial \rho_g \varphi_g}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left\{ R_s \cdot K \cdot r \cdot \frac{\partial \left(\rho_g \cdot T(t, r_{sub}) \right)}{\partial r} \right\} + J_{EP} \cdot \Gamma_{EP} \tag{4}$$

$$with \;\; J_{EP} = J_{EP}^0 \cdot \exp\left(-\frac{E_{EP}}{R_s T} \right), \quad P = \rho_g \cdot R_s \cdot T(t, r_{sub}) \quad and \quad K_g = K_0 \exp\left(50 \cdot \varphi_g^{\frac{1}{3}} \right)$$

The initial and boundary conditions are defined in formula 5, whereby r_{sub} maximum radius with T > 800 K is.

$$\rho_{r,0} = 0 \,, \quad \frac{\partial \rho_{0,t}}{\partial r} = 0 \quad and \quad \rho_{r_{Sub},0} = 0 \tag{5}$$

Most pyrolysis specific coefficients used from the literature of Dimitrienko (2000) e.g. the amount of pyrolysis volume in composite $\varphi_g = \varphi_0 = 0.07$ and the gasification coefficient of epoxy $\Gamma_{EP} = 0.78$ as well as $J_{EP}^{0} = 3.2 \cdot 106$ kg/(m³s).

The lift-off phenomena of a resin chip requires at least a total pressure in the resin-fiber-cavitiy over atmospheric pressure of 1 bar (100,000 Pa). Because the field of pressure is significantly depending on temperature, maximum pressure is where the highest temperature profile at the end of the pulse exists. The averaged pyrolysis pressure P_D in cavity can be calculated by an integral over the circular spot area in equation (6).

$$P_D = \frac{\int_0^{r_{sub}} P(r) 2\pi r dr}{\pi r_{sub}^2} \tag{6}$$

It is assumed that mechanical effects act in a very short time period, which makes a simplification to a static problem feasible. In that case a static equilibrium of forces in z-dimension regarding equation (7) is possible, but should be understood as “safe” assessment. It is assumed, that shear stress is the dominating mechanical effect for lift-off. Of course, adhesion to fibers, bulging of chip, bending of chip and tension stresses along the chip are happen simultaneously in reality. Furthermore, the existence of shockwaves, called by Domke et al. (2014), e.g. by internal thermal stresses can not be excluded.

$$\pi \cdot r_{Sub}^{\ 2} \cdot (P_D - P_u) = \sigma_S \cdot 2\pi \cdot r_{Sub} \cdot th_{\max} \tag{7}$$

If a lift-off has to occure the cavitiy pressure relative to atmospheric pressure should be high enough for surpass the resistance against shearing the chip. This resistance is depending on resin layer thickness and shear strength σ_s of the resin. The (lowest) maximum resin thickness th_{max} can be calculated based on the equilibrium. Reduction of shear strength by heat is neglected. Note, the shear strength of cross-linked epoxy resins vary from 9 MPa, Dimitrienko (2000), up to 50 MPa, Netravali et al. (1989).

For determination of the lateral dimension of the heat affected zone the thermal conduction of heat at carbon fiber surface after the end of the pulse is important. In relation to equation 2 the initial and boundary conditions are adjusted to describe a thermal energy balancing without external input, compare Weber et al. (2011).

$$T_{r,0} = T(r, t_{240ns}), \quad \frac{\partial T_{0\mu m,t}}{\partial r} = 0 \quad and \quad \frac{\partial T_{6000\mu m,t}}{\partial r} = 0 \tag{8}$$

Because of the confined laser ablation, compare Domke et al. (2014) – absorption on top carbon fiber layer without destroying of carbon fibers – there are difficulties in determination of heat affected zone in same direction as laser beam – here depth – compare analyzing of HAZ during laser cutting by Rose et al. (2015).

4. Modell results

Exemplary, the model is applied for the evaluation of change in fluence by spot defocusing. A variation of spot diameter by going out of effective focal spot area is chosen. Motivated for increasing the area-oriented ablation rates and identification of minimum puls energy, the existance of limiting effects should be identified for example the increasing of HAZ in width (Gaussian vs. flat-top profile) by the dilemma of overcome temperature interval between 450 K and 800 K. Expanding spots are attractive for improvement of homogenization and increasing accuracy for ongoing measurments of ablation crater. The model results in figure 3 are generated with the following input data: puls length of 240 ns, puls energy of 1 mJ and spot diameters of 269 µm for 1.8 J/cm², 202 µm for 3.1 J/cm² and 168 µm for 4.5 J/cm².

A efficient selection of allowed fluences for fiber exposing can be done based on the threshold of sublimation of carbon fiber. Peak intensity of a valid beam profile may not be above these value. The diagramm at the top right illustrates resulting temperatures in the fiber-resin-interface. The corresponding diagramm at the botton shows the resulting pressure generated by pyrolysis. For validation of the model is, especially, the lowest graph with the fluence 1,8 J/cm² of interest. There, the averaged pressure at 54 kPa is below 100 kPa – the atmospheric pressure. This should lead to a unstable ablation process with in best case only partial ablation over laser spot.

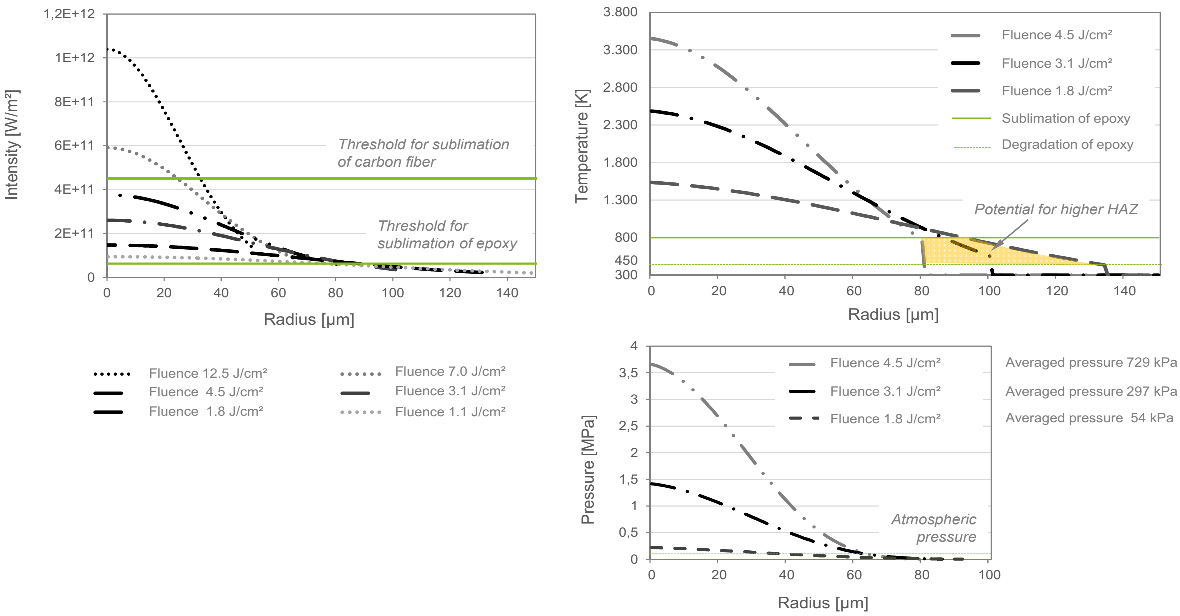


Fig. 3. Results based on the model illustrated in beam intensity, temperature at carbon disk and pressure within the fiber-near epoxy.

During puls absorption a great quantity of gases is generated in the interface between fibers and epoxy, which makes it possible to be heated up and creating considerable pore pressure. The level of pore pressure during indirectly-induced laser ablation in the spot center can be as high as 35 bar. These pressures have to be existing for a assumed pure lift-off by shearing of a 15µm thick chips.

5. Discussion and validation of model

Experiments for validation were done with a puls frequence of 25 kHz, beam speed of 20 m/s, puls length of 240 ns and puls energy of 1 mJ for provocation of single craters. By using an optical microscope VHX-1000 of Keyence, Neu-Isenburg, Germany the following aspects were measured: area of the thin top resin layer with turbidity (assumed as heat affected zone), area of strong carbon fiber degradation and area of fiber exposing. The results of the experiments are shown in figure 4 together with the results from the model.

The calculation results of ablation area and HAZ is close to results in experiments. Significant deviations between model and experiments only exists if average pressure is not over atmospheric pressure, see 1,8 J/cm². If this requirement is not fulfilled, no reproducible ablation is happen. Furthermore, in this case the largest amount of the spot area can be defined as HAZ. Based on this observation, the integration of the pressure calculation is important because purely thermal calculations does not indicate the process instability at low fluences.

An expanding of spot diameter means an expanding of crossing points between the temperature profile and the 800 K as well as the 450 K reference line, see figure 3. The exemplary pictures in figure 4 prove this finding. Based on these investigations for indirectly-induced ablation a near-top-flat intensity profile would be desirable. Calculations based on equation 8 about thermal conduction after the end of the pulse underline the low influence of expanding HAZ at low fluences. The reason is the relative low amount of accumulated energy in the carbon fiber top layer in the center of the spot at these low fluences.

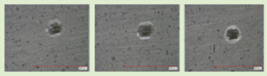
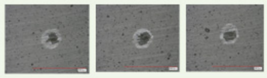
Averaged fluence	Pressure over 100 kPa	Radius of ablation (T>800K at end of pulse)	Radius of ablation	Radius of HAZ (T>450K after thermal conduction)	Radius of HAZ at experiment	Exemplary pictures
	Model	Model	Experiment	Model	Experiment	Experiment
4.5 J/cm²	yes	80 µm	89 µm	153 µm	160 µm	
3.1 J/cm²	yes	86 µm	75 µm	154 µm	133 µm	
1.8 J/cm²	no	91 µm	28 µm	154 µm	118 µm	

Fig. 4. Validation of model based on experiments for indirect induced ablation with short pulsed NIR laser.

The investigations disclose an accepted average fluence of 3 J/cm² up to 5 J/cm² with the laser system described above. Also for other laser sources a fluence of 3 J/cm² guarantee well processing, see Kreling et al. (2014). Additional, in this range of fluences high shear strengths could be reached between pretreated thermoset CFRP and subsequently overmolded thermoplastic compounds, Fischer et al. (2016) and Würtele et al. (2016).

6. Conclusion

Investigations on the indirectly-induced laser ablation lead to four requirements, whose fulfillment is essential for well selected process parameters using short-pulsed NIR laser sources. A fiber fair thermo-mechanical removal of thin top resin layer can be analytical described if:

- the intensity distribution of a laser pulse is located completely below the fiber damage threshold,
- the Gaussian beam zone with a temperature of more than 800 K at the top of the carbon fiber layer directly after the pulse duration is existing (potential fiber exposing area),
- the averaged pressure by pyrolysis in fiber-near region of resin is greater than the atmospheric pressure,
- the actual top resin layer thickness is smaller than the maximum liftable layer thickness.

The integration of the pressure calculation is important because purely thermal calculations does not reveal the process instability at low fluences. There are low deviations between experiments and model in the range of average fluences of 3 J/cm² up to 5 J/cm². Therewith the validity of the model is confirmed for this range of average fluences, although, potential for optimization is still existing. Nonetheless, the model can be used generating further findings for example regarding influence of puls duration.

Further validation of model will follow e.g. by time-resolved investigations. These investigations will identify further details about time sequence of mechanisms and details of mechanichal effects e.g. fracture mechanics or reduction of epoxy shear strength at higher temperatures. Also, the existence of plasma has to be investigated for indirectly-induced ablation.

The confined ablation process limits the possibilities in analyzing effects of HAZ in depth. Future investigations with the aim of research the HAZ effects in dimension of depth and influence of fiber destroying is only possible by the implementation of here used differential equations in a FEM simulation. This has to include the thermo-mechanical effects as well as the generation of a inhomogeneous model based on resin and real-scale carbon fibers, see Negarestani et al. (2009) or Wang et al. (2012).

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