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Comparison of UV- to M-IR laser for surface pre-treatment based on the ILSS-test

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Abstract

Carbon fiber-reinforced plastics (CFRP) offer a great potential for any lightweight construction. Besides there are also some challenges – especially the joining technology and the repair process. Adhesive bonding is a key factor to solve both challenges, namely and based on the common CFRP fabrication processes it requires a surface pre-treatment because of residues of release agents. For the removal of those contaminations and also for the ablation of whole fiber plies for the repair process, laser radiation is a suitable tool. However due to the thermal interaction of laser radiation and CFRP there is a tendency to cause delamination during laser treatment. This paper describes the approach of predicting the delamination tendency in bonded CFRP joints with a very simple mechanical test. It is shown that the results gained out of the ILSS-test are correlating with the amount of delamination in adhesively bonded and previously laser ablated CFRP-joints.

Keywords: CFRP; pre-treatent; ILSS; delamaniation

1. Motivation / State-of-the-Art

Carbon fiber-reinforced plastics (CFRP) are more and more used for structural components in aerospace and automotive applications. One major challenge for the application of CFRP-parts is the joining technology because on one hand conventional methods such as riveting weaken the structure and on the other hand the most promising technique (bonding) is limited due to the form-based production processes and the remaining contaminations such like release agents. These contaminations lower the adhesion between adhesive and adherent [1].

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So the structural and durable adhesive bonding of CFRP requires a surface pre-treatment to provide a reproducibly clean and slightly rough surface without any contaminations. State-of-the-Art methods in industrial applications to achieve these conditions are so called peel-plies which are laminated into the surface and removed before adhesive bonding or several mechanical treatments (e.g. grinding or grit blasting) [2,3]. While these methods show specific disadvantages the process speed is limited for all conventional applications. Laser radiation shows a high potential to remove contaminations and activate the surface prior to adhesive bonding, with a sufficient process speed [4,5].

However, depending of the applied laser source (especially with IR-laser sources) the process window to achieve a stable and secure pre-treatment is small. Due to the interaction of laser radiation, matrix resin and carbon fiber the ablation process is mostly thermally dominated [6]. With this the risk to cause delamination in the top fiber layers is high and even when the delamination cannot be detected with optical instruments (e.g. SEM) the fiber/resin adhesion can be weakened. Unfortunately these circumstances will not be visible until the pre-treated specimens are bonded and tested, see Fig. 1.

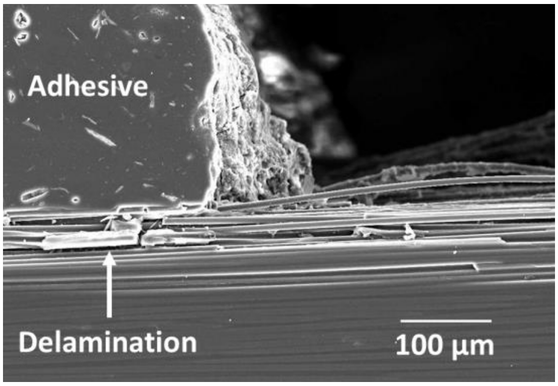

Fig. 1. SEM-picture of local delamination, which became visible during mechanical testing of a bonded specimen

This leads to a wide parameter study to achieve sufficient results for the pre-treatment. In addition, the challenge of process-caused delamination becomes more and more important since even non-replaceable parts (for example the fuselage structure of the Boeing 787) are made out of CFRP and repair technologies become necessary. As well conventional (grinding) as also innovative laser-based approaches [7] to produce the repair scarfs show a high potential to cause delaminations, when the ablation process is not specially cooled and sensitive designed [8].

One approach to reduce the test effort of bonded specimens and to easier qualify CFRP repair technologies by testing the delamination tendency with a simple mechanical test is presented in this paper.

The motivation is based on some observation during the laser ablation of CFRP with a CO_2-laser where significant delaminations correspond with very low values for the interlaminar shear strength (ILSS). Accordingly the main focus of this paper is to predict the delamination tendency of laser treated and adhesively bonded CFRP specimens by correlating this tendency with the test results of a relatively simple mechanical test method (ILSS test in order to DIN EN ISO 14130). Compared to the most common test method to qualify adhesive bonds (lap shear test e.g. DIN EN 1465) this test method reduce the effort through the elimination of the bonding process step and also through the reduction of pre-treated specimens surface by roughly one third.

2. Experiments

To get a solid base to prove the correlation of ILSS-test with the delamination tendency of laser treated specimens, different laser sources were used, to become more independent from the ablation mechanism, or prove their influence on the test results. Furthermore a numerical approach to link the laser parameter with the ablation process is applied.

2.1. Applied laser systems

During this study specimens were irradiated with laser radiation in the wavelengths of 355 nm, 1064 nm and 10600 nm. The laser source to work with UV-radiation was a Coherent ®AVIA™ Q-switched Solid State Laser emitting in the third harmonic (355 nm). The diode pump Nd:YAG rod is pumped by diode lasers and achieves a maximum average power of 23 W, depending on the repetition rate (common from 50 to 150 kHz). The repetition rate depending pulse duration was in the range of 30 ns for the presented study. The N-IR laser source was a SPI-G4 pulsed fiber laser (SP-070P-AHS-H-C-Y) emitting radiation with the wavelength of 1064 nm. With a maximum average output power of 70 W for repetition rates from 55 to 1000 kHz, while the pulse duration is in the range of around 50 ns. The third applied laser system was a Coherent ® Diamond E 400 CO_2-laser, operating with pulse frequencies of single shot to 200 kHz, due to the operating mode the pulse widths are significant higher (almost one scale with around 3.3 µs) than those of the other to laser systems. The maximum average output power is 400W.

All Systems where combined with scan systems to guide the laser spot over the surface, where several x- and y-overlapping laser spots achieve the desired treatment and the entire CFRP surface is covered. For the applied combinations of laser sources an optical instruments, the scan-speed could be up to 10000mm/s while the spot diameter vary from roughly 30 µm (UV), over 90µm (N-IR) to 180 µm (10600nm).

2.2. Laser parameter

Combining scan-speed, repetition rate, average power, pulse duration, x- and y-overlap and laser spot dimensions it is possible to calculate the applied energy per surface. There are different approaches to correlate the ablation with the numerical models to calculate the relevant ablation energy.

The model used in this study is published in [6]. This approach is based on the fact that the surface increments are hit different times by the laser pulse.

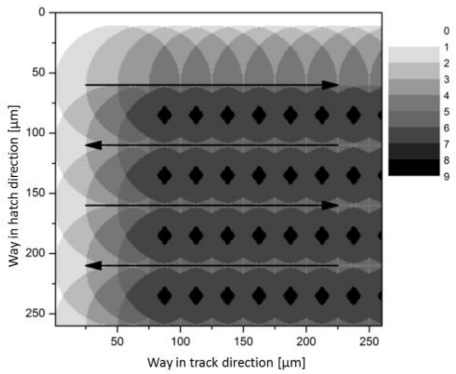

Fig. 2. Exemplary presentation for the distribution of increment hits (parameters: dspot = 100 µm; f = 40 kHz; v =1000 mm/s; dhatch = 50 µm) [6].

An exemplary representation of the mentioned effect is shown in Fig. 2. There the frequency of the increment hits and its distribution is shown for an exemplary laser treatment. Kreling also postulated an equation (1) to calculate the average number of laser pulses (round spot) per surface increment per hatch (n), which is depending on the repetition rate (f), the laser spot diameter (d_{spot}), the scan speed (v) and the hatch distance (d_{hatch}).

$$n(d_{spot}, d_{hatch}, v, f) \approx \frac{0.78 \cdot f \cdot d_{spot}^{\;2}}{v \cdot d_{hatch}} \qquad (1)$$

By multiplying the number of increment hits with the laser pulse fluence it is possible to calculate the surface energy per hatch. Due to the fact that the examinations for this study are based in the research topic of CFRP repair and the ablation with laser radiation, the surfaces were scanned multiple times to ablate a fiber ply of around 250 µm. Therefore the total accumulated energy is the product of the surface energy per hatch with the number of scanning cycles.

Table 1. An example of a table

Parameter	Pulse fluence [J/cm²]	Pulses / surface increment / hatch	Surface energy / hatch [J/cm²]	Cycles	Accumulated energy [J/cm²]
UV-L	28.06	4.16	116	16	1856
UV-M	29.75	5.82	173	11	1903
UV-H	42.38	5.82	242	10	2420
IR-L	6.88	12.29	84	10	840
IR-M	10.27	12.29	126	7	882
IR-H	15.72	12.29	193	6	1158
CO_2-H	14,03	25.65	218	13	2834

The parameters for this study are given in table 1. As mentioned before the first correlations were made with the specimens treated with the CO_2-laser, which has a high average output power. To achieve pulse fluencies in the same range, the other laser parameters were applied to prove the mentioned correlation for laser parameters, which are more corresponding to a laser surface pre-treatment. In addition, all examinations were performed with repetition rates of 70 kHz to avoid any influence of this parameter.

It is obvious that based on different ablation mechanisms the accumulated energies are different for the three laser sources but the internal comparison for each laser source show that the calculated accumulated energies are in the same range and therefore qualitative comparisons can be made. Some differences have to be accepted because the ablation of one ply can only be judged after one treatment cycle.

2.3. Materials and measurement

The CFRP plates for this study were made out of a typical aerospace prepreg which has a curing temperature of 175°C. They were made out of 8 plies in unidirectional orientation and finally cured in an autoclave with an applied pressure of 7 bar, while the vacuum was approx.-0.8 bar. To achieve typical surface contaminations the mold was coated with a polysiloxane-based release agent and no additional release film was applied. The specimens for the ILSS and the lap shear test were cut out of the manufactured CFRP-plates. A water cooled circular saw was used to cut the specimens and they were afterwards cleaned with isopropanol to remove any additional contaminations such like dust or residues of the cutting fluid. The joint specimens were bonded with a one component epoxy based adhesive and cured at 125°C for one hour.

The mechanical tests were performed with a universal tension/compression testing machine Instron 5567, Instron Deutschland GmbH. The testing rates were according to DIN EN 1465 5.0 mm/min for the testing of the bonded specimens (lap shear test) and in order to DIN EN ISO 14130 1.0 mm/min to characterize the apparent inter-laminar shear strength (ILSS).

3. Results and discussion

The aim of the experiments presented in this paper was to use a simple mechanical test method to predict the tendency of laser treated specimens for surface near delamination which can occur in adhesively bonded joints. Therefore two different test methods were used.

3.1. ILSS Test

The ILSS test is a variation of the classic three-point bending test in the fact that the specimen geometries are much smaller. So the standard geometry is 20 mm x 10 mm x 2 mm (length x width x height). In addition the support width is reduced to 10 mm. The interlaminar shear strength is calculated by equation 2.

$$ILSS = \tau = \frac{3}{4} \cdot \frac{F}{w \cdot h} \qquad (2)$$

So the ILSS is depending on the height (h) and width (w) in mm and maximum load (F) in Newton.

The mechanical test results show a strong dependency of the ILSS with the laser parameter. This effect is represented in Fig. 3.

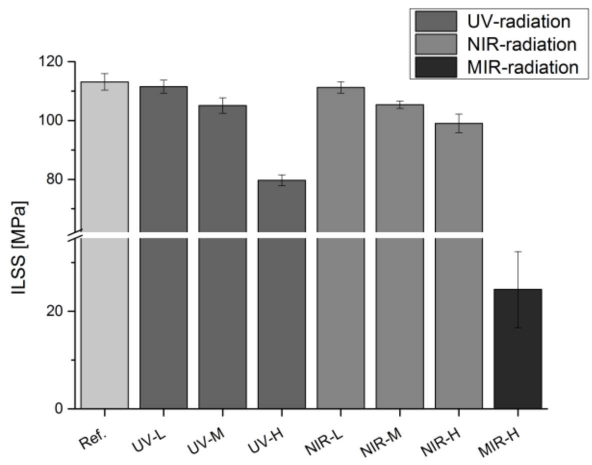

Fig. 3. The achieved ILSS of laser ablated CFRP specimens and reference strength.

The reference and untreated specimens show the highest ILSS of around 113 MPa (which is a typical value for the used prepreg system), while the ILSS of the laser ablated specimens decrease with increasing laser intensity. As mentioned before a big decrease is visible especially for the specimens, where the plies were ablated with high intensity and the CO_2-laser.

The effect of decreasing ILSS with increasing laser intensity can be found for both laser sources. Besides there are some differences in the gradient for the specific laser sources. The difference in the ILSS of specimens which are irradiated with UV radiation is almost 30 % between the highest ILSS of 111 MPa for the least intense treatment and around 80 MPa for the intense treated specimens. This big difference can not be detected for the other laser source. For the NIR-sources the least intense treated specimens achieve almost the strength of the reference and the intense treated specimens achieve strengths, which are around 10 % lower. The specimens which are treated with medium intensity are intermediary with around 5 % decreased ILSS. The initial approach with the CO_2-laser shows the lowest ILSS value. The ILSS value is remarkable because it is significant lower than those of the others. This can be correlated with the long pulse duration and therefore a significant thermal degradation of the matrix.

3.2. Lap Shear Test

While for the ILSS test the strengths are negatively correlating with the laser intensity, the lap shear strengths are fairly stable for all laser treated specimens, excluding the specimens which were ablated with the CO_2-laser – they show much lower lap shear strengths.

The effect of the laser ply ablation of CFRP prior to adhesive bonding was tested in order to DIN EN 1465, while on one specimen the top fiber ply was laser ablated and the other joining partner was mechanically abraded to achieve sufficient adhesion. The lap shear strength as the maximum achieved load divided by the joining area are shown in Fig. 4 for the differently treated specimens.

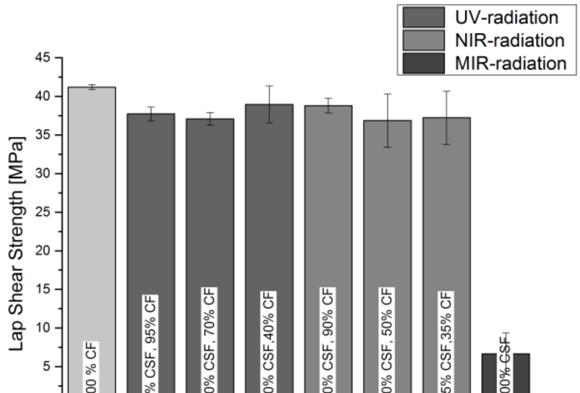

Fig. 4. Lap shear strengths of laser treated adhesive bonded CFRP specimens and the correlating failure patterns.

The reference specimens were abraded prior to adhesive bonding and achieve the highest lap shear strengths of around 40 MPa. The laser treated specimens reach sufficient lap shear strength between 35 and 40 MPa, expect those of the MIR treatment which achieve only around 7 MPa. For the main specimens (UV and NIR) of this study no significant dependency of laser intensity is identifiable (to ablate the top fiber ply) and the lap shear strengths are in the same range - all specimens fail inside one scatter band. The failure patterns show contrary. The reference specimens show complete cohesion failure (CF) inside the adhesive which is achievable for the optimal utilization of the adhesive properties. The laser treated specimens also show big parts of cohesion failure, but there are always some cohesive substrate failures (CSF) respectively delaminations which lower the strength. It can be noted that the amount of the cohesive substrate failure correlates with the laser intensity. Thus for all laser sources the laser energy during the ply ablation process has a big influence on the specimen's delamination tendency. Based on the ablation process and its high required laser energies (compared to pre-treatment) some percentage of CSF has to be accepted, but if the energy is too high, the substrate material is damaged because of degradation below the fiber layer. These circumstances are visualized in fig. 5 where the failure patterns of the NIR-treated specimens are shown.

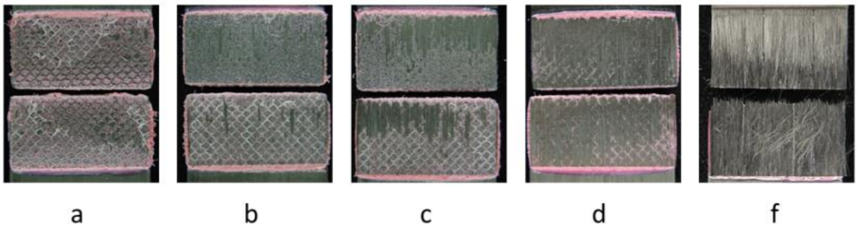

Fig. 5. Failure patterns of a representative reference- (a); NIR-L- (b); NIR-M- (c) and NIR-H–specimens (d). Additional the failure pattern of one of the MIR-specimens with 100% delamination (f).

The presented failure patterns in Fig. 5 can be correlated with the surface energy / hatch as parameter for the application of energy. The least amount of delamination (Fig. 5 b) was achieved with a surface energy per hatch of 84 J/cm². With increasing energy application the share of delamination was increased up to 65% in average for an treatment with a surface energy / hatch of 193 J/cm² (Fig. 5 c). A medium surface energy / hatch of 126 J/cm² also shows an intermediary amount of delamination (Fig. 5 d). The intense treated specimens in Fig. 5 f show a full delamination of the top plies, based on the ablation process of the CO_2-laser radiation. Figure 5 a) shows a complete cohesive failure of abraded and previously adhesively bonded specimens.

3.3. Correlation of both test methods

The mentioned correlation between the failure pattern and especially the amount of delamination respectively cohesive structure failure can also be linked with the ILSS. Here it can be stated that the tested ILSS correlates with the failure pattern and its amount of delamination. This effect is shown in Fig.6 where the ILSS, lap shear strength and the fracture patterns are shown for the representing specimens which were treated with NIR-radiation.

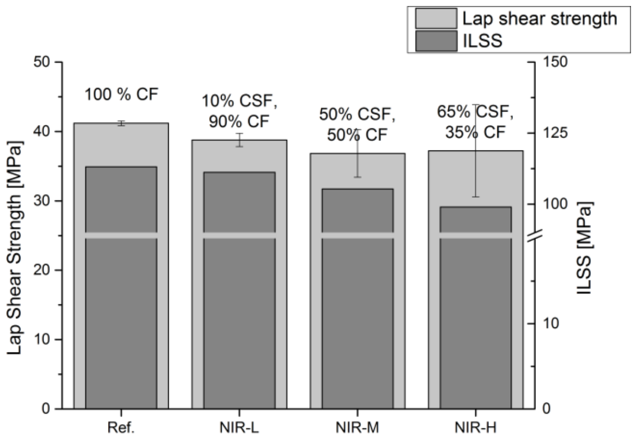

Fig. 6. ILSS, Lap shear strengths and correlating fracture patterns for NIR-laser treated specimens

The decreasing mechanical values in the ILSS and the corresponding increasing amount of delamination can be justified by the stress distribution inside the specimen. Besides the dominating shear stress there is also a bending stress because of the three point loaded specimen. While the delaminations are not highly relevant for the maximum achievable shear stress, they have a large influence on the bending behavior of any three point loaded specimen. Any fiber which is orientated longitudinal to the specimen length increases the bending stiffness, especially if they are in the top layers. In the case of visible delamination and a bad fiber/matrix adhesion which will result in delaminations, the top fiber layers are weakened and therefore the amount of bending strength in the measured mechanical value is decreased. This also justifies the small changes in the measured value, even if there are big differences in the fracture patterns of the lap shear specimens.

However based on this fact the ILSS-test can be used to predict the delamination tendency of laser treated specimens.

4. Conclusion

The presented examinations show the potential of a simple mechanical test method namely the ILSS test according to DIN EN ISO 14130 to predict the delamination tendency caused by a laser treatment prior to adhesive bonding of CFRP. The intensity of the laser treatment as well as the laser sources were varied to prove different effects, which can cause delamination.

Furthermore it is shown that the amount of cohesive substrate failure in CFRP-CFRP joints can be estimated by correlating the ILSS with the failure patterns of single lap shear specimens. However it was not possible to correlate the lap shear strength of the treated and later on adhesively bonded specimens with the measured ILSS.

Further investigations on the correlation between ILSS and delamination tendency are necessary. It has to be proven that the mentioned effects are also entirely contagious for the surface pre-treatment process, because the applied laser energies are lower than those used in this study for the laser-repair process. In addition the influence of specific laser parameter on the delamination tendency should be investigated. Especially the pulse width seems to have a big effect on the delamination tendency.

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