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Interface characterization of hybrid biocompatible fiber-metal laminates after laser-based surface treatment

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1. Introduction

Rising life expectancy and the significant aging of the population as a result of demographic change characterize modern society. It is not surprising that the number of repeated surgical interventions to replace implants also increases with a current implant life of around 20–25 years [1]. For example, the proportion of these so-called "revision surgeries" in the field of orthopedic hip implants increased by 4 to 26 % worldwide between 2010 and 2015, while forecasts suggest an increase of 137 % in the United States between 2005 and 2030 [2]. Consequently, the development of long-lasting and particularly biocompatible implants for use in orthopedic and reconstructive surgery is in the focus of science [3]. In addition to ceramic and polymeric materials, metals, and metal alloys and in particular titanium, show some benefits for medical applications, for example due to their excellent mechanical performance. However, when placed within the human body, metals are not completely inert. Furthermore, a deteriorated osseointegration ability can usually be observed with metals compared to ceramics and plastics [4].

In this context, biopolymers, such as polylactide or chitosan, have already been considered for use in temporary implants in recent years. The use of polylactide was particularly impressive in the form of biodegradable implants for a temporary fixation of bone fractures. On the other hand, the controlled release of pharmaceuticals incorporated into the polymer allows the medical regulation of the inflammation. Research has already shown, that the osseointegration capability of the implant could temporarily be improved using a bioactive polymer. Hence, service life of the implants could be increased [2,5,6].

Another promising material is (aliphatic) polyester. This polymer shows excellent binding properties to human tissue and is considered for scaffolds or thin coatings [7]. However, and especially due to the comparatively low mechanical strength, biopolymers are usually not suitable to be used in load-bearing orthopedic implants for a longer time-period [8]. A recent study reported on a trend to combine conventional metallic implants with bioactive materials to improve the implant performance within the human body as well as the implant life at the same time. Approaches include different processes such as injection molding, magnetron sputtering and the creation of an implant coating using laser-induced surface treatment [1].

Within the study at hand, another potential solution to realize long-term implants with an improved bio-performance, compared to conventional metal implants, is explored. The investigated combination of titanium and self-reinforced PLA is not only very promising to reduce metallic corrosion by means of a protective polylactide layer but also to provide sufficient mechanical performance of the polymeric component realized by fibrous reinforcement. Although the polymer might slightly

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degrade with time, the metal surface is only gradually exposed to harmful and corrosive environment within the human body. Hence, it might be possible to slow down the progress of corrosion and thus increase the life of the implant. To investigate the properties of titanium combined with self-reinforced polylactide, this work focuses on manufacturing of the fiber-metal laminates as a first approach considering a surface pre-treatment of the metallic component for increased bond strength. In this context, the suitability of laser-induced surface pre-treatment is investigated to identify suitable processing parameters.

Modification of surface properties of titanium has intensively been studied recently [9–11]. Vorobyev and Guo investigated possibilities to generate laser structures on flat titanium sheets with a femtosecond laser system at a frequency of 1 kHz and a central wavelength of 0.8 μm. By varying the spot diameter (in a range from 100 μm to 1200 μm) the repetition rate and energy, different homogeneous, mostly drop-shaped nanostructures down to a size of 20 nm could be generated. The roughness of the surface was in a range of about 1–15 μm [12].

In addition, influence of surface morphology on adhesive properties in fiber-metal laminates (titanium and fiber reinforced epoxy) were investigated by Kurtovic et al. [13,14]. Their work investigated the influence of a laser pre-treatment on surface morphology and adhesion properties of a titanium alloy (Ti-6Al-4 V) which was bonded to a fiber-reinforced epoxy (film adhesive system FM 73 M). The titanium sheets were pre-treated with a nano-second pulsed neodymium Yttrium orthovanadate laser (Nd: YVO4) with a wavelength of 1064 nm and then combined with the adhesive and fiber reinforced component using an autoclave. Wedge and roller peel tests were carried out to assess the influence of the laser-assisted surface pretreatment. The results showed that a laser pre-treatment contributed to a significant increase in the effective surface, whereby both chemical bonds and mechanical anchoring resulted within the interface. In addition, the evaporation of contaminants and the titanium dioxide layer which formed as a result of the pre-treatment led to improved adhesion properties [13,14].

Zinn et al. continued on the research results obtained by Kurtovic et al. and examined the development of a strong and aging-resistant connection between aluminum or a micro-alloyed fine-grain steel (1.0548) and a carbon fiber reinforced (CFRP) component based on a thermoset matrix material [15–17]. The fiber reinforced composite consisted of six alternating (0° / 90°) layers with a thickness of 0.3 mm each and an epoxy-based unidirectional carbon fiber fabric. The same laser system, as presented by Kurtovic [13,14], was considered in pulsed mode to pre-treat the metal sheets (thickness of 2 mm). Zinn also aimed to create a finely and uniformly structured surface with an open-pored surface morphology characterized by fine undercuts [15]. After the laser pre-treatment, the metal-CFRP composite was joined using a modified vacuum assisted resin transfer molding (VARTM) process and mechanically characterized by means of bending and shear edge tests [17]. It was possible to show that a laser pre-treatment of the metallic surface could increase the interface strength and deformability of the specimens. Depending on material, realized surface morphology and injection strategy interface strength increased up to 400 % compared to an untreated reference. All samples showed an interlaminar failure between the first and second CFRP layer. A highly open-pored surface enabled the adhesive to easily infiltrate the cavities to form chemical bonds and mechanical interlocking. The enlargement of the surface due to formation of open-pored and homogeneous structures were directly linked to the resulting adhesion properties in the boundary layer. In addition, to guarantee infiltration of the formed nanocavities, the laser-induced structure must be adapted to the viscosity of the adhesive system. Furthermore, as shown by Rechner et al. [18], Kurtovic et al. [13,14] and Zinn [15], the thickness of the oxide layers on aluminum or titanium increased significantly by a laser-induced pre-treatment and the contamination on surface was reduced. Hence, corrosion resistance and adhesion properties in the joining zone could be significantly improved [15,19].

Comparable results could also be achieved with thermoplastic-metal bonds [20–22]. Schulze et al. [23–25] for example investigated the adhesion and degradation behaviour at the interface between pre-treated titanium sheets and polyetheretherketone (PEEK) by means of tensile shear tests and mixed-mode bending tests. The effect of different mechanical and physical pre-treatment processes on initial and hydrothermal aging adhesion properties were analysed. To determine moisture resistance, the specimens were aged in 80 °C deionized water for up to 28 days. Ti-3Al-2.5V (grade 9) sheets with a thickness of 0.5 mm or 1.6 mm from Timet Germany GmbH were combined with a 100 μm thick semi-crystalline PEEK film Lite TK from Lipp Terler GmbH as well as 136 μm thick unidirectionally reinforced carbon fibre reinforced-PEEK tapes (CETEX®Thermo-Lite®TC1200) from TenCate and joined in a laboratory furnace (Nabertherm P330) or by means of a hot press (Voigt LaboPress P300S; 400 °C, 15 min, 15 bar). A laser pre-treatment using a pulsed Nd:YAG solid-state laser (Clean Laser CL20) from Clean Lasersysteme GmbH, Germany allowed homogeneously distributed surface structures on the micro level characterized by undercuts to be realized at a pulse frequency of 70 kHz and a scanning speed of 3.000 mm/s. In addition to the resulting surface structure on micro level, the formation of nanoporous oxide layers in the range of 20–100 nm could also be observed by means of transmission electron microscopy (TEM) images. PEEK residues in the undercuts and in the nanoporous oxide layer before and after hydrothermal aging indicated mechanical adhesion mechanisms. While the roughness and specific surface area, determined by Brunauer–Emmett–Teller (BET) measurement on the micro level, achieved by sandblasting (116.3 ± 1.79 %) and laser pre-treatment (125.6 ± 2.5 %) were in a comparable range, the laser pre-treatment additionally exhibited a significant surface increase on the nanoscale (781 %). The gain in surface roughness of the laser-pre-treated titanium strips could be noticed by the determined shear strengths. For example, while the tensile shear strengths of sandblasted (70 ± 4.3 MPa) and laser-pre-treated titanium sheets (77 ± 1.4 MPa) were in a comparable range for initially tested specimens, the shear strength of the laser-pre-treated titanium strips after 3 days of hydrothermal aging (62 ± 10.2 MPa) clearly exceeded the strength of sandblasted (31 ± 4.0 MPa) and of grinded specimens (18 ± 0.8 MPa). Due to the significantly increased surface reactivity, it was assumed that mechanical and chemical adhesion mechanisms overlap. Adhesion properties and aging in the joining zone could be significantly improved by laser pre-treatment.

A laser-based pre-treatment was also already proven to be a promising method to increase the bond strength of titanium hybrids [26]. The study at hand ties in with the aforementioned results and aims to investigate the effect of a laser-based surface pre-treatment of titanium sheets to be joined to self-reinforced PLA to form long-lasting and biocompatible fiber-metal laminates with appropriate mechanical performance. Starting from knowledge of hybrid fiber-metal laminates based on thermoset matrix systems, the questions arises if suitable surface structures to join these two materials are also suitable if a thermoplastic matrix material is considered.

2. Materials and manufacturing

Pure titanium (grade 1, (3.7025), Electronic Thinkgs) was considered for the manufacturing of the titanium-PLA laminate. The dimensions of the titanium sheets were 1 × 100 × 100 mm^3. A consolidated self-reinforced PLA organosheet (woven fabric design) with dimension 600 × 800 mm^2 in size and a thickness of ~1.3 mm was purchased from COMFIL®. The sheet consisted of 4 fabrics of 360 g/m^2 each and was manufactured at 158 °C.

The composite is made of PLA fibers with a lower melting temperature, which melt first when heated, and PLA fibers with a higher melting temperature, which maintain their integrity during processing and serve as reinforcing fibers in the consolidated composite. The sheet had a twill 2/2 weave design and the ingoing yarns consisted of 50/50 wt% PLA-PLA-160 (meaning a 50 wt% PLA filament and 50 wt% PLA matrix and yarn count of 160 tex). The twill 2/2 design implies that one

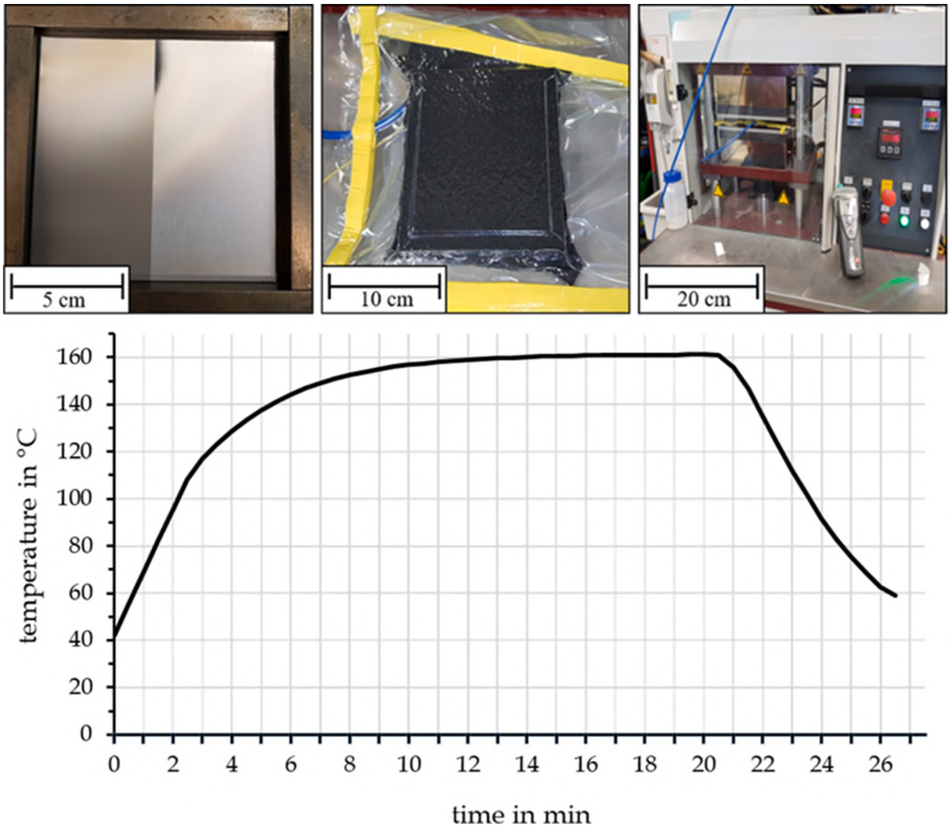

Fig. 1. Manufacturing of hybrid fiber-metal laminates (stacking, packing, and pressing) with representative temperature-time curve.

or more warp fibers alternatively weave over and under two or more weft fibers regularly and repeatedly, which results in a balanced fabric construction [27,28].

After a laser-induced pre-treatment of the titanium sheets (described in detail in section 3.1) the titanium and the polylactide sheets were degreased with isopropanol to guarantee comparable initial conditions. Then, the metallic and polymeric sheets were stacked and combined at elevated temperatures with a hot press (press type: Voigt, LaboPress P200S). A thermocouple allowed to monitor temperature evolution during manufacturing. To avoid leakage, the mold was covered with Polytetrafluoroethylene (PTFE) foil, packed vacuum-tight and connected to an external vacuum pump. Then, the mold was placed within the press at 161 °C and a nominal pressure of 10 bar. Real pressure fluctuated in the range of 9.5 to 11 bar. After reaching a laminate core temperature of approximately 157 °C, the mold remained closed for 10 min before starting a subsequent cooling process while pressure was maintained. At a core temperature of about 60° C, the hybridized polymer-metal laminate was removed from the press and set aside to cool down completely at room temperature. The individual steps to manufacture the fiber-metal laminates as well as a representative temperature–time curve are depicted in Fig. 1.

3. Methods

3.1. *laser-induced surface pre-treatment*

A pulsed ytterbium fiber laser (type: MOPA, YDFLP series from JPT Opto-Electronics Co, Ltd.) was considered to generate a laser-induced surface structure of the titanium sheets. To guarantee uniform structuring, the laser beam was guided by an integrated two-mirror system. Before starting the laser pre-treatment, the surfaces were degreased with isopropanol to ensure comparable initial conditions. A first parameter study in combination with investigation by means of scanning-electron microscopy (SEM) to qualitatively assess the resulting microstructure of the surface, aimed to define limits of suitable laser parameters in combination with titanium. This preliminary study pointed out, that the suitable laser power range was between 10 % and 100 %. In addition, scan track spacing of 30 μm matching a fixed spot size of 40 μm (due to a focal length of 160 mm) were suitable to enable the focal edge areas to overlap and thus guarantee a homogeneous structuring of the metal sheet over the entire surface. The respective area was structured with a linear fill pattern at an angle of 45° to the edges of the sheet. Scanning speed was set to 800 mm/s according to preceding investigations by Zinn [15] and Kurtovic et al. [13,14]. In subsequent parameter studies (study 1 – study 3), frequency and power of the laser have been modified according to Table 1. In each of the three studies the laser power was increased starting from 10 % of the maximum power up to 100 % by steps of 10 %. Similarly, the laser frequency was increased in equal steps in each interval up to the specified maximum frequency. Pre-treated sheets with structures based on different parameter-sets are shown in Fig. 2.

Table 1
Parameter-sets of parameter studies.

	Power in %	Frequency in kHz	Velocity of scan in mm/s	Jumping velocity in mm/s	Spot size in μm	Distance of lines in μm
Study 1	10–100	10–100	800	10.000	40	30
Study 2	10–100	110–500	800	10.000	40	30
Study 3	10–100	1–10	800	10.000	40	30

3.2. *Specimen preparation and shear-edge testing*

The specimens for mechanical characterization, with dimensions of 15 × 15 × 2.3 mm^3, were extracted with a circular saw (type: DIADISC 5200,

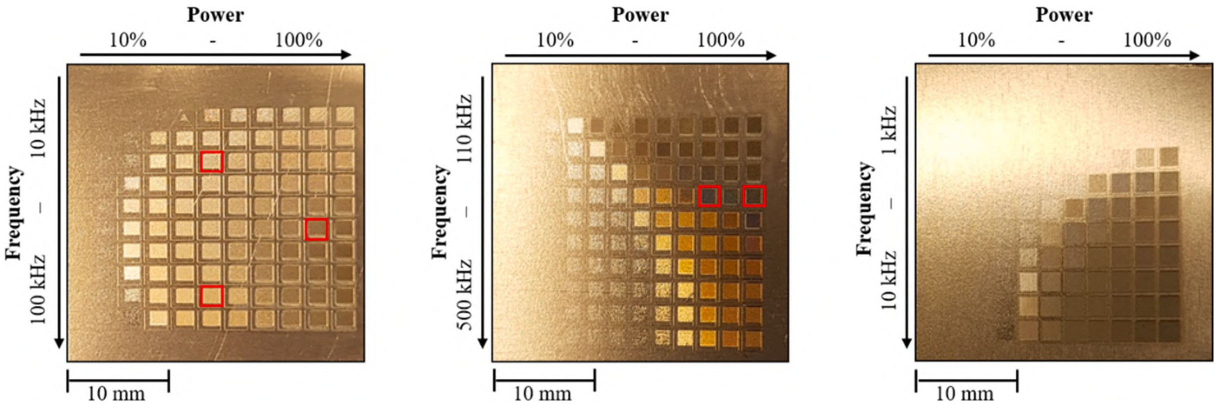

Fig. 2. Laser induced pre-treated titanium sheets, showing sections of different parameter-sets. Marked sections indicate parameter-sets which were considered to manufacture the hybrid fiber-metal laminates.

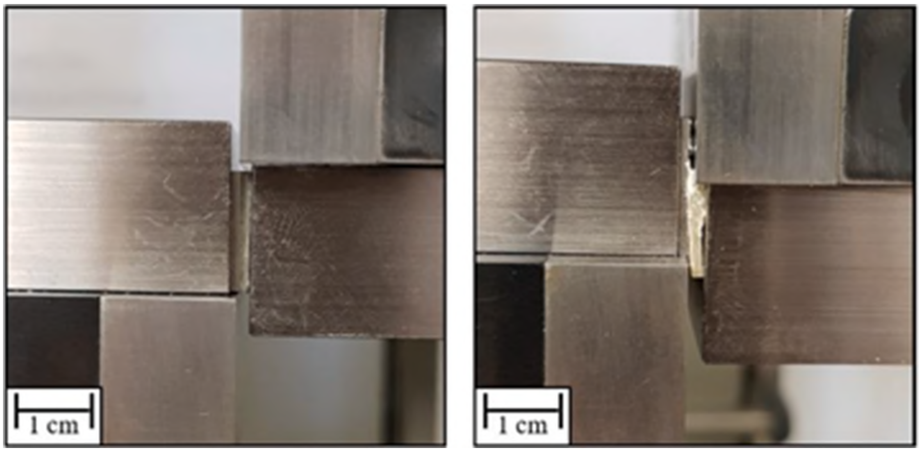

Fig. 3. Shear edge testing: initial condition (left) and sheared specimen (right).

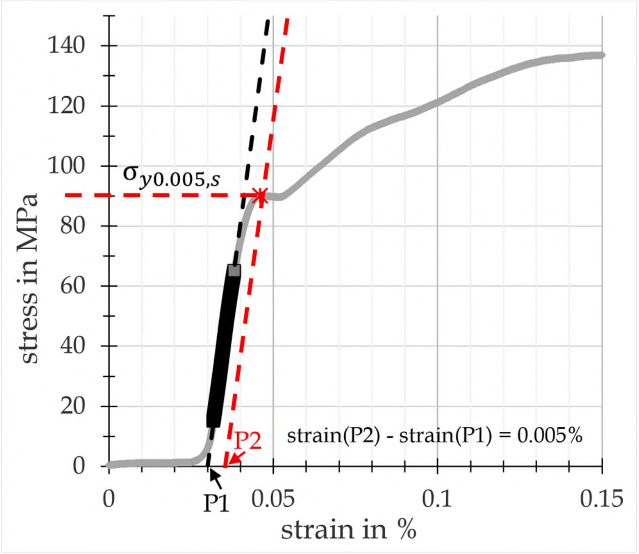

Fig. 4. Procedure to define shear modulus and yield shear strength ($\sigma_{y0.005,s}$) of hybrid fiber-metal laminates with shear edge testing.

Mutronic GmbH & Co KG 2020). The hybrid laminate, based on a non-pretreated titanium sheet, delaminated after being removed from the mold. Hence, it was not possible to derive interface properties of hybrid fiber-metal laminates consisting of non-pre-treated titanium sheets.

To mechanically characterize interface properties, shear edge testing (cf. Fig. 3) was carried out according to [29]. The specimens were clamped into the testing device which was installed in a Zwick universal testing machine. After positioning, specimens were preloaded with a force of 15 N. Then, shear loading was induced by opposite movement of the two blocks on the metallic and polymeric side of the specimen with a speed of 1 mm/min. Force and deflection were recorded during loading to subsequently determine the mechanical behavior. In detail, shear failure strength ($\sigma_{f,s}$), shear yield strength ($\sigma_{y0.005,s}$) and shear modulus were determined. Shear failure strength resulted from maximum recorded force divided by the surface of the specimen. To determine the shear modulus a linear regression was carried out in a stress range from 15 MPa to 65 MPa and the slope was calculated. The intersection of the regression line with x-axis was referred to as origin to determine a 0.005 % yield shear strength ($\sigma_{y0.005,s}$) in accordance to the definition of a 0.2 % yield strength (cf. Fig. 4) [30]. For the characteristics determined, arithmetic mean value and standard deviation were considered.

4. Results and discussion

4.1. Laser-induced pre-treatment

The laser induced pre-treatment power and pulse frequency were varied over a wide range in order to generate different surface

Table 2
Definition of selected parameter-sets to pre-treat the titanium sheets.

Name of specimen	Power in %	Frequency in kHz
P50_F30	50	30
P50_F90	50	90
P80_F250	80	250
P90_F60	90	60
P100_F250	100	250
Untreated	–	–

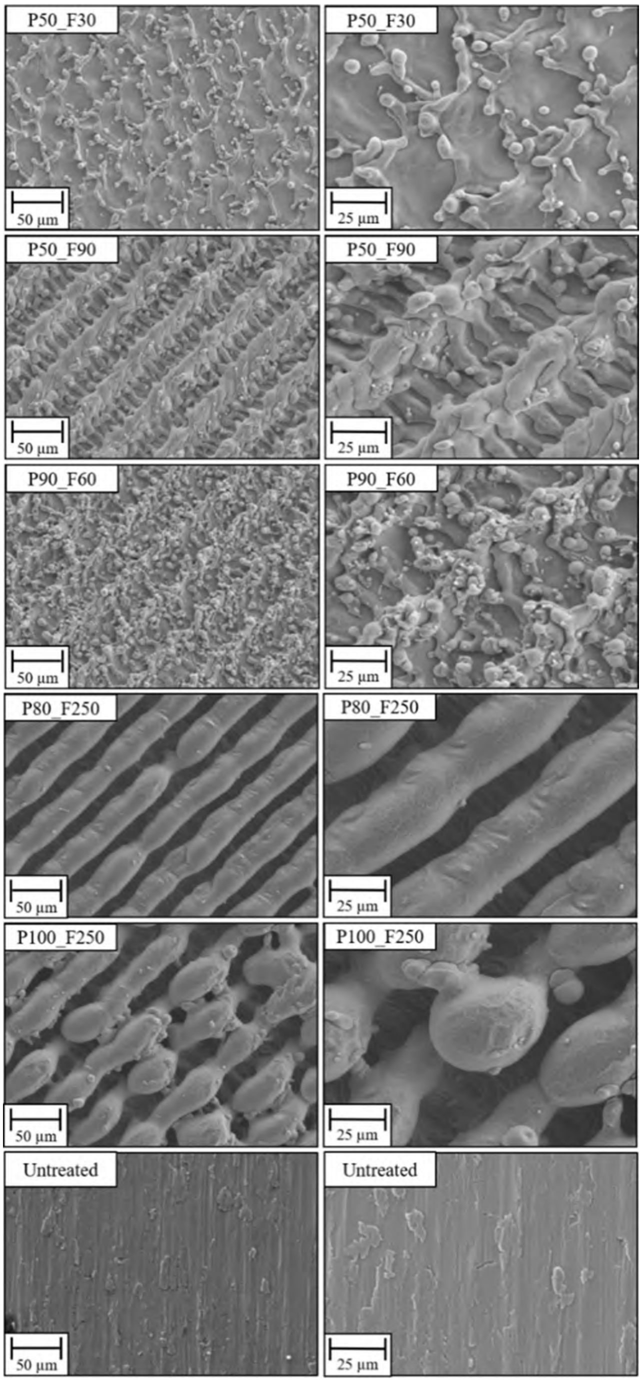

Fig. 5. Scanning electron microscopy images of pre-treated surfaces resulting from a laser pre-treatment with different values of power (P) and frequency (F). In detail, power (P) was equal to 50 %, 80 %, 90 % or 100 % of maximum power and frequency (F) was set to 30 kHz, 60 kHz, 90 kHz, or 250 kHz.

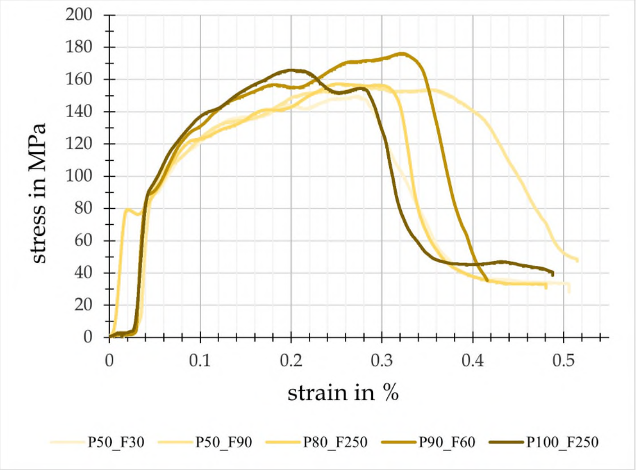

Fig. 6. Representative stress–strain curves.

morphologies and to investigate their influence on adhesion. The aim of the structuring was a uniform, fine surface morphology characterized by undercuts and, if possible, open porosity. An examination based on scanning electron microscopy highlighted five different parameter sets (listed in Table 2), resulting from study 1 – study 3 (cf. Fig. 2) that showed satisfying surface morphologies of the titanium sheets. Representative images, showing the different microstructures, are depicted in Fig. 5.

In the following the letter P indicates the power in percentage of the maximum power and F the frequency in kHz.

Comparing surface topology resulting from a laser pre-treatment with parameter sets P50_F30 and P50_F90, the increasing overlap of the individual spots within a scan track because of the increased pulse frequency is clearly visible. Due to the accumulation of melted and re-solidified substrate, the edges are more pronounced. A similar trend is observed when comparing the parameter sets P80_F250 and P100_F250. While a slightly lower laser power of 80 % with the same pulse frequency led to a uniform linear structure, an increase of power led to a more irregular surface structure with a significantly increased proportion of undercuts. A simultaneous increase of power and frequency (parameter set P90_F60) led to a surface structure with a higher degree of overlap of the individual focal points within a scan track and, as a result of the increased amount of energy, to more pronounced pulse edges compared to the parameter set P50_F30.

It holds generally true that the higher the power, the more pronounced the material removal and the more structured the surface topography. For all five selected parameter-sets a homogeneous structuring of the titanium surfaces in the micrometer range and thus an increase in the effective surface could be achieved. The surfaces were characterized by a high proportion of undercuts. Furthermore, all surface structures (especially P80_F250 and P100_F250) showed an open pored structure, which might have a positive effect on infiltration of the melted thermoplastic polymer.

4.2. Interface properties

Shear edge testing aimed to determine the effect of a distinct surface morphology on the adhesive properties of the hybrid fiber-metal laminate. During mechanical loading, shear forces were introduced into the interface and force and displacement of the movable edge was recorded. As depicted in Fig. 6 the initial part of the obtained stress-strain curve of the respective parameter sets are comparable and show a flat start-up behavior, (possibly resulting from an alignment of the specimen within the fixture) before curves started to rise steeply in a linear way. Following this linear increase, a kinking of different severity can be observed. From this point, scrapping of the polymeric component

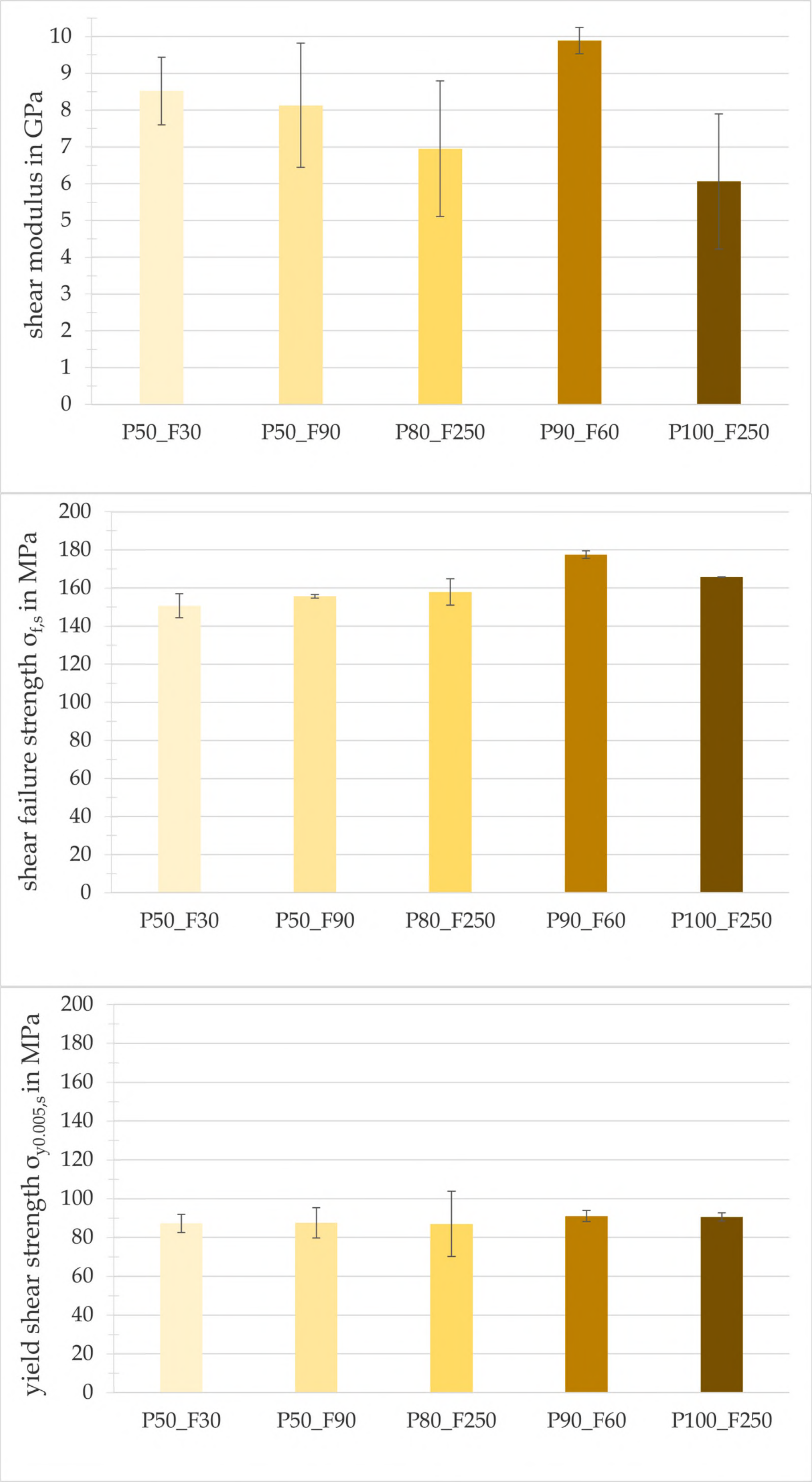

Fig. 7. Resulting mechanical properties. Depicted are arithmetic mean value and standard deviation.

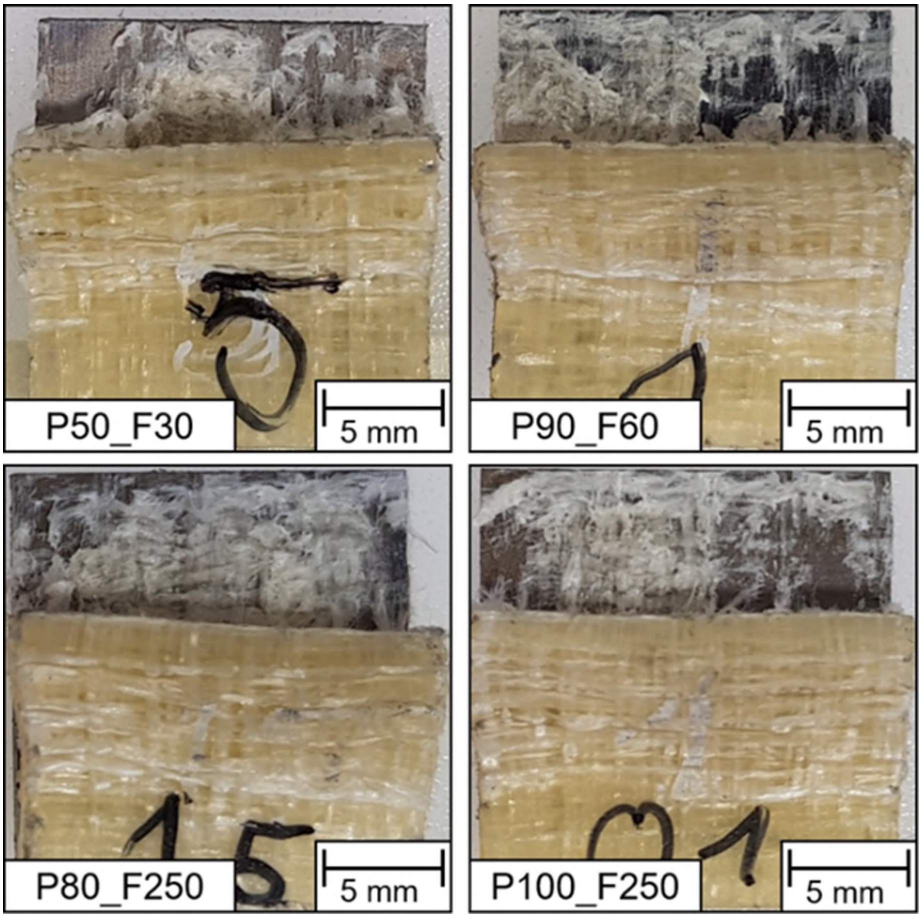

Fig. 8. Representative specimens after shear edge testing.

started. Force increased up to a maximum value before it suddenly dropped and final failure occurred.

When comparing the arithmetic mean values of the yield shear strength, no significant difference was observed. Similarly, shear failure strength only showed a slight trend to higher values for maximum power, hence for parameter sets P100_F250 and P90_F60 (cf. Fig. 7). Compared to the untreated titanium sheet, all laser structures led to significantly improved adhesion properties and an increase in bond strength (measured as shear failure strength). As expected, due to the orthotropic reinforcing architecture of the polymeric component, no difference could be determined between longitudinal and transverse specimens.

It is important to highlight that failure was characterized by a local shearing-off of the polymeric component, instead of pure interface failure between the polymeric and metallic part (cf. Fig. 8).

Due to the suspected material failure, interlaminar shear strength could not be determined. A similar behavior was already evident for different thermoplastic fiber composite materials used by Baumgärtner [31].

4.3. Post-mortem observation of interface

Following the destructive testing, one specimen of each parameter-set was examined by scanning electron microscopy (cf. Fig. 9) that enabled an initial assessment of failure mechanisms. For all investigated specimens, polymer residues on the metallic component were visible. The fiber-like structure of these polymeric parts indicated that the PLA (high melting) fibers did not melt during the joining process and the selected process temperature is suitable to not to destroy the fibrous reinforcement. In the case of untreated titanium, hardly any polymer residues can be seen.

The polylactide residues on the laser-pretreated metal sheets allow the conclusion, with a comparative view of the untreated titanium surface, that laser structuring of the metal surface leads to a qualitative improvement in the adhesion properties in the boundary layer. The comparison of the laser pre-treated surfaces before and after the joining process are shown in Fig. 10. Whereas laser structuring with parameter-set P50_F30 did not show a significant difference before and after joining, all other observed specimens show a distinct change in the initially generated structures. The surface structuring of parameter P50_F90 appears compressed, while with the laser structures P80_F250 and P100_F250 the metallic surface appears comparatively smooth except for the remaining clearly recognizable lines.

5. Summary and conclusion

The hybrid fiber-metal laminate, consisting of titanium and self-reinforced PLA, which was investigated in the study at hand, might represent a possible solution for extending the current implant life cycle. Surface pre-treatment aimed to achieve a significantly enlarged surface to enhance interaction of the two materials within the interface. In the context of extensive parameter studies, suitable laser settings, such as scan speed, scan track spacing and jump speed, can now be defined for the specific material combination titanium and self-reinforced PLA. Exposed to shear loading, failure of the hybrid laminate occurred mostly at the interface. Post-mortem observations by means of scanning electron microscopy also showed a cohesive material failure within the polymeric component.

To summarize main findings one can conclude:

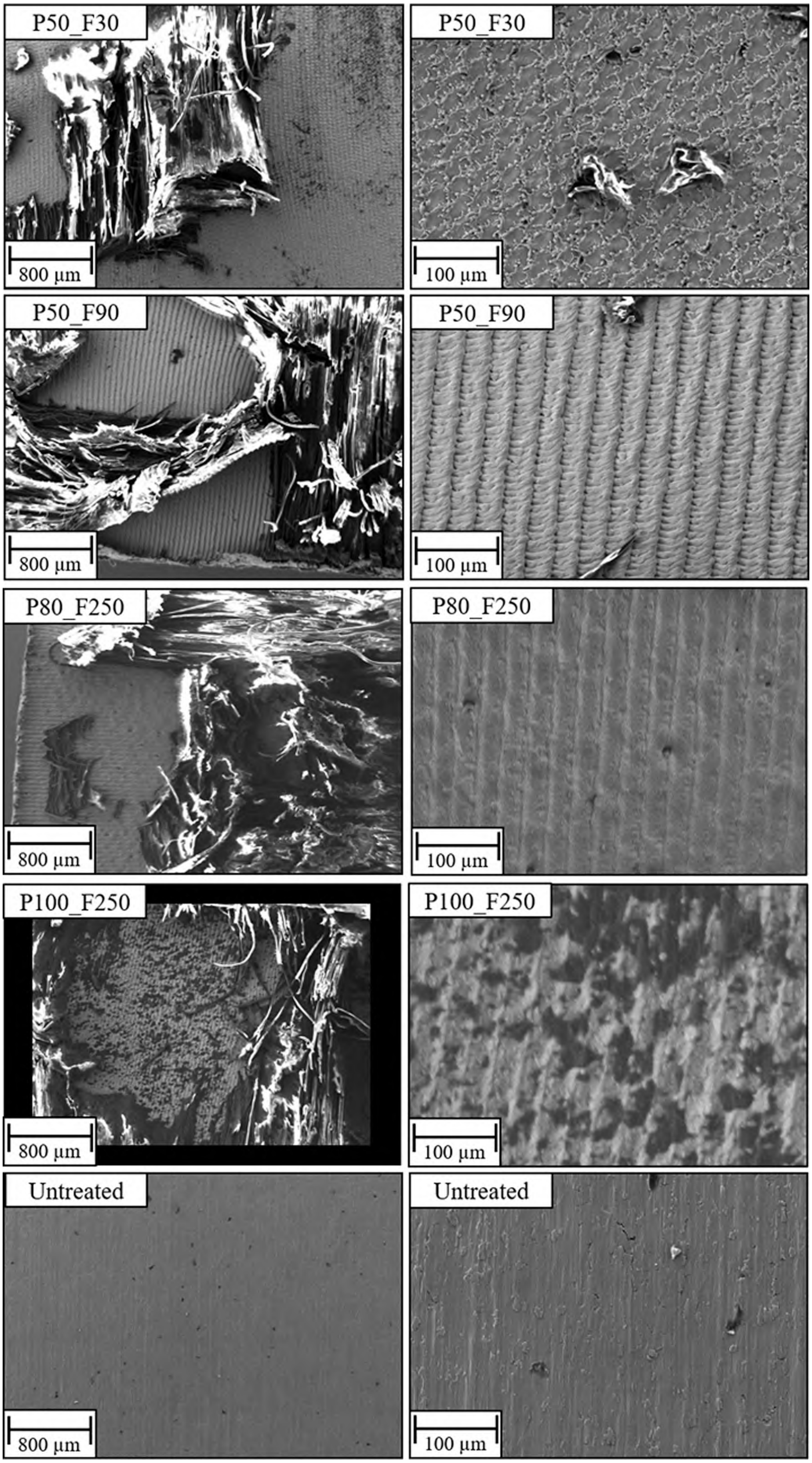

Fig. 9. Scanning electron microscopy images of selected specimens after testing, extracted from sheets manufactured with different parameter-sets, hence, different values of power (P) and frequency (F).

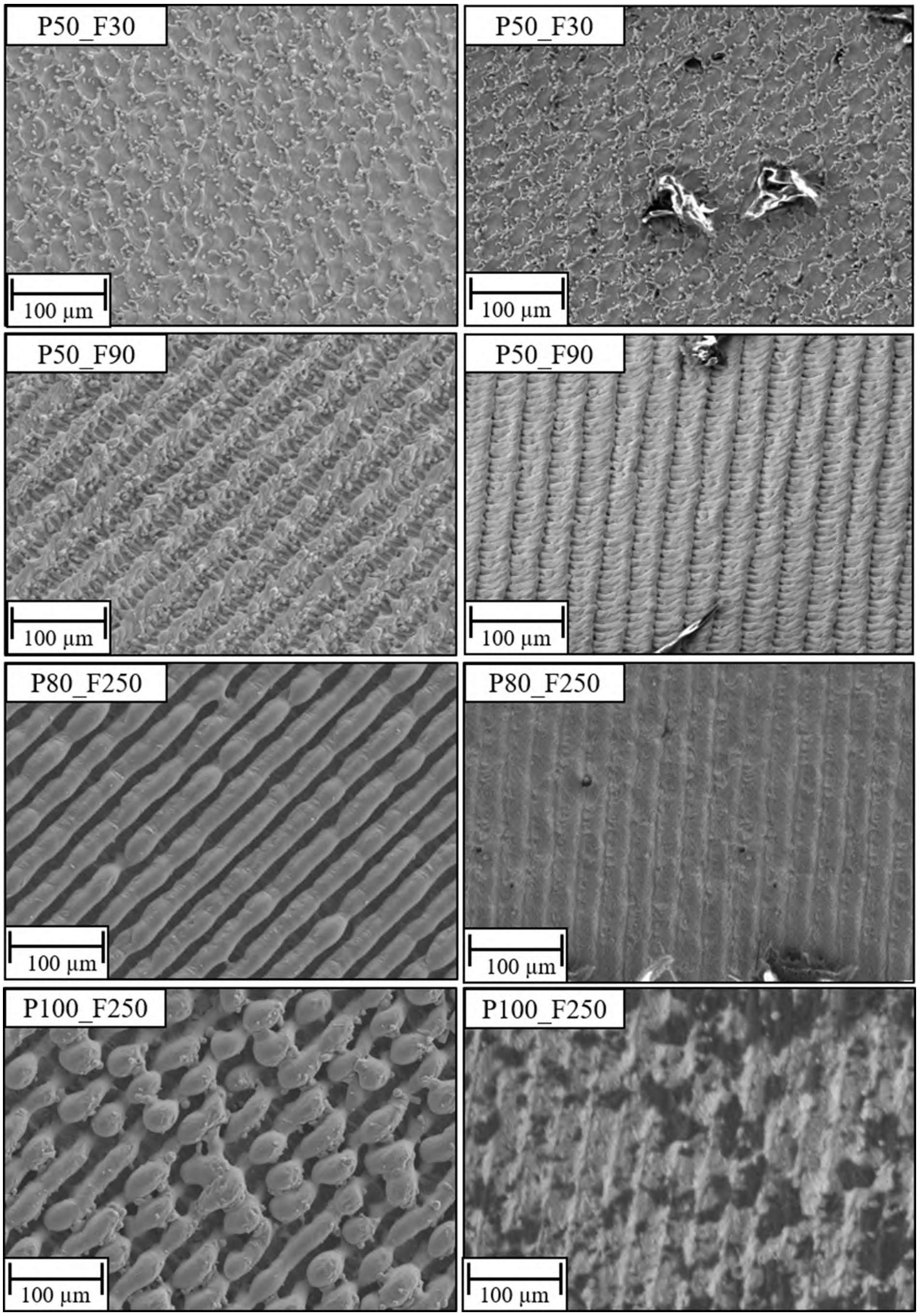

Fig. 10. Comparison of the laser structuring before (left) and after shear edge testing (right).

- Without a laser pre-treatment titanium and self-reinforced-PLA could not be joined by means of hot pressing.
- In comparison to the unstructured reference sheet, a significant increase in adhesion was achieved for all specimens, regardless of the laser structures produced. A laser-induced surface structuring can therefore be classified as a suitable pre-treatment process for the selected material combination to achieve increased bond strengths.
- Regardless of the surface topographies all examined specimens showed a similar failure behavior.
- Failure occurred partly within the polymeric component (cohesive failure) and partly at the interface (adhesive failure).
- To obtain the highest mechanical interface properties within the shown parameter range the titanium sheet must be pre-treated with a laser power of 90 % and a frequency of 60 kHz. However, to propose the best parameter set for the use of medical implants inside the human body further investigations (e.g., on corrosion, long-term biocompatibility, long-term mechanical properties) must be done.

Data Availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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