Suitability of roughness parameters for the interlaminar strength prediction of mechanically interlocked polymer-metal-interfaces

E Saborowski1, P Steinert2, A Dittes1, T Lindner1, A Schubert2 and T Lampke1

1 Materials and Surface Engineering Group, Faculty of Mechanical Engineering, Chemnitz University of Technology, Erfenschlager Straße 73, D-09125 Chemnitz, Germany
2 Micromanufacturing Technology Group, Faculty of Mechanical Engineering, Chemnitz University of Technology, Reichenhainer Straße 70, D-09126 Chemnitz, Germany

*e-mail: erik.saborowski@mb.tu-chemnitz.de

Abstract. The interlaminar strength of mechanically interlocked polymer-metal-interfaces strongly depends on the metal surface topography. Hence, this contribution assesses standardized surface roughness parameters as well as fractal dimension for interlaminar strength prediction of EN AW-6082/polyamide 6 polymer-metal-hybrids. Seven different metal surface topographies generated by mechanical blasting, alkaline etching, thermal spraying as well as laser structuring are investigated. Interlaminar strength testing is carried out with regard to tensile and shear strength using butt-bonded hollow cylinder specimens. The surface roughness profiles are recorded with a tactile stylus profiler. The fractal dimension of the metal surface is derived from cross-sectional images. The measured interlaminar strength values show good correlation to the fractal dimension, moderate correlation to the root mean square slope of the surface roughness profile and no correlation to surface roughness height parameters.

1. Introduction

Thermoplastic polymer-metal-hybrids (PMH) offer great potential for automotive mass production due to their quick processability. If reinforcing fibers are added to the polymer, they also provide high specific strength and stiffness. Thermal contact joining using micro-scale mechanical interlocking is a common approach for connecting both dissimilar material groups. Thereby, the thermoplastic polymer is used as hot melt adhesive as it infiltrates and interlocks the roughness features of the metal surface. For joining PMH, metal and polymer are put together under pressure, while the contact area between both materials is heated until the polymer melts. The necessary thermal energy can e.g. be generated by direct laser heating [1], indirect laser heating [2, 3, 4], induction heating [5, 6], ultrasonic oscillations [7, 8], friction [9] or heat conduction [10, 11, 12]. The achievable adhesion depends mainly on the surface topography of the metallic joining partner. Therefore, the precise micro-structuring of surfaces offers promising possibilities for increasing the interlaminar strength of dissimilar material joints. Possible surface structuring techniques are mechanical blasting [2, 13, 12, 14], laser structuring [3, 8, 15, 16, 17, 18], thermal spraying [6, 16, 19, 20] and etching [12, 21].

According to the state of the art, strength-enhancing properties of the surface structure are the structure density and the aspect ratio of the roughness features [15, 17, 22, 23] as well as the presence of undercuts [21, 22] and sub-structures [18]. Consequently, an appropriate measure for predicting the interlaminar strength needs to consider these specific properties. Standardized roughness height
parameters like $Ra$, $Rz$ and $Rc$ do not consider any of these aspects and therefore, no considerable accordance to the interlaminar strength was found in various studies [2, 13, 21]. On the other hand, the root mean square slope $R∆q$ showed good accordance to the shear strength of steel-bone cement joints [23] since this measure is directly connected to structure density and aspect ratio of the roughness features. However, undercuts cannot be recorded using tactile surface roughness measurement. Another approach for roughness description is the fractal dimension. The surface topography can e.g. be derived from cross-sectional images, whereby undercuts and sub-structures are taken into consideration as far as the image resolution allows. A study investigating the adhesion of thermally sprayed ceramic coatings on blasted steel substrates showed good correlation between fractal dimension and bonding strength [24].

This contribution is the continuation of our previous work [12, 16] on the relation between surface topography and interlaminar strength of mechanically interlocked PMH. The aim of this work is the assessment of the suitability of standardized surface roughness parameters and comparison to the fractal dimension [16] for predicting the interlaminar strength. To this end, seven different surface topographies are applied to EN AW-6082 aluminium alloy specimens. The structured surfaces are joined to polyamide 6 (PA6) by heat conduction hot pressing. Interlaminar strength values are determined using butt-bonded hollow cylinder specimens, since this method allows interlaminar shear and tensile strength testing with one single specimen geometry [11, 25].

2. Experimental and materials

2.1. Materials

For all experiments, hollow cylinders made up from EN AW-6082 aluminum alloy were used as metal part. Hollow cylinders made up from extruded Ultramid® B3 PA6 (BASF, Ludwigshafen, Germany) were used as polymer part. Table 1 shows the relevant material data.

|                | EN AW-6082 | PA6 (humid condition) |
|----------------|------------|-----------------------|
| Density [kg/m³] | 2.7        | 1.14                  |
| Young’s modulus [MPa] | 70000 | 1800                  |
| Poisson’s ratio [-] | 0.34 | -                     |
| Yield strength [MPa]  | 260        | 60                    |
| Ultimate strength [MPa] | 310  | -                     |
| Elongation to failure [%] | 7    | 200                   |
| Melting temperature [K] | 933   | 496                   |
| Thermal expansion coefficient $[10^{-6}/K]$ | 23.4 | 70                    |
| Thermal conductance $[W/(m·K)]$ | 170 - 220 | 0.23                |
| Specific heat $[J/(kg·K)]$ | 898    | 1700                  |

2.2. Surface pretreatment

In total, seven different surface topographies were applied to the aluminum surface using grit blasting (F16), grit blasting and additional etching (F16-NaOH), thermal spraying (NiAl5) and laser structuring (deterministically distributed pin structures: L-8, L-11, L-14; stochastically distributed pin structures: L-Pin). Figure 1 shows scanning electron (SE) microscopy topography images as well as cross-sectional back scattering detector (BSD) images of the generated surfaces.
Figure 1. SE images of topography (left) and cross-sectional BSD images (right).
F16 surfaces were generated with WFA F16 corundum (particle size distribution: 1000–1400 µm) using a blasting pressure of 0.2 MPa, a blasting distance of 100 mm, a blasting angle of 75° and a blasting time of 10 s. In addition to the F16 specimens, alkaline etching was carried out on grit blasted surfaces to modify the coarse roughness features with much finer sub-structures. For generating the F16-NaOH specimens, a blasting pressure of 0.3 MPa was applied to increase the vertical extent of the structures. Alkaline etching was conducted with 2 % NaOH solution (343 K / 5 min). Afterwards, the sheets were submerged into 50 % nitric acid solution (ambient temperature / 2 min) to clean the surface.

For generation of the NiAl5 surfaces, a nickel-aluminum 95 wt%-5 wt% coating was deposited on the aluminum. Prior to the coating process, WFA F24 corundum (particle size distribution: 600–850 µm) was applied with a blasting distance of 100 mm, a blasting angle of 75°, a blasting pressure of 0.2 MPa and a treatment time of 10 s to enhance the adhesion of the coating. The coating was applied by electric wire arc spraying, using a VISU ARC 350 arc spray system with a Schub 5 spraying gun (Co. Oerlikon Metco, Wohlen, Switzerland). Thereby, a current of 150 A, a voltage of 30 V, a spraying distance of 130 mm, an air pressure of 3.5 bar, a feed speed of 0.6 m/s and a row spacing of 5 mm were used.

The laser structuring processes were conducted by a Nd:YVO4 nanosecond laser system (specifications: wavelength = 532 nm, pulse duration = 10 ns, max. mean power = 13 W, focus diameter = 15 µm; manufacturer: Co. Spectra Physics®, Santa Clara, USA). Multiple, line-wise scanning of the specimen’s surface area with overlapping single pulses was performed for the realization of the surface micro structures. For generating the L-Pin structures, a defined energy input above the material-specific threshold laser fluence was applied to the aluminum surface. Therefore, a laser intensity of 3–6 J/cm² was realized by using a defocused laser spot measuring about 55 µm in diameter. Applying the aforementioned conditions, stochastically arranged micro structures (cone-like protrusions - CLP) were generated on the specimen surface. L-8, L-11 and L-14 surfaces were processed with a focused laser beam, a pulse frequency of 200 kHz and a number of 8, 11 and 14 scans. The resulting material ablations on the aluminum surface measured approximately 14 µm in diameter. By setting the distance between the material ablations slightly below their diameter (13 µm), the material in between was perforated. The resulting surface structure was determined by pin-like profile elements distributed in a grid. Thereby, the number of scans adjusted the height of the pins.

2.3. Specimen geometry and strength testing

Figure 2 shows the geometry of the hollow cylinder specimens. The outer diameter \( d_o = 28 \) mm and the inner diameter \( d_i = 23 \) mm. The length of the metal cylinder \( l_m = 40 \) mm and the length of the polymer cylinder \( l_p = 60 \) mm. For the metal cylinder, the free testing length \( l_{m,f} = 20 \) mm. For the polymer cylinder, the free testing length \( l_{p,f} = 30 \) mm. In case of the laser-structured specimens, \( l_{p,f} \) was reduced to 10 mm since the maximum twist angle of the testing machine (90°) would have been exceeded otherwise. The specimens were manufactured using a heat conduction hot-pressing process. Figure 3 illustrates the hot-pressing tool. During the hot pressing process an isobaric pressure (0.2 MPa) was applied normal to the interfacial zone of the specimen. The copper block was heated until the polymer in the interface melted. Afterwards, the copper block was actively air-cooled. After the joining process, the specimens were reworked by turning to ensure equal dimensions as well as proper centricity for testing. [11, 12] provide a detailed description of the manufacturing process.

The strength testing was conducted with a PTT 250 K1 hydraulic testing machine (Co. Carl Schenck AG, Darmstadt, Germany). The applied testing speeds were determined from the desired strain- and shear-rate of the PA6, whereby the much stiffer aluminum was considered rigid. The shear rate was set to 0.002 1/s (laser-structured: 5 °/min, others: 15 °/min) and the strain rate was set to 0.0002 1/s (laser-structured: 0.12 mm/min, others: 0.36 mm/min). The interlaminar shear strength \( \sigma_{max} \) and the interlaminar tensile strength \( \tau_{max} \) were calculated from maximum torque \( T_{max} \) and maximum force \( F_{max} \) before fracture, respectively (Equations (1, 2)). [11] provides a detailed description of the specimen testing procedure.
\[ \tau_{\text{max}} = \frac{16 T_{\text{max}} d_o}{\pi (d_o^4 - d_i^4)} \quad \sigma_{\text{max}} = \frac{4 F_{\text{max}}}{\pi (d_o^2 - d_i^2)} \]  \hspace{1cm} (1, 2)

Figure 2. Hollow cylinder specimen [16].

Figure 3. Heat conduction hot pressing tool [16].

2.4. Determination of surface parameters
The surface roughness measurements were carried out with a Hommel-Etamic® T8000 stylus profiler (Co. Jenoptik AG, Jena, Germany). Figure 4 illustrates how the height signal \( y \) is recorded as function of distance \( x \). Five measurements with a sampling length of \( l_s = 2.5 \) mm and an evaluation length of \( l_e = 5 l_s \) were recorded and evaluated for each surface structuring process. A stylus tip with 2 \( \mu \)m radius and 60° tip angle was used for capturing a high amount of profile details.

\[ R_z = \frac{1}{5} \sum_{i=1}^{5} R_{z_i} \] \hspace{1cm} (3)

\[ R_a = \frac{1}{l_e} \int_{0}^{l_e} |y(x)| \, dx \] \hspace{1cm} (4)

The root mean square slope \( R_{\Delta q} \) (Equation (5)) indicates the root mean square of the local gradient \( dy/dx \) along \( l_e \).
\[ R\Delta q = \sqrt{\frac{1}{\ln n} \int_0^L \left( \frac{dy(x)}{dx} \right)^2 \, dx} \]  

(5)

Figure 5 illustrates how the fractal dimension was derived from the interface line using the box-counting algorithm. Fifteen cross-sectional images were recorded per treatment using a LEO1455VP SE microscope (Co. Carl Zeiss Microscopy GmbH, Jena, Germany). The images were further processed and binarized with GIMP 2 image manipulation software. Embedding resin and metal surface were separated from each other by the fuzzy select function that allows area selection based on colour similarity. Afterwards, a MATLAB® algorithm was used for the identification of the interface line. Finally, the box-counting algorithm was applied. Thereby, the image is divided in squares of size \( r \). For each \( r_i \), a certain number of squares \( n_i \), containing at least one black pixel, exists. Beginning from the smallest possible box size of \( r_{\text{min}} = 1 \) pixel, the box size is increased stepwise by a power of 2 till the maximum box size \( r_{\text{max}} \), covering the complete image, is reached. [16] provides a detailed description of the fractal dimension determination approach in connection with PMH-interfaces.

![Figure 5](image)

**Figure 5.** Process of image analysis for determination of fractal dimension (a) Cross-section (b) Binary image (c) Interface line (d) Box-counting algorithm.

The local dimension between individual box sizes \( d_i \) is calculated by Equation (6).

\[ d_i = \log_2 n_{i+1} - \log_2 n_i \]  

(6)

The fractal dimension of the interface line \( D \) equals the mean value of all local dimensions (Equation (7)).

\[ D = \frac{1}{k} \sum_{i=1}^{k} d_i \quad \text{with} \quad k = \log_2 r_{\text{max}} \]  

(7)

3. Results and discussion

Table 2 summarizes the test results as well as surface characteristics for all investigated surface pretreatments (\( SD \) denotes the standard deviation). Thereby, the shear strength varied in a broad range from 11.5 MPa to 32.5 MPa and the tensile strength from 2.02 MPa to 26.0 MPa. F16 Treatment offered the second lowest interlaminar strength in both load directions. Additional etching (F16-NaOH treatment) even lowered the strength; presumably due to a leveling of sharp edges and undercuts at the roughness features. NiAl5 treatment provided higher strength than the blasting treatment, but lower strength than every laser treatment. For the L-8, L-11 and L-14 treatments, the strength increased with the number of scans. The highest strength was achieved with the L-Pin treatment.
Table 2. Roughness parameters, fractal dimension and interlaminar strength of the investigated surface structuring methods, mean values ± 1 SD.

| Treatment | $Ra$ / $\mu$m | $Rz$ / $\mu$m | $Raq$ | $D$ | $\tau_{max}$ / MPa | $\sigma_{max}$ / MPa |
|-----------|---------------|---------------|-------|-----|---------------------|---------------------|
| F16-NaOH  | 20.5 ± 2.4    | 124 ± 9       | 0.773 ± 0.024 | 1.093 ± 0.032 | 11.5 ± 2.0         | 2.02 ± 0.55         |
| F16       | 22.0 ± 1.9    | 131 ± 8       | 0.806 ± 0.020 | 1.102 ± 0.016 | 14.2 ± 4.4         | 4.16 ± 0.69         |
| NiAl5     | 11.6 ± 0.7    | 80.4 ± 4.2    | 0.828 ± 0.031 | 1.123 ± 0.016 | 17.0 ± 0.1         | 5.81 ± 0.03         |
| L-8       | 4.95 ± 0.15   | 32.2 ± 0.4    | 0.919 ± 0.115 | 1.153 ± 0.012 | 21.9 ± 0.8         | 7.92 ± 0.24         |
| L-11      | 4.92 ± 0.21   | 31.0 ± 1.1    | 0.970 ± 0.121 | 1.195 ± 0.013 | 26.7 ± 0.8         | 10.2 ± 1.3          |
| L-14      | 4.38 ± 0.21   | 27.5 ± 2.2    | 0.929 ± 0.164 | 1.231 ± 0.011 | 29.6 ± 0.2         | 17.3 ± 0.8          |
| L-Pin     | 5.59 ± 0.17   | 45.6 ± 1.2    | 0.971 ± 0.025 | 1.233 ± 0.015 | 32.5 ± 1.4         | 26.0 ± 1.6          |

Figure 6 shows the interlaminar shear and tensile strength in relation to the evaluated roughness parameters. A linear fit was carried out in each case to obtain an additional indication how suitable the respective parameter is to predict the interlaminar strength. For both roughness height parameters $Rz$ and $Ra$, the strength decreases with increasing values for two reasons. Firstly, the surface roughness measurement underestimates the profile height difference especially for the high strength providing laser treated specimens, since the stylus tip is not able to penetrate the tight profile valleys completely. Despite the real profile height of L-8 and L-14 differs around factor two (see Figure 1), the measured surface roughness values ($Ra$, $Rz$ and $Raq$) are almost identical. Secondly, $Ra$ and $Rz$ do not consider undercuts, sub-structures as well as structure density and aspect ratio of the profile elements. The coarse and widely spaced F16 / F16-NaOH roughness features have high $Ra$ and $Rz$, but provide only low strengths. Hence, the coefficients of determination $R^2$ are low in all cases. Thus, as already stated in state-of-the-art literature, surface roughness height parameters are not suitable for strength prediction.

The root mean square slope $Raq$ shows much higher accordance ($R^2 = 0.9$ for $\tau_{max}$, $R^2 = 0.62$ for $\sigma_{max}$) since it is a direct measure for structure density as well as aspect ratio. Both $\tau_{max}$ and $\sigma_{max}$ increase with $Raq$. Especially for F16, F16-NaOH and NiAl5 treated specimens, a monotonic increase can be observed. However, the loss of detail when recording the surface roughness profile also leads to an underestimation of $Raq$ and thus, the tightly spaced L-8, L-11, L-14 and L-Pin structures show considerable deviation from the fitted function. Moreover, the anisotropic orientation of the L-8, L-11 and L-14 profile elements leads to high standard deviations in $Raq$.

Fractal dimension $D$ shows the highest accordance of all evaluated parameters ($R^2 = 0.98$ for $\tau_{max}$, $R^2 = 0.83$ for $\sigma_{max}$). In contrast to the tactile surface roughness measurement, the determination of the interface line from cross-sectional images captures all details as far as the image resolution allows. Thus, undercuts, sub-structures and structure density are considered when calculating $D$. 
Figure 6. Interlaminar shear- and tensile strength in relation to $Ra$, $Rz$, $R\Delta q$ and $D$, mean values $\pm 1$ SD.
4. Conclusions
In the present work, the suitability of tactile measured surface roughness parameters and fractal dimension for surface topography assessment and corresponding interlaminar strength prediction of mechanically interlocked polymer-metal-hybrids has been investigated. The surface topographies were generated by corundum blasting, combined corundum blasting and etching, thermal spraying and laser structuring. For the determination of the interlaminar strength in shear as well as tensile direction, butt-bonded hollow cylinder specimens were used. The joining of specimens was carried out by heat conduction hot pressing.

The results indicate that the interlaminar strength can be predicted from the surface topography by the help of appropriate roughness parameters. Assuming that the adhesion resulting from mechanical interlocking mainly depends on structure density and the aspect ratio of the roughness features as well as the presence of undercuts and sub-structures, surface roughness height parameters alone are not able to give reliable predictions. This is confirmed by the poor correlation between $Ra$ as well as $Rz$ and the interlaminar strength. The surface roughness slope parameter $R_{\Delta q}$ can lead to good accordance since it is directly connected to structure density and aspect ratio of the roughness features. However, the roughness measurement device must be able to record the actual roughness profile adequately. The tactile measurement of tightly spaced, laser-structured topographies yielded a massive underestimation of the actual $R_{\Delta q}$ since the stylus tip was not able to penetrate the profile valleys completely.

Fractal dimension, though, resulted in the best correlation to the interlaminar strength for all investigated specimens. Since the interface line is derived from cross-sectional images, undercuts and small-scale roughness features can be considered. Therefore, fractal dimension is most likely to be recommended for interlaminar strength prediction of mechanically interlocked polymer-metal-hybrids.

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