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Microbond data analysis: comparative assessment of different approaches to determine IFSS

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Abstract. Studies on fiber surface modifications typically utilize micromechanical characterization methods, such as microbond tests, to assess the adhesion of a resin on a fiber. The fiber surface is thought to cause the majority of the deviation seen in the data since the surface treatment methods rarely produce uniform surfaces on all treated fibers. A realistic number of droplets must be tested to achieve reliable and statistically significant data of the fiber-matrix interface. Therefore, it is crucial to understand how the interfacial shear strength (IFSS) behaves as a function of the dataset size. An extensive microbond data analysis was conducted for a dataset consisting of approximately 1600 epoxy droplets on a commercial glass fiber (based on 53 filaments). The IFSS was calculated using three approaches to evaluate how each IFSS value behaves as a function of increasing dataset size. It was evident that the droplet's embedded area has a significant effect on the apparent IFSS, which is troublesome if the deposited resin volume and thus the droplet size distribution cannot be controlled well during sample manufacturing. According to the results, linear regression based IFSS should be preferred but apparent IFSS may be used for comparative analysis. However, the linear regression method where a single fit is made to the whole dataset requires a high number of filaments to provide reliable results.

1. Introduction

Studies on fiber surface modifications typically utilize micromechanical characterization methods, such as microbond tests, to assess the adhesion of a resin on a fiber. The methods try to evaluate the load transfer capacity of the interface of a single filament that is separated from a roving. The microbond method provides interfacial shear strength (IFSS) by measuring the embedded area A_{emb} and the maximum force F_{max} required to debond a droplet from a single filament. Conventionally, the load transfer capacity of the interface is evaluated by calculating apparent IFSS (IFSS_{app}), denoted also as τ_{app} , using Equation 1:

$$\tau_{app} = \frac{F_{max}}{A_{emb}} \quad (1)$$

One challenge the microbond community faces is the lack of standards for sample preparation, testing, and data analysis. The lack of standardization has led to diverse practices which reduce the repeatability of results and, in some cases, produce misleading conclusions. One of these data analysis practices involves forcing a linear regression fit of $F_{max} - A_{emb}$ to pass through the origin. The IFSS_{app} is then calculated from the shifted data with Equation 1. The challenges are



further complicated by the many microbond devices that have been developed independently by different research groups. Configuration changes have been made to universal testing machines, thermo-mechanical analyzers (TMA), dynamic-mechanical analyzers, and micromechanical testers to enable microbond measurements [1][2][3][4]. Specific devices designed to measure droplets on fibers are also available [5][6]. While different microbond device configurations exist, it is possible to obtain comparable IFSS values across setups. Minty et al. [7] compared a modified universal testing machine and a TMA microbond device resulting in 39 MPa and 40 MPa for a glass fiber-epoxy system. Instead of using $IFSS_{app}$ in the comparison, they used the slope of the linear regression fits of F_{max} vs. A_{emb} data as the IFSS value, referred to herein as $IFSS_{slope}$. Furthermore, consistent results were obtained for glass fiber-polypropylene system with the same microbond devices, with $IFSS_{slope}$ values of 7.97 MPa and 7.67 MPa [3].

Various interfacial testing methods have been compared in a round robin program where four laboratories did microbond testing for untreated and surface treated carbon fibers with an epoxy resin [8]. Most of the laboratories tested between 22-52 droplets with varying embedded areas and compared the $IFSS_{app}$ values. To this day, this is a typical number of tested droplets. Each laboratory was able to observe the difference between untreated and treated fibers and the scatter within the results of each laboratory were in acceptable range. However, between the laboratories the $IFSS_{app}$ values exhibited significant scatter, as indicated by the coefficients of variation, namely 29% for untreated fibers and 28% for treated fibers. In the end, differences in the droplet loading were concluded to cause the scatter, not the data reduction scheme. Also, the small number of tested filaments was thought to cause some scatter to the results since few carbon fiber filaments were selected from a 12000-filament tow.

A dataset of 1600 microbond measurements on a commercial glass fiber-epoxy system has been analysed to explore the error sources in the microbond test of a specific microbond test device [5]. Most of the variation seen in the IFSS results was concluded to originate from the differences in the surface properties between individual filaments, not from the measurement itself, as is typically thought. Rarely do the surface treatments make uniform surfaces to all treated fibers. Laurikainen et al. used $IFSS_{app}$ and two $IFSS_{slope}$ approaches to assess the load transfer capacity. The same microbond data will be further analyzed here. In contrast the prior publication using the same dataset, the present study investigates how different approaches to calculate IFSS respond to variations in the dataset size. This helps to address questions such as what constitutes a sufficient dataset size and which method of determining IFSS should be preferred. As a conclusion, a set of recommendations is given for microbond data analysis to improve the reliability and reproducibility of the results.

2. Methodology

An epoxy resin system Araldite 5052 LY and curing agent Aradur 5052 CH were used to make droplets on commercial glass fibers. The constituents were mixed in a weight ratio of 100:38. The droplets were deposited HiPer-tex W 2020 (Ahlstrom-Munksjö Glassfibre Oy) by pulling a filament through a resin well. The glass fibers were taken from a length of 135 cm of the roving. The resin droplets were cured at room temperature for 24 h, followed by a further post-curing at 80 °C for 8 h. In total, 53 filaments and approximately 1600 droplets were tested with a high-throughput microbond device (FIBRObotics Oy). Approximately 30 droplets were tested from each filament. Details regarding the device specifications and associated data can be found in [5]. Tested embedded lengths of the droplets varied in the range of 50-180 μm resulting in an embedded area range of 0.002 - 0.010 mm^2 (filament diameter 18 μm).

Three different approaches were used to calculate the IFSS from the F_{max} required to debond a droplet and the measured A_{emb} . Each approach is illustrated in Figure 1.

- 1) IFSS_{app}: an average of the apparent IFSS (τ_{app}) values calculated for each droplet separately,
- 2) IFSS_{slope, fiber}: an average of the linear regression slopes fitted to each filament data separately, and
- 3) IFSS_{slope, all}: a linear regression slope fitted to the whole data set from different filaments.

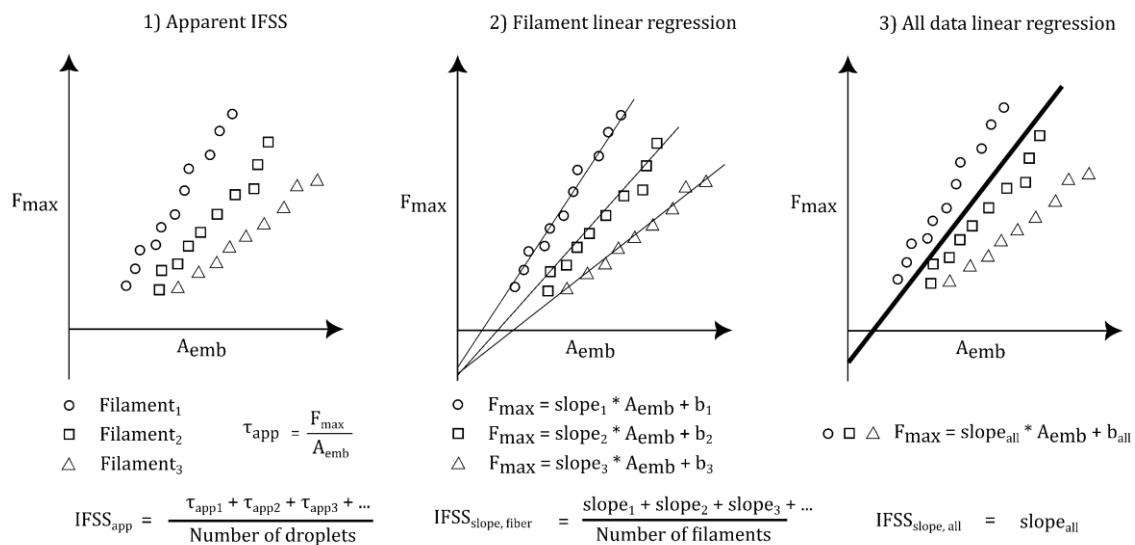


Figure 1. Three approaches used to calculate IFSS 1) the average of the conventional apparent IFSS, 2) the average of the slopes from linear regression for each filament, and 3) the single slope from linear regression fitted into the whole data set.

3. Results and discussion

3.1 Impact of dataset size on IFSS values

Fiber bundles consist of thousands of individual filaments which can have varying surface properties as well as non-constant fiber diameters. Evaluating how each IFSS approach behaves as the dataset size increases is essential for understanding how data sampling affects the results. For this purpose, spaghetti plots were created for each IFSS approach. To generate the plots, random subsets of the dataset were incrementally constructed by adding data points from each filament at a time in a randomized order as is seen in Figure 2.a. This process was repeated 100 times, each with a different random ordering to get the spaghetti plot in Figure 2.b.

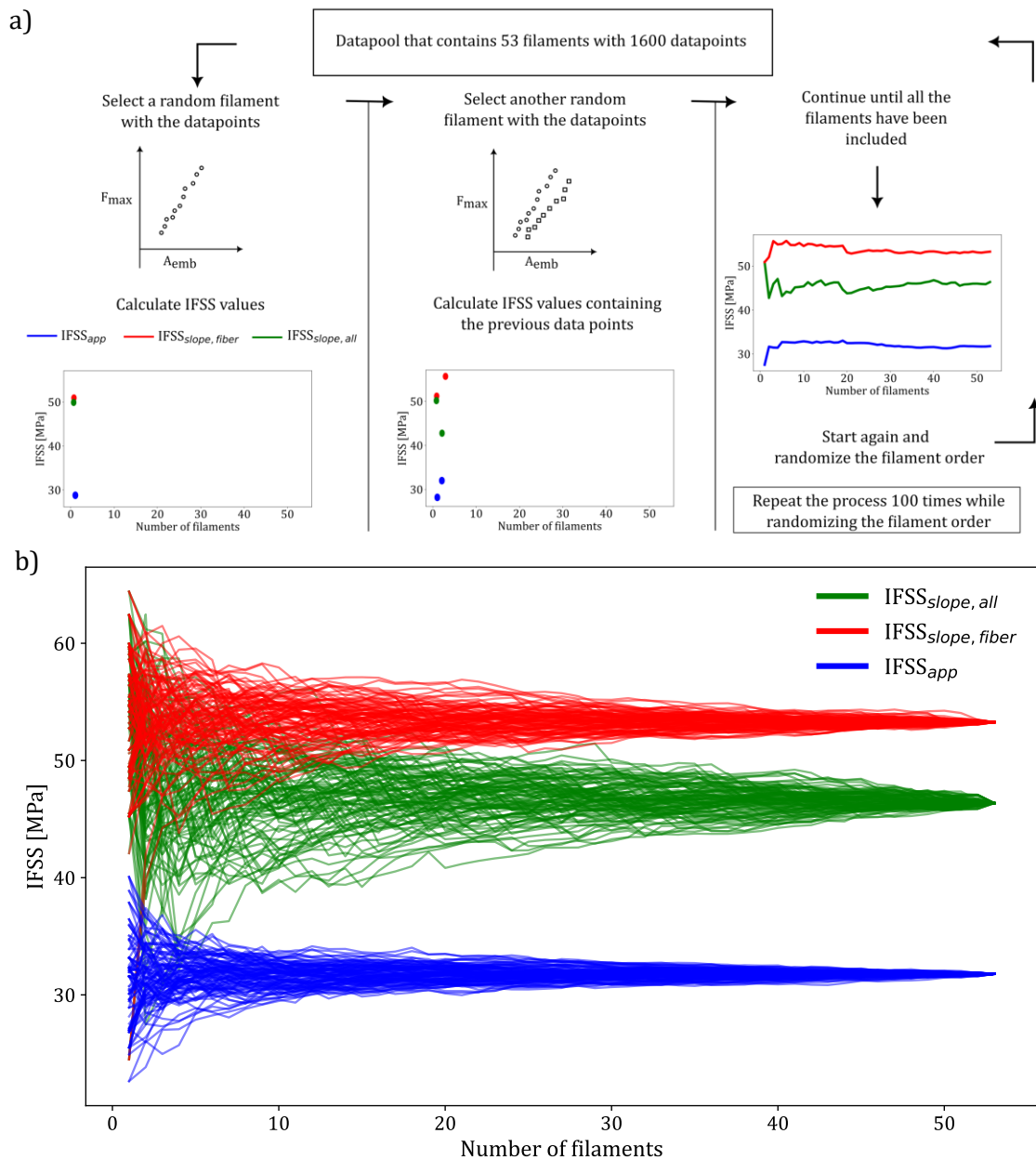


Figure 2. a) A flowchart illustrating the analysis steps used to construct the spaghetti plot, b) spaghetti plot showing $IFSS_{app}$ (blue), $IFSS_{slope, fiber}$ (red), and $IFSS_{slope, all}$ (green).

Each approach behaves a little differently compared to one another. The most significant differences are the evolution of the variation of the IFSS values for each approach and the final IFSS values of the full datasets. The variation between the subset IFSS values, especially with a low number of filaments, is relatively high for all different approaches, but clearly highest for the $IFSS_{slope, all}$. $IFSS_{app}$ exhibits slightly lower variation compared to $IFSS_{slope, fiber}$. It is evident that measuring only few filaments from a bundle can cause an erroneous impression of the IFSS value despite the approach of calculating the IFSS. Having a representative sampling from a fiber bundle is of the utmost importance in microbond measurements. The need for measuring more filaments is emphasized since a single filament tends to have similar surface quality [5]. It should be noted

that the analysed data was collected from a length of 135 cm which likely increases the variation in each IFSS approach.

After adding the whole dataset from the 53 filaments the IFSS values were 32 MPa for $IFSS_{app}$, 53 MPa for $IFSS_{slope, fiber}$ and 46 MPa for $IFSS_{slope, all}$. The $IFSS_{app}$ has a significantly lower end value compared to the linear regression methods which is expected outcome since rarely the linear regression fits pass through the origin. Other studies have reported $IFSS_{app}$ values of 50 MPa for the same glass fiber and for DER 332 bisphenol-A diglycidyl ether cured with triethylenetetramine [9], it is essential to consider the role of data analysis and the embedded area in contributing to the seen differences. A similar effect where $IFSS_{app}$ is lower than linear regression slopes has also been observed for a glass fiber-polyamide 6 system [1].

3.2 The effect of embedded length on IFSS values

Pearsson's correlation coefficient for $IFSS_{app}$ as a function of A_{emb} yields a positive correlation of 0.55. Evidently, this means larger droplets give larger $IFSS_{app}$ values. The behaviour is seen in Figure 3 which represents all $IFSS_{app}$ data points as the function of A_{emb} . From a data analysis point of view the embedded area of the droplet should not have a significant effect on the IFSS.

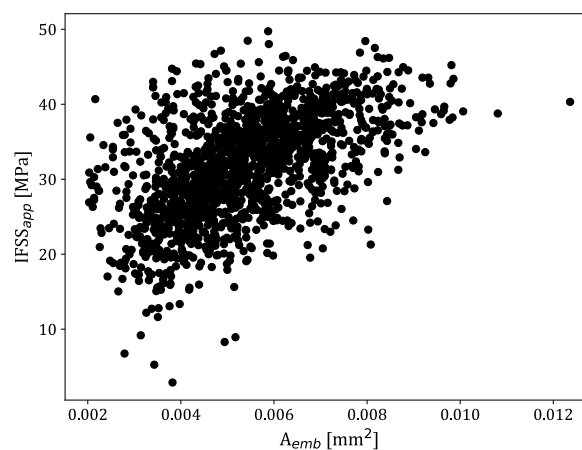


Figure 3. $IFSS_{app}$ as a function of A_{emb} .

The positive correlation between $IFSS_{app}$ and A_{emb} is a direct consequence of negative y-axis intercepts of linear regression fits. Figure 4.a and b. show how simulated $F_{max} - A_{emb}$ data behave with different intercepts and how it affects the $IFSS_{app}$ values. The data was simulated by generating $F_{max} - A_{emb}$ values within equal intervals that correspond to typical microbond results. Data with negative intercepts show similar non-linear behaviour of the $IFSS_{app}$ when approaching small A_{emb} values as seen in the experimental data in Figure 3. On the other hand, if the linear regression fits of $F_{max} - A_{emb}$ passes through the origin, the average of $IFSS_{app}$ and the corresponding linear regression slope are equal. This behaviour is exemplified with the experimental data from two selected filaments (Figures 4.c-d). Thus, to prevent concluding erroneous $IFSS_{app}$ values when evaluating the effect of surface treatments, similar embedded length ranges and number of droplets should be measured for different sample sets.

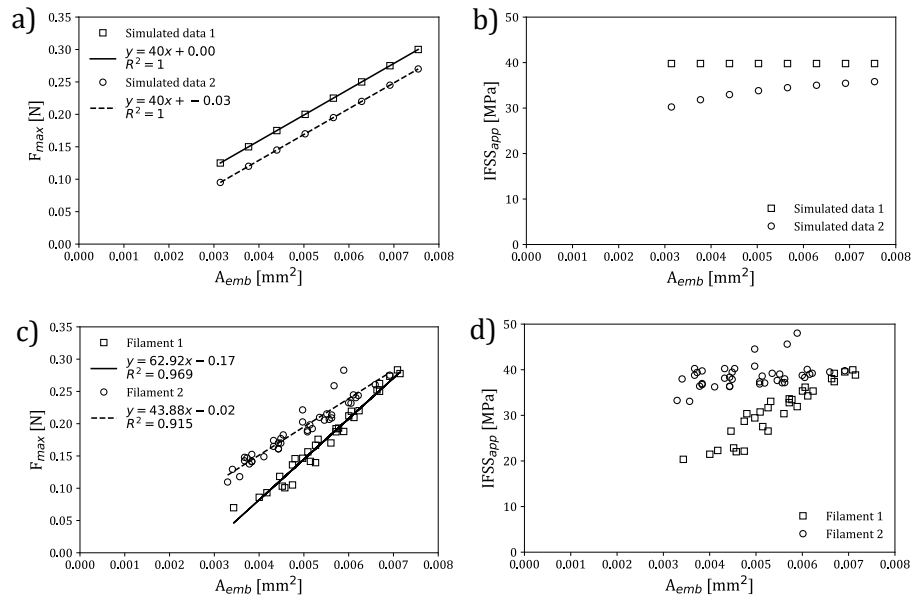


Figure 4. The effect of negative intercept on simulated microbond data: a) F_{\max} – A_{emb} and b) IFSS_{app} – A_{emb} . Experimental microbond data: c) F_{\max} – A_{emb} and d) IFSS_{app} – A_{emb} .

It has been argued that the linear regression should pass through the origin because Equation 1 predicts it [10]. IFSS_{app} inherently assumes a linear relationship between F_{\max} and A_{emb} intersecting the origin. While technically correct, the IFSS_{app} is a very simple model for a complex interface system. Many phenomena affect the measured F_{\max} , such as chemical bonds at the interface, interfacial friction, cure shrinkage, and droplet manufacturing technique [10][11]. These effects are not explicitly accounted for in Equation 1. Therefore, compensating for them by shifting the linear regression through the origin is a highly questionable practice. While such manipulation may reduce IFSS_{app} scatter, as seen in Figure 5, it does so at the cost of distorting the original data. Moreover, a critical issue arises when such data compensation is not explicitly reported, leading to potentially misleading interpretations and limiting the reproducibility of the results. For these reasons, any form of compensation must be justified, transparently described, and applied with great caution. Thomason et al. have argued that the linear regression fit and the y-axis intercept could be used to identify unknown errors in the microbond measurements [10]. They noticed atypical negative intercepts for the linear regression slopes for epoxy droplets on glass fibers. The debonded droplets had left the meniscus on the fiber and therefore the embedded length was evaluated too high. By considering the real embedded length, the linear regression was close to the origin. Negative intercepts have also been observed for glass fiber polypropylene samples where thermal degradation had occurred [3].

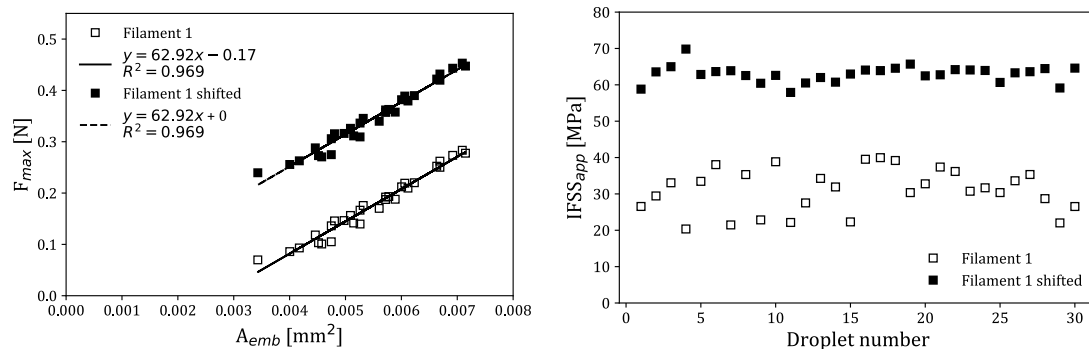


Figure 5. Experimental microbond data (filament 1) with and without shifting it to go through the origin and its effect on the $IFSS_{app}$. Note the different x-axis on the left and right figures.

One challenge that arises when going through the literature where microbond has been used to evaluate IFSS values is that crucial information is left out. These include the measured embedded length range of the droplets, the number of filaments where droplets have been made, and the y-axis intercept calculated from the linear regression. In some publications, it is not clearly reported if the F_{max} - A_{emb} data has been shifted to go through the origin. Meticulous reporting is essential for result comparison between laboratories, repeatability, and enabling standardization.

5. Conclusions

This study investigated the reliability and consistency of different approaches for calculating the interfacial shear strength (IFSS) from microbond tests, focusing specifically on the effects of dataset size, embedded length, and regression method. Three primary methods, apparent IFSS ($IFSS_{app}$), IFSS obtained by averaging slopes from individual filament regressions ($IFSS_{slope, fiber}$), and a linear regression applied to the full dataset ($IFSS_{slope, all}$), are compared to determine their robustness and sensitivity. It was observed that the $IFSS_{slope, all}$ becomes unstable when droplets from only a few filaments is used. This highlights the critical importance of sufficient filament sampling to obtain representative results. Both $IFSS_{app}$ and $IFSS_{slope, fiber}$ were more stable throughout the dataset size and exhibit symmetrical variation. A positive Pearson correlation coefficient of 0.55 was observed between $IFSS_{app}$ and the embedded area, indicating that larger droplets produced higher $IFSS_{app}$ values. This correlation was traced back to non-zero y-axis intercepts in the linear regression of maximum force vs. embedded area. When the linear regression fit did not pass through the origin, even small intercept values significantly distorted the $IFSS_{app}$ values relative to linear regression slope. These findings underlie the risks of using $IFSS_{app}$ as the measure for interfacial shear strength. Instead, linear regression based IFSS should be preferred since it provides a more robust comparison between filaments and different microbond devices. However, $IFSS_{app}$ may be used for comparative analysis when measurements are conducted with the same device and within a similar embedded length range, and when linear regression intercepts are comparable. To advance the standardization of microbond testing and improve cross-laboratory comparability, a new round robin campaign should be initiated and conducted.

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