Study of Polymer-Metal Adhesion Mechanisms in Fused Filament Fabrication Process

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Abstract

Additive Manufacturing (AM) technologies began fabricating end-use products instead of the simple parts and prototypes primary produced with it. The development of more sustainable products include the use of multi-materials and, comparing with others, Polymer/Metal Hybrid (PMH) components take the upper hand in manufacture easiness and increased weight-to-strength ratio. Then, this work intends to study the adhesion mechanisms between polymer/metal parts manufactured with Fused Filament Fabrication (FFF). Single-lap joints with two different adherends: a metallic base substrate and a polymeric top substrate, were manufactured and tested by optical profilometry measurements, Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy's (EDS) analysis and mechanical tests. Optical profilometry revealed more irregularities in non-abraded and grit blasted surfaces, comparing with that abraded by P120 sandpaper, while grit blasting technique showed higher abrasion's level. For the single-lap joints' mechanical characterization, five specimens of each pair of polymer/metal were manufactured and tensile tests were performed. The shear stress-displacement curves reflects the surface roughness and fibers' influence on adhesion strength, which translates on the higher result values obtained for Nylon CF15 polymer. Despite the phenomenon of adhesion among the list of materials used in this work not being fully elucidated, an enhanced adhesion after abrading the surface of the aluminium adherend is evident. The importance of fibers in adhesion mechanisms was also demonstrated and the successful fabrication of several PMH joints possible.
Keywords: Fused Filament Fabrication, Polymer/Metal Hybrid Component, Single-Lap Joint, ABS, Nylon, Nylon CF15

1. Introduction

Current European policies plan to make European Union's (EU) economy sustainable, decreasing our greenhouse gas emissions by at least 55% below 1990 levels by 2030, creating a path to become climate neutral by 2050 [1]. Europe's new industrial way clearly sets a common goal for every enterprise within EU, where ecology and digital transformation come together towards an enhanced portfolio of European sustainability leading companies [2].

This ever-increasing demand for sustainable and complex components opens a window of opportunity for Additive Manufacturing (AM) technologies [3] that, after decades of improvement, began to fabricate end-use products instead of the simple parts and prototypes primary produced with it [4]. Due to its flexibility in producing different designs easily without increased costs, it arises as an interesting option for many companies worldwide, for improvement of their market share [5]. Indeed, Dumitrescu et al. [6] stated an estimated potential of $10.8 billion for global 3D Printing (3DP) Industry by 2021. This value follows the "21.2% growth to $11.867 billion" grow in Global 3DP Industry, published in Wohlers Associates' 2020 report [7]. Such data strengthen AM technologies' socioeconomic influence.

The development of more sustainable products include the use of multi-materials, due to its weight saving potential being one of the most important factors in carbon dioxide emission's reduction [8]. However, combining dissimilar materials, such as metals and polymers, still present many challenges for each industry's sector. When these challenges are overthrown, *automotive, power production, aerospace, medical* and many other sectors, may all benefit from this improved technology [9].

Multi-material components, specifically polymer/metal ones, urge to control the new manufacturing industry that still has to come. Comparing with others, polymer/metal components take the upper hand in manufacture's easiness and increased weight-to-strength ratio [10]. Furthermore, the ability to fabricate such parts, added to industry's highly demand for such, come as a strong advantage to some AM technologies [11, 12]. In this context, Fused Filament Fabrication (FFF) is now perceived as one major AM technology platforms for the production of polymer/metal multi-materials components.

The main goal of this thesis is the study of adhesion mechanisms between polymer/metal parts manufactured by FFF. Our project comes in pursue of recent developments by Falck et al. [13], where a new technique called *AddJoining*, successfully combines polymer filaments with Aluminium. To pursue this objective, the following tasks are required:

- Surface treatment of the metallic samples with abrasion techniques;
- Creation of a physical apparatus for fixing metallic samples on a 3D printer;
- Optimization of 3DP parameters for each pair of polymer/metal materials;
- Manufacturing of PMH components combined in a single-lap joint;
- Evaluation of the quasi-static mechanical behaviour of multi-material single-lap joints;
- Assessment of surface treatment and fibers content's effect on adhesion strength;
- Correlate the fracture's surface of the joints with its mechanical behaviour;
- Discuss the applied adhesion techniques and its feasibility for future applications in the context of the FFF technologies.

2. Literature Review

In 1987, *3D Systems* introduced AM technology to the market. The first process to be introduced was Stereolithography (SL). During the 90's, Selective Laser Sintering (SLS) came to life [14]. Meanwhile, the American company *Stratasys* created the trademark Fused Deposition Modeling (FDM), usually called Fused Filament Fabrication (FFF) and the technology used in this thesis.

Increasing product's functionality and manufacturing's efficiency is highly demanding. This enhances the need for using multi-materials' components, hence the focus in joining dissimilar materials [9], a fact already recognized by almost every industry.

Adhesive joining is the most appropriate technique for bonding metallic and non-metallic structures where strength-to-weight ratio must be maximized [15]. This technique utilizes polymeric substrates to join a large variety of material combinations. The literature identifies six main theories of adhesion [16, 17]. Absorption theory: the adhesive completely wets the substrate; eletrostatic attration theory: suggests the development of an electrostatically charged double layer of ions at the adhesive-adherend interface [18, 19]; chemical bonding theory: along with intermolecular forces, polar groups' presence characterize the strength of the adhesive joint between a pair of the same material or dissimilar materials [20]; mechanical interlocking theory: entails the physical interlocking of the adherend surface and the cured adhesive at the macroscopic level, facilitating chemical bonding and wettability of this coupling [21, 22, 23]; diffusion theory: a successful union between two non-polar polymeric substrates results from the interdiffusion of the macromolecules at the interface [24]; weak boundary layer theory: in the event of failure on an adhesive joint, a cohesive break of a weak boundary layer is the most likely scenario to have caused it [25].

A mixture of theories is usually responsible for a given system [26], since researches have not yet reached a conclusion on which mechanisms best suit adhesive bonding.

A proper compatibility of the interface between both substrates desired is of extreme importance to form the adhesive joint. Surface treatment is used to improve this adhesion. An increase in friction coefficient correlates with an increment in surface roughness of the material [27, 28], a highly important parameter in our research. In addiction, some researches point to an increase in wettability when combining the incorporation of polar functional groups to a polymer joined with a silicon coated steel, influencing positively the adhesion strengh with steel [28, 29]. Grit-blasting technique is referred as the ideal surface treatment on metallic mediums [30, 31, 32]. Despite these surface treatments, some substrates may still provide only negligible adhesion. At this point, primers or adhesive promoters may come to aid with further joining strength.

Previous researches have presented various AM of multi-material components. Among them, Shaped Deposition Model (SDM) gives an insight of multiple applications with Polymer/Metal Hybrid (PMH) in industries such as automotive and eletrical systems [33] and in AddJoining technique [13], single-lap joints of Aluminium and polymer filaments are joined with FFF only. Many other techniques can be found in literature [33, 34, 29].

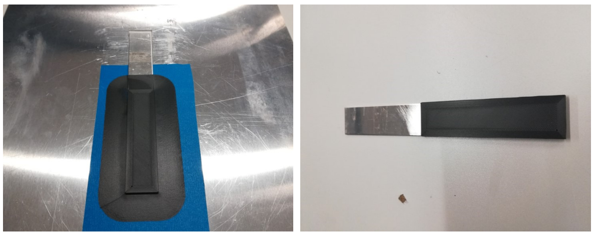
The type of adhesive and joint geometry also influence the strength of the bond. Due to the popularity and widely use of single-lap joints, adding to the limitations of AM, we found in this type of joint an ideal choice for our project.

3. Materials and Methods

The specimens used in this work were adapted from the ASTM D3163 – 01 standard [35], a single-lap joint with two different adherends: a metallic base substrate and a polymeric top substrate. A metallic substrate sheet with a thickness of $2\ mm$ was chosen from a laboratory sample and characterized. For the top polymeric adherend, ABS, Nylon and Nylon CF15's $1.75\ mm$ filaments were used.

The 3D printer employed in our work was a Raise3D Pro2, which makes part of Lab2Prod 3D printers' portfolio. The original build plate was replaced by a customized Aluminium one made from the same batch of the specimens, with a slot to insert them.

FFF started by determining optimum printing parameters for the polymeric top adherend's manufacturing. The surface of the overlap area of the base material was then mechanically changed, to study its influence on adherence strength. For each specimen, the surface of the overlap area was cleaned from any type of grease or impurity with isopropyl alcohol and later abraded by sandpaper or sandblasting technique. For Aluminium-ABS's joints, primer was manually spread with a paintbrush, whereas for Aluminium-Nylon and Aluminium-Nylon CF15's joints, primer's application was done by FFF. For the later, primer was then remelted to better infiltrate Aluminium's surface cavities. Finally, each top adherend was manufactured by FFF, as illustrated in Figure 1.



(a) (b)

Figure 1: (a) Nylon CF15 specimen's resting time after printing and (b) after removal of the brim.

In the wake of comparing the different surface treatments and its effects on the lap-shear strength of the PMH joints, an optical profilometry measurement was done on the top surface of the base material with a profilometer Profilm 3D with a 20X objective. A Scanning Electron Microscopy (SEM) and an Energy Dispersive Spectroscopy (EDS) analysis were conducted to characterize the base material and measure primer's thickness. The mechanical properties of the base material and PMH joints were determined by uniaxial tensile tests in an INSTRON 3369 universal testing machine with a $50\ kN$ load cell and a crosshead speed of $1.0\ mm/min$. Microindentation hardness tests were conducted on a Shimadzu HMV-2 microhardness tester.

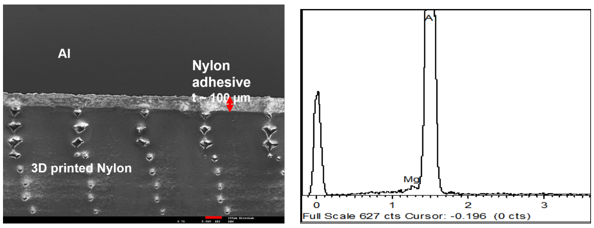
4. Results and Discussion

Optical profilometry revealed more irregularities in non-abraded and grit blasted surfaces, comparing with that abraded by P120 sandpaper. Sandpaper's utilization contributed to a homogenization of the non-abraded surface, increasing however its surface's roughness. Grit blasting technique shows an exceptionally degree of abrasion, when compared with non-abraded and P120 sandpaper's treatment. For each base material's surface condition, the measured surface roughness values, R_a and R_c are given, Table 1.

Table 1: Surface roughness measurements R_a and R_c values of aluminium adherend after surface treatment.

Surface Treatment	$\mathbf{R_a}\,[\mu m]$	$\mathbf{R_c}\,[\mu m]$
Non-abraded	0.16 $\pm$ 0.04	0.58 $\pm$ 0.12
Abraded by P120	0.32 $\pm$ 0.05	1.12 $\pm$ 0.22
Abraded by grit-blasting	1.65 $\pm$ 0.46	6.20 $\pm$ 1.67

SEM's results and EDS spectra validated the programmed $0.1\ mm$ primer's thickness of PMH joints and helped characterize the base material, respectively, Figure 2. However, a proximity between each element energy levels made it very hard to properly identify the microstructure of the base material. It only shows a probable existence of Magnesium inclusions ($1.12\,wt\%$) within the Aluminium domain.



(a) (b)

Figure 2: (a) SEM micrography analysis and (b) EDS spectra of the base material.

Following tensile loading and Vickers hardeness tests, average hardness value of the base material was found to be equal to 127 (VH), complemented by its remaining mechanical properties: $\sigma_{\mathbf{UTS}} = 265.38 \pm 30.89\ MPa$, $\sigma_{\mathbf{y}} = 235.98 \pm 25.27\ MPa$, $\mathbf{E} = 78.8 \pm 1.68\,GPa$ and $\varepsilon_{\mathbf{F}} = 3.92 \pm 1.54\%$. These results suggest that the base material is part of the 5000 Aluminium alloy's series [36].

For the single-lap joints' mechanical characterization, five specimens of each pair were manufactured and tensile tests were performed. The shear stress-displacement curves are shown below, Figures 3 to 7, for sandpaper and grit blasting's abrasion techniques.

Sandpaper's abrasion technique was validated for Aluminium-Nylon and Aluminium-Nylon CF15's joints only, Figures 3 and 4, respectively. The maximum Ultimate Lap Shear Stress (ULSS) achieved for the first was $3.02\ MPa$, whereas the last failed at a maximum ULSS equal to $3.51\ MPa$. Some specimens required a higher removal force than others from the printing plattform, which translated in a lower failure load. A higher disparity in results can be observed for Aluminium-Nylon CF15's joints, Figure 4. In fact, an explanation for this might be an increased adherence of this polymer to the blue masking tape, possibly due to the presence of milled carbon fibers.

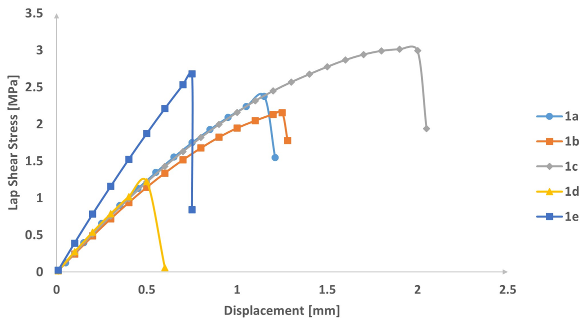


Figure 3: Aluminium-Nylon joints, previously abraded by sandpaper.

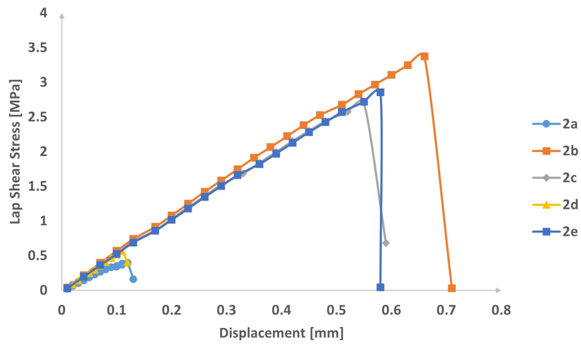


Figure 4: Aluminium-Nylon CF15 joints, previously abraded by sandpaper.

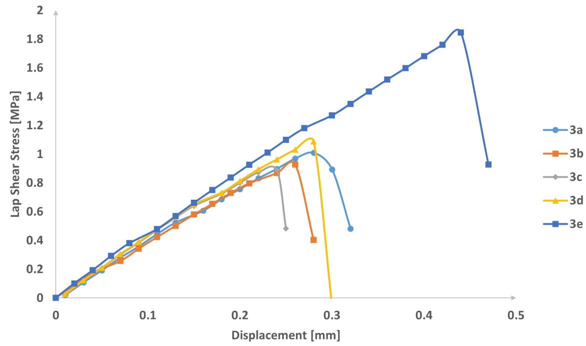


Figure 5: Aluminium-ABS joints, previously abraded by grit blasting.

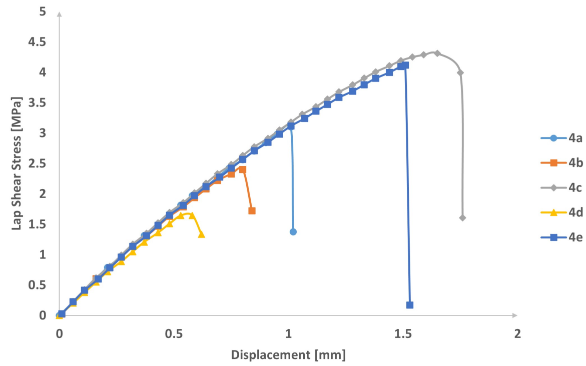


Figure 6: Aluminium-Nylon joints, previously abraded by grit blasting.

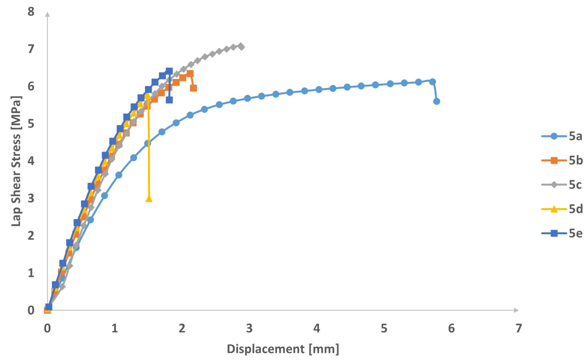


Figure 7: Aluminium-Nylon CF15 joints, previously abraded by grit blasting.

The results for Aluminium-ABS specimens treated with grit-blasting technique, Figure 5, show an average ULSS of $0.99 \pm 0.08\ MPa$ and a maximum ULSS of $1.92\ MPa$. This difference in roughness values contrast with the ones obtained by Falck et al. [13] for the same pair of materials: ULSS $= 5.3 \pm 0.3\ MPa$ for Aluminium-ABS joints. However, their joints showed an average peak to valley roughness $R_c = 89.0 \pm 0.3\ \mu m$, contrasting with our $R_c = 6.20 \pm 1.67\ \mu m$. This variance might explain the greater ULSS achieved by them.

Examining the results for Aluminium-Nylon specimens abraded with grit-blasting, Figure 6, a maximum ULSS equal to $4.34\ MPa$ was achieved. Primer's application processes might explain the differences found between the maximum ULSS values of Aluminium-ABS and Aluminium-Nylon single lap joints. When applying it manually, as it was the case for ABS, the spreading of the polymer into Aluminium's surface cavities was less efficient than with Nylon. The higher temperatures of the nozzle and the contact force it exerted on Aluminium's surface might be the cause for this decrease in viscosity of Nylon's, promoting a better adhesion between Aluminium and Nylon. Now, the effect that this method had in adhesion strength may be explained by a combination of the absorption theory with the mechanical interlocking.

The final experiment revealed the highest tested failure load encountered and a maximum ULSS equal to $7.10\ MPa$, Figure 7. Aluminium-Nylon CF15's tensile tests results may be compared with others found by Falck et al. [13] for Aluminium with alternate layers of Nylon and Continuous Carbon Fibers. With an average peak to valley roughness $R_c = 89.0 \pm 0.3\ \mu m$, the tests conducted to these joints, previously abraded by grit blasting, resulted in a maximum ULSS $= 21.9 \pm 1.1\ MPa$. This results' disparity may be explained by multiple factors: a larger surface profile was already mentioned has a vehicle for good adhesion among polymer/metal joints and the resource for continuous carbon fibers can also enhance the rigidity of the joint and elevate its ULSS.

A previous study conducted by Boutar et al. [37] had already measured similar roughness values to our sandpaper's abraded ones, in which we achieved an ULSS equal to $2.33 \pm 0.57\ MPa$ for Nylon specimens and $2.05 \pm 1.29\ MPa$ for Nylon CF15's ones, which are in line with their resulting ULSS equal to $2.92\ MPa$. Further along, the results found in our study for grit-blasting specimens can be compared with the ones found in theirs for P180 abraded specimens (an average ULSS equal to $3.52\ MPa$), where Aluminium-ABS specimens performed well below (ULSS $= 1.18 \pm 0.38\ MPa$), contrasting with similar values for Aluminium-Nylon's (ULSS $= 3.14 \pm 1.00\ MPa$) and better results for Aluminium-Nylon CF15 specimens (ULSS $= 6.36 \pm 0.43\ MPa$). Boutar's P180 specimens showed a $R_a = 1.5 \pm 0.14\ \mu m$. For Aluminium-ABS specimens, a lower result might be due to primer's application process and the nonexistence of a thermal post-processing of the metallic adherend prior to adhesion with ABS's top adherend.

Figure 8 illustrates surface roughness and fibers' relevance in adhesion among PMH joints. For specimens previously abraded by sandpaper, with an average surface's roughness, R_a, equal to $0.32 \pm 0.05\ \mu m$, Nylon CF15 specimens' single lap-shear maximum result was $3.51\ MPa$, 16% more than the maximum value obtained for Nylon specimens. Moreover, when surface's roughness increases to a R_a equal to $1.65 \pm 0.46\ \mu m$, after a grit-blasting treatment, the maximum ULSS of Nylon CF15 specimens overcome Nylon ones by 64%, with a maximum ULSS equal to $7.10\ MPa$ against $4.34\ MPa$, respectively.

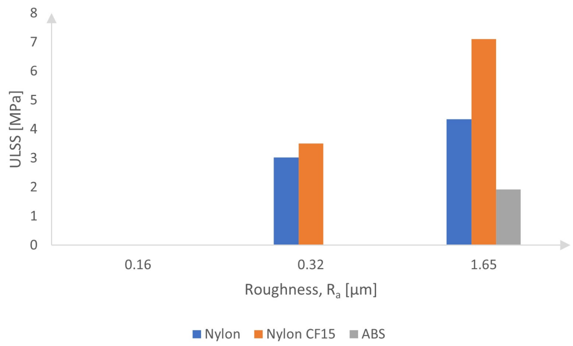
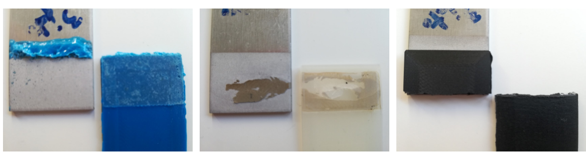


Figure 8: Surface's Roughness and Fiber's Content Effect on Ultimate Lap Shear Strength.

Comparing both variables, fibers' content appears to have a strong effect on adhesion strength between Aluminium and 3D printed polyamides, whereas Aluminium's surface roughness effect is only secondary. Along this, fibers' content also visually improved the quality of the print, adding to less shrinkage on the final specimen once it cooled down. This shrinkage's decrease could also result in an increase on shear strength, as previous researches concluded [38].

The observation of failure's surface was also part of this work. It was found interesting to examine each experiment and evaluate the adhesion performance from the occurred failure mode. An example of each failure mode obtained in this experiment is illustrated in Figure 9, which exemplifies (a) an adhesive failure mode for ABS type specimens, (b) a mixed adhesive/cohesive failure mode for Nylon type specimens and (c) a substrate failure mode for Nylon CF15 type specimens, all previously abraded by grit-blasting.



(a) (b) (c)

Figure 9: Main failures' mode: (a) adhesive failure mode, (b) mixed adhesive/cohesive failure mode and (c) substrate failure mode.

Aluminium-ABS and Aluminium-Nylon's joints abraded by sandpaper and grit blasting, respectively, showed evidence of a characteristic adhesive failure mode and the poorest ULSS's results. For Aluminium-Nylon and Aluminium-Nylon CF15's joints abraded by grit blasting and sandpaper, respectively, a principal cohesive/substrate failure mode is shown and its ULSS's results are also increased. Aluminium-Nylon CF15's joints abraded by grit blasting show a predominant substrate failure mode, which agrees with its greater ULSS.

5. Conclusions

This work laid hold of previous researches in metal-polymer adhesion mechanisms in FFF. It was studied the feasibility of adhesion among Aluminium and three polymeric substrates: ABS, Nylon and Nylon CF15. Additionally, it has been studied the effect of surface roughness and fibers content on the adhesion strength between each pair of material, which are the highlights of this work and a further addition to the scientific literature.

The results found in here are incapable of completely explaining the phenomenon of adhesion among the list of materials used in this work. However, an enhanced adhesion after abrading the surface of the Aluminium adherend is evident, which can be anticipated by mechanical interlocking theory, increased contact surface area and formation of a clean surface. These results also demonstrate the suitability of milled fibers for PMH components fabricated by FFF.

Future researches would most likely benefit from an accurate strain field analysis, to better understand the failure mechanism of these joints, adding to further microscopical's tests to study fibers' inclusion into Aluminium's surface. To prevent peeling stresses in the joint, a more suitable removal mechanism should be implemented and a heating control unit should be added to the customized build plate for the improvement of the remelting process.

Overall, this work validated a novel FFF technique for additional combination of materials, other than those studied previously. Aluminium-Nylon CF15 specimens exhibited the best mechanical properties, which reiterates the importance of fibers in adhesion mechanisms. Thermal post-treatment revealed extremely advantageous, an element that could be easily added to a standard FFF process, when looking for PMH component's fabrication.

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