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**Improvement of automotive joining  
technologies by surface treatment processes**

*Ph.D. dissertation booklet*

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## **1. INTRODUCTION**

In the automotive industry, the greening of reducing consumption and thus emissions, is also emerging [1–3]. One of the most tangible ways to achieve this is through light weighting of vehicles. Reducing the kerb weight of vehicles significantly reduces the energy demand for propulsion, fuel consumption and therefore emissions [1,3]. One suitable way to achieve this could be to use high-strength steels and advanced polymer materials in potential applications [4–7]. However, the design of joints between elements with different properties still poses problems and challenges for vehicle manufacturers [8–12].

In my opinion, it is advisable to concentrate on developing a bonding method that can be used on all types of materials at the same time without increasing the mass. One such timely and economical way of non-solvent bonding of different materials is adhesive bonding [13–18]. The strength of bonded joints is mainly due to two types of forces: 1) the cohesion of the adhesive and the base materials, and 2) the combination of forces at the boundary of the material to be bonded and the adhesive, called adhesion. In a well bonded joint, the adhesion forces are at least as large as the cohesive forces [19]. However, they are affected by the challenge of today's bonding technology, the wetting characteristics of the surfaces to be bonded. The strength of the bond can be traced back to the contact that the adhesive makes with the surface of the materials to be bonded [20–22]. The higher the surface energy of our material of interest, the higher will be the strength of the adhesion technology applied to the surface. In most cases, the materials used in the modern automotive industry have poor wetting properties. Bonding them in the base state would lead to strength limits and barriers. However, by using the right preparation and surface treatment process, we can influence the wettability values in a way that is favourable to us [23–25].

High energy density beam surface treatments such as plasma and laser beam can be a suitable solution to optimise surfaces for bonding. They are the most advanced surface treatment methods for the automotive industry today, but have been little addressed. In my thesis, I therefore aim to multiply the shear strength of automotive adhesive bonding technology on different material types using high energy density surface treatment techniques.

## **2. LITERATURE REVIEW**

In the automotive industry, trends in the use of materials are shifting towards modern high-strength steels and high-impact polymers, but in parallel with the development of materials, there is also a need to improve the bonding quality of materials. One of these emerging bonding methods is adhesive bonding. In order to improve adhesive technology,

the aim is to increase the surface adhesion of different base materials by modifying the boundary of the surface. Closely related to surface tension is the concept of wetting, which refers to the change in the interface. The most common and widely used surface energy calculation theory is the Fowkes method [26–29]. By measuring the wetting contact angle and then calculating the Fowkes surface energy, the wetting property of a material can be determined. However, it is expected that this can be varied. This can be achieved by different surface treatment methods. There are two emerging technologies that have not yet been widely used for this type of wettability enhancing surface modification, but have great potential, and are emerging from traditional mechanic-chemical surface treatments, such as plasma and laser beam surface treatment.

Vesel et al. applied oxygen plasma treatment to PMMA (Poly(methyl methacrylate)) surfaces and achieved a reduction of the wetting angle from 83° to 42° in the untreated condition [30]. Hsu and his team also performed atmospheric pressure plasma surface treatment on PMMA surface and improved the surface wetting to 30°, and then used the cleaned and improved surface for further measurements [31].

In a similar study, Kehrer et al. performed a pure oxygen and nitrogen plasma surface activation on a PP (polypropylene) surface, but the surface was definitely burnt, with a strong aesthetic change in the tested parameter window, but the shear strength of the bonded joint increased from 31 MPa to only 39 MPa [32].

Rodríguez-Villanueva and his team used plasma jet surface cleaning on metals. The experiment involved deliberately applying a given volume of oil to the surface and using a plasma beam to test its cleaning and hence its effect on adhesion. The technology tested included a plasma treatment contact time at a constant distance. The plasma jet was a nitrogen gas plasma jet. With this surface cleaning and treatment, the shear strength of the silicone-based adhesive bonds increased from 1.9 MPa to 2.3 MPa. It is noticeable that plasma surface treatment of metals is less effective compared to polymers [33].

An innovative application of ultrafast laser pulsed surface treatments is their wetting enhancement. Giorano et al. studied the wettability of titanium and mild steels using picosecond pulses, where only partial wettability enhancement was achieved [34]. Zhao et al. used pulsed Nd:YAG lasers to improve the wettability properties of copper. In their results, the surface energy of copper was improved to 55 mN/m by laser beam treatment at an average power of 1500 W and 2000 W. In the best case, the wetting contact angle was reduced from 42° to 31° on the surface treated with 2000 W laser beam [35]. József Hlinka experimented with Nd:YAG lasers on copper surfaces, where he achieved a

wetting modification effect when investigating brazed joints [36]. Demir et al. studied the wettability of AZ31 Mg alloy against water, concluding that by laser surface treatment, increasing the size of the oxidized surface allows a decrease in the wetting contact angle [34].

Several authors, such as Ngo and Chun, have proposed a method for the low-temperature annealing of metals to form hydrophobic surfaces [37]. This has also been observed by Kietzig et al. who found that the formation of the hydrophobic wetting state is related to the change of carbon molecular groups due to the slow decomposition of CO<sub>2</sub> at room temperature [38]. Ngo and Chun supported this theory and showed that low-temperature annealing accelerated this transition due to the more intense decomposition of CO<sub>2</sub> [37]. Bizi-Bandoki et al. argued that the improvement in wettability could be explained by the appearance of new functional molecular groups. Thus, different heat treatments on the surface achieved wettability enhancing effects through heat-induced changes in surface molecular groups [39].

The results reported by Y. Boutar in their paper suggest that there is a close relationship between the wetting angle, the shear strength of the adhesive and the surface roughness in adhesive technology. There is an inverse proportionality between wetting angle and shear strength, while the relationship between roughness and shear strength is parabolic. In their case, it was possible to reduce the wetting angle from 75° to 62°, which increased the shear strength of the bonded joint from 2.9 MPa to 3.5 MPa. The 13° reduction in the contact angle resulted in a slight increase in strength, but this is already a significant change [40].

Giovanna and his co-workers, treated DP500 high strength and AISI 304 corrosion resistant steel with a trans-laser. Their aim was to improve wetting, whereby they achieved an improvement in bond shear strength. In their results, they succeeded in reducing the untreated 50° wetting edge angle of the substrate to 10°, and they also observed an intense deterioration of surface roughness with the improvement of wetting [41].

### **3. OBJECTIVES OF THE RESEARCH**

The effect of changing the surface wetting phenomena on the performance of bonding technologies (expected increase in shear strength), however, the effect of different surface treatments on the surface energy is not sufficiently well understood. Advances in new beam technologies are enabling new types of approaches to altering surface energies, which will affect the wettability of the surface, which will affect bond strength. Knowledge of the surface energy of radiation technology processes is less studied and therefore there is little reference to the relationship between wetting properties and bond strengths. The uptake of

plasma and laser beam processes for surface treatment could be greatly aided if systematic research can demonstrate that the mechanisms of action of the treatments significantly affect the bond strength properties of adhesion bonds through changes in surface energies. Which, in turn, are essential for the uptake of practical applications. The results of the literature survey show that high strength steels and high impact polystyrene (HIPS) have become the materials of choice for modern vehicle structures. On these materials I plan to investigate the effect of cold plasma and laser beam processes on the surface energy, the wettability and consequently the shear strength of the bonded joints.

Accordingly, the research will focus on:

1. Comparison of plasma and laser surface treatment processes, based on their mechanism of action, in terms of surface energies. With the aim of identifying the technological data sets that allow the preparation of optimisation.

2. On high impact polystyrene, it is necessary to determine how cold plasma jet process data affect the wetting properties and how they affect the shear strength of the bonded joints, which will allow the optimisation of the process data set.

3. On DP600 incremental strength steel, it is necessary to determine how cold plasma and laser beam process data affect surface topography and wetting properties, which can be used to optimise the setting of process data and how they affect the shear strength of bonded joints.

4. To investigate the temporal effects of the time elapsed after plasma and laser beam surface treatments until the formation of bonded joints (environmental effects) on properties important for the bonding technology, such as wetting.

#### **4. DESCRIPTION OF EXPERIMENTAL MATERIALS, EQUIPMENT AND PROCEDURES**

##### **Test materials**

For the first test, I chose high impact polystyrene as the base material. The base material is a high impact PS sheet from A-Plast Ltd., white, 1000 x 2000 x 5 mm, 744, with a glossy and protective film on one side and a matt surface on the other side. This material represents the research carried out on automotive polymers.

Among the metals I used for my investigations is DP600 type uncoated steel, which is also widely used as a vehicle body material. Dual Phase steels (DP600) consist of hard martensite islands embedded in soft, malleable ferrite, depending on the desired mechanical properties. In my experiments, I used 1 mm thick cold rolled DP600 steel plate.

The area of the test specimens was defined as 25 x 55 mm<sup>2</sup>. This is sufficient for surface treatment, wetting, microscopy and adhesive technology tests.

### **Cold plasma surface treatment system**

For the surface treatment, I used PlasmaTreat's OpenAir® atmospheric pressure, compressed air system. The cold plasma was generated by a plasma generator with a maximum output power of 1000 W. A plasma head and a high voltage transformer were connected to the generator. The machine was operated at 21 kHz. The exit point of the plasma was eccentrically positioned at the edge of the head and rotated at 2500 rpm to dissipate the energy uniformly in the scattering radius. Variable process settings were the distance between the plane of the material to be treated and the plasma emitting head and the speed of the main straight line motion.

### **Ultrafast pulsed femtosecond laser system**

For the surface treatments I used a Coherent Monaco 1035 nm laser operating in femtosecond pulse mode. The machine can output a maximum power of 60 W with a pulse duration of 300 fs and a wavelength of 1035 nm. The frequency was 188 kHz, the focal spot beam diameter was 80  $\mu\text{m}$ , and the fill was 60  $\mu\text{m}$ .

### **Room-temperature droplet method for measuring the contact angle**

Immediately after the surface treatments, I measured the wetting contact angle on the specimens at room temperature using the stilling-drop method. Using a micropipette, 5  $\mu\text{l}$  drops were used for the measurement. Two liquids were used for the droplet analysis to apply the Fowkes method, distilled water and  $\geq 99\%$  pure ethylene glycol.

### **Scanning electron microscopy investigations**

I use scanning electron microscopy (SEM) to investigate the topography-changing ability of surface treatments. The SEM equipment used for the studies were Zeiss Sigma and Tescan MIRA systems.

### **Presentation of bonding technology experiments, process of determining shear strength**

In a series of experiments with HIPS bonding technology, I used Loctite 4080 hybrid adhesive, a cyanoacrylate/acrylic structural adhesive. The bonded area was 25 x 30  $\text{mm}^2$ .

For the DP600 steel, Loctite 9466 two-component epoxy structural adhesive was used for the bonding experiments. This type of adhesive is particularly suitable for high strength bonding of metals. The bonded area for the metal sheets was 25 x 25  $\text{mm}^2$ .

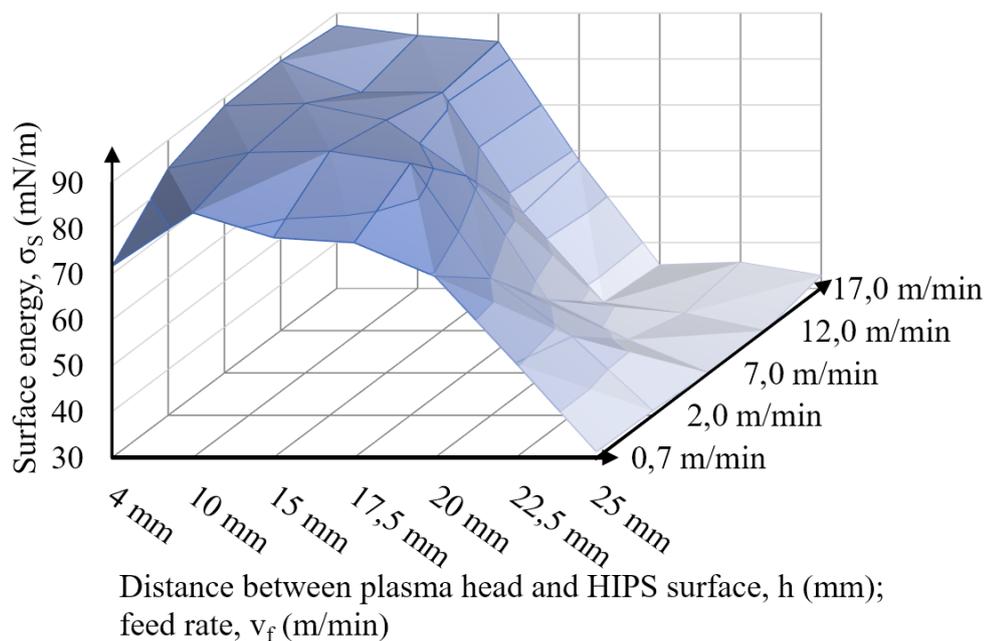
The bonded joints were tensile tested using an INSTRON 5900R 4482 universal testing machine. In order to avoid side pulling, I used inserts during the clamping to ensure that the axis of the pull was parallel with the bonded surface. The shear strength of the bonded joints of the overlapped specimens was characterized by the average stress at the bonded surface.

## 5. RESULTS

In the results, I will present the surface modification effects achieved in my research on HIPS polymer and DP600 high strength steel and their implications for the strength increase of bonded joints. I will also present a study of the time dependence of the surface treatments and a comparison of the mechanism of action of the two surface treatment methods.

### Experiments on plasma surface treatment of HIPS polymer

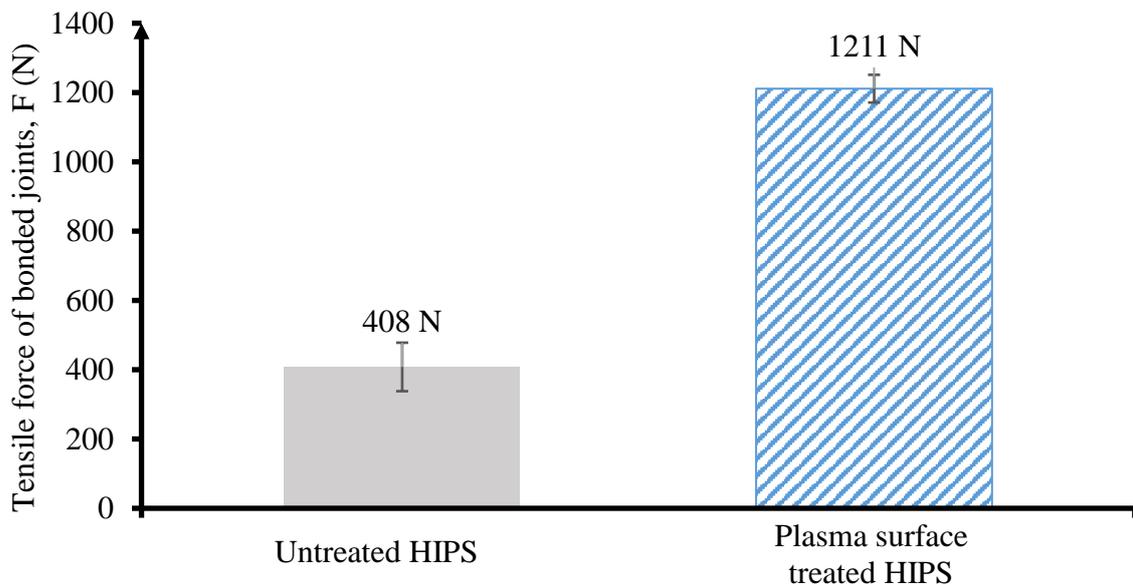
The untreated surface energy of poorly wetted HIPS is only 27.4 mN/m. With the cold plasma jet test data set, my aim is to evaluate the surface activation effect of plasma jet surface treatment on HIPS substrate and to optimize the treatment configurations. Thus, a total of 5 scan speeds and 7 scan distances were tested, resulting in 35 combinations. The polymer test specimens were treated under the same conditions prior to each measurement and I removed the intact protective film immediately prior to surface treatment, ensuring that the only surface interaction factor in my research was the plasma surface treatment. I treated five plates in each combination. When presenting the results, it is typically useful to use the total surface area value, i.e. the sum of the polar and dispersive energy values, to characterise the variation in wetting as a function of different surface treatment data. The surface energy map shown in Figure 1 can be used to determine the wetting variation trend of plasma surface treatment configurations. The trend of the diagrams mainly shows the surface activating effect of the plasma surface treatment and hence the decreasing effect on the wetting contact angle.



1. figure: Map of surface energy variation on HIPS material as a function of plasma surface treatment data

The trend of the chart can be divided into 3 areas. Firstly, at the beginning of the plasma surface treatment, when we are either moving too fast or too far away from the HIPS surface, the surface energy values start to increase gradually. The higher the plasma surface energy on the surface, the more the surface activation effect is enhanced. The reason for this increase in surface energy values is the change in the surface molecular groupings brought about by the excess energy of the plasma. In the second stage, a full hydrophilic state on the surface is achieved, such as surface treatment at a scanning depth of 7 m/min and a distance of 10 mm. In these cases, I have communicated enough energy to the HIPS surface to achieve functionalisation of the surface molecular groups and this is reflected in the achievement of full hydrophilicity on the surface compared to the initial conditions. In the third stage, the hydrophilic state is degraded by slow and proximal treatment due to excessive heat exposure, resulting in deterioration of the wetting properties. An optimum for the improvement of wetting and surface energy can be defined, this is a plasma head feed rate of 7 m/min and a surface-to-plasma head distance of 10 mm. At this condition, all boundary angle values were  $0^\circ$ , so the surface hydrophilic while the surface energy values increased to  $88.7 \pm 5$  mN/m.

In a series of HIPS bonding experiments, untreated HIPS specimen pairs, surface-treated after removal of the protective film only and with optimum settings, were bonded in interleaved bonding and their shear strength was tested. The results are shown in Figure 2 with the mean and standard deviation of the results obtained.

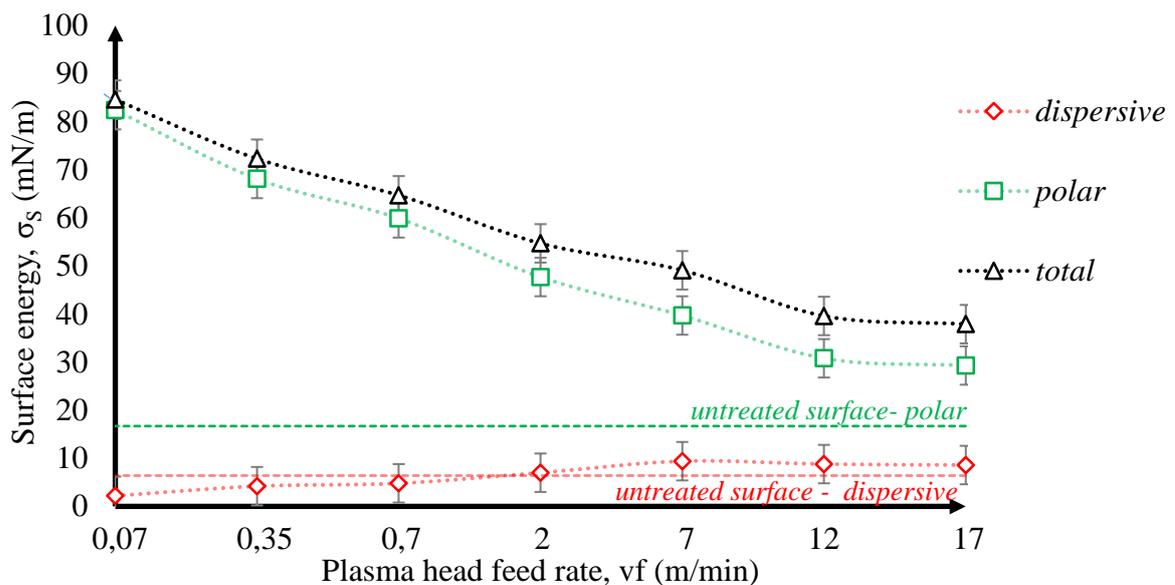


2. figure: Results of interleaved rupture of HIPS specimens surface-treated with plasma beam with untreated and optimized process data

It can be seen that the untreated samples had an average tensile strength of  $408 \pm 74$  N, whereas the average tensile strength increased to  $1211 \pm 28$  N during bonding of the plasma treated samples to the hydrophilic surface. This represents an increase of 296% in shear strength of  $0.54 \pm 0.09$  MPa for untreated samples and  $1.61 \pm 0.04$  MPa for HIPS surface-treated with the optimal technology.

### Experiments on plasma surface treatment of DP600 high strength steel

In my experimental design, I chose a constant distance of 4 mm between the plasma scanning head and the steel plate. The power of the equipment was applied at 100% and thus the variable setting was the plasma beam travel speed. Here, I used speeds of 17, 12, 7, 2, 0.7 and 0.35 and 0.07 m/min. From the surface energy value of the untreated substrate, I determined that the degreased, cleaned surface of DP600 steel has poor wetting characteristics and a low surface energy of 26 mN/m. From the trend of the results, it can be observed that the surface treatment at 17 m/min already gave a result compared to the initial surface energy, with a total surface energy value of 38 mN/m (Figure 3).

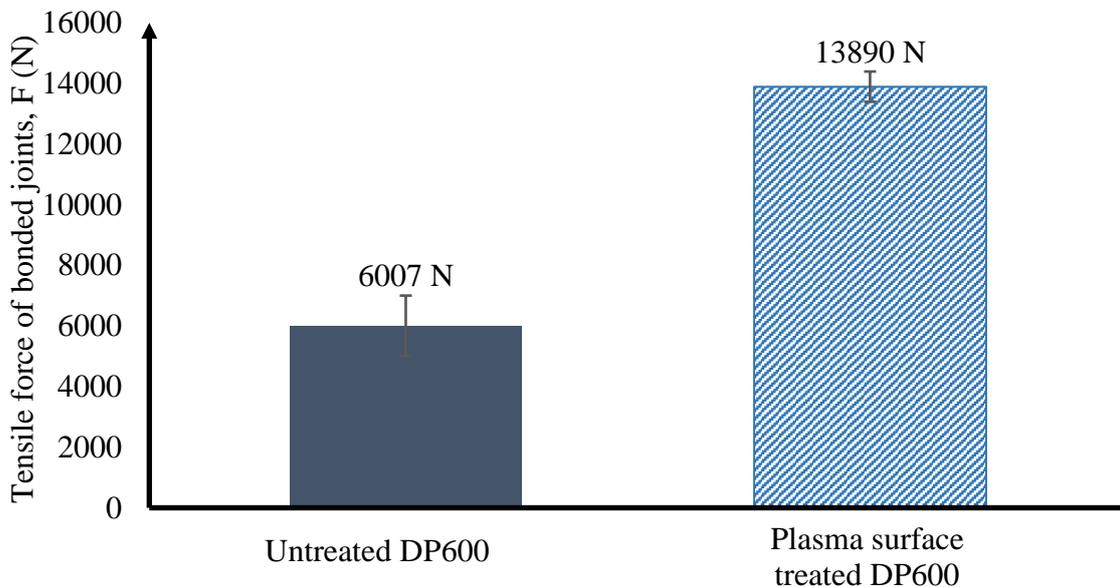


3. figure: Variation of surface energies of DP600 steel plate surface treated with plasma jet for different feed rates

To improve the wettability of the steel surface of the DP600, I slowed down the speed, concluding that up to a feed rate of 0.7 m/min, there is no reduction in the contact angle to  $0^\circ$ , with a 59% reduction in contact angle values compared to untreated values. This may be due to the fact that in the first stage, where the speed is higher than 2 m/min, the plasma jet on the steel surface is cleaning the surface, removing the impurities, but the energy of the plasma jet is not sufficient to activate the molecular groups on the surface, which is

completely different for DP600 steel compared to the processes that take place in HIPS. However, by applying a velocity of 0.7 m/min, the surface is irradiated with excess plasma energy, which improves the surface energy values. I slowed the process down to 0.07 m/min (70 mm/min), which is an engineering low speed that can still be applied to the manufacturing process. In this case, the process slowed down drastically and the steel specimen was heated due to the proximity and speed of the excess plasma energy at 4 mm. No hydrophilic condition was reached during the wetting measurement process for the plasma treated DP600 steel plate. The highest surface energy value was  $84\pm 5$  mN/m.

Bonding experiments were carried out on the surface treated DP600 steel plate at the slowest feed rate of 0.07 m/min and a plasma jet surface treatment of 4 mm distance using a 2 component epoxy. From the results shown in Figure 9, it can be concluded that the plasma jet surface treatment greatly increased the shear strength of the bonded joint of the steel.



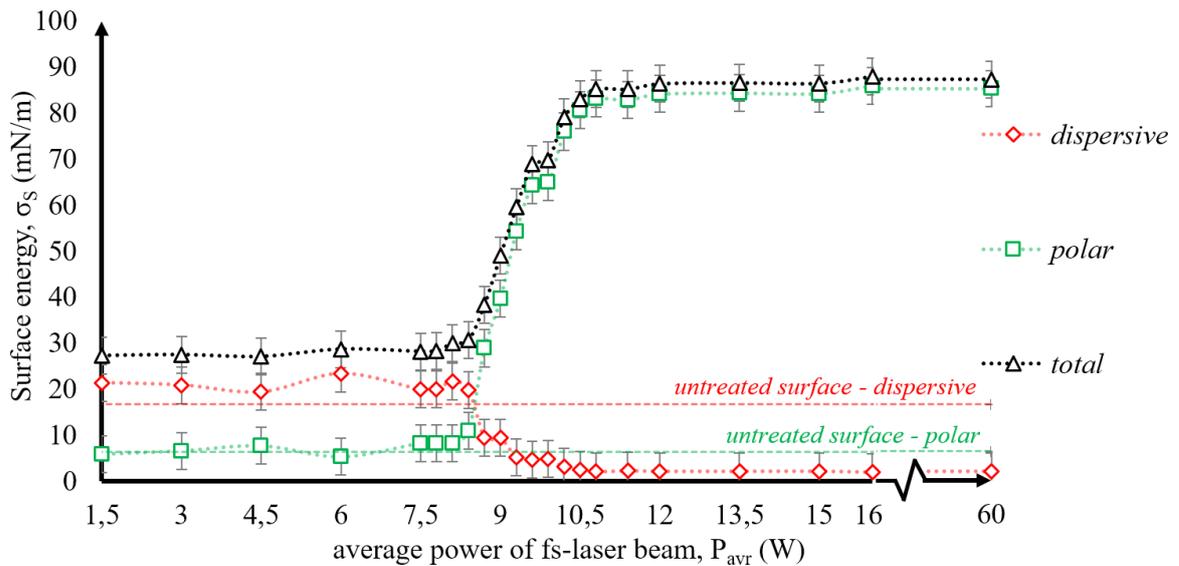
4. figure: Results on bond shear strength of untreated and plasma treated DP600 steel pairs

Untreated DP600 steel overlap bonded at  $6007\pm 734$  N, while specimens prepared with a feed rate of 0.07 m/min and a plasma surface treatment at a distance of 4 mm withstood  $13890\pm 400$  N. The shear strength for the untreated specimens was  $9.6\pm 1.2$  MPa, while for those surface-treated with plasma beam it was  $22.2\pm 0.6$  MPa, an increase of 231%.

#### **Experiment on ultrafast pulsed laser surface treatment of DP600 steel**

Each specimen was treated with different process data and after treatment, its wetting properties were investigated by measuring the contact angle with water and ethylene glycol. Based on preliminary experience, I chose a scanning speed of 300 m/min. The diameter of

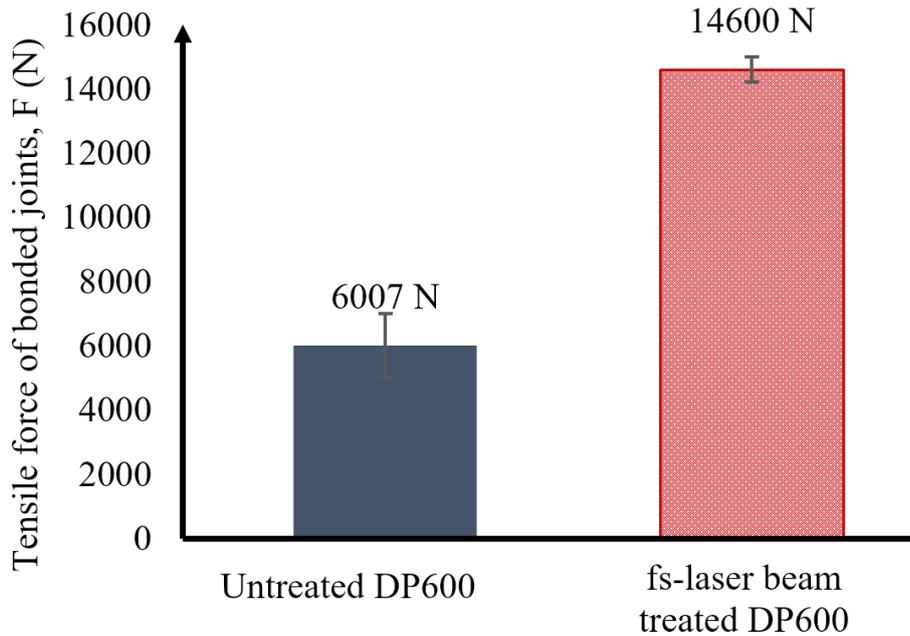
the laser beam on the scanned surface was 80  $\mu\text{m}$ . The laser beam source was applied at 188 kHz, with a pulse width of 300 femtoseconds and a step size of 60  $\mu\text{m}$  to ensure proper filling of the treated surface. The variable process data was the average power of the pulsed laser beam, which was measured at high resolution from 0 to a maximum of 60 W. The wetting tests were performed on the modified surfaces and the Fowkes equation system was used to calculate the surface energy variation due to each treatment using the measured boundary angles, as shown in (Figure 5). The values measured on the untreated surface are indicated by a horizontal line.



5. figure: Variation of surface energy on DP600 steel as a result of femtosecond laser beam surface treatment

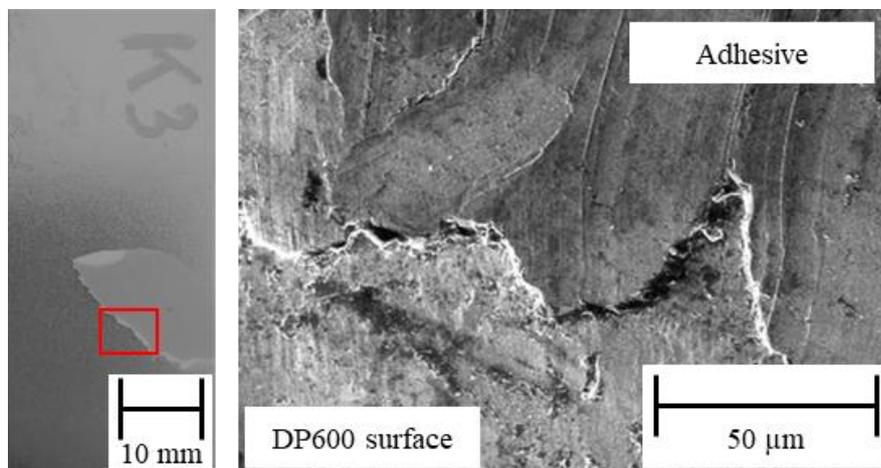
Figure 5 shows that as the average power of the fs-laser beam increases, the surface energy also increases. Compared to an untreated surface energy of 26 mN/m, the energy of a plate treated above 10 W average power increased to 85 mN/m. The best wetting was achieved at the treated surface for an average power of 51 W and this was defined as the optimum surface treatment data. Based on the results, it can be said that it was possible to improve the wetting properties with femtosecond laser beams.

I performed a bonding experiment on untreated and femtosecond laser beam treated DP600 steel plates. I performed bonding on untreated and 51 W treated plates. The untreated is the steel plate with poor wetting, the 51 W average power treated steel plate is the hydrophilic with the best area. Five pairs of each of the two types of plates were bonded together and tensile tested. From the tensile test results, Figure 6 shows that the shear strength of the bond more than doubled as a result of the treatment.



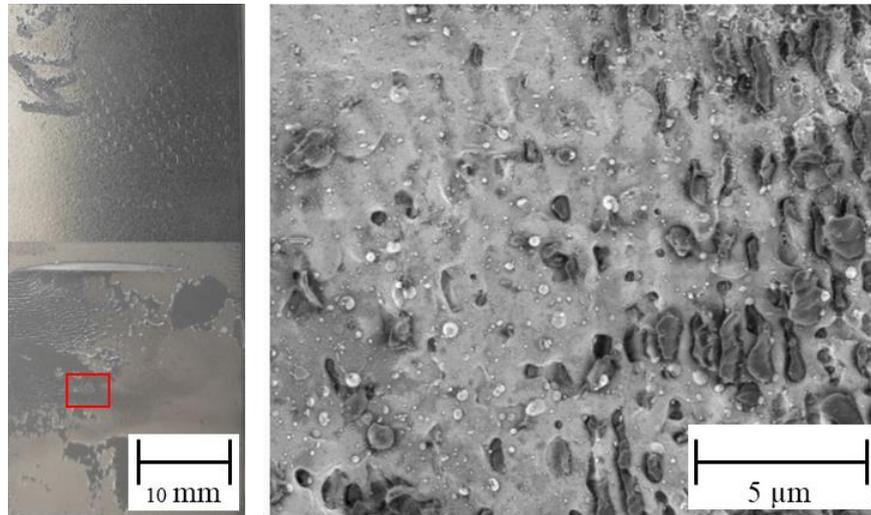
6. figure: Results on the bond shear strength of surface-activated DP600 steel pairs by femtosecond laser beam

In the untreated case, the 25 x 25 mm<sup>2</sup> lap joints ruptured at an average of 6007±734 N, while in the 51 W treated case they withstood 14600±288 N. The shear strength of the bonded joint was thus 9.6±1.2 MPa for untreated and 23.4±0.6 MPa for surface treated, an increase in shear strength to 240%. In addition to surface energies, I believe that the laser-induced periodic surface structures (LIPSS) on the surface also play a role in the development of tensile strength. In order to investigate the role of the surface in the formation of adhesion of the bonded joints, I used an electron microscope to examine the adhesive material remaining on the surface of the already torn specimens. As a basis for comparison, the surface of an untreated DP600 steel after rupture is shown in Figure 7.



7. figure: Untreated DP600 steel post-gluing tear image, SEM image location marked on the left specimen

In this case, the surface adhesion was significantly lower, so the adhesive was accidentally peeled off the DP600 steel surface in places. Neither any deformation of the steel surface during the adhesion of the adhesive nor any deformation of the adhesive on top of the adhesive in the lower region is visible. In contrast, a post-rupture image of a bond on a treated surface is shown in Figure 8.

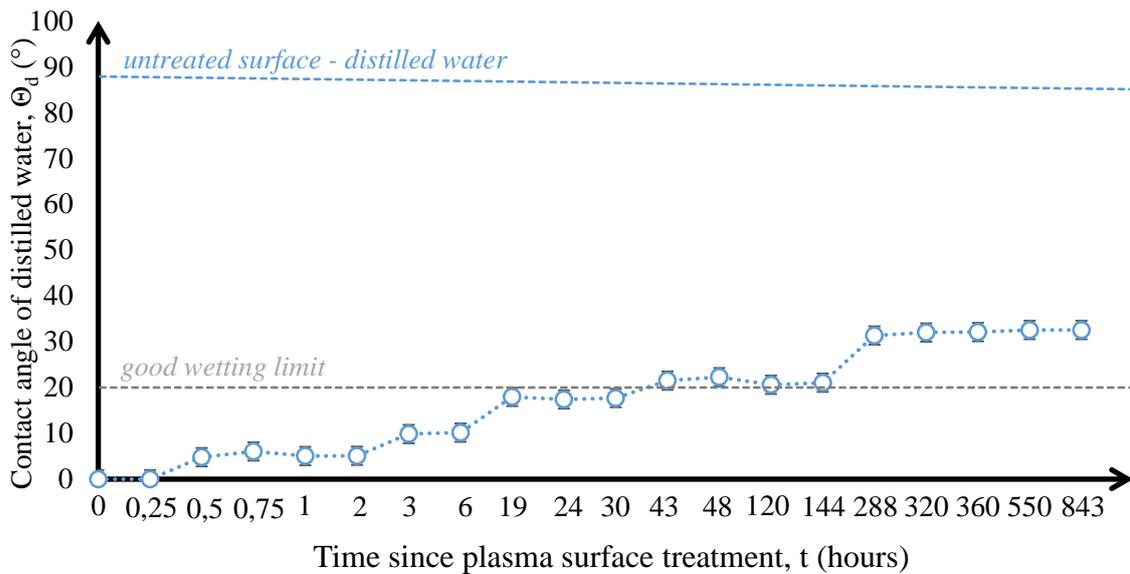


*8. figure: LIPSS post-bonding separation image of DP600 steel, SEM image position marked on the left sample*

It can be seen that the 2-component epoxy on the surface of the hydrophilic wetted surface and LIPSS modified DP600 steel has largely infiltrated and created adhesion bonding with the shape of the structure. The adhesive residue released during the tearing process shows that the adhesive has covered the structure. In the SEM image, the grey smooth surface patches are the residual adhesives, their longitudinal arrangement parallel to the shape of the LIPSS indicates the ability of the epoxy adhesive to flow into the microstructure and form an adhesion with it. This combination of strong adhesion and high surface area adhesion could increase the shear strength of the bonded joint of the overlapped samples.

### Investigating the time dependence of surface treatments

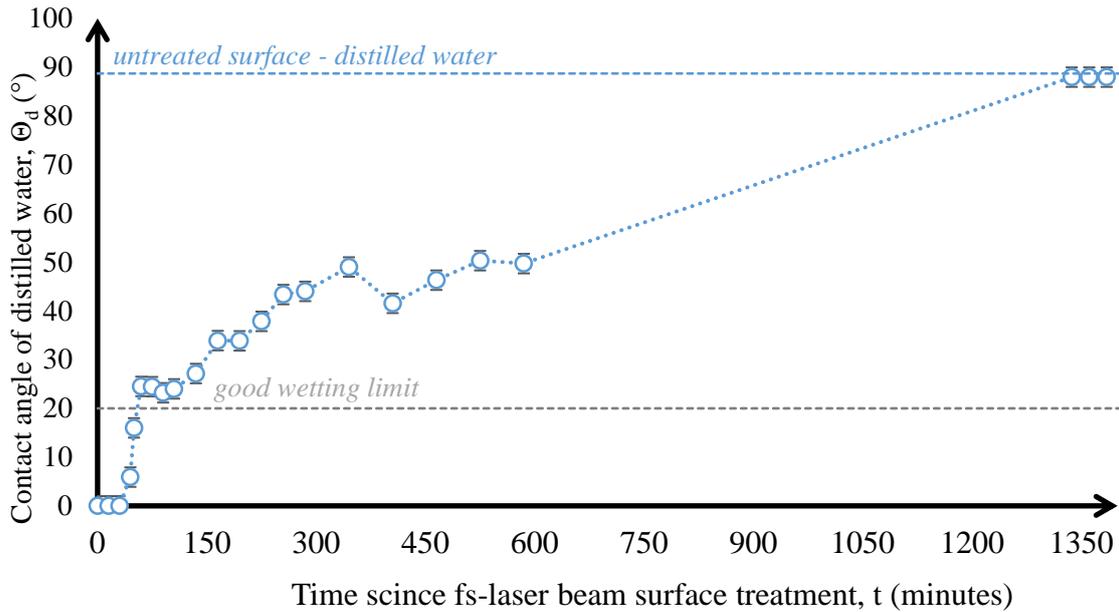
The plasma treated surface loses its good wetting properties over time, so I measured the duration of the treatment. For the test, water was dropped on pre-treated specimens prepared with the optimized treatment. After treatment, the specimens were stored in a dark, completely enclosed room at  $25\pm 2^\circ\text{C}$  and 50-55% humidity. A graph (Figure 9) plots the measured contact angle values versus time after treatment.



9. figure: Duration of action of plasma-treated HIPS from the hydrophilic state, measured with distilled water, on a logarithmic scale

As more time passed after the treatment, the water's edge angle began to increase. This could be because the free radicals on the surface started to react with the surrounding air, atoms started to sit in the empty spaces, in other words the surface started to become contaminated. The HIPS polymer passed the  $20^\circ$  or below range, which is considered a good wetting angle range, after 43 hours, so this may be the limit for the practical application of the surface treatment technology until bonding can be performed.

Based on literature results and experience, it can be stated that the effect of laser surface treatment also wears off with time. To find out how long the good wetting effect of a plate treated with an optimal average power of 51 W lasts, I carried out a test to measure the edge angle of the plate by dripping water at given intervals, the result of which is shown in Figure 10.



10. figure: Change in wetting angle of hydrophilic DP600 steel as a function of time

From Figure 10, it can be seen that the water is fully spread over the surface for 30 minutes and reaches the 20° contact angle in about 45 minutes. However, gradually over time, the good wetting condition is lost. Again, this can be due to two things, one is the deposition of dust and particles in the air on the surface and the other is the activated surface reacting with the air and thus gradually losing its functionality.

## 6. CLAIMS – NEW SCIENTIFIC RESULTS

New scientific findings in the field of the materials and devices under investigation, in the range of data presented in the thesis.

### 1. Claim:

Based on the study of the effect mechanism of the cold plasma and femtosecond laser beam processes, I found that for the same plasma surface treatment data (4 mm surface-to-plasma head distance and 7 m/min feed rate), a surface energy value of  $40 \pm 5$  mN/m could be achieved on DP600 and  $88.1 \pm 5$  mN/m on HIPS. The plasma beam technologies evaluated significantly enhanced the shear strength of the bonded joints through changes in surface energy on both HIPS and DP600 selected materials, while the femtosecond laser beam technology significantly improved the shear strength of the bonded joints only on DP600. Surface treatment with laser beam with a wavelength range of 1035 nm and a pulse duration of 300 fs to improve wetting was not achieved for the HIPS material [S2,S4,S6,S9-S12].

**2. Claim:**

I have determined the process data range with the highest surface energy using a high impact polystyrene cold plasma surface treatment technology. The optimized data defined are a plasma jet head feed rate of 7 m/min and a surface-to-plasma jet head distance of 10 mm at a maximum power of 1000 W. This configuration increased the surface energy to  $88.7\pm 5$  mN/m. The maximum shear strength of the adhesive bonds with overlapped joints increased from an average of  $0.54\pm 0.09$  MPa to  $1.61\pm 0.04$  MPa for the HIPS polymer with the plasma surface treatment tested compared to the untreated sample [S2,S6,S9-S11,S19].

**3. Claim:**

I evaluated the surface energy variation on DP600 high strength steel using cold plasma surface treatment technology at plasma head feed rates of 0.07, 0.35, 0.7, 2, 7, 12 and 17 m/min, surface-to-plasma head spacing of 4 mm and power of 1000 W. Using a feed rate of 0.07 m/min, I achieved a surface energy value of  $84\pm 5$  mN/m. I increased the maximum shear strength of the bonded overlapped joints of DP600 steel from an average of  $9.6\pm 1.2$  MPa untreated to  $22.2\pm 0.6$  MPa after surface treatment [S6,S10,S11].

**4. Claim:**

Using a femtosecond impulse laser, I have investigated the effect of process data on the wetting variation of DP600 high strength steel. I established the optimum process data to achieve hydrophilicity without aesthetic degradation using a laser beam with a wavelength of 1035 nm, a diameter of 80  $\mu\text{m}$ , a scanning speed of 300 m/min, a fill of 60  $\mu\text{m}$ , a repetition rate of 188 kHz, a impulse time of 300 fs and an average power of 51 W. The maximum shear strength of the bonded overlapped joint of DP600 steel with hydrophilic surface condition achieved by optimized laser beam surface treatment was increased from an average untreated  $9.6\pm 1.2$  MPa to  $23.4\pm 0.6$  MPa. I have shown that for untreated surfaces, the failure is adhesion failure, whereas for treated surfaces the failure is predominantly cohesive [S4-S7,S10,S12].

**5. Claim:**

I determined that both HIPS polymer and DP600 steel lose their hydrophilic state over time. When the treated surfaces were exposed to normal light conditions in air and stored at room temperature, starting from a hydrophilic wetting condition, the  $20^\circ$  wetting angle tested with distilled water was exceeded by the HIPS polymer in 43 h, while the DP600 steel exceeded it after 45 min [S2,S6,S9,S13].

## 7. LIST OF PUBLICATIONS RELATED TO THE PHD THESIS RESEARCH

- S1. M. Berczeli, G. Marsi, Analysis of the effect of different laser beam technology parameters when cutting stainless steel sheets with ytterbium fiber laser machine, *GRADUS*. 8 (2021) 11–323. <https://doi.org/10.47833/2021.1.ENG.015>.
- S2. M. Berczeli, Z. Weltsch, Enhanced Wetting and Adhesive Properties by Atmospheric Pressure Plasma Surface Treatment Methods and Investigation Processes on the Influencing Parameters on HIPS Polymer, *Polymers (Basel)*. 13 (2021) 901. <https://doi.org/10.3390/polym13060901>. IF=4,329 , FI=3
- S3. M. Berczeli, Wettability Changing of FINEMET Substrates Using High-Energy Femtosecond Laser Impulses, *ACTA Phys. Pol. A*. 137 (2020) 864-867 PG–4. <https://doi.org/10.12693/APhysPolA.137.864>. IF=0,545, FI=1
- S4. F. Tajti, M. Berczeli, Development of high power femtosecond laser microstructures on automotive stainless steel, *IOP Conf. Ser. Mater. Sci. Eng.* 903 (2020) 12025. <https://doi.org/10.1088/1757-899X/903/1/012025>.
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