SURFACE PREPARATION FOR APPLICATION OF STRUCTURAL ADHESIVES – AN OVERVIEW

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Abstract: The use of structural adhesives has increased in recent years as an alternative to intrusive bonding methods. In order for an adhesive joint to be successfully executed, it is necessary to prepare the surface of the substrate, which can be done by different methods. The objective of this work was to make an overview of the main existing surface preparation methods for adhesive bonding and the range of parameters of interest, such as roughness and wettability, that can be obtained.

Keywords: structural adhesives, adhesive joint, roughness and wettability, surface preparation methods.

PREPARAÇÃO DE SUPERFICIES PARA APLICAÇÃO DE ADESIVOS ESTRUTURAIS - OVERVIEW

Resumo: O uso de adesivos estruturais vem aumentando nos últimos anos como alternativa para métodos de união intrusivos. Para que uma junta adesivada seja executada com sucesso é necessário a correta preparação do da superfície do substrato, que pode ser feita por métodos variados. O objetivo deste trabalho foi fazer um apanhado geral os principais métodos de preparação de superfície para colagem adesiva existentes e a faixa de parâmetros de interesse, como rugosidade e molhabilidade, que podem ser obtidos.

Palavras-chave: Adesivos estruturais, junta adesivada, métodos de preparação de superfície, rugosidade e molhabilidade.

1. INTRODUCTION

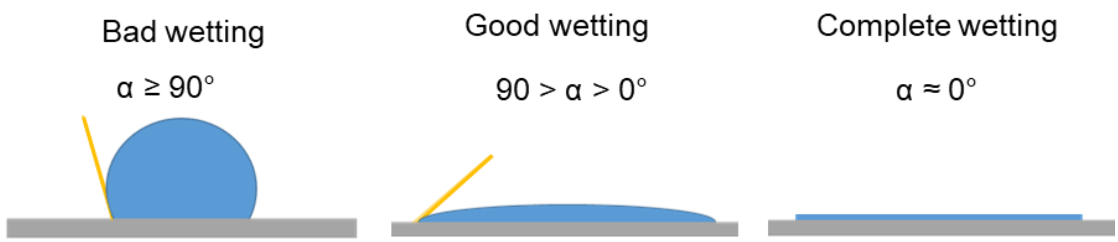
The use of structural adhesives has increasingly replaced conventional mechanical fastening systems (bolts, welds, and rivets). The acceptance of adhesives is by the industry comes from advantages as joining thick and/or thin shaped materials, similar and dissimilar materials, resisting fatigue, insulating against heat transfer and electrical conductance, minimizing electrochemical corrosion, uniform distribution of stress, good vibration absorption and hot work avoidance [1], [2].

However, adhesive bonding requires careful surface preparation due to low surface energy of polymers. Also, the environmental conditions can affect the joint quality [3]. For that reason, surface preparation is crucial to ensure good adhesion between the adhesive and the substrate, avoiding premature failure [3].

While the internal strength of the adhesive and the adherent are characterized and controlled, the interface between them is the result of a combination of mechanical, chemical, and physical interactions and its properties can be strongly affected by contaminants [3].

Surface cleaning and surface preparation are both important steps of an adhesion process, being crucial for the quality of the interaction of the surface with the adhesive as discussed by [3]. The adhesion capability is measured by a wide range of parameters for the characterization of the substrate surface that controls the interaction of substances such as roughness R_a or R_z, wettability and surface energy [4]. Wettability is a parameter that control the capability of adhesion of a surface by analyzing the contact angle formed by a droplet of distilled water. It characterizes the level of interactions of a substance and the capability of adhesion of the treated surface [5]. The surface wettability can be measured considering the contact angle shown in Figure 1. A desirable output when analyzing surface wettability is that the degrees get lower than 90° preferably as closer to 0° as possible [3],[5].

Figure 1. Example of wettability degrees



Source: [3]

On different literature, there is an optimum surface roughness for maximum strength in steel and aluminum joints [6]. Examination of the fracture surface when applied loads, made by SEM - Scanning electron microscopy, showed evidence of adhesive deformation after failure [6]. Surface roughness is linked also with wettability. The contact angle can increase continuously with higher surface roughness [7],[8]. For lower roughness values, the wettability is decreased as the surface energy is reduced and the result is a lower interaction between substrate and adhesive [7],[8]. A lower interaction between substrate and adhesive leads to interfacial failure instead of cohesive failure of adhesive [6].

Despite the higher contact angle, therefore lower wettability, the shear strength of treated surface joints tends to improve compared to the non-abraded adherend joints [9]. This implies that wettability does not interfere with supported shear loads meaning it cannot be explained only by increased roughness characteristics, such as mechanical interlocking, surface texture, and increased bonding area [6].

Even though surface roughness is an important parameter for joint strength, the proper selection of adherent-adhesive material combination should not be ignored for maximum performance of adhesively bonded joints. In addition, some specimens with similar average roughness values but altered morphology can show further improvement in the initial adhesion strength. The porous morphologies of grit blasted, and etched surfaces can show superior adhesion performance and appear to be decisive for triggering possible mechanical interlocking. Which results from the formation of a micro-composite interphase zone due to penetration of the adhesive into the surficial pores. [10]

2. ANALYSIS OF SURFACE PREPARATION METHODS

Certifying that the adhesion of the structural adhesive is correct is an important step to grant a safe and reliable use of systems that need high payload so, for this, is necessary to choose a surface preparation method that can change surfaces characteristics in an assertive and controllable way.

To find the ideal surface preparation method among the existing methods, there was an overview of the performance over different substrates while using different control parameters for obtaining an analysis of the surface roughness and wettability judging the quality of the adhesion through strength tests.

2.1. Milling

The process of milling is categorized as the condition of fabrication, but even in this process, the surface roughness can vary depending on rotations per minute (rpm) and the speed of the toll in mm per second for the removal of the substrate [11]. The variation of the parameters of milling was observed by [11] as presented, at each condition on a substrate of Ti-6Al-4V, and the surface roughness (R_a) were observed, as presented at Table 1.

Table 1. Milling roughness

Milling	Roughness R_a (μm)	Wettability (degrees)
v=10mm/min and 4000rpm	0.135	98.3°
v=40mm/min and 1000rpm	0.173	92.7°
v=60mm/min and 500rpm	0.546	94.7°

Source: [11]

2.2. Polishing

The process of polishing or sanding is a common procedure for preparing the surface for adhesion. It has a diverse variation on sandpaper grades, which usually are used in sequel with different grades. Other than the sandpaper grades the process can be controlled by the pressure applied and the speed of grinding (rpm) [11]. In [11] research it can be seen an increase in roughness and a lowering in wettability as the grade of the sandpaper (data in Table 2).

Table 2. Polishing roughness

Sandpaper	Roughness R_a (μm)	Wettability (degrees)
P600	0.162	83°
P320	0.187	73.7°
P220	0.446	66°

Source: [11]

In [12] is shown the performance of the process using a disk sander, on a surface of ASTM A36 and achieved the best performance in tests using a tensile strength, following ASTM D 4541-02, using the lower grit size as presented at Table 3.

Table 3. Disk sander Roughness

Surface preparation	Surf. Profile R_a (μm)	Tensile strength (MPa)
Disk sander (36)	6.6	9.6
Disk sander (60)	4.3	5.5
Disk sander (80)	2.6	5.9

Source: [12]

2.3. Blasting

Blasting treatments are commonly used with a vast range of blasting components such as steel shots, steel grit, aluminum cut wire, zinc cut wire, brown corundum, glass beads, baking soda, or plastic soda abrasive, each one has different proposes for applications [13]. With the objective to prepare the surface for adhesion changing the surface profile on a smaller scale and removal of oxides for adhesion proposes studies with glass beads and soda abrasives, such as the ones shown by [11],[14],[15],[16],[17].

The control over the effectiveness of the change in the surface is based on the distance of blasting, the pressure of the jet, and the size, hardness, and speed of the particle. The faster and bigger the size of the particle, bigger changes occur in the substrate worked directly to the energy disposed of by the blast [13],[14].

The effect of this treatment on a substrate of ASTM A36 in the surface roughness parameter and the performance of adhesion of an epoxy resin culminated in the characteristics presented in Table 4.

Table 4. Blast characteristics

Surface preparation	Surface Profile Ra (μm)	Tensile strength (MPa)
Garnet (16-40)	23.9	13.6
Garnet (30-60)	20.9	14.7

Garnet (80)	22.1	14.3
Garnet (120)	16.5	12.7

Source: [15]

The data presents an optimal performance at the garnet size of 30-80 and shows an influence of the roughness of the surface in the performance of the adhesive but more parameters need to be analyzed for better judgment.

2.4. Dry Ice Blasting

Dry ice blasting treatment is based on the difference of dilatation from materials present on the substrate surface removing rust and paint while changing the surface characteristics such as surface roughness [18],[19]. The pellets are at temperatures of $-78{,}5\ °C$ so, using this treatment needs precautions because of the risk of surface fracture [18],[19]. The parameters of control are the ice pellet size (IPS), speed of the pellet jet pressure, and distance of jet [18],[19]. A study, made by [18], analyzed the performance of a surface of AlMg4,5Mn0,4 in adhesive interactions of 3 different Two-components using dry ice blasting varying control parameters and then submitting the treated surface to a shear strength test. In more detail, the surface was mapped before the shear test and showed the performance presented at Table 5. There is also a parameter of wettability that can be obtained of 87.3°, in Aluminum Surfaces.

Table 5. Surface characteristics of dry ice treated surface

$v\ (m/min)$	IPS(mm)	$R_a \pm s\ (\mu m)$
0.1	0.5	0.43 ± 0.15
0.5	0.5	0.42 ± 0.11
1.0	0.5	0.41 ± 0.11
0.5	0.1	0.43 ± 0.07
1.0	0.1	0.46 ± 0.10

Source: [18]

2.5. Laser Ablation

Laser ablation is a new process introduced in the procedures of surface cleaning or preparations of steel substrates, which usually are composed of chemical or mechanical treatments [3]. This process uses irradiation for changing the interface substrate by heating the undesirable substances while changing the surface characteristics [20]. In some cases, for preventing the heating process of the surface the laser is pulsed at high frequencies. Some studies made by [20],[21], and [22] show the performance of laser ablation in different scenarios such as in the removal of micro biofouling and performance in adhesion in flat, dimple, groove, and grid patterns.

When analyzing the process of preparation of the surface for adhesion the laser energy density is the control parameter, as well as the diameter of the laser head [22]. The process using laser influence between $0{,}17\ to\ 5{,}71\ J/mm^2$ in surfaces of AA 6082 T6 and AISI 304 was mapped by [22] in parameters of roughness and performance on lap shear strength test and the data is presented at Table 6.

Table 6. Laser-cleaned surface characteristics

Density (J/mm²)	Diameter (μm)	Substrate	Roughness Ra (μm)
0.14	50	AA 6082 T6	0.98
0.51	50		4.24
0.51	100		2.60
1.14	50		5.18
5.71	50		11.78
0.14	50	AISI 304	1.14
0.51	50		0.94
0.51	100		1.31
1.14	50		1.81
5.71	50		7.54

Source: [22]

After cleaning the substrate, [22] submitted the treated substrate to a shear strength test and all energy densities tested had a better performance than the usual ones such as degreasing and grit blasting.

In the case of laser cleaning the best performance, with a cohesive failure mode, was with the energy densities of $0{,}51\ and\ 1{,}14\ \ J/mm^2$ that had the best performance, with shear strength values varying from $41.61 \text{ to } 46.02\ \text{MPa}$, showing of the best substrate/adhesive interaction with a roughness of 0.94 to 1.81 µm at a steel substrate.

3. DISCUSSION

The surface characterization, such as R_a, has great importance on adhesion conditions so it is very relevant that the treatment used not only removes impurities (rust, biofouling, etc.) but also changes these values. The change of surface characteristics is dependent on the control parameters of the treatment and substrate of work. After analyzing those characteristics each treatment has an R_a achieved derived from each condition the surface was exposed.

The surface generated by milling is considered a surface with poor wetting, which is not ideal for adhesion. With the increase in the velocity of the tool, this parameter is even worse, as shown in Table 1.

As pointed out by the characterization of wetting of the surface, the treatment by alkaline etching and acid cleaning was the most efficient in the adhesion in comparison to the substrate cleaned by degreasing, while also presenting a lesser variation of the data. It was seen that the time of attack changes the surface profile for a rougher as time increases.

In comparison between surface treatments of steel substrate, it was noted that polishing had a better performance than laser ablation in achieving a lower surface roughness. But as this isn't the only relevant parameter, for a better comparison it should be done more test studies of each treatment using the same substrate and adhesive for better evaluation of this procedure.

Although the authors use different control parameter for qualification of adhesive bonding, after obtaining an adequate surface roughness the principal value is the wettability degree as can be seen at Table 7 that compiles the best results from each test the treatment that had the best result was the laser ablation judging by nanometric surface roughness and one of the lower wettability obtained.

Table 7. Treatment performance overview

Treatment	Wettability (degree)	Roughness (µm)
Milling	92.7	0.173
Polishing	66	0.446
Blasting	19.7	20.9
Dry Ice	87.3	0.41
Laser Ablation	25	0.94

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