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Free surface energy evaluation in the laser texturing of a carbon steel s275

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Abstract

Currently, the manufacture of hybrid structures of dissimilar materials is generating great interest. These allow to combine mechanical properties of different materials to achieve a higher functional performance. To obtain these structures, different joining technologies are used, especially the use of adhesives. Nevertheless, for the correct application of an adhesive, the surface of the metal to be bonded must be prepared. Laser texturing has become a widely studied technology for this purpose because it allows very defined textures to be obtained. This technology allows to activate the surface and improve the application of the adhesive afterwards. In that sense, hydrophilic surfaces are generated and evaluated by wettability tests to know the surface free energy obtained in the texturing.

In order to improve the bonding of dissimilar materials in the form of hybrid structure, a study has been carried out on the laser texturing of a carbon steel S275 and the relationship between the texturing parameters with its surface activation through wettability and surface free energy tests. Thus, the energy density obtained by the combination of power and scanning speed allows the generation of very defined textures that increase the surface activation of the steel. This has made it possible to establish a mathematical relationship between the texturing parameters and the results obtained.

Keywords:

1. Introduction

The industrial sector is currently searching to use high-performance materials in order to obtain high quality products. This is the case of the civil sector, which seeks to apply light and high-strength structures to replace the materials used in recent years. These structures must have an excellent ratio between weight and mechanical properties, achieving a high functional performance (Sheng, Jiao, Du, Wang, & Wang, 2018).

A new application of these materials is thus emerging in the form of hybrid structures. These structures consist of joining dissimilar materials to combine the advantages they offer separately. One example is the joining of steel alloys with carbon fiber reinforced thermoplastic matrix composites. (Jiao, Wang, Wang, Zan, & Zhang, 2017; Jiao, Xu, Wang, Sheng, & Zhang, 2018). This kind of hybrid structure is of great interest for the construction sector as it combines the mechanical properties of steel with the lightness of the composite material.

Within the wide range of carbon steels this work is going to focus on the study of a structural steel S275. This kind of steel is called low carbon structural steel and is widely used in various industrial sectors due to its mechanical properties (Rodriguez-Vidal et al., 2020). It is a steel with a minimum yield strength of 275 N/mm^2, being the carbon steel with the highest yield strength among the most common ones (Aldeeb T., 2018; Çevik, 2018).

Nevertheless, the process of joining two materials of different nature becomes a challenge that must be controlled and improved. In this sense, the correct surface preparation of the steel prior to joining is an essential step. Several publications highlight the importance of activating the surface of the metal in order to obtain a hydrophilic surface, which allows the adhesive to spread over the entire surface, thus achieving a quality bond (Campos Bernardes et al., 2012; Islam, Tong, & Falzon, 2014). Several methods are used for this activation, being laser texturing one of the most studied in recent years due to its speed, flexibility and control of the

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surface obtained (Vazquez Martinez, Del Sol Illana, Iglesias Victoria, & Salguero, 2019).

This proposal aims to activate the surface of a carbon steel S275 by laser texturing through an experimental design (Wu et al., 2017). The objective is to perform a parametric study in order to activate the surface of the steel and determine which combination of cutting parameters generates a higher surface free energy. Contact angle evaluation tests in wettability tests will be performed to determine the surface free energy generated (Lawrence & Li, 1999).

2. Methodology

In order to increase the surface activation of an S275 carbon steel (Table 1), laser texturing was performed on different samples by means of an experimental design.

Table 1. Characteristics of the carbon steel used.

%C	%Mn	%P	%S	%Si
0.25	1.60	0.04	0.05	0.05
Yield Strength (MPa)	**Tensile Strength (MPa)**	**Thickness (mm)**		
275	450	3		

For this purpose, a Rofin Easymark F20 Ytterbium laser texturing machine was used (Table 3). Different levels of power and scanning speed have been modified based on previous tests. This experimental design seeks to establish a relationship between these parameters and the surface activation of the steel and to determine an optimal combination. The parameters used in the texturing by means of an experimental design are shown in Table 2. All tests were performed with a wavelength of 1062 nm and a focal length of 185 mm, resulting in a spot diameter of 60 µm and a distance between lines of 1 µm. The texturing area has been 10 mm x 10 mm and all samples have been generated maintaining a bidirectional shading distribution without laser beam overlapping in a pattern of straight parallel lines.

Table 2. Parameters used for laser texturing of surfaces.

P (W)	F (KHz)	Ed (J/cm^2)	Vs (mm/s)
5	20	8.84	10 100 250
10	20	17.68	10 100 250
20	20	35.37	10 100 250

After the texturing tests, the surface free energy generated in all the samples was determined by wettability tests. The contact angle between liquid and solid phases is measured by depositing a drop on the horizontal surface.

For this purpose, a phase contact angle measurement system was used (Figure 1), consisting of a high-resolution CCD camera positioned on the axis that crosses the drop, while a back illumination point provides contrast to capture the geometry.

Table 3. Characteristics of the laser equipment used in the methodology.

Maximum power (W)	20
Maximum workpiece weight (kg)	10
Maximum texturing area (mm^2)	120x120
Z-axis displacement (mm)	120

To perform the test, a 2µl droplet is deposited on the textured area and the contact angle is determined using image processing software (Figure 2). The shape that the droplets can take when deposited is of great importance in the study of the wettability of the surface.

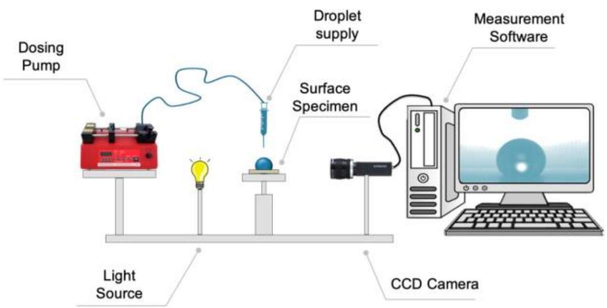

Figure 1. Graphical representation of the equipment used for the contact angle measurement (Vázquez Martínez, 2016).

Contrary to the wetting test, where only one drop of a known liquid is deposited, to determine the surface free energy, it is necessary to apply three liquids of different polarity in order to determine the surface free energy (Cai et al., 2018; Pou et al., 2019).

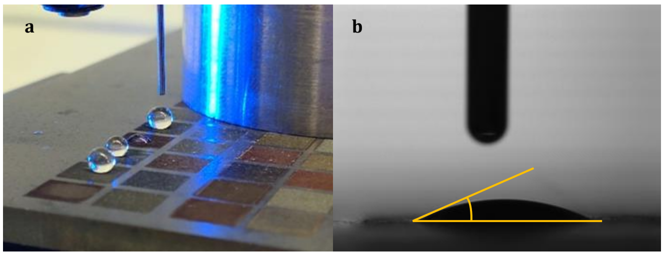

Figure 2. Contact angle measurement process using image processing software: a) Deposition of the liquid on the textured surface; b) Measurement of the contact angle.

In order to calculate this value, the Owens-Wendt-Rabel-Kabele (OWRK) equation is used. This method applies the evaluation of the contact angle by applying various liquids to the surface of the solid, which have different polarities. Therefore, the total polar component of all liquids and their free energy dispersion are obtained, the final value being the sum of both.

By using equation 1, the surface free energy of a solid that has been surface modified can be calculated:

$$\sqrt{\gamma_{SV}^{d}} + \sqrt{\gamma_{SV}^{p}}\sqrt{\frac{\gamma_{LV}^{p}}{\gamma_{LV}^{d}}} = \frac{1}{2}\frac{[\gamma_{LV}(1+coscos\,\theta\,)}{\gamma_{LV}^{d}} \quad (1)$$

The presented equation has a structure close to a linear equation of the type y=mx+b where m=γ_{SV}^{p} y b=$\sqrt{\gamma_{SV}^{d}}$. On the other hand, being tabulated liquids, the values of γ_{LV}, γ_{LV}^{d} y γ_{SV}^{p} are known values. Thus, by determining the contact angle of the liquids used and, through their graphical representation, the polar and dispersive component of the modified solid can be calculated.

Thus, the liquids used for the evaluation of this parameter consisted of distilled water, diiodomethane and ethylene glycol, whose polarity values were previously known (Table 4). For each evaluation test, three replicates were carried out with a percentage variation of less than 10%.

Table 4. Polar and dispersive components of liquids used to evaluate surface free energy.

	γ_{LV} (mJ/m2)	γ_{LV}^{d} (mJ/m2)	γ_{LV}^{p} (mJ/m2)
Distilled water	72.8	21.8	51
Diiodomethane	50.8	50.8	0
Ethylene glycol	47.7	30.9	16.

3. Results

Figure 3 shows the evolution of the contact angle when depositing a drop of water on the textured surface of the steel at a fixed power of 10 W and increasing the scanning speed of the laser.

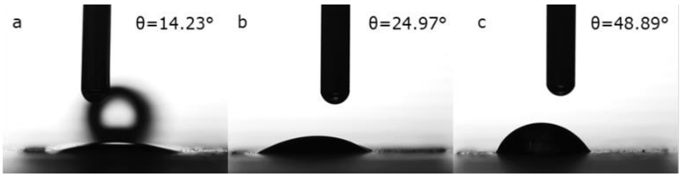

Figure 3. Contact angle obtained for a power of 10 W and: a) Sweep speed of 10 mm/s; b) Sweep speed of 100 mm/s; c) Sweep speed of 250 mm/s.

The influence of the scanning speed parameter on the modification of the steel surface is appreciated when generating a hydrophilic surface when using a speed of 10 mm/s compared to the increase in the contact angle when using a speed of 250 mm/s. This is due to the fact that the time the laser beam is incident on the surface is considerably reduced as the speed increases, reducing the ability to penetrate the material and generating a less rough surface. This decreases the ability of the liquid to expand over the channels created by the laser.

On contrary, for a given speed, increasing the laser beam power generates a similar result in the formation of a hydrophilic or hydrophobic surface (Figure 4).

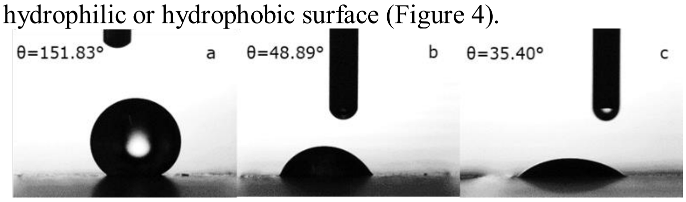

Figure 4. Contact angle obtained for a scanning speed of 250 mm/s and: a) 5 W power; b) 10 W power; c) 20 W power.

Thus, for a power of 5 W at the maximum scanning speed, the ability of the laser beam to modify the surface is considerably reduced. With this combination of cutting parameters, the existing interaction between the substrate surface and the liquid (γSV< γSL) is being reduced, creating a higher surface tension that repels the liquid on the surface itself. Thus, an unsuitable surface for adhesive deposition is being generated since, by generating this surface tension, the adhesive is being prevented from fully expanding on the surface and filling the cavities generated by the laser.

On the other hand, if the laser power is increased, its ability to penetrate the material and modify its surface increases significantly, generating deeper and more defined cavities. This causes a strong interaction between the substrate and the liquid, allowing a reduction of the surface tension and a reduction of the contact angle obtained (Yang, Deng, He, & Özel, 2021).

Thus, in order to seek the best possible wettability of the surface and to obtain a maximum surface free energy and a better mechanical anchorage of the adhesive, a power of 20 W with a sweeping speed of 10 mm/s should be used. This is shown in Figure 5 where the surface difference obtained for the most unfavorable combination compared to the optimum combination in terms of wettability is shown.

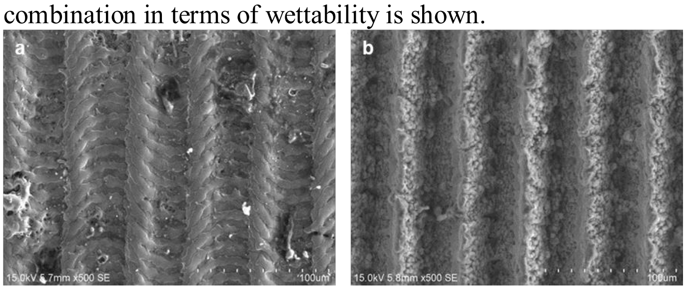

Figure 5. SEM images of the surface obtained for: a) 5 W and 250 mm/s; b) 20 W and 10 mm/s.

The wettability of the steel for each texturing test is shown in Figure 6. The influence of the texturing parameters on the surface morphology and on the surface tension generated is shown in this graph.

On the one hand, an increase in power provides greater energy to the laser beam allowing a greater amount of material to be fused and removed, generating a more porous and rougher surface. This results in a lower surface tension and a decrease in the contact angle. Thus, for the three applied scanning speeds, a constant reduction of the contact angle is observed when the power is increased from 5 W to 20 W.

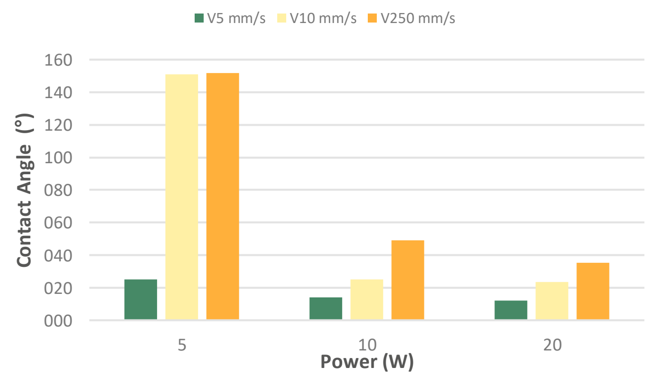

Figure 6. Contact angles obtained for laser texturing tests.

On the other hand, this energy density is in turn influenced by the scanning speed of the laser itself. In the results obtained, an increase in this speed produces a smoothing of the surface because the laser energy is reduced in each pass. The case of a power of 5 W is particularly noteworthy. For this power, the contact angle increases from 20° to almost 160° as the scanning speed increases, changing from a hydrophilic surface to a hydrophobic surface. Therefore, if the aim is to generate a surface activation on the metal, reduced scanning speeds should be combined with powers close to 20 W to reduce the surface tension and increase the surface porosity.

Finally, by means of the contact angle values obtained when depositing the three liquids of different polarity on the textured surfaces and by means of the OWRK equation, the surface free energy value for each texturing can be determined (Figure 7).

Surface free energy values are related to the surface activation of the metal for subsequent bonding with other materials. Textured surfaces or surfaces modified by different technologies allow a better mechanical anchorage of the adhesive enhancing the manufacturing of hybrid structures in the industry (Bañon, Sambruno, Batista, Simonet, & Salguero, 2020, 2021).

The relationship between the wettability of the surface and its surface activation is established. Thus, sweeping speeds of 10 mm/s are those that produce a higher surface activation with surface free energy values that exceed 60 mJ/m^2 when applying a power of 20 W. This combination gives a higher energy density to the laser beam, reducing the surface tension of the steel and creating a more porous surface. This allows the deposited liquid to fully expand on the surface, indicating that it presents a surface suitable for the application of subsequent adhesives.

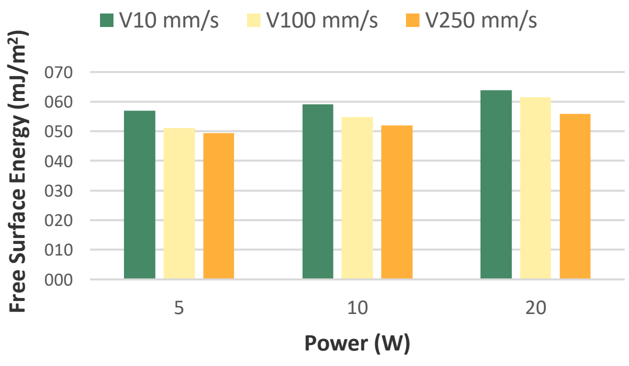

Figure 7. Surface free energy values for laser texturing tests.

On the other hand, the relationship between hydrophilic and hydrophobic surfaces with surface active or not very active surfaces is corroborated. For the same power, the increase of the sweeping speed generates a smoother surface with a minimum porosity which increases the contact angle of the deposited liquid. This is corroborated in the surface free energy values which decrease with this increase in speed.

Therefore, in order to prepare metal surfaces for subsequent bonding operations by laser texturing, it is required to combine a power of 20 W with a scanning speed of 10 mm/s in order to generate a hydrophilic surface with a high porosity that favors the expansion of the adhesive and improves the mechanical anchorage.

In order to establish a statistical influence, an ANOVA statistical analysis has been developed (Table 5). Thus, the modified parameters show a statistical significance in relation to the surface free energy. Power is the most dominant parameter, followed by sweep speed. This corroborates the influence of the laser beam power and its penetration capability in generating deeper and well-defined cavities.

Finally, a response surface model relating this parameter to the input variables has been obtained with a fit of 97.82% (Figure 8). It is corroborated that, maximum values of surface free energy are obtained for a combination of a power close to 20 W and a sweeping speed of 10 mm/s.

Table 5. ANOVA analysis for surface free energy results

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	181.122	36.2244	26.94	0.011
Power (W)	1	90.661	90.6615	67.43	0.004
Sweep speed (mm/s)	1	84.878	84.8783	63.13	0.004
Error	3	4.033	1.3444		
Total	8	185.155			

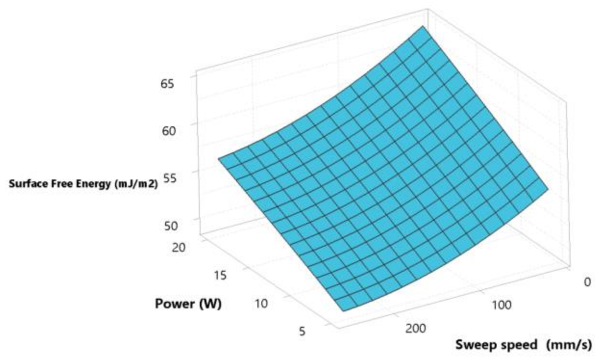

Figure 8. Response surface model relating surface free energy to power and sweep speed

4. Conclusions

A parametric laser texturing test has been performed on a carbon steel S275 in order to evaluate the surface activation prior to joining operations in terms of wettability and surface free energy.

The results obtained corroborate the effectiveness of laser texturing as a surface activation process. This allows to increase the surface free energy of the steel and to enhance the final bonding quality with other materials of different nature in the form of hybrid structures. The importance of a correct selection of parameters to generate a defined texture that allows the subsequent expansion of the adhesive for a better mechanical anchorage is confirmed.

The importance of scanning speed and power in the laser beam to obtain hydrophilic or hydrophobic surfaces has been determined.

Increasing the scanning speed reduces the penetration capacity of the laser, reducing the porosity obtained and generating smoother surfaces that increase the contact angle values and reduce the surface free energy.

Increasing the power gives a higher energy density to the laser beam allowing more material to be melted and generating more defined cavities with higher porosity. This in turn increases the surface activation of the steel in terms of surface free energy indicating that there should be more mechanical anchorage when applying an adhesive to this surface.

Finally, the combination of texturing parameters that generate a higher surface activation on carbon steel is a sweep speed of 10 mm/s with a power of 20 W.

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