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Influence of cold plasma treatment parameters on the mechanical properties of polyamide homogeneous bonded joints

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ABSTRACT

This study reports a systematic investigation of the effects of various cold plasma treatment parameters on adhesion characteristics of two polyamide substrates.

Polyamide 6 and polyamide 6.6 specimens were treated with a low pressure radio-frequency discharge plasma using different treatment time, power inputs and working gas (air, argon and oxygen). Contact angle measurements with sessile drop technique were carried out for estimation of surface wettability, as well as surface roughness evaluation and X-ray Photoelectron Spectroscopy (XPS) analysis. Then, untreated samples and cold plasma treated samples were adhesively bonded together to form overlap joints. Single lap shear tensile testing of these adhesively bonded joints was performed to investigate the effect of different surface treatments on the joint strength.

The experimental results show that the optimized plasma process may remarkably increase wettability properties of polyamide surface and shear strength of the bonded joints.

1. Introduction

Adhesive bonding techniques present a large number of benefits and advantages compared to mechanical fastening and welding processes: no stress concentration or thermal stresses in the joints; complete sealing of the joint area; excellent aesthetic finishing of the structure with the absence of holes or extra material on the substrates; possibility of realizing joints with controllable and repeatable properties as a function of substrates, adhesive and geometry of the joint; flexibility to join almost any material regardless of type, shape or form [1–4].

All these aspects make this technique an optimal choice for a type of substrates increasingly used in industrial applications: polymeric materials.

Polymer based materials have been applied successfully in several industrial applications and they are gradually replacing metallic and ceramic materials in many industrial sectors, from electronics, to food, up to automotive and aerospace industries [5]. Plastic materials have good mechanical properties compared to their specific weight, high chemical inertness as well as a good processability and low manufacturing costs.

In particular, thanks to their high mechanical properties under severe service conditions, polyamides are used for the manufacture of machine components, such as bearing cages, pneumatic connectors or to make a variety of fixing products, such as cable ties, fasteners, staples and drills [5,6].

However, they have some disadvantages, such as poor wettability, printability, and low adhesion properties, due to the hydrophobic characteristics of their surfaces. Surface properties, such as chemical composition, hydrophilicity and roughness are often mandatory for successful applications. Surface treatment techniques, which can modify plastic in order to create finished products, have become an important part of the research for polymer industries [7–9].

For this kind of substrate physical or chemical treatments such as sanding and solvent wiping are usually applied in order to remove the glossy finish and all traces of dirt, grease, mould release, or other contaminants from the bonding surfaces. To overcome both the possible surface contamination and the environmental problems, new surface treatment methods based on physical principles have been developed to introduce oxygen-containing functional groups.

These methods, such as corona and plasma discharges [10–15], ultraviolet irradiation [16,17], and electron bombardment have been used in order to introduce polar groups onto the polymer surfaces and increase their surface energy and wettability.

In particular cold plasma treatment has proven to be an excellent, efficient and eco-friendly alternative to traditional methods for many substrates. A treatment in cold plasma allows obtaining more reactive surfaces, facilitating the adhesion between material and adhesive; furthermore, the mechanical properties of the base material are not affected due to the low operating temperature [18–24].

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In spite of this, literature on polyamide plasma treatment is not very wide and it mainly focuses on the surface modification of thin films or fibres [25–27].

Tušek et al. in their work evaluated surface modification of polyamide 6 foils as a function of treatment time with NH_3 plasma treatment. Their results showed that the introduction of N-containing groups increased with longer treatment time only to a certain extent where the negative effect of surface destruction prevails over the positive effect of introduction of functional groups [28].

Etching rate of polyamide 6 films was examined by Gao et al. investigating various plasma parameters, such as treatment duration, output power, oxygen gas flux, jet to substrate distance, and moisture regain, using atmospheric pressure plasma. The etching rate increased as the output power, oxygen gas flux, and moisture regain increased. As the treatment time increased, the etching rate first increased and then decreased. T-peel strength showed an improved bonding strength between the PA 6 films and an adhesive tape after plasma treatment [29].

Hnilica et al. treated polyamide 12 films using a microwave plasma jet at atmospheric pressure. It was found that significant change in wettability is achieved very rapidly, after only 25 ms of treatment. It was concluded that the increase in wettability was caused by both chemical and morphological changes [30].

The aim of this study was to evaluate the possible effect of a cold plasma treatment on the adhesion characteristics of two kind of polyamide 2 mm-substrates.

The first step was the identification of the machine parameters (power, exposure time to the treatment and the process gas) that allow adhesively bonded joints characterized by high mechanical strength to be obtained.

To assess the effects of the plasma treatment four types of tests were performed: the surface wettability, then the tensile test of the joint realized with differently treated substrates, the surface roughness evaluation and finally the X-ray Photoelectron Spectroscopy (XPS) on untreated and plasma treated material.

2. Materials and methods

2.1. Materials

The polymers used as substrates were polyamide 6 and polyamide 6.6, supplied in 2 mm-thick sheets. Samples were cut to 100 mm × 25 mm, in order to allow all the tests, both on surfaces and on the correct joint configuration, according to ASTM D3163 standard [31]. The samples were all cleaned using acetone before any kind of surface treatment.

A bi-component acrylic adhesive supplied with commercial name of DP8810 by 3M™ was used to realize bonded-joints. In Table 1 some technical information is reported.

It contains glass microspheres of 0.25 mm-diameter, so that the thickness of adhesive remains constant throughout the joint. Its suggested full cure time is between 8 and 24 h but, since this time is greatly influenced by environmental conditions, it was decided to wait about 72 h before testing. In this way there was the certainty that the adhesive was completely cross-linked, ensuring the greatest adhesion strength of the joint.

Table 1
Technical information about the acrylic adhesive used for the bonded joints.

Property	Component	
Colour	Base (B)	Off-white
	Accelerator (A)	Blue
Viscosity	Base (B)	90,000 cP
	Accelerator (A)	35,000 cP
Density	Base (B)	1.08 g/cm3
	Accelerator (A)	1.08 g/cm3
Mix ratio (By volume)		10 Parts B: 1 Part A

2.2. Surface pre-treatment

Plasma treatment was carried out in a radio frequency (RF) low pressure plasma. In particular, a glow discharge RF generator operating at 13.56 MHz (Gambetti Kenologia, Italy) was used. The chamber volume is approximately 5.5 l and the chamber dimensions were a diameter of 150 mm × 330 mm in length.

The power, as measured on the RF supply, can be selected in the range of 10–200 W. The operating conditions led to little or no significant heating of the plates on their removal shortly after the plasma was extinguished. The plasma can work with different gases. Here, air and standard grade argon and oxygen were used as working gases and a flow rate of 0.025 SLM was employed. The process pressure was set to 0.6 mbar.

In order to investigate in depth the effect of plasma treatment on polyamide substrates, different set-up parameters were employed and compared to abrasion standard treatment, that is the one suggested by ASTM D2093 standard. In Table 2, a summary of all the treatments performed is reported.

2.3. Evaluation of contact angle

A calibrated water drop of 4 μl was released on the substrate surface. All the measures were performed using a Leica Digital Microscope and X-Pro Software. Ten drops were measured and averaged on the samples treated. The measurement was carried out at room temperature.

2.4. Lap shear test

Rectangular specimens, having dimensions 100 mm × 25 mm × 2 mm, prepared with different types of treatment and parameters, were adhesively bonded in order to evaluate adhesion properties of the polyamide surfaces through single lap-shear tests.

The test was carried out using an Instron Testing Machine at a test speed of 1.3 mm/min. Geometry of the test specimen (Fig. 1) and test conditions followed ASTM D3163 standard [31]. For each sample, five

Table 2
Surface treatment performed in this study.

Surface treatment	Description	
Degreasing (D)	Acetone wiping	
Abrasion (DA)	Acetone wiping + P240 grain carbide paper abrasion + acetone wiping	
Plasma	Acetone wiping + plasma discharge (with different set-up parameters)	
Set-up parameters	Power input (W)	Exposure time (s)
	50, 150, 200	10, 60, 180, 300, 450, 600

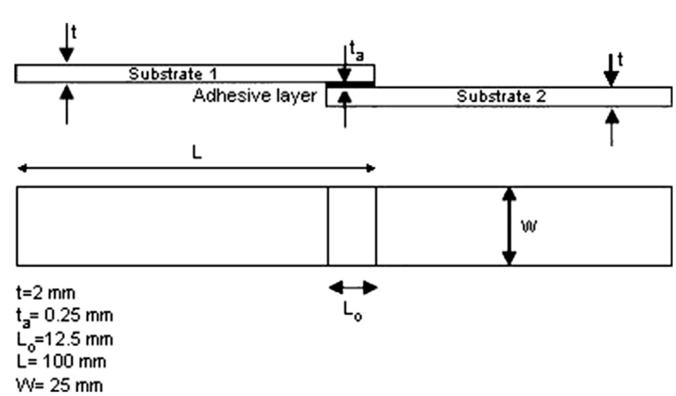

Fig. 1. Lap-shear joint configuration according to ASTM D3163.