Plasma Reactors for the Surface Treatment of Fibers in Composite Materials

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1. Introduction

Composite materials of lignocellulosics (natural fibers) and plastics will be in great demand in the near future, due to the shortage and high cost of petroleum based synthetic materials. Indoor panels of automobiles are already being made of these composites, and a great deal of study is still necessary for optimizing the composition for different applications, requiring different physical and chemical properties. The immense variety and availability of natural fibers in Brazil makes this field a very promising research and investment area. The problem with lignocellulosics-plastics composites is the lack of compatibility between natural (with polar, hydrophylic surfaces) and synthetic (non polar, hydrophobic surfaces) fibers, making their adhesion very poor. Chemical treatments of these fibers are currently in use but they are either expensive or environmentally hazardous due to the large amount of solvents involved in the process. Plasma treatment is thus a good alternative for modifying these fibers surface and improving adhesion. In this work we present two plasma reactors, one being a conventional planar parallel plate capacitive reactor (40 kHz) for the quantitative study of adhesion improvement, and the other a cylindrical rotating reactor (also driven capacitively at 13.56 MHz), for the treatment of fibers in their original irregular forms for the production of composite materials and testing of their mechanical properties.

2. The Planar Reactor

A plane parallel plate capacitive reactor was built for treating the surfaces of plane samples of plastics or fibers (paper, wood etc), in order to measure the adhesion force through peel-testing.

Figure 1 shows the schematics of the vacuum vessel, made of stainless steel. The gases are injected controlling the flux with the needle valve of a flowmeter. A pre-chamber mixes the gases that diffuse to the main chamber through a series of equally spaced holes so that a cylindrically uniform gas injection is made. The reactor has two 20cm diameter electrodes, the upper one in a "drum type" configuration, isolated form the vessel through a ceramic ring. The distance between the electrodes can be changed by introducing spacers to the upper electrode or by moving the lower electrode which is held through a system of dynamic o-rings. The main body of the chamber can be lifted using a counterweight, and separated from the base and lower electrode, opening the chamber for loading/unloading the samples. The vacuum system consists of a mechanical pump, filter and

liquid nitrogen trap, which prevents contamination of the vessel by the oil pump. A base pressure of 20 mTorr is reached within a few minutes of pumping, as measured with an absolute pressure gauge (Baratron). A 2500 W maximum power supply at 40 kHz is connected to the upper electrode, while the rest of the chamber is grounded. Although above 300W of power the temperature of the electrodes is above 100 ° C, there is so far no cooling system, since lower powers is usually best for fibers treatment according to the literature.

To measure electron density and temperature a single Lagmuir probe was used made of a tungsten wire 7mm long and 0.5mm in diameter. Electron temperature was measured through the usual way of the inclination of the characteristic curve in its exponential part. Plasma density was measured using the radial theory (ABR - Allen, Boyd, Reynolds)[1], and the Sonin plot calculated by Chen[2,3], since the electron temperatures of (1 to 2) eV and densities of (10^9 - 10^{10}) cm^{-3} are not suitable for using the thin sheath approximation where the ion saturation current is well defined, giving the density in a straightforward way. Temperatures and densities were measured for working pressures of 200 mTorr to 600 mTorr and power from 50W to 500W. Temperature (between 1 to 2 eV for 200W of power) and density (from 1 to 2 $x10^{10}cm^{-3}$ also for 200W) are not significantly affected by the working pressure. The increase of power leads to a higher density which increases from 1 $x10^{10}cm^{-3}$ at 50W to 4.5 $x10^{10}cm^{-3}$ at 400W with no significant changes in temperature, which means that it only affects the ionization at this working power range. The electron temperature and density profiles show that while density decreases with the radius, the electron temperature increases towards the edge of the electrodes. These last measurements were made from the three access windows, separated by 90 degrees from each other, showing the cylindrical symmetry of the reactor.

3. Treatment of Polypropylene with Oxigen Plasmas for Adhesion Improvement to Cellulose.

Quantitative adhesion measurements are made by peeling the treated polypropylene film from a sheet of filter paper (pure cellulose) to which it was hot pressed, and measuring the peeling force using a universal EMIC testing machine.

The polypropylene (PP) film supplied by Polibrasil (Koppol CG30) is 30 microns thick, and was treated with aditives to improve the packing process to which it was destined. Laminates 4cmx 15cm were cut and washed with acetone and isopropilic alcohool before treatment. The filter paper was from Binzer & Munktell (Germany) and is made of pure cotton cellulose with ash content bellow 0,007%.

The PP films were treated with oxygen plasmas at pressure of 200 mTorr (flux of 30 cc/min), 50W of power and treatment times of 10, 30 and 60 seconds. Three samples were treated for each case. After treatment the films were hot pressed between two sheets of filter paper at (139 ± 9)°C and 8 MPa of pressure for 10 minutes. The filter paper is oven dried before pressing.