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IMPROVEMENT OF MECHANICAL PROPERTIES BY COLD PLASMA TREATMENT OF BONDED SURFACES

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Abstract: In nature, matter is found in solid, liquid or gaseous form. At very high temperatures, molecules can absorb an enormous amount of energy, causing them to move in a disordered way. With the absorbed energy, they can dissociate into atoms, and because of their disordered motion, these molecules or atoms can split into ions and electrons. Excessive heating of matter is therefore always accompanied by ionisation, leaving the medium electrically neutral. This ionised state is known as plasma. It is sometimes called the fourth state of matter. Plasmas are therefore mixtures of neutral and/or excited ions, electrons, atoms, radicals or demodules with an internal energy above the neutral state. Sources of plasma can be as follows: naturally occurring in the ionosphere; produced by nuclear reactions; generated by a hyperfrequency source; formed by direct heating followed by confinement in a region of space by a magnetic field; and produced by electrical discharges. Plasma is characterised by the state of its components: density, which is defined by the number of particles per unit volume; kinetic temperature; and degree of ionisation. Plasma can be thermal or out of thermal equilibrium, or 'cold plasma'. Cold plasma is formed, for example, when a gas passes through strong electric fields - electrons are dissociated from the molecules and the gas becomes electrically conductive and glows blue. The paper describes the equipment used to create cold plasma, which was used to treat different surfaces. After treatment, they were bonded with different adhesives and tested for tensile strenght. An improvement in the mechanical properties mentioned above was observed when the surfaces to be bonded were treated with cold plasma compared to surfaces bonded normally. When the impact of cold plasma treatment on elongation, or ε, is examined, it is found that for Fiber Wood samples, cold plasma treatment considerably boosts elongation, increasing it from 0.35% for untreated samples to 0.81% for samples treated with cold plasma. For samples that have been treated with cold plasma, the average value increases significantly from 0.03% to 0.63% when ArbofillFichte material is used. In addition, the application of cold plasma treatment to the surfaces results in an increase in the modulus of elasticity. For example, the modulus of elasticity doubles for Arbofill Fichte samples, going from 113.56MPa for untreated samples to 227.43MPa for cold plasma treated samples; for Fiber Wood, it increases from an average value of 789.52MPa for untreated samples to an average value of 931.62MPa for treated samples.

Key words: cold plasma, tensile, bonding, adhesive

1. INTRODUCTION

The three states of matter that can be found in nature are solid, liquid, and gaseous. The molecules are able to take in a large amount of energy when they are subjected to extremely high temperatures, which causes them to move in an unorganized manner. They have the ability to disintegrate into atoms as a result of the energy that they have received, and as a result of the chaotic motion that they have, these molecules or atoms can divide into ions and electrons. Ionization is always accompanied by excessive heating of materials, which results in the medium remaining electrically neutral for the duration of the process. Plasma is the name given to this hyperionized state. There are occasions when it is referred to as the fourth state of matter. In light of this, plasmas are mixes of neutral and/or excited ions, electrons, atoms, radicals, or demodules that have an internal energy that is higher than the neutral state. Plasma can originate from a variety of sources, including but not limited to: naturally occurring in the ionosphere; produced by nuclear processes; generated by a hyperfrequency source; made by direct heating followed by confinement in an area of space by a magnetic field, and produced by electrical discharges. Density, which is defined by the number of particles per unit volume, kinetic temperature, and the degree of ionization are the three characteristics that constitute plasma. Plasma is distinguished by the condition of its constituents. Cold plasma refers to plasma that is either thermal or out of thermal equilibrium. Another example of the formation of cold plasma occurs when a gas is subjected to intense electric fields. This causes the electrons in the gas to become dissociated from the molecules, which results in the gas becoming electrically conductive and emitting a blue glow, [1, 2, 5, 12, 13].

Plasma, often regarded as the fourth fundamental state of matter, is composed of a combination of ions, electrons, atoms, radicals, and molecules, each possessing energy levels above their neutral counterparts. In the case of non-thermal plasma, there is a lack of thermodynamic balance as the temperature of the electrons is increased, whereas the larger particles like protons and neutrons remain at lower temperatures. The creation of a cold plasma jet involves the transition of matter from a gaseous state through an electric field, leading to the generation of active species and metastable radicals as a result of the gas ionization. Cold plasma has proven highly effective in killing a wide range of pathogens such as bacteria, viruses, and fungal spores. This is primarily due to the generation of reactive species such as ozone, peroxides, and free radicals during plasma treatment. These reactive species can disrupt microbial cell walls and damage nucleic acids, leading to the sterilization of the surface. This makes cold plasma a valuable tool for disinfecting medical tools and environments without the need for harsh chemicals or high temperatures that could damage sensitive instruments or materials, [6, 7, 10]. Cold plasma treatment can alter the physical and chemical properties of various surfaces to improve their functionality, [8, 11]. For example, plasma can increase the surface energy of polymers, metals, and other materials, leading to better adhesion properties. This is particularly beneficial in the medical field where enhancing the wettability and biocompatibility of implants, like dental implants or prosthetics, is crucial for their successful integration into the body.

The current technology on the market relies on using a mixture of noble gases because the types of high-voltage sources currently in use do not allow the use of atmospheric air as an ionization medium. Over time, various types of sources have been proposed, some based on direct current, as seen in the work, [3, 9]. Others are based on the use of radio frequency/microwave generators as demonstrated in the study, [4]. This method involves using two inductively coupled coils tightly wrapped around a non-magnetic core. By employing an electronic circuit that enables rapid voltage switching, an alternating current is generated in the primary coil. This current creates a magnetic field, which in turn induces a current in the secondary coil. Due to the resonance phenomenon, when the frequency of oscillations in the primary circuit matches the natural frequency of the secondary coil, an efficient energy transfer occurs. This leads to the generation of extremely high voltages at the end of the secondary coil, manifested by electrical discharges capable of easily ionizing atmospheric air. This approach successfully addresses the challenge of maintaining a stable atmospheric cold plasma jet, using atmospheric air as the ionization medium. The main advantage of using atmospheric air as the ionization medium is the significant reduction in technology usage costs within the industry and the elimination of the logistical complexity required for the supply and storing of noble gases.

The paper presents the equipment used to generate cold plasma, which was used to treat different surfaces. After treatment, they were bonded with different adhesives and tested for tensile strength. An improvement in the above-mentioned mechanical properties was observed when the surfaces to be bonded were treated with cold plasma compared to untreated surfaces.

2. MATERIALS AND METHODS

3D-printed Fiber Wood samples and injected Arbofill Fichte samples were used. The samples are shaped like dumbbells and dimensioned according to ISO 527:2, [14].

The *tensile test* was performed according to ISO527 on an INSTRON 3382 universal electromechanical testing machine. The test parameters used are: traverse displacement speed V_d=0.5 mm/min; deformation temperature 23±0.5°C (air-conditioned laboratory).

Injection of Arbofill Fichte samples (figure 1a) were performed using the SZ-600H injection moulding machine (SHEN ZHOU, Zhangjiagang, China) at the Laboratory of Fine Mechanics and Nanotechnologies, Technical University "Gheorghe Asachi", Iași, Romania. The main technological parameters were: melting temperature 150°C, injection pressure 80MPa, injection angle 90° and cooling time 18s.

3D printing of Fiber Wood samples (figure 1b) were performed on Raise Pro2 Plus equipment with the following printing parameters: layer thickness 0.1mm; printing speed 40mm/s; sample orientation was flat on the printing table; nozzle temperature 190°C and printing table temperature 50°C.


a) b)

Fig.1. Test samples: a) obtained by injection moulding from Arbofill Fichte; b) obtained by 3D printing from Fiber Wood

The cold plasma generating device consists of a relaxation oscillator that generates oscillations with a frequency tuned to the natural resonant frequency of the secondary coil. In addition, the device incorporates a switch circuit to control the intensity of the cold plasma jet by varying the number and length of electrical discharges every second (figure 1). Figure 1 shows the oscillator signal set to the natural frequency of the secondary coil, which will be amplified by the Gate Driver integrated circuit. This integrated circuit holds the function of interrupted operation, making it possible to produce cold plasma in pulses. Figure 2 shows this pulsed operation, within one pulse period set by the switch circuit hundreds of thousands of oscillations will be produced in the inverter. These diagrams were generated using the LTSpice 17.1.15 application, an electrical and electronic circuit simulation software developed by Analog Devices used to design, analyze and optimize circuits.

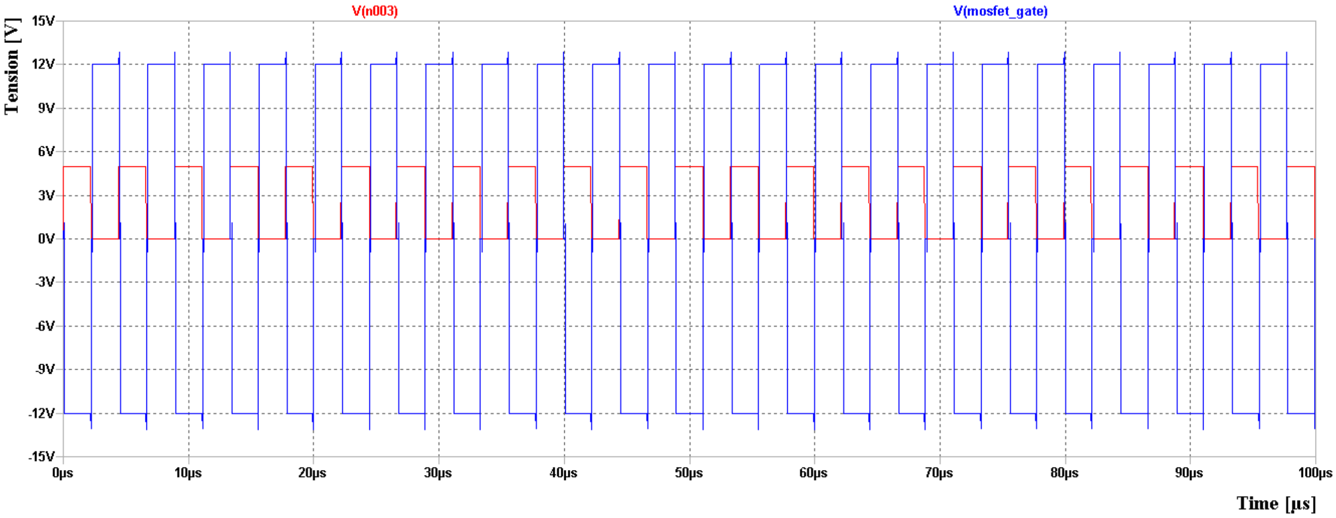


Fig. 1. Circuit operation, red line is the natural resonant frequency oscillator signal, blue line is the inverter signal

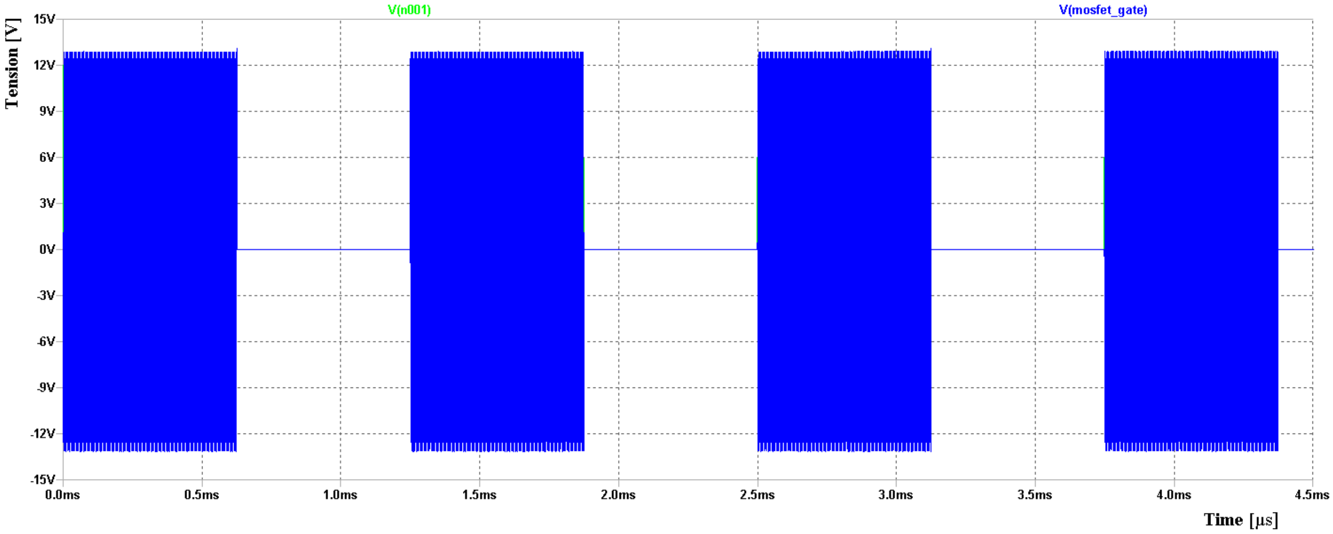
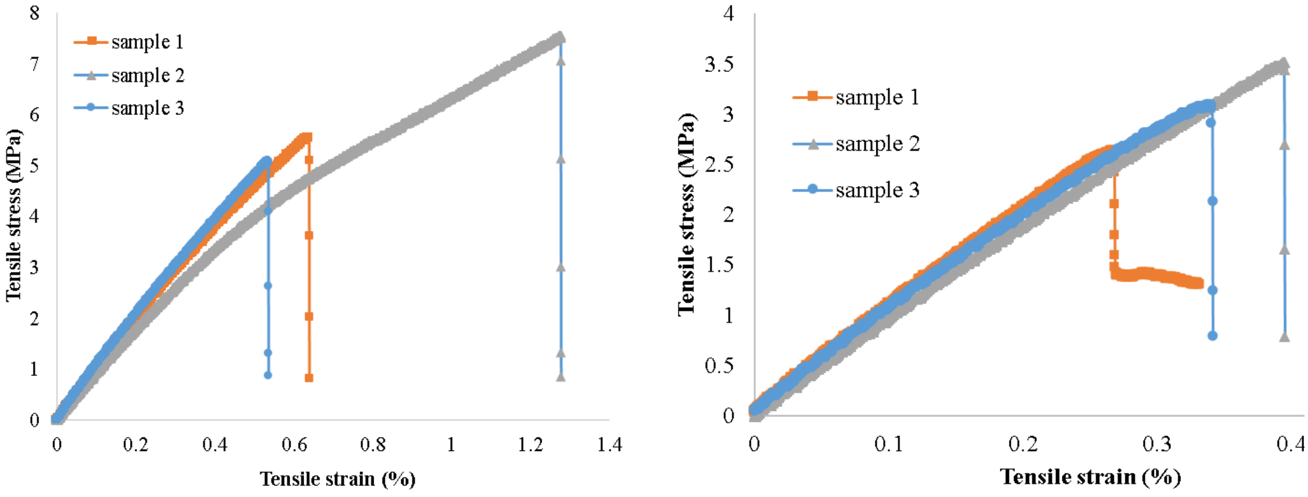


Fig. 2. Interrupted circuit operation, each blue rectangle is composed of thousands of oscillations that are triggered by the circuit breaker output

The parameters used in cold plasma formation are as follows:
- resonant frequency of approximately 500 kHz using a primary and secondary coil assembly;
- switch circuit frequency of 1.6 kHz with a duty cycle of 50%.
- the supply voltage applied to the inverter by an adjustable voltage source with which the tests were performed was 60V DC, with a current limit set by a ballast resistor limiting the consumption to 100W;
- the surface exposure time, which was 5 minutes, with the cold plasma generation head at a distance of (3-5) mm.

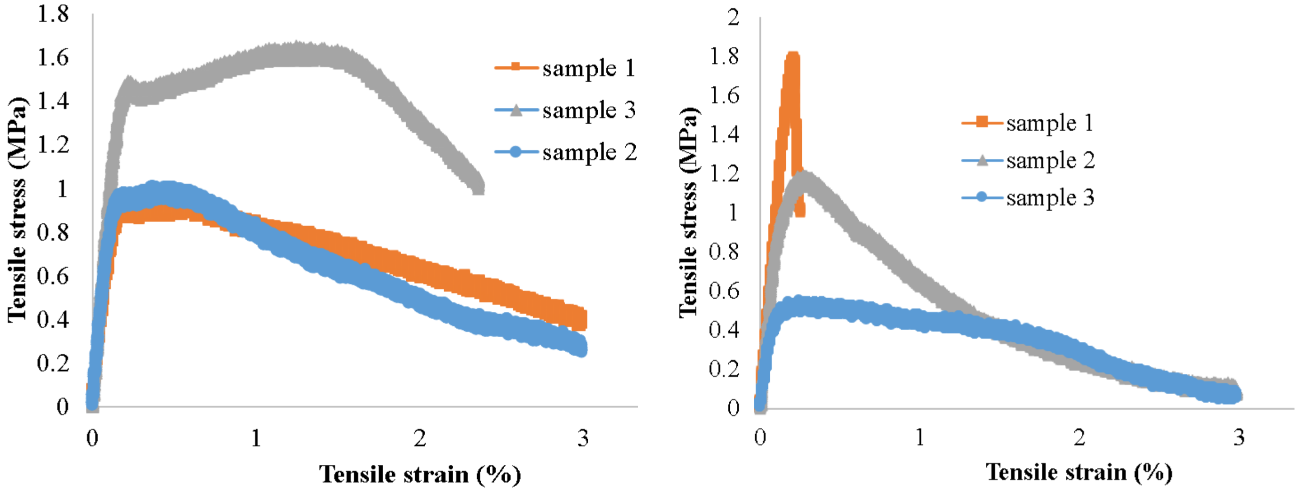
3. RESULTS AND DISCUSSION

Figure 3 shows the tensile diagrams for 3D-printed Fiber Wood specimens bonded with Bison Super Glue (BSG) adhesive with and without cold plasma bonded surface treatment, and Figure 4 shows the tensile diagrams for Arbofill Fichte injection molded specimens with and without cold plasma bonded surface treatment bonded with Bison Vinyl Plastic (BVP). The maximum tensile strength values for cold plasma treated surfaces are higher than the values obtained when bonding surfaces without surface treatment (Table 3).



a). b).

Fig.3. Tensile strength of Fiber Wood samples bonded with BSG adhesive: a) with cold plasma surface treatment; b) without cold plasma surface treatment



a). b).

Fig.4. Tensile strength of Arbofill Fichte samples bonded with BVP adhesive: (a) with cold plasma surface treatment; (b) without cold plasma surface treatment

Table 3. Experimental results of uniaxial tensile tests of tested samples: tensile strength, σ_{max} [MPa]; elongation, ε [%]; modulus of elasticity, E [MPa]

Material	No. exp.	σ_{max}, [MPa]	ε, [%]	E, [MPa]
Fiber Wood_BSG_ Treated surfaces	1	5.57	0.63	960.38
	2	7.53	1.27	846.70
	3	5.08	0.53	987.77
	Valori medii	**6.06**	**0.81**	**931.62**
	Standard Deviation	1.30	0.40	74.80
Fiber Wood_BSG_ Untreated surfaces	1	1.33	0.32	555.82
	2	3.50	0.39	902.35
	3	3.09	0.33	910.39
	Valori medii	**2.64**	**0.35**	**789.52**
	Standard Deviation	1.15	0.04	202.43
Arbofill_BVP_ Untreated surfaces	1	1.79	0.02	40
	2	1.18	0.01	237.81
	3	0.32	0.05	62.88
	Valori medii	**1.10**	**0.03**	**113.56**
	Standard Deviation	0.74	0.02	108.21
Arbofill_BVP_ Treated surfaces	1	0.91	0.66	174.01
	2	0.96	0.25	154.83
	3	1.60	0.97	353.44
	Valori medii	**1.16**	**0.63**	**227.43**
	Standard Deviation	0.38	0.36	109.55

4. CONCLUSIONS

Cold plasma treatment can alter the physical and chemical properties of various surfaces to improve their functionality. For example, plasma can increase the surface energy of polymers, metals, and other materials, leading to better adhesion properties.

Our air-based cold plasma device that is significantly cheaper and easier to use, eliminating the need for bulky gas tanks, is a revolutionary breakthrough in plasma science due to multiple factors. Firstly, it drastically reduces operational costs by removing the need for expensive noble gases, enabling more economical production processes and competitive pricing. Secondly, the simplicity and accessibility of using freely available atmospheric air streamline operations, potentially broadening the adoption of cold plasma technology across different scales of the fabrication sector. Thirdly, such devices reduce the environmental impact by cutting down on the carbon footprint associated with transporting and storing gas cylinders and minimize dependency on chemical gases, aligning with sustainable manufacturing practices. Fourthly, safety and space efficiency are markedly improved as the inherent risks and spatial requirements associated with gas tank storage are eliminated, making cold plasma technology safer and more suitable for various environments. Additionally, maintenance and logistics become more straightforward, ensuring uninterrupted fabrication processes free from the complexities of noble gas supply chains. Finally, the affordability and ease of use of these air-based devices could spur innovation and expand applications within the industry, such as in advanced material processing and surface treatments, further enhancing the value and versatility of cold plasma in fabrication settings.

The tensile tests show, in the case of Fiber Wood samples with surfaces treated with cold plasma before bonding, an average tensile strength value, σ_{max}, of 6.06 MPa while the average tensile strength value, σ_{max}, for samples bonded without surface treatment of 2.64 MPa. In the case of Arbofill Fichte samples obtained by injection moulding the average value of the maximum tensile strength, in the case of surfaces treated with cold plasma before bonding, is 1.16MPa, while in the case of surfaces bonded without surface treatment with cold plasma 1.10MPa was obtained.

Analyzing the influence of cold plasma treatment on elongation, ε, it is observed that in the case of Fiber Wood samples, cold plasma treatment significantly increases elongation from the value of 0.35% for untreated samples to 0.81% for cold plasma treated samples.

When using ArbofillFichte material there is a major increase from 0.03% average value for untreated samples to 0.63% average value for cold plasma treated samples.

Cold plasma treatment of the surfaces also causes an increase in the modulus of elasticity: in the case of Fiber Wood, from an average value of 789.52MPa for untreated samples to an average value of 931.62MPa for cold plasma treated samples; for Arbofill Fichte samples, the average value of the modulus of elasticity doubles from 113.56MPa for untreated samples to 227.43MPa for cold plasma treated samples.

Conflicts of Interest: There is no conflict of interest.

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