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Influence of dry etching plasma treatment energy on the Young's modulus and surface appearance of bamboo fibres

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ABSTRACT

We analyzed the effect of dry etching plasma treatment on the Young's modulus and surface appearance of *Guadua angustifolia* fibres. The fibres were bombarded with argon ions at different energy levels and exposure times through a dry etching method. Scanning electron microscopy (SEM) analysis revealed that ion bombardment modified the fibre surface morphology, increasing roughness. Using higher plasma energy with shorter exposure times produced similar effects to those obtained with lower energy and longer exposure times. The increase in Young's modulus was directly related to changes in the fibre's surface structure. These findings confirm the influence of plasma treatment on the mechanical properties of the fibres and open new possibilities for their application as a reinforcement in composite materials.

1. Introduction

Natural fibres are becoming a valuable resource for the composite industry. The use of natural and synthetic fibres as reinforcement of polymeric matrices has many advantages. They have better mechanical properties per unit of weight, they decrease the cost of manufacturing per unit of volume and, among other things, are environmentally friendly. Despite these advantages, the low compatibility between the constituents can result in poor mechanical behaviour. However, plasma treatments can modify the surface of the fibres used as reinforcement, improving the connections between the different phases of the composite (Alsubari et al., 2021; Elseify et al., 2021; Mulenga et al., 2021; Kuram, 2022; Zhan et al., 2021).

From an environmental perspective, the main advantages of using natural fibres for reinforcement include that they are derived from renewable resources, their production requires a minimal amount of energy, and they contribute to CO_2 absorption, (Galan-Marin et al., 2016). Their low density, high specific strength and stiffness, and cost-effectiveness are noteworthy technical benefits. Nonetheless, there are disadvantages, such as the high variability in properties, moisture absorption, low strength and durability, and limited fibre / matrix bonding (Sanjay et al., 2019; Halip et al., 2019; Pickering et al., 2016; Hosseini et al., 2023). Most authors state that the primary disadvantage of using natural fibres as a reinforcement in polymeric matrices is the inadequate bonding between composite phases (Mousavi et al., 2022; Acosta et al., 2021; Pramudi et al., 2021) due to the hydrophilic nature of fibres and the hydrophobic properties of most polymers (Peças et al., 2018; Halip et al., 2019; Hashim et al., 2017; Rohit and Dixit, 2016). This issue could be addressed by modifying the physicochemical properties of natural fibres through chemical or physical methods (Faruk et al., 2012; Fernandes-P et al., 2015; Islam et al., 2013).

The limited use of natural fibres is mainly due to the low compatibility with polymeric matrices, although this can be overcome by treating the fibres using chemical or physical procedures. Chemical methods have been used to reduce the hydrophilic properties of natural fibres by removing hemicellulose and lignin from their surfaces. However, chemical methods can degrade the fibres and reduce their mechanical strength, generating chemical waste, which entails environmental impact and additional treatment costs (Luna et al., 2024).

Physical methods such as plasma etching have been used primarily to modify the structural and surface properties of fibres without affecting their internal structure and with minimal impact on volumetric properties. This is intended to improve compatibility with the polymer matrices without impairing the mechanical properties of the fibres. These modifications, which can be achieved by plasma treatments such

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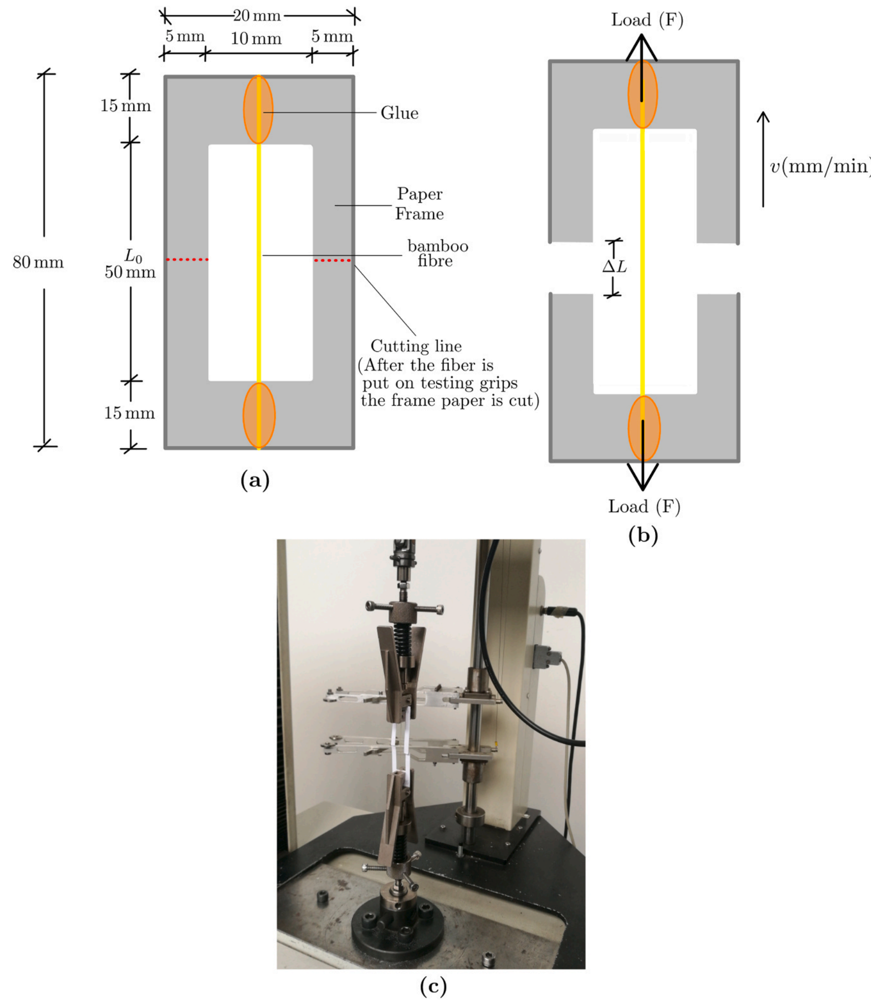


Fig. 1. a) and b) show the method used to perform fibre tensile testing, ASTM C1557 Standard. (ASTM-C1557, 2020), c) Load cells used to perform tensile tests on fibres (Luna et al., 2016; Mosquera et al., 2022).

as dry etching, mainly modify the surface characteristics of the fibres through an ion bombardment process and could therefore substantially increase the adhesion between natural fibres and polymeric matrices (Luna et al., 2016, 2018; Mosquera et al., 2022; Luna et al., 2024).

We investigated the effect of dry etching plasma treatment on the Young's modulus and surface morphology of *Guadua angustifolia* Kunth fibres. Unlike previous studies, we analyzed different plasma energy levels and exposure times to determine their influence on the mechanical properties of the fibres. This work will contribute to the development of sustainable treatments to enhance the bonding of natural fibres in composite materials without compromising their mechanical performance.

One of the objectives of this research was to obtain technical data to advance the utilization of *Guadua angustifolia* bamboo fibres as reinforcement in composite materials.

2. Experimental

Fibres were extracted from 3 to 6 year old culms of the bamboo *Guadua angustifolia*. A chemical-mechanical process enabled fibres of approximately 90 mm in length to be obtained, with the main objective of the chemical procedure being to soften the bamboo fibres to separate the lignin, cellulose and hemicellulose. The mechanical process enabled the separation of the fibres, see (Luna et al., 2016, 2024).

The bamboo fibres were subsequently subjected to a bombardment with argon ions at room temperature. Two different powers were used: 0.2 W and 1.1 W. Fibres bombarded at 0.2 W were treated for either 900 or 1500 s, and those bombarded at 1.1 W were treated for 90 or 180 s. A commercial dry etching system (BAL TEC model SCD 050) was used. In this case, all the samples were placed inside a vacuum system, which was evacuated until reaching a vacuum of approximately 10^{-3} mbar. Special care was taken to obtain similar treatment conditions for all the samples.

SEM analysis using a scanning electron microscope, was undertaken to study the surface appearance of the fibres following the bombardment with Ar ions. The objective of this analysis was to evaluate changes in roughness, surface irregularities, and potential damage induced by ion bombardment. SEM images were obtained using a Tescan Vega 3 SB scanning electron microscope operating in secondary electron mode.

The acceleration voltage was set between 5 kV to 15 kV to ensure optimal resolution and contrast. The fibres were coated with a thin layer of gold using a plasma sputtering technique. Images were captured at magnifications ranging from 3kx to 35kx to analyze the different scales of surface modification.

Tensile test and cross-sectional measurements were carried out on 5 replicates for each treatment, to evaluate the effects on the mechanical properties of the fibres subjected to the bombardment. Tensile tests were performed following ASTM C1557–14 using a Shimadzu universal testing machine equipped with a 50 N load cell. Each specimen was carefully mounted between mechanical grips, ensuring proper alignment to minimize slippage. The gage length of the fibres was set to 50 mm. A constant crosshead speed of 1.0 mm/min was applied during

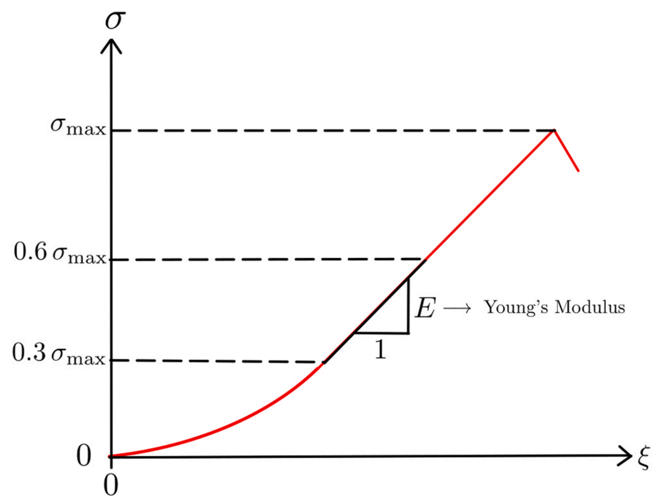


Fig. 2. Shows the stress (σ) vs. strain (ξ) curves used to determine Young's modulus, (Luna et al., 2016).

testing. The failure mode and stress-strain behaviour of the fibres were recorded for further analysis. A representative image of the fibre mounted in the testing machine is shown in Fig. 1 (Luna et al., 2016; Mosquera et al., 2022).

The Young's modulus was calculated from the slope of stress (σ) vs strain (ξ) in a material from the expression (1)

$$E = \frac{\Delta\sigma}{\Delta\xi} \tag{1}$$

where E is the Young's modulus, as shown in Fig. 2.

A cross-sectional image of each bamboo fibre was taken after failure using a Nikon SMZ800N stereoscope, and their area was subsequently measured using special software (Image-J, 2020).

Considering that the fibres had an irregular cross-section, the area was calculated taking into account the cross-section of the point where the fibres failed. The fibres were placed in red clay to provide contrast and an average area was determined for each fibre (Image-J, 2020; Mosquera, 2023).

3. Experimental results and discussion

Figs. 3a and 4a) display the SEM micrographs of untreated fibres.

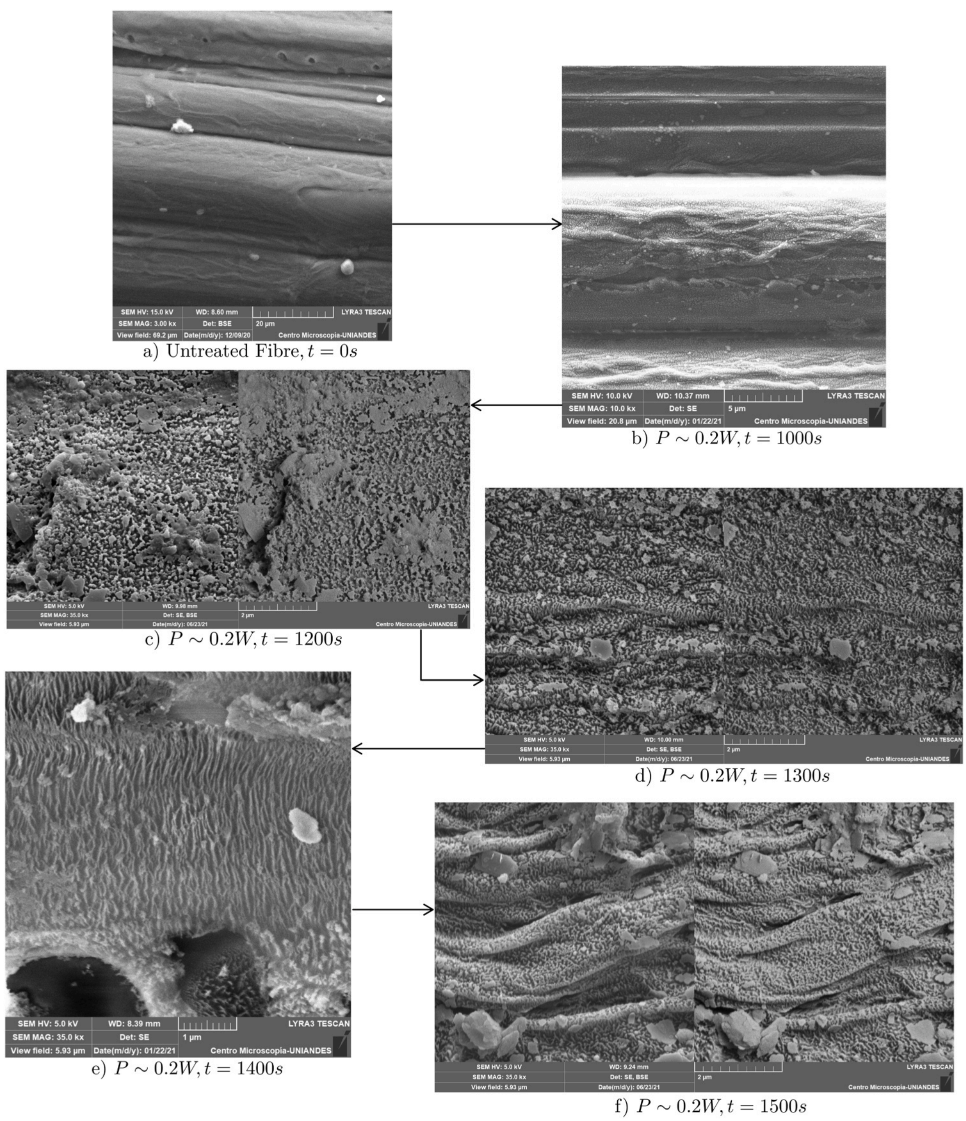


Fig. 3. a) An SEM micrograph for an untreated fibre. Figures b), c), d), e) and f) show the corresponding SEM micrographs for ion bombardment powers of 0.2 W and different times. The surface of the fibres was damaged by the ion bombardment.

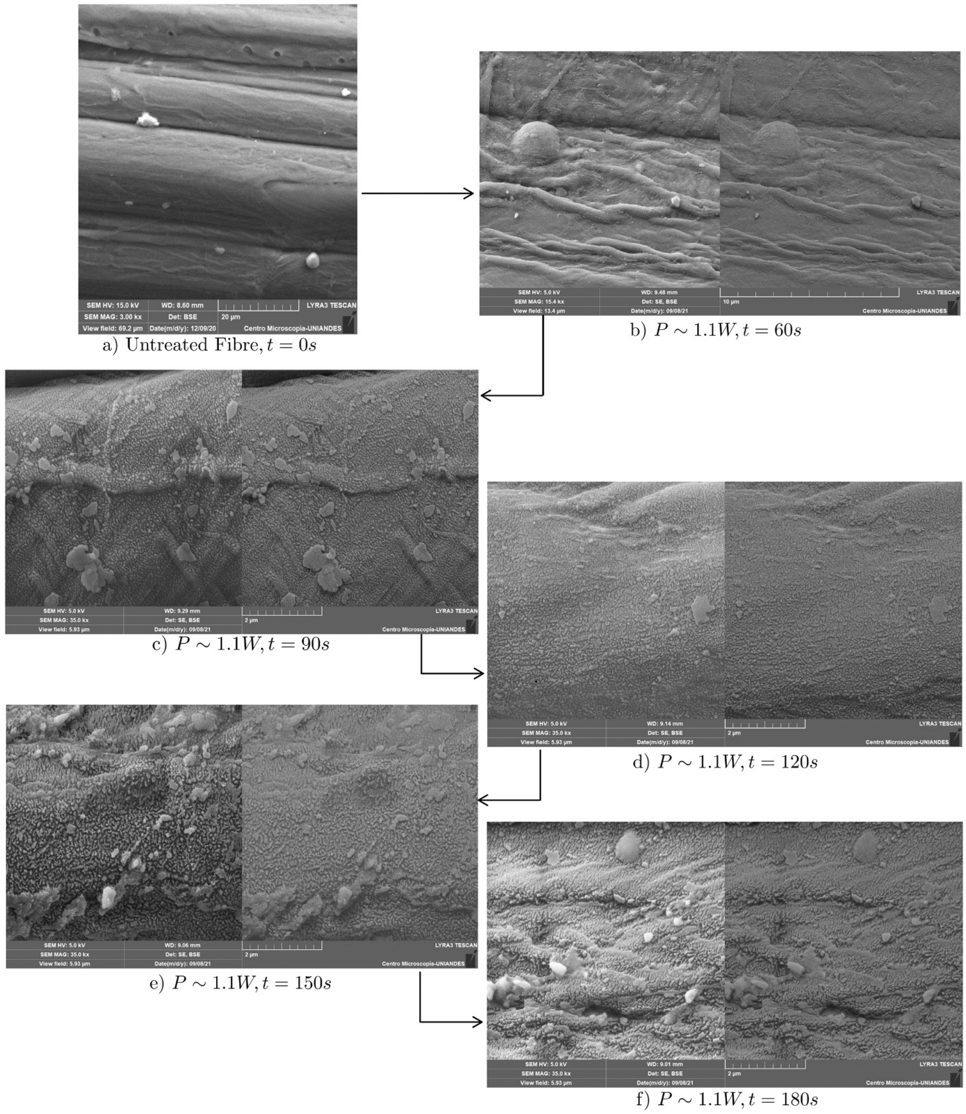


Fig. 4. a)An SEM micrograph for an untreated fibre. Figures b), c), d), e) and f) show the corresponding SEM micrographs for ion bombardment powers at 1.1 W and different times. The ion bombardment damaged the surface of the fibers.

Figs. 3b) to 3f) and 4b) to 4f) show treated fibres with ion bombardment powers of ~0.2 W and ~1.1 W, respectively, for different bombardment times.

In both cases all fibres treated with ion bombardment powers of 0.2 W (Fig. 3b to 3f) and those subjected to ion bombardment of 1.1 W (Fig. 4b to 4f) show rough surfaces compared to untreated fibres. The roughness on the surfaces of the fibres increased with ion bombardment time. This behaviour correlates well with the increase observed in Young Modulus for bombardment times $t < 1200$ s (Fig. 5a) and $t < 150$ s (Fig. 5b) for 0.2 W and 1.1 W, respectively.

For both treatment strengths, the SEM micrographs also displayed greater damage to the surfaces of fibres subjected to longer ion bombardment times. Additionally some roughness was observed on the surfaces of fibres subjected to ion bombardment at higher powers and shorter times in contrast to those obtained with lower powers and longer bombardment times.

Figs. 5a) and 5b) display the Young's modulus as a function of bombardment time at 0.2 W and 1.1 W, respectively. Young's modulus was determined using Eq. 1 from the stress-strain curves as shown in Fig. 2 (Mosquera, 2023). The magnitude of the increases in the Young's Modulus was similar in both treatments. ΔEmax was around 10 %, despite the lower damage observed in the fibres treated with P ~ 1.1 W and the shorter times.

In both cases, there was a decrease in the value of Young's modulus as the treatment time increased. Possible causes of this decrease may be the damage to the surfaces of the fibres, the formation of voids, partial melting or other causes.

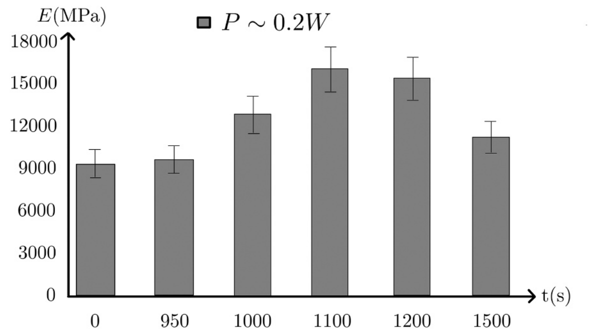
4. Conclusions

Argon ion bombardment affected the surfaces of the bamboo fibres, resulting in surface roughness. At both treatment strengths, the Young's modulus decreased with longer bombardment times. Exposure at a higher power for a shorter time produced similar results (~ 10 %) to those obtained at a lower power for a longer time damaged the surface of the fibres less than when a lower power was used for a longer bombardment time.

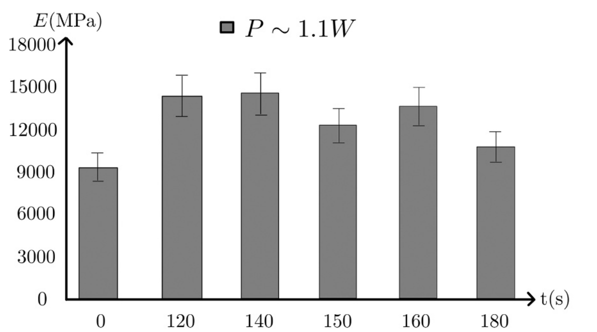
The observed decrease in Young's Modulus over longer bombardment times may be associated with the damage to the fibre surfaces, or to the formation of voids, partial melting or other causes.

The roughness produced on the fibre surfaces by the ion bombardment could enhance mechanical bonding within composite phases.

Future research should focus on comparing this technique with conventional fibre treatments, including FTIR, and should use XRD



a)Young's modulus as a function of bombardment time with $P \sim 0.2W$.



b)Young's modulus as a function of bombardment time with $P \sim 1.1W$.

Fig. 5. a) and b) display the Young's modulus as a function of bombardment time for power of 0.2 W and 1.1 W, respectively.

characterization to better understand the chemical and structural effects of argon ion bombardment.

CRediT authorship contribution statement

S. Mosquera G.: Investigation, Data curation, Conceptualization, Methodology, Writing – original draft, Formal analysis. **A. Mariño C:** Supervision, Writing – review & editing, Methodology, Formal analysis. **P. Luna T.:** Supervision, Writing – review & editing, Formal analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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