


The potentialities of ultrasound as an alternative to chemical etching for proton beam writing micropatterning

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Abstract

In this paper, we explore the potentialities of using ultrasound waves as a post-irradiation treatment after proton beam writing (PBW) patterning of PTFE. To that end, 2-mm-thick foils of PTFE were irradiated with 2.2 MeV protons with an average current of 50 pA. Straight line structures were patterned on the polymer with a $3.5 \times 3.5 \mu\text{m}^2$ focused proton beam. Post-irradiation polymers were placed in a heat bath at 60 °C and immersed either in distilled water or in a 6 M solution of NaOH under the action of 40 kHz ultrasound waves for developing the patterned structures. The results indicate that distilled water submitted to ultrasound waves is very efficient for removing rough structures created by the proton irradiation and thus providing a good aspect ratio to the PBW microstructures. On the other hand, the use of 6 M NaOH instead of water did not improve the quality of the structures patterned with the proton beam. The results are discussed in terms of the parameters that characterize the interaction of ultrasound waves with the liquid media and the polymer.

KEYWORDS

PBW, proton beam writing, PTFE, ultrasound

1 | INTRODUCTION

The patterning process of materials through focused ion irradiation is generally known as proton beam writing (PBW) despite ions other than protons can be employed for generating 2D and 3D microstructures.¹ In the last decade, this technique has been extensively explored and has

proved to be an effective patterning process for several positive and negative resists.² In this process, the materials under study are usually patterned with a focused proton beam of a few MeV which weakens and damages their chemical structure at those places irradiated by the beam. Finally, the irradiated material can be developed through the use of chemicals in order to polish and provide

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structures with enhanced aspect ratios.³ The patterning of ultimate high-quality structures depends on several factors including focusing ion system, beam fluence, and the choice of an appropriate material.^{2,4} Other factors including the ion fluence, the etchant or developer and the etching time play an important role as well.^{5,6}

Polytetrafluoroethylene (PTFE) is a fluorinated polymer widely used by the industry for several applications.⁷ This polymer, known as teflon[®], exhibits a high degree of inertness as it is remarkably resistant to chemical agents and is not soluble in most solvents. This polymer features distinct characteristics like high crystalline melting point, high melt viscosity, and high thermal stability among others.⁸ Given the technological potential this material offers, PTFE is the source of several innovations in segments of electronics, photonics and biomedicine⁹ due to its hydrophobic properties and biocompatibility. However, the attempt to explore ways of manufacturing microstructures through a lithographic process remains a challenge.

The use of PBW as a direct writing process of PTFE without any post-irradiation treatment has been demonstrated through the fabrication of highly porous 3D structures.¹⁰ Moreover, Nishikawa and Hozumi showed that a thermal treatment carried out after the patterning of PTFE through PBW turns rough structures into smooth ones with good aspect ratio.¹¹ Smooth structures have been obtained as well once the PWB process takes place in the presence of an oxygen-rich environment. Therefore, this kind of experiment can be carried out under ambient pressure (in air) with an external beam setup¹² if good aspect ratio structures are needed.

Sound waves with frequencies above the hearing range of humans (about 16 kHz) are classified as ultrasonic waves. Such waves found a great number of applications in different fields including materials science, chemistry and biology among others.¹³ For instance, sonochemistry is the science that explore different uses of ultrasonic waves to enhance chemical reactions following the cavitation process.¹⁴ Moreover, ultrasonic waves have proved to be a valuable tool for the synthesis process of nanoparticles and nanocomposites.¹⁵ In general, each particular use of ultrasonic waves is optimized according to the wave frequency. While ultrasonic cleaning employs frequencies from about 20 kHz up to 40 kHz, medical imaging applications use sonic waves with frequencies of a few MHz.¹³

Due to its properties, the functionalization of PTFE is of major interest for a wide range of industrial applications. Studies have been showing the potential of this material in fields such as microfluidics for the production of super hydrophobic surfaces.¹⁶ Besides, this material has been targeted for the production of membranes for biomedical applications^{17–19} and for proton exchange studies²⁰ among others.

The aim of this work is to develop a methodology for the preparation of patterned microstructures on PTFE

with high aspect ratio and minimal sample handling. To that end, 2 mm foils of PTFE were submitted to the proton beam writing (PBW) process and subsequent treatment of the irradiated structures using ultrasound waves. Distilled water and a solution of 6 M NaOH were employed as the wave propagating media during the post-irradiation treatment. The final results were interpreted in terms of the parameters of the liquid media and the PTFE.

2 | MATERIALS AND METHODS

The microprobe system from Oxford Microbeams Limited[®] operating in triplet mode was employed for the PBW experiments. Microstructures with dimensions of 1×100 pixels (line pattern) were fabricated on polytetrafluoroethylene (PTFE) through pixel-by-pixel exposure to the proton beam. In the present case, the scan size ($256 \times 256 \mu\text{m}^2$) matches the number of pixels programmed in the scanning system (256×256), thus leading to a pixel size of $1 \times 1 \mu\text{m}^2$. Since the beam spot size on the target is $3.5 \times 3.5 \mu\text{m}^2$, a considerable overlap of the beam takes place when the beam moves from one pixel to the next one, thus giving rise to a nonuniform irradiation of the polymer as discussed elsewhere.⁶

Commercial polytetrafluoroethylene (PTFE) foils of about 1 cm^2 area and (2.0 ± 0.4) mm thick were irradiated with 2.2 MeV protons with an average current of 50 pA. According to simulations carried out with the SRIM/TRIM software package,²¹ such protons have a range of about 53 μm in PTFE. Therefore, the proton beam deposited all its energy in the polymer. Finally, two proton fluences were used, namely $6 \times 10^{14} \text{ H}^+ \cdot \text{cm}^{-2}$ and $1 \times 10^{15} \text{ H}^+ \cdot \text{cm}^{-2}$. Fluences equal or lower than $1 \times 10^{14} \text{ H}^+ \cdot \text{cm}^{-2}$ proved to be insufficient for the proper polymer functionalization.

After irradiation, the PTFE foils were immersed horizontally in the bottom of an adapted beaker filled with 50 mL of either distilled water or a solution of sodium hydroxide 6 M (NaOH Merck 99% P.A.). The beaker was placed inside a heat bath (Figure 1) at a constant temperature of $(60 \pm 1)^\circ\text{C}$ and submitted to a 100 W ultrasound system consisting of two transducers. The frequency of the ultrasound waves was fixed at 40 kHz. The polymer foils were kept in the liquid medium during 5 min.

The post-treatment microstructures were observed under a scanning electron microscope (SEM—Jeol JSM-6060) using a 10 keV electron primary beam. Micrographs were obtained through the detection of secondary electrons.

3 | RESULTS AND DISCUSSIONS

Figure 2 shows the results of polytetrafluoroethylene (PTFE) foils line-patterned with 2.2 MeV protons with a

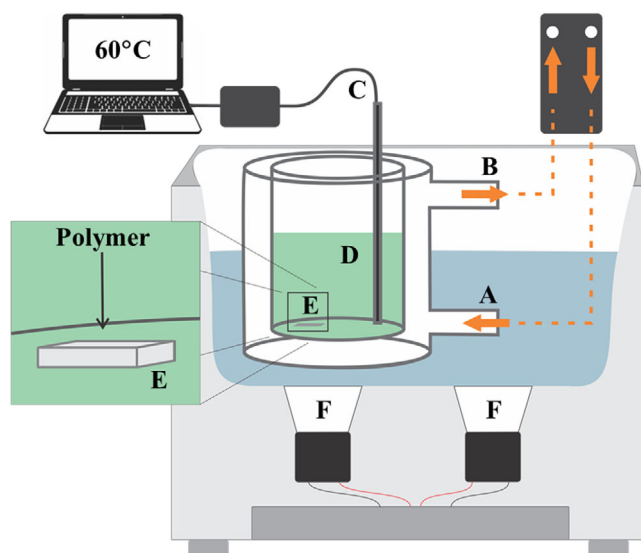


FIGURE 1 Schematic view of the experimental setup used for the post-irradiation treatment. (a) Hot water inlet; (b) hot water outlet; (c) thermostat; (d) 50 ml of either distilled water or 6 M NaOH; (e) irradiated PTFE sample; and (f) ultrasound transducers [Color figure can be viewed at wileyonlinelibrary.com]

fluence $1 \times 10^{15} \text{ H}^+ \cdot \text{cm}^{-2}$. Figure 2a depicts the microstructure as-irradiated, that is, with no post-irradiation treatment. The radiation damage induced by the proton beam on the polymer is clearly visible, leading to a quite rough structure filled with irregular ridges. The irradiated regions of the polymer can be smoothed out by the removal of the modified material through a proper post-irradiation treatment. For the present case, the efficient removal of material depends on several factors including the type of polymer, the irradiation parameters and the etching technique adopted as a post-irradiation treatment. For PTFE, previous studies showed that techniques like a thermal treatment carried out after the patterning through PBW¹¹ and in-air patterning¹² turns rough structures into smooth ones with good aspect ratio. For other polymers such as polyethylene terephthalate (PET, Mylar[®]), an etching procedure consisting of a 6 M NaOH solution in a thermal bath with magnetic stirring has been used successfully for the removal of the damaged structures.⁶

In the present study, two similar routes employing ultrasonic waves were followed as post-irradiation treatment. One of them made use of a 6 M NaOH solution as the wave propagating medium, while another one employed just distilled water. The results of these post-treatment procedures are shown in Figure 2b,c, respectively. Figure 2b suggests that the use of a 6 M NaOH solution at 60°C submitted to 40 kHz ultrasound waves is not appropriate for the development of proton-induced microstructures on PTFE. Indeed, a closer look at the

structure (Figure 2b-2) reveals that instead of removing the broken structures, the etching procedure seems to have melted them in a quite irregular manner. This result shows that although the 6 M NaOH solution works for polyethylene terephthalate polymers, it is not suitable for developing structures fabricated on polytetrafluoroethylene under the present circumstances.

Figure 2c shows the results obtained with distilled water at 60 °C instead of a NaOH solution as a propagating medium for the ultrasound waves. In this case, the post-irradiation treatment resulted in sharper and well-defined walls with good aspect ratio. However, Figure 2c-2 reveals that the bottom of the structure was not fully smoothed out during post-irradiation treatment.

Control experiments were carried out in order to verify the effectiveness of the proposed procedure for the production of good aspect ratio structures after proton bombardment of PTFE. First of all, several independent measurements were carried out in order to test the reproducibility of the post-irradiation treatment with water and NaOH under ultrasound waves. To that end, the irradiations and the post-irradiation treatments were performed in different days, thus assuring the independence of the experiments. The results shown in Figure 3 indicate that the proposed procedure is quite effective as far as water is used as a propagating medium for the ultrasound waves.

Additionally, post-irradiated polymeric foils were immersed either into water or 6 M NaOH solution for 5 min at a temperature of 60 °C without any influence of ultrasonic waves. The results shown in Figure 4 suggest that there are no changes in the structures regardless the liquid medium under study. Therefore, the application of ultrasound waves on the heat bath is of utmost importance as far as cleaning purposes are concerned.

The impact of different ion fluences on the post-irradiation treatment was investigated as well. Figure 5 depicts the results obtained with a fluence of $6 \times 10^{14} \text{ H}^+ \cdot \text{cm}^{-2}$. The removal of material by the post-irradiation treatment was not totally effective for this fluence. This result indicates that a fluence of $6 \times 10^{14} \text{ H}^+ \cdot \text{cm}^{-2}$ does not functionalize the polymer as the higher fluence does. Moreover, experiments carried out with fluences less or equal than $1 \times 10^{14} \text{ H}^+ \cdot \text{cm}^{-2}$ proved to be insufficient to produce patterns on PTFE.

All results indicate that a post-irradiation treatment using water instead of NaOH is much more efficient for smoothing out structures through the removal of the irradiated material. Since the experimental procedure was standardized for all experiments, the only difference between the two procedures is the propagating medium of the ultrasound waves. All relevant parameters concerning both propagating media are shown in Table 1.

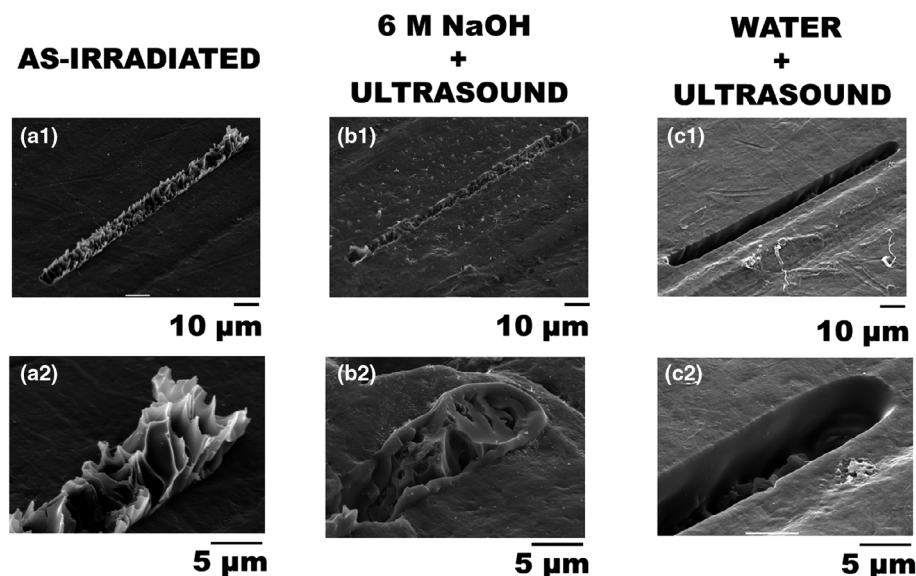


FIGURE 2 Line microstructures of 1×100 pixels patterned on 2-mm-thick PTFE with 2.2 MeV protons with a fluence of 1×10^{15} ions.cm⁻². (a-1) polymer as-irradiated; (b-1) irradiated polymer after treatment with a 6 M NaOH solution at 60°C under the influence of a 40 kHz ultrasound waves; (c-1) irradiated polymer after treatment with distilled water at 60°C under the influence of a 40 kHz ultrasound waves. Panels (a-2), (b-2), and (c-2) depict a zoom of the respective upper panels

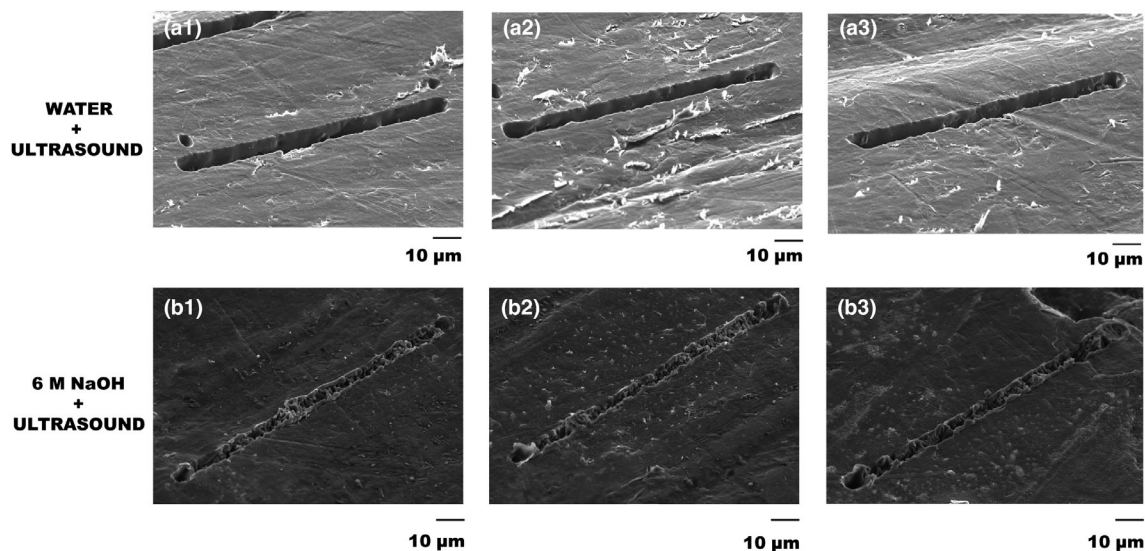


FIGURE 3 Line microstructures of 1×100 pixels patterned on 2-mm-thick PTFE with 2.2 MeV protons with a fluence of 1×10^{15} ions.cm⁻². Panels (a) and (b) depict post-irradiation results obtained either with distilled water at 60°C under the influence of a 40 kHz ultrasound waves or with a 6 M NaOH solution under the same experimental conditions, respectively. Each panel represents independent results using different samples irradiated and treated at different days

The values of the wavelength λ were obtained from the tabulated results of the longitudinal velocity of sound in water and NaOH assuming 40 kHz as a wave frequency through the equation.

$$\lambda = \frac{v}{f} \quad (1)$$

The interaction of ultrasonic waves with liquids depends on several parameters such as the wave frequency f , the medium density ρ , and the viscosity η . As an

ultrasonic wave with longitudinal velocity v propagates in the medium, it is attenuated as it transfers energy to it. According to Kirchoff,¹⁴ the absorption coefficient α_k of sound waves by the liquid medium in the limit of an incompressible fluid is given by.

$$\alpha_k = \frac{2\pi^2 f^2 \eta}{\rho v^3} \quad (2)$$

where η is the dynamic viscosity. Equation (1) neglects the effects of shear viscosity and is related to the Stoke's classical attenuation coefficient α_s ²² through the simple relation.

FIGURE 4 Line microstructures of 1×100 pixels patterned on 2-mm-thick PTFE with 2.2 MeV protons with a fluence of 1×10^{15} ions.cm⁻². Panels (a) show the results of as-irradiated samples. Panels (b) show the results of post-irradiation treatment with distilled water at 60°C without the action of ultrasound waves. Panels (c) show the results of post-irradiation treatment with 6 M NaOH at 60°C without the action of ultrasound waves. Each panel represents results obtained from independent measurements and post-irradiation treatments

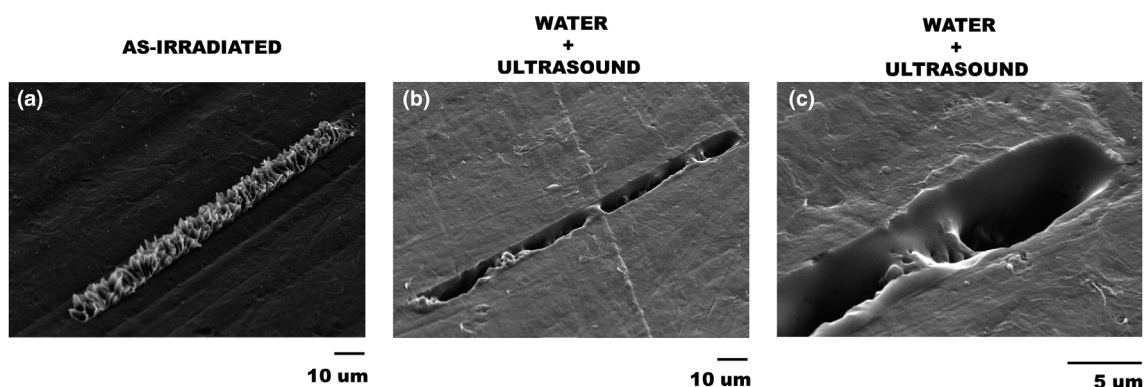
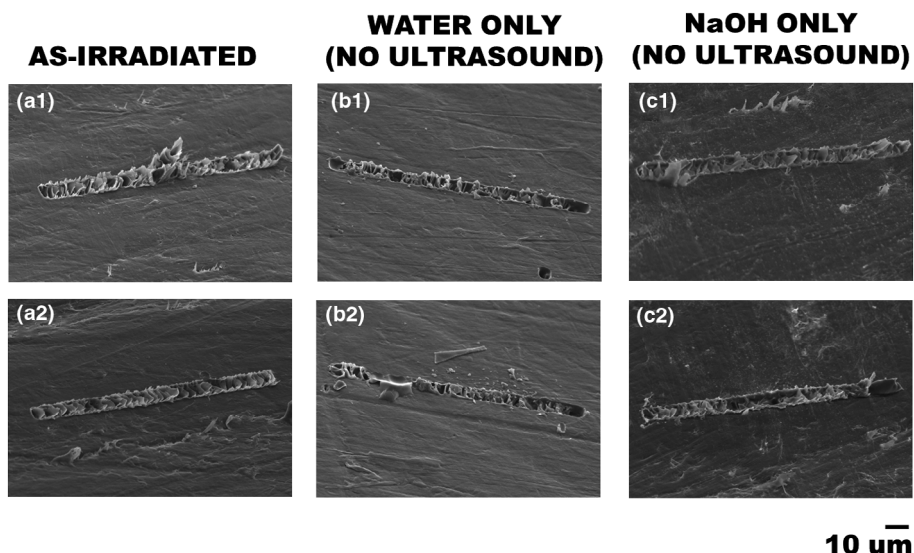


FIGURE 5 Line microstructures of 1×100 pixels patterned on 2-mm-thick PTFE with 2.2 MeV protons with a fluence of 6×10^{14} ions.cm⁻². (a) Sample as-irradiated; (b) irradiated polymer after treatment with distilled water at 60 °C under the influence of a 40 kHz ultrasound waves; (c) zoom of the top end portion of the structure shown in panel (b)

TABLE 1 Values of density ρ , vapor pressure VP, viscosity η , speed of sound v , and wavelength λ of the materials used in this work at particular temperatures T

Material	T (°C)	ρ (kg/m ³)	VP (kPa)	η (μPa.s)	v (m/s)	λ (m)
Water	60	983.4 ^a	19.932 ^d	466 ^g	1551.0 ⁱ	0.0388 ^k
NaOH 6 M	60	1196.0 ^b	14.665 ^e	1700 ^e	2284.2 ^j	0.0571 ^k
PTFE	21	2168.9 ^c	1.2×10^{-8f}	2.3×10^{14h}	1329.0 ^c	0.0332 ^k

^aReference 24.

^bReference 25.

^cReference 26.

^dReference 27.

^eReference 28.

^fReference 29.

^gReference 30.

^hReference 31.

ⁱReference 32.

^jReference 33.

^kEquation (1).

$$\alpha_s = \frac{4}{3}\alpha_k \quad (3)$$

Moreover, when ultrasound waves travel through different materials, they can be reflected in the interface between these materials through the relation²³

$$R = \left(\frac{Z_1 - Z_2}{Z_1 + Z_2} \right)^2 \quad (4)$$

where Z represents the acoustic impedance of materials 1 and 2 and R is the reflection factor. The acoustic impedance is simply given by

$$Z = \rho v \quad (5)$$

Finally, the transmission factor T through the interface of materials 1 and 2 is related to the reflection factor R by

$$T = 1 - R \quad (6)$$

With these parameters, the absorption coefficients and the acoustic impedances were calculated through Equations (2), (3), and (5) and are shown in Table 2. Comparing water with NaOH, Table 2 reveals that the power of absorption is slightly higher for water while the acoustic impedance is about twice higher for NaOH. Finally, PTFE has much higher absorption coefficients than water and NaOH and a similar acoustic impedance of NaOH.

Table 3 shows the reflection and transmission factors calculated with Equations (4) and (6) for the interfaces water/PTFE and NaOH/PTFE. Basically, there is no reflection on the NaOH/PTFE interface since their acoustic impedances are practically the same. On the other hand, about 9.5% of the waves are reflected on the water / PTFE.

Table 4 summarizes the comparison between those parameters from water and 6 M NaOH through the relative difference between them given by

$$\Delta P = \frac{P_{\text{water}} - P_{\text{NaOH}}}{P_{\text{water}}} \quad (7)$$

Material	T (°C)	α_k (m ⁻¹)	α_s (m ⁻¹)	Z (kg/m ² /s)
Water	60	4.011×10^{-6}	5.348×10^{-6}	1.525×10^6
NaOH 6 M	60	3.767×10^{-6}	5.022×10^{-6}	2.732×10^6
PTFE	21	1.4268×10^{12}	1.902×10^{12}	2.882×10^6

Note: The calculations were carried out with the values shown in Table 1.

where P_{water} and P_{NaOH} are the parameters shown in Tables 1–3 for water and 6 M NaOH, respectively. This table reveals that the relative differences of vapor pressure, reflection factor and Kirchoff absorption coefficient parameters are larger for water than NaOH. On the other hand, NaOH relative difference parameters including density, viscosity, wavelength, acoustic impedance and transmission factor are larger than those of water. Certainly, these differences are responsible for the remarkable difference in the performance of both liquids as far as post-irradiation treatment is concerned.

The primary mechanism behind the development of the patterned structures with ultrasonic waves is cavitation, which consists of nucleation, creation of cavitation bubbles, growth, and collapse.¹⁴ Cavitation is essentially a mechanical process and therefore it could generate heat during its interaction with the propagating medium. In the present case, molecules from the liquid media vibrate under the influence of viscous interactions and therefore some mechanical energy is indeed transformed into thermal energy. However, due to the low compressibility of the media, little or no heating of the media is to be expected. Moreover, Table 2 suggests that the media absorption coefficients are several orders of magnitude smaller than that of PTFE. Therefore, most energy deposited in the system is retained by the PTFE in the interface polymer/liquid, thus allowing the cavitation process to take place. In this way, very little energy remains in the liquid media to raise their temperature significantly.

For the present study, the presence of vapor nuclei and gases trapped in the crevices created by the proton beam are potential seeds for the creation of the cavitation process. Despite this mechanism depends on parameters like temperature of the medium and the frequency of the

TABLE 3 Reflection and transmission factors calculated with Equations (4) and (6), respectively, considering the interfaces water/PTFE and NaOH/PTFE

Interface	Reflection (%)	Transmission (%)
Water/PTFE	9.48	90.52
NaOH/PTFE	0.072	99.93

Note: The calculations were carried out with the acoustic impedances shown in Table 2.

TABLE 2 Values of Kirchoff absorption coefficients α_k (Equation (2)), Stokes absorption coefficients α_s (Equation (3)), and acoustic impedances Z (Equation (5))

TABLE 4 Relative differences between water and NaOH parameters with respect to the water values

$\Delta\rho$	ΔVP	$\Delta\eta$	$\Delta\nu$	$\Delta\lambda$	$\Delta\alpha_k$	ΔZ	ΔR	ΔT
-21.6%	+26.4%	-264.8%	-47.3%	-47.2%	+6.1%	-79.1%	+99.2%	-10.4%

Note: The results were calculated with Equation (7) using those values presented in Tables 1–3.

ultrasound, these parameters are the same for both water and NaOH solution. Conversely, parameters like viscosity and vapor pressure play important roles in the cavitation process as well. Indeed, smaller viscosities and larger vapor pressures enhances the production of cavitation.¹⁴ In this respect, Table 4 favors water's performance as a post-irradiation treatment. That is the reason water is considered a medium with great potential for the cavitation process to take place.¹³

Table 2 indicates that the NaOH acoustic impedance is larger than that of water. That means that the impedance mismatch is larger between water and PTFE than NaOH and PTFE (Table 2). The larger the impedance mismatch the stronger the scattering of waves in the interface is, thus leading to a pronounced wave multiple scattering in the interface.¹³ This process could help dissipate the energy in the crevices created by the proton beam, thus enhancing the cavitation process when water is used as a propagating medium. The much larger reflection factor of water and PTFE shown in Table 3 is directly related to the differences observed for the acoustic impedances shown in Table 2.

4 | CONCLUDING REMARKS

Polytetrafluoroethylene (PTFE) foils were micropatterned using the proton beam writing (PBW) technique in order to evaluate the etching process through a combination of either 6 M NaOH or distilled water under the action of 40 kHz ultrasound waves. For patterns produced with a 2.2 MeV proton beam with a fluence of $1 \times 10^{15} \text{ H}^+ \cdot \text{cm}^{-2}$, the results indicate that distilled water and ultrasound waves constitute a very efficient, simple and relatively cheap post-irradiation treatment for this polymer. This etching procedure is capable of removing rough ridges and producing good aspect ratio structures. On the other hand, the use of 6 M NaOH solution instead of distilled water under the same experimental conditions does not promote any substantial result on the post-irradiated structures. The effectiveness of water as an ultrasound wave propagation medium is due to important parameters including viscosity, vapor pressure and acoustic impedance among others.

In short, the present study offers a simple and efficient solution as a post-irradiation treatment of PTFE free of any chemical agents. Applications like the production

of PTFE membranes could profit from the present results.

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AUTHOR CONTRIBUTIONS

Deiverti de Vila Bauer: Conceptualization (equal); formal analysis (lead); investigation (lead); methodology (equal); validation (lead); visualization (lead); writing – original draft (supporting). **Rafaela Debastiani:** Visualization (supporting); writing – original draft (lead); writing – review & editing (equal). **Claudia Telles de Souza:** Conceptualization (equal); methodology (equal); resources (equal); supervision (equal); validation (equal); visualization (equal). **Livio Amaral:** Conceptualization (equal); data curation (supporting); investigation (equal); methodology (equal); supervision (supporting); visualization (equal); writing – original draft (supporting). **Johnny Ferraz Dias:** Conceptualization (equal); funding acquisition (lead); methodology (equal); project administration (lead); resources (lead); supervision (lead); visualization (equal); writing – original draft (equal); writing – review and editing (lead).

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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