

Scanning Electron Microscopy Analysis of Argon Plasma Treated Jute Fibre

Md. Masroor Anwer, Md. Abdullah Kayser, Shuranjan Sarkar, S M Mahruf Hossain, Sharmin Akter, Tahnin Bintay Kamal, Fatema Nusrat Jahan

Abstract— Low temperature plasma (LTP) treatment, a kind of environmentally friendly surface modification technique, was applied to biodegradable and ligno-cellulosic jute fibre with the use of nonpolymerizing argon (Ar) gas at various discharge power levels of 50, 75 and 100 W and exposure times 5, 10, 15 and 20 min. with a flow rate of 0.2 L/min. By means of scanning electron microscopy (SEM), the influence of treatment time and discharge power on the surface morphology of the surface of LTP treated jute were studied and were compared with that of raw jute. SEM microphotographs reveal that the roughness of the fibre surfaces increases with the increase of discharge power and exposure time. This is caused due to the bombardment of high energetic ions on the fibre surface and the fibres become sputtered.

Index Terms— Nonpolymerizing gas, Jute fibre, Plasma treatment, Exposure time and Discharge power.

I. INTRODUCTION

Scanning electron microscopy (SEM) is used for inspecting topographies of specimens at very high magnifications using a piece of equipment called the scanning electron microscope. SEM is a special type of microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity (1). SEM magnifications can go to more than $500,000 \times$ but most semiconductor manufacturing applications require magnifications of less than $3,000 \times$ only. SEM inspection is often used in the analysis of die/package cracks and fracture surfaces, bond failures, and physical defects on the die or package surface. SEM produces types of signals that include secondary electrons, back-scattered electrons (BSE), characteristic X-rays, light, specimen

current and transmitted electrons. Secondary electron detectors are common in all SEMs. Interactions of the electron beam with atoms produce signals at or near the surface of the sample. In the most common or standard detection mode, secondary electron imaging or SEI, the SEM can produce very high-resolution images of a sample surface, revealing details about less than 1 to 5 nm in size. A wide range of magnifications is possible, from about 10 times to more than 500,000 times, about 250 times the magnification limit of the best light microscopes (2-3).

In a typical SEM, an electron beam is thermionically emitted from an electron gun fitted with a tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be heated for electron emission and because of its low cost. The electron beam, which typically has an energy ranging from 0.5 to 40 keV, is focused by one or two condenser lenses to a spot about 0.4 to 5 nm in diameter. The beam passes through pairs of scanning coils or pairs of deflector plates in the electron column, typically in the final lens, which deflect the beam in the x and y axes so that it scans in a raster fashion over a rectangular area of the sample surface (4). When the primary electron beam interacts with the sample, the electrons lose energy by repeated random scattering and absorption within a teardrop-shaped volume of the specimen known as the interaction volume, which extends from less than 100 nm to around $5 \mu\text{m}$ into the surface. The energy exchange between the electron beam and the sample results in the reflection of high-energy electrons by elastic scattering, emission of secondary electrons by inelastic scattering and the emission of electromagnetic radiation, each of which can be detected by specialized detectors. The beam current absorbed by the specimen can also be detected and used to create images of the distribution of specimen current. Image magnification in the SEM is not a function of the power of the objective lens. In SEM condenser and objective lenses, has a function of focusing the beam to a spot, and not to image the specimen. In a SEM, magnification results from the ratio of the dimensions of the raster on the specimen and the raster on the display device (5).

II. MATERIALS AND METHODS

A. Low Temperature Plasma Treatment

Jute fibres (*Corchorus Olitorius* or Tossa jute) were collected from the local market in Bangladesh. The fibres were introduced into a bell jar type capacitively coupled glow discharge reactor as shown in figure 1.

Md. Masroor Anwer, Principal Scientific Officer, Textile Physics Division, Bangladesh Jute Research Institute, Dhaka-1207, Bangladesh, Cell No. +8801552342233

Md. Abdullah Kayser, Senior Scientific Officer, Textile Physics Division, Bangladesh Jute Research Institute, Dhaka-1207, Bangladesh

Shuranjan Sarkar, Senior Scientific Officer, Chemistry Division, Bangladesh Jute Research Institute, Dhaka-1207, Bangladesh

S M Mahruf Hossain, Scientific Officer, Chemistry Division, Bangladesh Jute Research Institute, Dhaka-1207, Bangladesh

Sharmin Akter, Scientific Officer, Textile Physics Division, Bangladesh Jute Research Institute, Dhaka-1207, Bangladesh **Tahnin Bintay Kamal**, Scientific Officer, Mechanical Processing Division, Bangladesh Jute Research Institute, Dhaka-1207, Bangladesh

Fatema Nusrat Jahan, Scientific Officer, Pilot Plant and Processing Division, Bangladesh Jute Research Institute, Dhaka-1207, Bangladesh.



Fig. 1 Schematic diagram of jute fibre and position of it in the glow discharge reactor

To sustain a glow discharge i.e. for getting proper and uniform plasma, the conductive electrodes are separated 0.035 m apart from each other. In order to exposed all through uniform LTP treatment on the samples surface, the fibres (length of each fibre: 0.08 m) were inserted in between the two metallic electrodes by a carrier. After placing jute fibres between pair of electrodes, the glow discharge chamber was evacuated by a rotary pump at a pressure of 1.33 Pa. Ar was considered as plasma gas for treating the jute fibre. In all treatments, both process gases were introduced separately into the reaction chamber by a flowmeter at a flow rate of 0.2 L/min. which is maintained by a needle valve. The discharge powers were adjusted at 50, 75 and 100 W at a line frequency of 50 Hz with the duration of exposure times of LTP treatment of fibres were 5, 10, 15 and 20 min. Figure 2 shows a flow chart of a plasma treatment system which was used in this experiment.

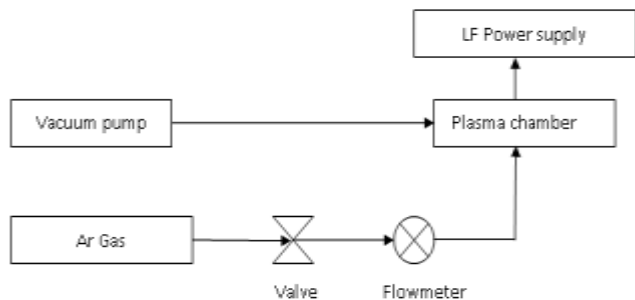


Fig. 2 Flow chart of the plasma treatment set-up

After plasma treatment has been finished, and the vacuum chamber was vented, jute samples were then removed and handled carefully in order to avoid possible surface contamination to the fibres. Later, the plasma treated fibres were immediately placed into a desiccator with the silica gel.

B. Scanning Electron Microscopy

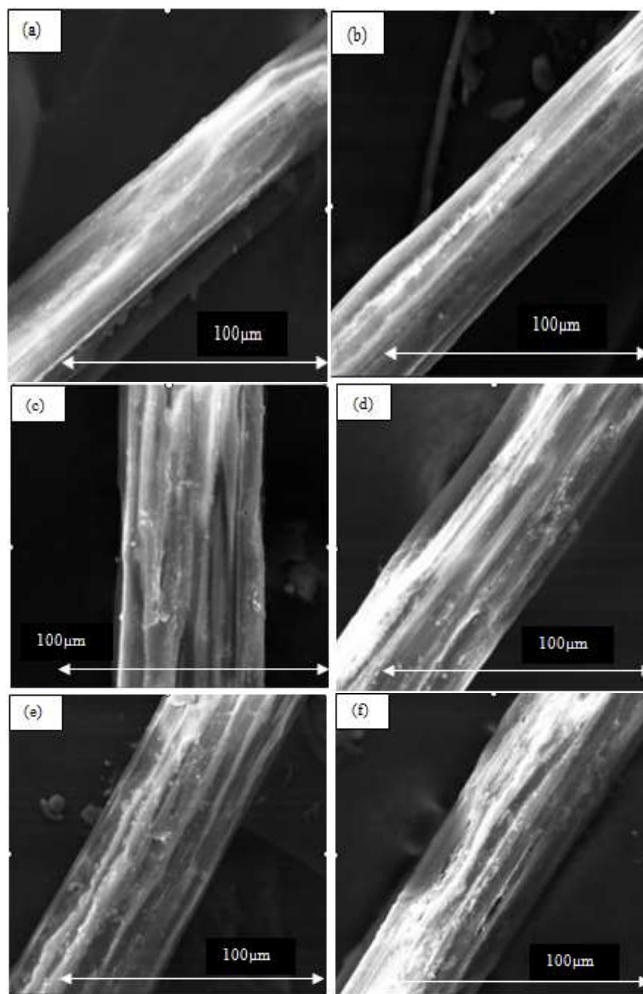
The surface morphology of the raw jute and LTP treated jute fibres were examined with a SEM (Model: S50, FEI Quanta Inspect, The Netherlands) operated at 25 kV in low vacuum control at 50 Pa. The surfaces of the jute fibres were examined at 2000× magnification. Figure 3 shows a photograph of SEM machine which was used in this experiment.



Fig. 3 The system photograph of scanning electron microscope

III. RESULTS

The SEM microphotographs of raw jute fibres and LTP treated jute fibres with Ar gas at various discharge powers (50, 75 and 100 W) and exposure times (5, 10, 15 and 20 min.) are taken in this experimental work and are presented in figure 4.



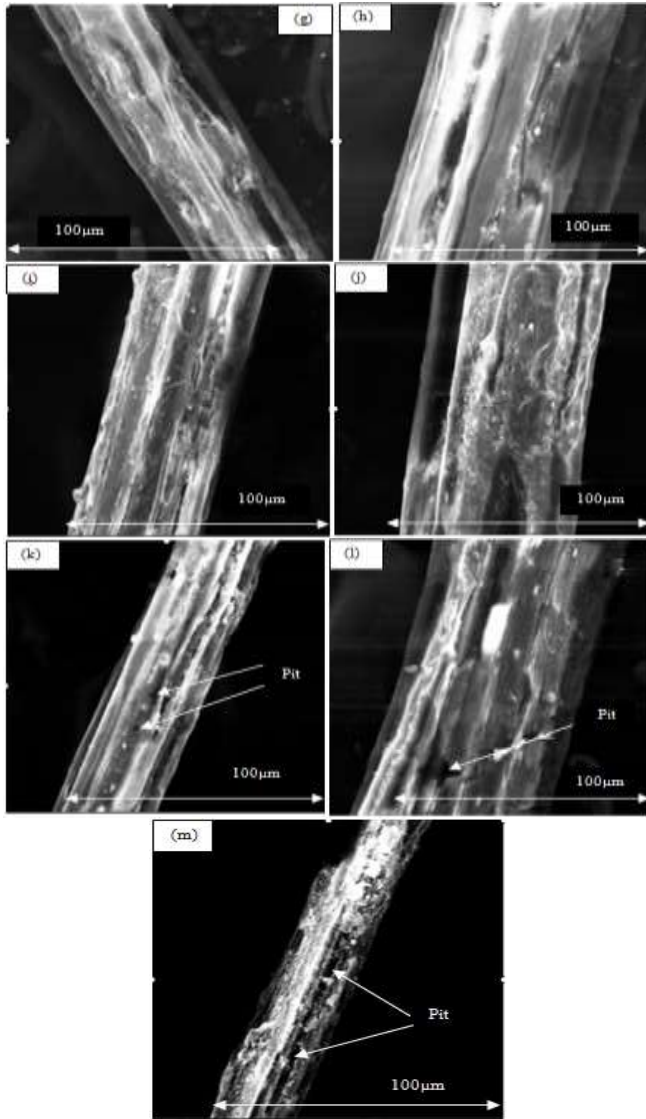


Fig.4 Surface morphologies of Ar plasma treated jute fibres: (a) Raw jute (b) 50 W, 5 min. (c) 50 W, 10 min. (d) 50 W, 15 min. (e) 50 W, 20 min. (f) 75 W, 5 min. (g) 75 W, 10 min. (h) 75 W, 15 min. (i) 75 W, 20 min. (j) 100 W, 5 min. (k) 100 W, 10 min. (l) 100 W, 15 min. and (m) 100 W, 20 min.

IV. DISCUSSION

It is seen from SEM photographs of raw jute as well as LTP treated jute fibres that physical changes such as micro-deformations as well as voids and cracks on the surface morphology of the fibre caused by LTP treatment. It can be visually verified that the surface of raw jute in figure 4 (a) is smooth and shows no roughness on the surface, whereas figures 4(b-m) illustrate the action of Ar plasma treatment on the fibre surface for different exposure times and discharge powers. Figures 4(b-h) associated with different discharge powers and treatment times present a surface like rough and fragmented. Figures 4(i-j) associated with different discharge powers and treatment times show more roughness and also degradation on fibre surface. Besides, figures 4(k-m) show the formation of pits on the fibre surface, may be due to the sputtering mechanism of plasma causing more degradation on the fibre surface (6-8).

According to this sputtering mechanism, the longer the duration of LTP treatment, the more severe the modification of the fibre surface is. Also, the higher the discharge power

applied, the more kinetic energy the plasma species will carry, resulting in strong intensity of plasma action (9). Hence, there will be a change in the total amount of the excited particles inside the plasma and their energy level accordingly when the input power increases under a constant pressure, resulting in the increase in the charged ion concentration (10-13). As a result, the fibres are more sputtered with exposure times as well as discharge powers. Some areas of plasma treated fibre are ruptured due to energetic gaseous ion bombardment to the surface. It is to be noted that jute fibre contains organic molecules. Gaseous plasmas may also introduce degradation and chain scission in the fibre (14). Plasma gases may also react with some polymer constituents inside the fibre and ultimately cause rupture and increase the surface roughness, voids and spaces in the jute fibres. Thus, plasma treatments result in significant change of morphology of the fibre surface.

ACKNOWLEDGEMENT

We would like to express our heartfelt thanks to the authority of Bangladesh Atomic Energy Centre, Dhaka, Bangladesh and the Bangladesh University of Engineering and Technology (BUET), Dhaka, Bangladesh for the conduction of our experimental work.

REFERENCE

- [1] Goldstein, G. I., Newbury, D. E., Echlin, P., Joy, D. C., Fiori, C. and Lifshin, E. 1981. Scanning Electron Microscopy and X-ray Microanalysis, New York: Plenum Press, 35(1):10-36.
- [2] Jeffree, C. E. and Read, N. D. 1991. Ambient and Low-temperature Scanning Electron Microscopy, London: Academic Press, 313-413.
- [3] Conley, T. R. 1975. Infrared Spectroscopy, 2nd edition, Allyn and Bacon, Inc., Boston, U.S.A.
- [4] Silverstein, R. M., Bassler, G. C. and Morrill, T. C. 1981. Spectrometric Identification of Organic Compounds, 4th edition, John Wiley and Sons, New York.
- [5] Sharma, B.K. 1993 Spectroscopy, Krishna prakashna Media Ltd, Meerut, India.
- [6] Sinha, E 2009, Effect of cold plasma treatment on macromolecular structure, thermal and mechanical behavior of jute fiber, *J. Indust. Text.*, 38(2): 317-338.
- [7] Yoldas, S., Kutlay, S., Mehmet, S., Haci, A. G. and Ismail, H. T., 2008. The influence of oxygen plasma treatment of jute fibres on mechanical properties of jute fibres reinforced thermoplastic composites, *Surf. Coat. Technol.*, 55(1): 45-50.
- [8] Molina, R., Canal, C., Bertran, D., Tascon, E. and Erra, L., 2009. Low temperature plasma modified wool fabrics: surface study by SEM, *J. Fib. Polym.*, 10(2): 781-786.
- [9] Chi-wai, K., Kwong, C., and Yuen, M., 2008. Surface characterization of low- temperature plasma treated wool fibre, *Text. Res. J.*, 8(3): 512-518.
- [10] Hatice, A. K., Esen, O., Asli, D., Ismail, C. K., Tulin, O. and Hakan, A., 2009. Effects of atmospheric pressure plasma treatments on some physical properties of wool fibers, *Surf. Coat. Technol.*, 203(1): 3178-3183.
- [11] Navancetha, N. P. and Selvarajan, K. V., 2007. Non-thermal plasma treatment for hydrophilicity improvement of grey cotton fabrics, *J. Mater. Process. Technol.*, 35(3): 48-55.
- [12] Pandiyaraj, N. K. and Selvarajan, V., 2007. Non-thermal plasma treatment for hydrophilicity improvement of grey cotton fabrics, *J. Mater. Process. Technol.*, 199(2): 130-139.
- [13] Sinha, E. and Panigrahi, S., 2009. Effect of plasma treatment on structure, wettability of jute fiber and flexural strength of its composite. *J. Compos. Mater.*, 43(2): 1791-1802.
- [14] Socrates, G. 2001. Infrared and Raman Characteristic Group Frequencies, Wiley, West Sussex, England, 3(1): 58-62