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Conductive Silver Coating on Polyether Ether Ketone (PEEK) for Thermoplastic Textile Composite

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Abstract

The aim of this work is to develop the electric conductivity on inert Polyether ether ketone (PEEK) fiber surface by applying metallic layers with the help of wet chemical procedure. The wet-chemical functionalization and atmospheric plasma modification of the inert fiber surface of PEEK is done before it is metalized by using polyamine with coating procedure.The wet-chemical procedure in this research practical is based on the use of amines, which consist of at least two amine groups, and which are well-suited to form a silver diamine complex.The PEEK fiber was coated by cationic silver. After the chemical reduction, the cationic silver turns into metallic particles on the surface of PEEK fiber. The measurement of electrical resistance after silver coating is also done.

Keywords: PEEK, Silver coating, Wet chemical method, Plasma.

I. Introduction

Polyether ether ketone (PEEK) is an organic semi crystalline thermoplastic fiber with excellent mechanical, thermal and chemical properties. PEEK polymer has a glass transition temperature at around 143°C and melts at around 343°C. PEEK polymer is used as a matrix for thermoplastic composite prepregs made of carbon, glass or aramid continuous fibers. Its outstanding properties make it an excellent substitute for metals and thermoset applications PEEK carbon composites are capable of withstanding continuous operating temperatures of up to 260°C in low stress applications and 120°C wet for aerospace structural application PEEK Aryl polyether ether ketone (PEEK) is first introduced by Imperial Chemical Industries (ICI) in 1978. Its excellent properties have allowed for its use in a variety of high performance applications. This polymer has recently been adopted in the aviation and automotive industries where conventional thermally durable materials are being replaced by lighter weight, high thermal stability polymers *Su Weifeng, et al., 2013;*

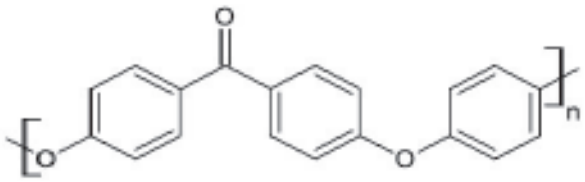

Figure 1: Chemical structure of PEEK

Poly (aryl ether ether ketone) PEEK is an aromatic polymer. Its molecular structure is shown in Figure 1. PEEK has an oxygen atom between phenyl groups. Plasma treatment enhances the PEEK/Ag interface adhesive strength. Many reports have indicated that oxygen plasma radiation on polymer surfaces and subsequent metallization improves adhesive strength (Kazuo Narushima & Hiroki Ikeji *et al;* 2009)

The target of this work is to bring metallic substances on the fiber surface. As the surface of PEEK is inert so the surface is modified with atmospheric Plasma. Plasma acts only on the surface and does not change their internal structure. Atmospheric plasma produces oxygen functional groups and etches the surface, which contributes to adhesive strength. For this reason, plasma treatment is a suitable method for the metallization of polymers using silver (Ag). The silver particles are bonded to each other by van

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der Waals force and they are agglomerated into large particles. Although the van der Waals force in comparison to the covalent chemical bonds or ionic bonds seems to be weak, the silver nano particles can be fixed on the fiber surfaces. With this property and by varying the experimental parameters and functionalizations, cation deposition and reductive metallization, it is possible to obtain PEEK fiber surfaces that are completely covered with silver nano particles (T. Onger *et al;* 2011) As metal coated PEEK fibers are conductive, their electrical behaviour can provide information about the strain, damage and temperature acting on them in real time via electrical measurement (M. M. B Hasan *et al;* 2021)

II. Materials and Methods

A. Materials

• PEEK yarns (25 Filaments, 46 Tex) collected from Zyex (A part of the multinational ICI) were used for this work.

• For the Fiber surface activation Formic acid (85%) (Kremer pigmente Gmbh) was used.

• The silver diamine complex was formed from silver nitrate (Grüssing, Germany) and 25% of ammonia (VWR Prolabo, Germany). Tetraethylenpentamine (TEPA) (Fluka, Germany) was used for corresponding coating.

• For the reductive metallization L (+) ascorbic acid (Appli chemistry, Germany) was considered.

B. Methods

Plasma modification:

The plasma treatment was carried out by using atmospheric pressure. The principle was "Corona pretreatment system-AS coating star (ASCS). For the Plasma treatment of the PEEK fiber surface, parameters were electrode distance 1.7 mm, 2 kW, Room temperature, 16-33 kHz, 3 Minutes were considered. The working principle of this plasma system is based on the dielectric barrier discharge (DBD) system.

Silver coating with amine:

For the direct silvering, a solution consisting of amine (TEPA) and silver diamine complex (silver nitrate and ammonia solution) was prepared (Equation 1).

$$Ag(NO_3) + 2NH_3 = [Ag(NH_3)_2]^+ + [NO_3] \quad (1)$$

1%, 3%, 5% AgNO3 solution have been used for silver coating in this work. The direct silvering of the surface of the pre-treated PEEK fibers was carried out for one hour at room temperature. After that the specimens were dried at room temperature for one hour. This way, ionic silver in a wet-chemical process was fixed on the fiber surface and reduced to a metallic form with a suitable L (+) ascorbic acid solution. A second silvering step was applied with a silver diamine complex (5% $AgNO_3$ + 50 ml Ammonia solution) and L (+) ascorbic acid ($C_6H_8O_6$) to cause a second reduction. Ionic silver has been reduced by the use of L (+) ascorbic acid to metallic silver (Equation 2) [4]

$$2Ag^+ + C_6H_8O_6 = 2Ag^0 + C_6H_8O_6 + 2H^+ \quad (2)$$

After the second silvering and second reduction, the specimens were rinsed with cold water then dried at room temperature. After that the specimens were thermofixiert at 120°C for 3 minutes. The principle of the connectivity possibilities of applied silver particles on the fiber surface is shown in figure 2.

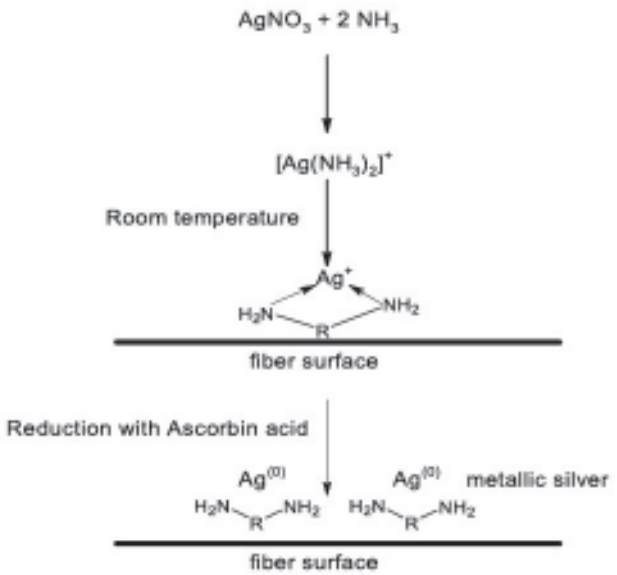


Figure 2: Reaction possibilities among $AgNO_3$, NH_3, and Fiber

III. Result and Discussion

A. Ninhydrin test

To proof the generated primary amine groups on the PEEK fiber surface, a dyeing test was carried out with 1% of ninhydrin solution. The colours of PEEK fibers were changed. In figure 3 a) and b) it has been shown. In case of PET fiber after ninhydrin test the colour becomes blue violet T. Onger *et al;* 2011) The colour of PEEK fiber became dark green after ninhydrin test.

Microscopic analysis

The fibers specimens were wetted with Glycerin and The changes of topography has been investigated by Axiocam MRC 5.

Proof of functionality

Ninhydrin test was carried out for the proof of generated primary amine groups on the PEEK fiber surface.1% ninhydrin solution (3 to 5 drops) was applied on the fiber surface.

Resistance measurement

Resistance was measured at different lengths of silver coated PEEK fiber by Ohm Meter.

SEM analysis

To assess the changes in surface topography of surface modified PEEK filaments, the scanning electron microscopy (SEM) is done to determine the functional groups and wettablility on plasma treated PEEK filament more precisely contact angle and surface energy have been measured before and after plasma treatment. The details of plasma treatment as well stress-strain behavior have also been measured (A.B.M Foisal *et al;* 2014), (A.B.M Foisal *et al;* 2014)

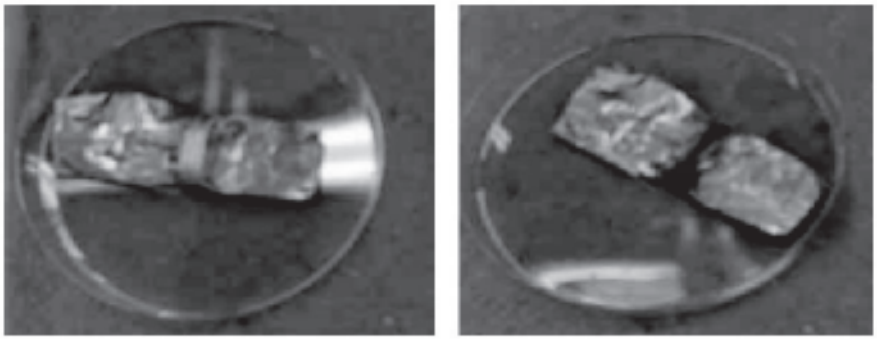


Figure 3: a) Before ninhydrin test the PEEK fibers, b) After ninhydrin test the colour were changed to dark green.

B. Microscopic analysis

The images of untreated fiber and treated fiber show changes in surface characteristics (figure 4 and figure5). The modified surfaces are identified from the following images enlarged at 200 times.

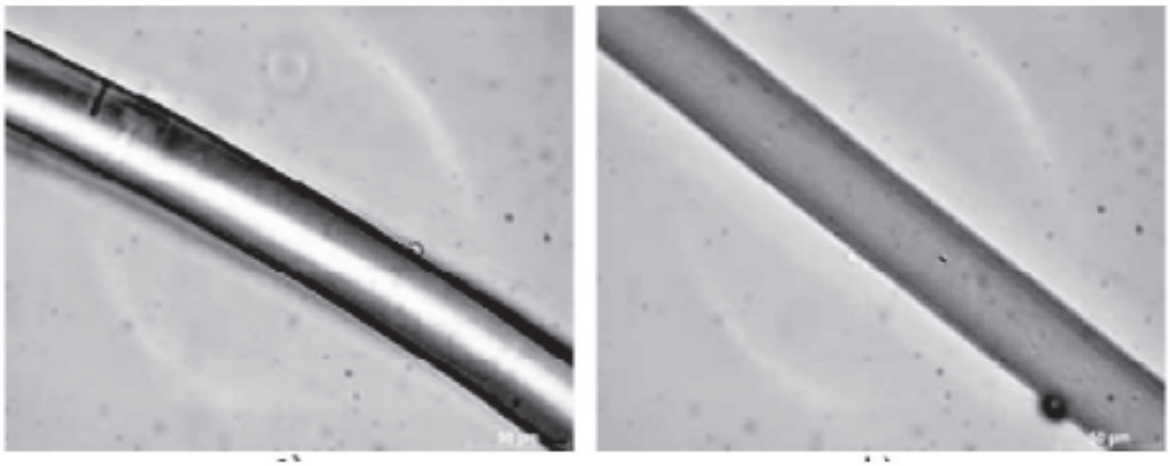


Figure 4: microscopic view (200 fold magnification) a) Original PEEK and b) Plasma treated PEEK

Figure 4 a) shows the image of original PEEK fiber (as received from Zyex), which presents a clean, smooth,and homogeneous surface. There were no obvious grooves and ruts.

Figure 4 b) shows the plasma treated PEEK fiber surface. The surface was dull, rough and etched effected due to oxidised.

In Figure 5 a) it is seen that the silver particles are distributed on the PEEK fiber surface. The silver particles are reflecting light in black ground.

Figure 5 b) shows that the silver particles on the fiber surface. The particles are distributed not uniformly on the surface.

In Figure 5 c) it is seen that the silver particles are distributed on one side of fiber. The silver particles are attached with the fiber surface.

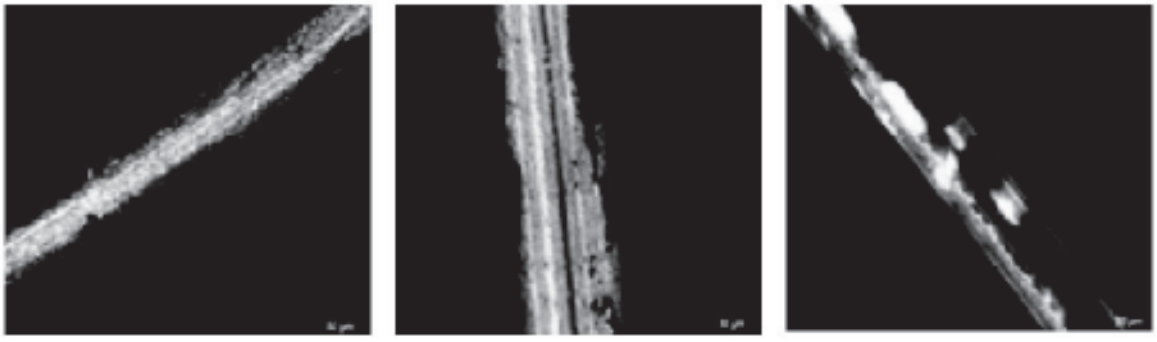
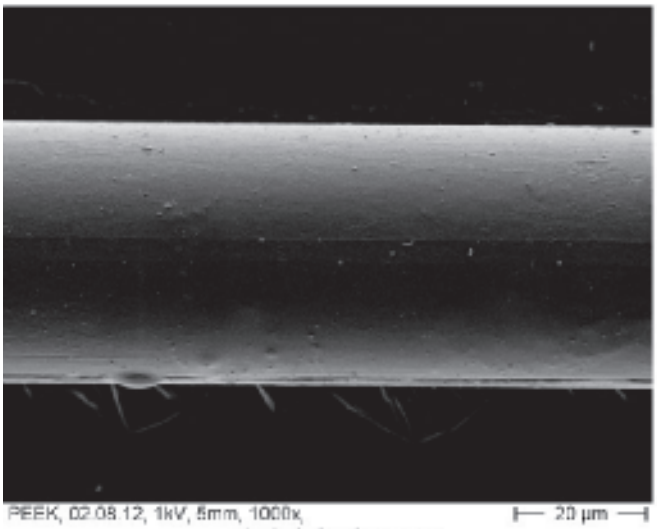
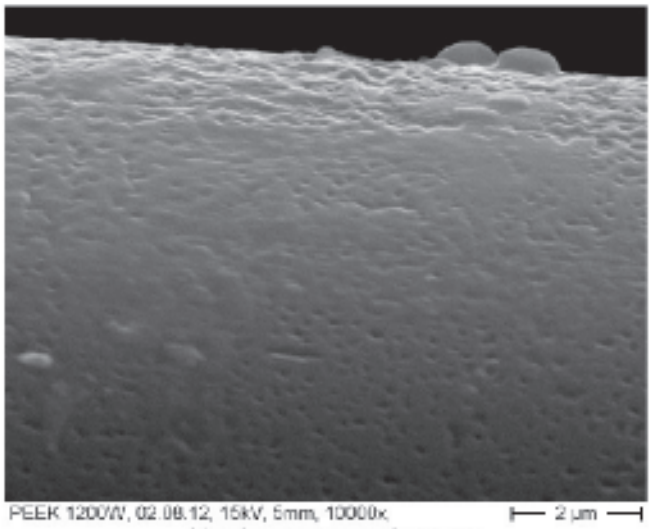


Figure 5: microscopic view (200 fold magnification) a) 5% AgNO3 coated PEEK b) 3% AgNO3 coated PEEK c) Plasma treated 3% AgNO3 coated PEEK

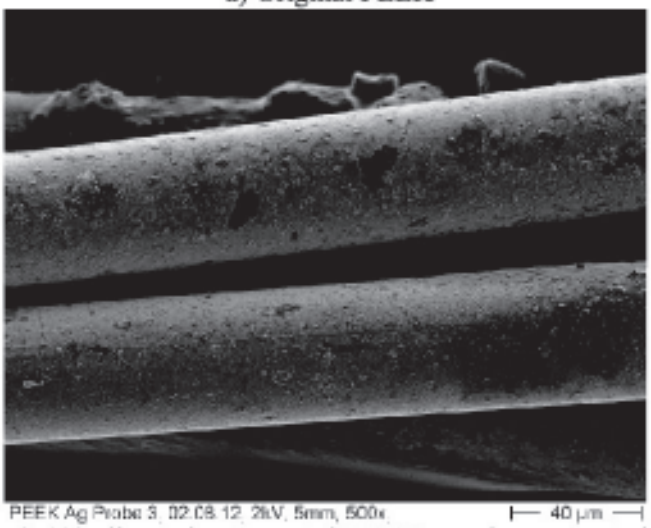
C. SEM analysis



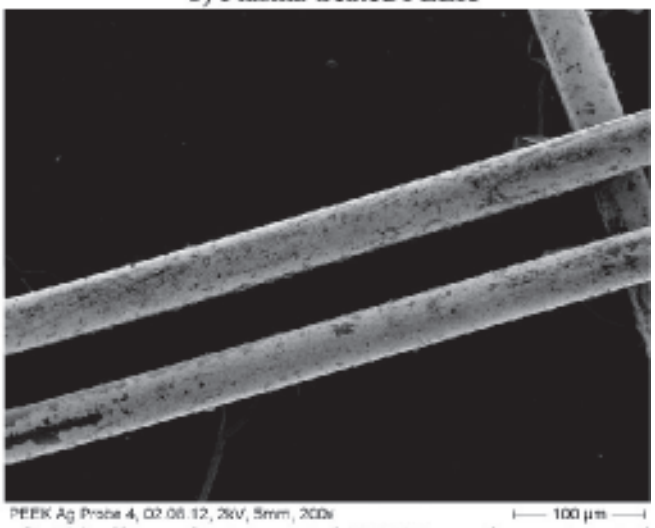
a) Original PEEK



b) Plasma treated PEEK



c) 3% silver nitrate coated PEEK on plasma treated PEEK



d) 5% silver nitrate coated PEEK on plasma treated of PEEK

Figure 6: Scan electron microscopic images of PEEK filament a) Original PEEK (1000x), b) Plasma treated PEEK (10000x), c) 3% silver nitrate coated PEEK on plasma treated PEEK, d) 5% silver nitrate coated PEEK on plasma treated PEEK.

Figure 6 a) shows that the surface of PEEK is smooth and without the roughness but after the plasma treatment the surface roughness is obvious and clearly understandable (in fig. 6 b).The silver particles coated on plasma treated PEEK filament surface which formed silver layer in 3% and 5% silver nitrate coated (in fig. 6c and 6d) respectively.

D. Resistance measurement

The comparison between without Plasma treated and plasma treated PEEK coated with silver (Ag) can be seen from the resistance measurement. Generally resistance increased with the increased

of length and decreased with the increase of cross sectional area of the coated fiber (equation 3)(M. H. Sadreay *et al;* 2013)

$$R_{yarn} = \rho_{coating}.L / A_{coat} \qquad (3)$$

R_{yarn} indicates the resistance, $\rho_{coating}$ indicates specific resistivity of coated yarn, L is length, and A_{coat} is cross sectional area of coated yarn. In comparison to Fig. 6 a) to 6 b) it is clearly seen that plasma treated PEEK has increased the resistance uniformly with the increase of length and up to 19 cm length resistance have been measured. However, in case of without plasma treated PEEK; resistance can be measured up to 6 cm. In figure 7 a) it is found that the electrical resistance increased with the increased of length at 5% $AgNO_3$ coated PEEK in Amine method and at 6 cm length resistance increased rapidly. Above 6 cm length there was no electric contact. As the coating was done at a small scale of fibers and metallic coating was not uniform on the fiber surface. In fig.7 b) after the treatment of plasma on the PEEK fiber surface and then coated with 5% $AgNO_3$ the electrical resistivity was increased with the increased of length uniformly. It indicates that the surface of fiber was functionalized by plasma and adhesion between fiber surface and silver (Ag) particles were uniform. Resistivity was found up to 19 cm length.

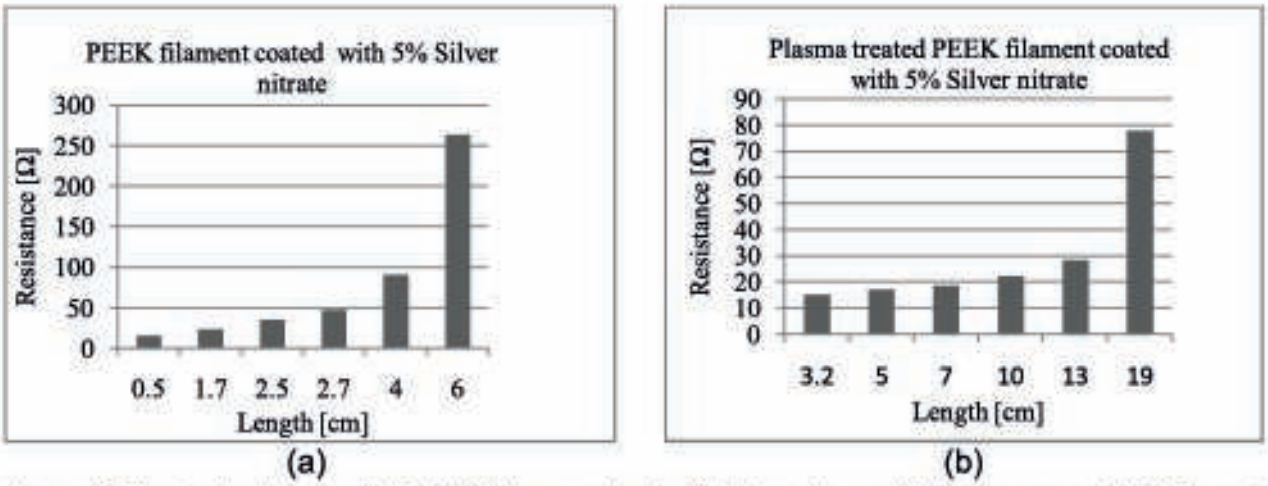


Figure 7: Electrical resistance of (a) PEEK fibre coated with 5% silver nitrate and (b) plasma treated PEEK coated with 5% silver nitrate

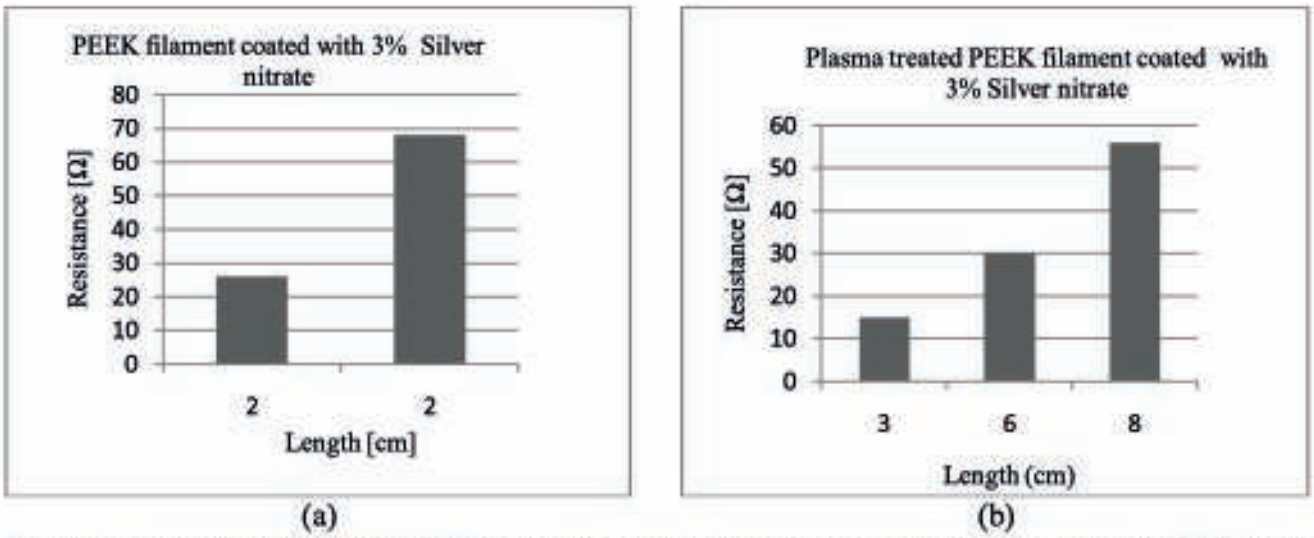


Figure 8: Electrical resistance of (a) PEEK fibre coated with 3% silver nitrate and (b) plasma treated PEEK coated with 3% silver nitrate

Figure 8 a) and b) show the comparison of resistance between without plasma treated and plasma treated PEEK fibers coated with 3% $AgNO_3$. The resistances of 2 cm length at different places are not uniform (in fig.8a). In fig.8 b) it is seen that the resistance increased linear with the increase of length.

IV. Conclusion:

This work has led the following conclusions.

i. The wet-chemical methods are characterized by the use of polyamines to achieve the PEEK fiber surface covered with evenly distributed silver particles.

ii. Plasma treatment produces oxygen functional groups and an etching action on the surface of PEEK. An etching action certainly contributes to adhesive strength at Ag/PEEK interface.

iii. Electrical conductivity depends on the uniformly distribution of silver particles on the PEEK fiber surface. The decrease of Ag layer thickness on the PEEK fiber increases the sensitivity. So it must be considered not only the uniformly distribution of silver particles but also the layer thickness. Electrical resistance is inversely proportional to the cross sectional area of the coated PEEK fiber.

iv. Measurements of electrical resistance of Ag coated PEEK filament yarns are performed before and after their integration into the textile reinforced thermoplastic composite (M.M.B. Hasan *et el;* 2013) (A.M.B. Foisal *et el;* 2017)

v. The resistance change (%) due to abrasion shows that the silver coated PEEK filaments can also withstand the rubbing with metal surface (A.M.B. Foisal *et el;* 2012)

Further investigation of silvered PEEK filaments to characterize the silver layers and the thermal and hydrothermal behavior (shrinkage%, wash fastness, rubbing fastness, mechanical deformation of silver layer, and tensile and shear strength) is needed in the future. It also includes the electrical conductivity through the silver coated PEEK filament in terms of cross sectional area and volume need to be investigated.

References

http://en.wikipedia.org/wiki/PEEK

http://www.victrex.com/de/products/victrex-peek-polymers/victrex-peek- polymers.php

P.Patel, T.Richard Hull, R. E. Lyon, S. I. Staliorov, R. N. Walters, Sean Crowley, N.Safronava. Investigation of thermal decomposition and flammability of PEEK and its carbon and glass-fibre composites; J.polymdegradation and stability 96(2011)12-22

Kazuo Narushima and Hiroki Ikeji, SENT GAKKAISHI Vol.65.No.5 (2009) Shizuoka University, Hamamatsu, Japan.

T. Onger,Tong Cheng, Heike Hund, R. D. Hund, C.Cherif. Metallization of inert PET textile materials: Wet chemical silvering with natural and synthetic polyamine, Part I; Textile Research Journal ; 26 September 2011

M. M. B. Hasan, Ch. Cherif, A. Matthes. Early predictiction of failure of textilereinforced thermoplastic composites using hybrid yarns; j.compscitech. 2012.04.007

A.B.M. Foisal, Surface Modification of Polyether ether ketone (PEEK) filament yarn. Institutional Engineering and Technology (IET), ISSN 2076-3972(online), Green Global Foundation. Inst. Engg. Tech. 4(2):1-7(December 2014)

A.B.M. Foisal; Conductive coating of Polyether ether ketone (PEEK) filament yarn. 1st Textile Research Conference (TRC), 16 August 2014, TRC book of papers 2014, pp 1-4, ISSN 2413-9629 Dhaka, Bangladesh.

J.Rausch, E. Mäder. Health monitoring in continuous glass fibre reinforced thermoplastics: Manufacturing and application of interphase sensors based on carbon nanotubes; j.compscitech.2010.05.0018

M.M.B. Hasan, Ch. Cherif, A.B.M. Foisal, T. Onggar, R.D. Hund, A. Nocke,; Development of conductive coated Polyether ether ketone (PEEK) filament for structural health monitoring of composites; Composite Science and Technology 88 (2013) P-76-83.

M.M.B. Hasan, A.B.M. Foisal, T. Onggar, R.D. Hund, A. Nocke, Ch. Cherif; Conductive coated Polyether ether ketone (PEEK) filament for functional applications in Thermoplastic composites; 13th AUTEX world textile conference, 22nd -24th May, 2013 Dresden, Germany.

A. B. M. Foisal, Conductive Coated Polyether Ether Ketone (PEEK) Filament for Functional Applications in Thermoplastic Composites, 3rd International PEEK Meeting, April 27-28, 2017, WASHINGTON, D.C., USA

A.B.M. Foisal, Conductive coating of Polyether ether ketone (PEEK) filament yarn, Masterarbeit, Nr. 1420, ITM, TU Dresden, Germany 2012