USE OF CO_2 LASER IRRADIATION TO CARBONIZE ACRYLIC COATED GLASS MATS

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Abstract

Polyacrylonitrile (PAN) is commonly used as a raw material for making carbon fibers. This work is about dissolving waste acrylic fibers in suitable solvent to make thick acrylic solution. This solution is coated onto glass fiber mats. Acrylic coated glass mats are irradiated with CO2 laser and possibility of carbonizing acrylic is studied. These coated samples are preheated at different temperatures from 300 °C to 500 °C to stabilize Polyacrylonitrile before converting it to carbon structure. It is found that stabilization at 300°C works well in order to stabilize acrylonitrile prior to laser irradiation. Heat treated and non-preheated samples were irradiated by laser for studying possibility of carbonization of PAN polymer. It is found that laser treatment of PAN coated glass fiber mats produces electrically conductive lines on the surface of the material. Moreover the conductivity is increased when the material is preheated to 300°C before laser irradiation. It is also found that conventional heat treatment is not suitable for carbonizing PAN coated surface.

Keywords: PAN, Carbonization, Conductive nonwoven

1. INTRODUCTION

Acrylic fibers also known as Polyacrylonitrile (PAN) is a common material used as a precursor in manufacturing of carbon fibers. Carbonization of acrylic produces carbon fibers. Nowadays about 90% of carbon fibers are manufactured by thermal conversion of PAN precursor [1]. Usually acrylic precursors are heat treated at various temperatures for converting them into carbon fiber. The conversion is done in three steps which are oxidative stabilization, high temperature carbonization and graphitization.

Oxidative stabilization is done in temperature range 180~300°C [2,3]. Some researchers found 270°C the best suitable temperature for oxidative stabilization [4] however some studies also recommended higher temperatures exceeding 300°C for stabilization process [5]. If the temperature is very high the fiber can fuse or burn, but at very low temperatures incomplete stabilization can be the result. Once the fiber is stabilized it will not melt again when exposed to high temperatures.

Carbonization is the step in which the fiber is exposed to temperature ranging from 800°C to 3000°C. This heating can produce up to 95% carbon content [6]. Heating around 1000°C can produce carbon fibers of high tensile strength but higher temperature is necessary to manufacture high modulus carbon fibers [7].

Graphitization is the step of heating the fiber in the temperature range 1600~3000°C. It means graphitization is a carbonization process at high temperature. When heating is done around 1600°C to 3000°C about 99% polymer is converted to carbon [8,9,10]. Carbon fibers manufactured by this process are high modulus carbon fiber and classified as type-1 carbon fiber.

In the recent years most PAN based carbon fibers are being used in composite manufacturing [11].

Besides other benefits presence of carbon or graphite imparts better electrical conductivity. Moreover better resistance to corrosion and lighter weight materials with good strength find applications to replace metals. Conductivity is also a desirable property in manufacturing of electromagnetic and radio frequency interference

(EMI/RFI) shielding for electronic devices, moderate conductivity is necessary for electrostatic dissipation (ESD) [2]. Better physical and mechanical characteristics of PAN based composites have given them a good space in automotive and aerospace applications [12]. Finding solutions to challenges in aerospace applications can be done by optimization of the acrylic (PAN) fiber which can prove it the ideal fiber for aerospace applications [13,14]. In this study the acrylic fibers from waste are dissolved in solvent to make acrylic fiber solution (acrylic solution) and then this solution is applied on glass fiber mats (in the form of nonwoven sheet). Subsequent thermal and laser treatment of the prepared samples is studied to impart electrical conductivity in the material.

2. EXPERIMENTAL

2.1. Materials

Acrylic fibers waste from Gurund Industries, Czech Republic.

DMF (by RCI Labscan Limited)

Glass fiber mats of 270 g/m^2 (provided by SEPAT Specialni Papirenske Technologie, Czech Republic)

2.2. Methods

Main purpose of the study was effective utilization of acrylic fiber waste. Since acrylic fibers can be dissolved in dimethyl-formamide (DMF), acrylic fiber solution was prepared using DMF. The samples were produced by coating glass fiber non-woven mats in acrylic solution keeping 100% pickup. In this way the glass fibers in the nonwoven sheet were covered and coated with the acrylic solution. SEM image of PAN coated glass fiber is shown in **Figure 1**.

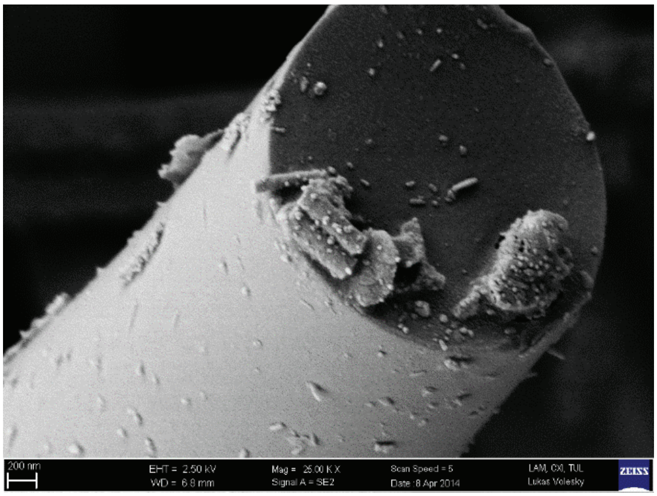

Figure 1 SEM Image of PAN coated Glass fiber

3. RESULTS AND DISCUSSION

The acrylic coated samples were treated in oven at different temperatures to study the effect of heat treatment on their properties. Heat treatment was carried out between 200°C to 450ºC. Weight of each sample before and after heat treatment was determined using electronic balance. Based on these observations percent weight loss of each sample was determined using equation 1.

$$\text{Weight loss (\%)} = [\text{ (initial weight} - \text{final weight) / initial weight}] * 100 \quad (1)$$

Table 1 shows the results of weight loss (%) of acrylic coated glass mats.

It is clear from the results summarized in **Table 1**, that increasing temperature increases weight loss. At 450ºC almost half of the samples weight is reduced which means that major amount of the applied acrylic polymer is evaporated of the sample. This implies that the conventional thermal pyrolysis process is not suitable to convert the coated acrylic polymer to carbon or graphite structure.

Table 1 Weight loss (%) of acrylic coated nonwoven after heat treatment

Sample	Treatment Temperature	Initial Weight	Final weight	Difference	weight loss
Sr. no.	°C	(gm)	(gm)	(gm)	%
1	200	1.00	0.89	-0.11	-11
2	250	1.00	0.87	-0.13	-13
3	300	1.00	0.81	-0.19	-19
4	350	1.00	0.64	-0.36	-36
5	400	1.00	0.59	-0.41	-41
6	450	1.00	0.51	-0.49	-49

Since the ideal oxidative stabilization temperature of acrylic is 270~300 ºC [4,5]. The samples heat treated at 300ºC is selected for laser irradiation to study the possibility of carbonization using laser treatment.

Infrared laser irradiation was performed with a commercial pulsed infrared laser (Marcatex 150 Flexi, Easy-Laser). The gas laser operates by carbon dioxide and produces a wavelength of 10.6 μm. The laser device contains the following parts (**Figure 2**): laser resonator (1), optical tube (2), optics box (3), the marking head (4), personal computer unit (5), central personal computer module (6), direct current source (7) and radio frequency source (8).

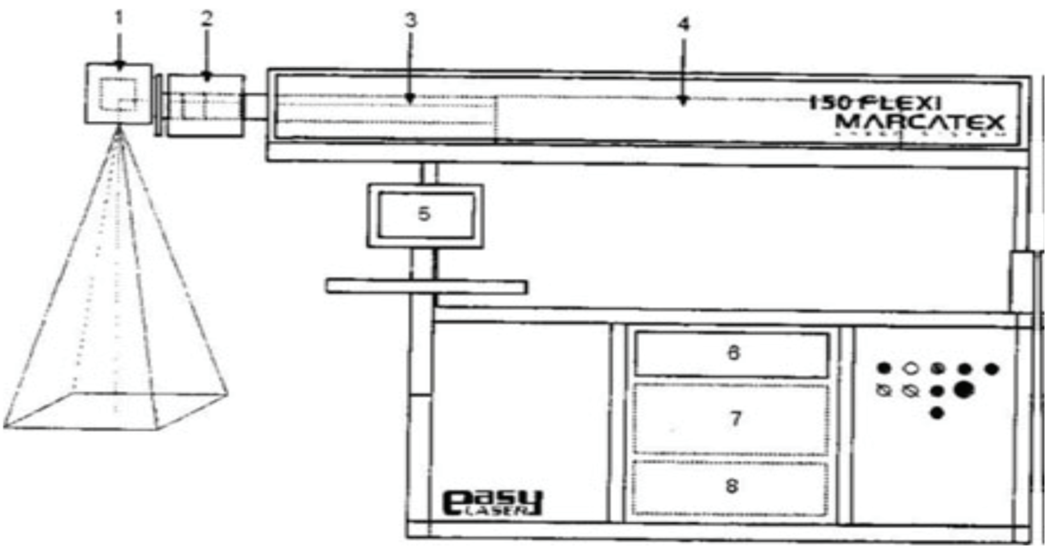

Figure 2 Diagram of laser system Marcatex 150 Flexi

Parameters that determine marking power of laser are: marking speed [bits/ms], duty cycle [%] and frequency [kHz]. Lower value of marking speed presents longer marking time. During the process of laser treatment, the marking speed of laser beam was set in 2, 4, 6, 8, 10, 12, 14, 16, 18, 20 and 22 bits/ms. In this study, the duty cycle (DC) was set at 50 %. The used laser power was 100 W at 50 % of DC and 5 kHz.

Two set of experiments were performed in order to check the suitability of laser treatment for checking possibility of carbonization and imparting electrical conductivity in the material. Firstly samples after drying and curing were directly irradiated by laser beam at different marking speed. Secondly samples after curing were preheated at 300oC for one hour in an oven for stabilization [4], and were then exposed to laser

The **Figure 3** shows that samples preheated at 300oC before laser treatment shows very less electrical resistance (good electrical conductivity) after laser treatment as compared to that of non-preheated samples.

High energy laser beam is carbonizing the acrylic to impart electrical conductivity. This means electrical conductivity is possibly imparted in the irradiated samples due to the presence of carbon or graphite structure [2] which is responsible for conductivity. Preheating the samples at 300oC stabilizes the acrylic [4] on the surface of the glass mats hence after laser irradiation more carbon or graphite is responsible for better conductivity.

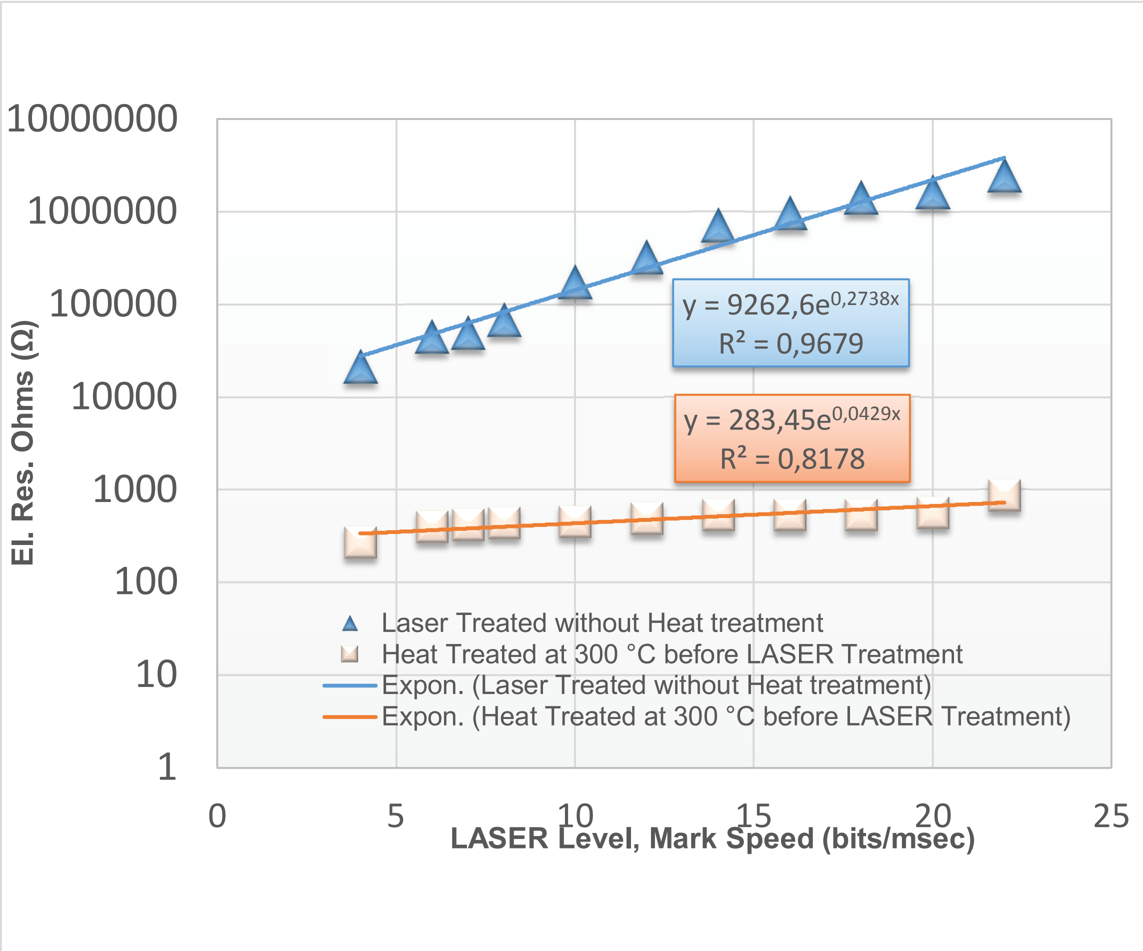

Figure 3 Electrical resistance of acrylic coated glass nonwovens at varying laser parameters

More powerful laser irradiation at lower mark speed has shown less electrical resistance and good conductivity. However at very lower mark speeds more intensity of laser irradiation can altogether destroy the sample.

CONCLUSION

It is concluded that laser irradiation of acrylic coated glass mats is capable of carbonizing the material surface and imparts electrical conductivity in the material. More is the powerful laser beam level, more is the electrical conductivity and vice versa. Hence using lower marking speed, but without destroying the sample, is necessary to get less electrical resistance and good conductivity.

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