Effects of Oxygen Plasma Treatment on Microstructure and Tensile Deformation of Nano SiO_2 Coated Vectran

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Abstract: The strain rate sensitivity of Oxygen plasma treated Nano Silicon Dioxide particles sol-gel coated Vectran and concurrent microstructural evolution was investigated. Both the materials are strain rate sensitive and the change in index parameter of strain rate sensitivity with strain reflects the change in micromechanisms of deformation and modes of fracture. Helium plasma can reduce the surface energy of vectran fiber. Oxygen plasma and nano SiO2 particles will enhance the surface energy.. The activation volumes rise with Oxygen. Although either Helium or Oxygen has made the activation volumes exceed that of the neat Vectran, the SiO2 nano-particle makes the activation volume rise rapidly.

Keywords: Strain rate, deformation, silicon dioxide, plasma jets, liquid crystalline polymers.

1. Introduction

Vectran is a thermoplastic liquid crystal polymer. In addition, these polymers are particularly suitable for high strength applications ranging from high performance fibers to reinforcement components of composite materials [1]. Extensional flow (e.g. during extrusion) results in significant orientation of the nuematic regions along the direction of flow [2], which yields higher mechanical strength and modulus [3].

The interest in developing novel organic–inorganic hybrid coatings in recent years is due to their unique properties derived from the inorganic and organic components. These coatings offer the opportunity to combine the desirable properties of organic polymers (toughness, elasticity) with those of inorganic solids (hardness, chemical resistance). Therefore, sol–gel methodology provides an easy, cost-effective and excellent way to incorporate inorganic compounds into an organic binder. Sol–gel processing can be used to produce a wide range of oxide and multi-component oxide coatings [4]. Sol–gels prepared from organo-functional silanes have been widely used to improve the adhesive bonding of different surfaces and to enhance the resistance to metal corrosion by means of surface coating [5]. From the previous study, many researchers had focused on using the SiO_2 coating [6].

As other nano-materials, agglomeration of SiO_2 nanoparticles has experienced the most obstacles in their wide applications [7] since the unsaturated dangling bonds and hydroxyl groups are extremely active and they saturate as soon as the SiO_2 is exposed to air. Therefore, the modifiers find it difficult to react with the active groups of SiO_2. Moreover, partial agglomeration usually occurs before the modification. With the APPJ treatment, SiO_2 nanoparticles are distributed homogeneously [8-11].

In order to determine what deformation mechanisms operate in nano-structured material, there has been a resurgent interest in equations of state involving activation energies and activation volumes. Activation volume is an important parameter if one is concerned with the time and temperature stability of recording inter-phase effect on strength in nano SiO_2 coated Vectran. By taking advantage of the simple kinetics in a nano-crystal system, we obtain insight into the mechanisms and microscopic processes of structural phase transitions.

The purpose of this paper is to report the effect of APPJ treatment on microstructure and tensile deformation of nano SiO_2 coated Vectran at room temperature. Tensile tests were performed to evaluate the mechanical performance. The SEM micrographs were used to investigate the surface morphologies, and FTIR tests to find out the chemical structure. In order to know the surface energy, contact angle tests were done.

2. Experimental methods

2.1 Material

Silicon dioxide (SiO_2,) was obtained from Hong Sheng Materials Technology Co., Ltd. Vectran is a manufactured fibre, spun from a liquid crystal polymer

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created by Celanese Acetate LLC and now manufactured by Kuraray Co., Ltd. Chemically it is an aromatic polyester.

2.2. Preparation of Vectran

The surface of Vectran filaments was deposited with Nano-SiO_2 particles by sol-gel dip-coating technique. Nano-SiO_2 sol-gel with a primary size of 14 nm in acetone was employed. After filtering the diluted solution, it was coated on the surface of Vectran filaments by dip coating. By changing the concentration of SiO_2 sol-gel and the speed of dip coating, the coverage of SiO_2 nanoparticles on Vectran filaments can be tuned. In this paper, the concentration of SiO_2 colloid was about 0.5 wt %, and the speed of dip-coating was 0.13 mm/s.

After coating, these filaments were treated by an APPJ (Atomflo-R, Surfx Company, USA) with 100% helium+O_2 gas. The APPJ is a small plasma jet with a length of around 15cm, consisting of two concentric electrodes with a 1.6mm gap through which the working gas helium+O_2 flows. The gas discharge was ignited by applying a low 13.56MHz radio frequency power, which enabled the jet to produce a stable discharge and avoid the arc transition.

The distance between the nozzle and the top of the filament was 5mm. The substrate circumrotated underneath the plasma jet at a speed of 10mm/s. Other plasma treatment parameters were set as follows: flow rate of helium gas was 20L/min, output power was 40W, treatment nozzle temperature was 60 C and sample treatment or stationary time was 3.3s.

2.3. Scanning electron microscope (SEM)

The surface morphology and microstructure of coatings composite was observed with scanning electron microscopy (SEM).

2.4. Fourier transforms infrared (FT-IR) spectroscopy

The surface chemical compositions of the Vectran filaments were studied using an FTIR spectrometer model Nexus-670 (Nicolet, USA).

2.5. Contact angle measurements

Contact angles of Vectran fibers were done with POWEREACH JC 2000A contact angle meter (Shanghai zhongchen digital technic apparatus co., ltd.). Care was taken to ensure that the contact angles were measured inside the Vectran fibers. The deionized water was loaded in a watering can, and while spraying the water to the testing fiber, real-time pictures were captured by digital camera. The contact angle was tested with JC200A software.

2.6 Mechanical measurements of a single fiber

The stress–strain curves for the fibers were measured on a tensile testing machine (YG001A) at strain rates of 0.02083 s^{-1}, 0.1 s^{-1}, 0.20833 s^{-1} and 0.625 s^{-1}. The reported values of all the mechanical properties were averaged over at least 50 independent measurements.

3. Results and discussions

3.1 Morphological studies

Figure 1 (a-d) show the SEM micrographs of the surface of the treated and untreated Vectran fiber in 20,000X. Figure 1 (b) show the Vectran with helium plasma treatment, with the electrons sprayed to the surface, and made the surface rougher than before. Figure 1 (c) is the picture of Vectran with helium and oxygen plasma treatment, the surface of the fiber is much smoother than the untreated Vectran. Comparing these pictures, Figure 1 (d) shows the photo of Vectran with helium and oxygen plasma treatment and nano SiO_2 coating, where the nano SiO_2 particles are spread evenly. It is clear that the reactions happened at the time of treatment.

3.2 Fourier transforms infrared (FT-IR) spectroscopy

In order to investigate the chemical structure of Vecrtan fiber with different treatments, the FTIR test was done, the spectra obtained is shown in Figure 2. The helium plasma treated Vectran is compared with helium and oxygen plasma treated Vecrtan, it can be seen that 3560-3903cm^{-1} and 2361, 2341cm^{-1} are vanished, which attributed to O-H and CO_2, respectively. In addition, the H_2CO, the stretching vibration situated at 2967 cm^{-1} is shown. This is due to the treatment of oxygen plasma, and the oxygen plasma must react with the fiber.