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Numerical and experimental approach to testing the adhesive properties of modified polymer blend based on EVA/PMMA as coatings for optical fibers

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ABSTRACT

The adhesive properties of polymer blends based on poly(ethylene-co-vinyl acetate) (EVA) and poly(methyl methacrylate) (PMMA) were studied. Two samples were prepared, whereby one kind was a physically mixed blend of commercial polymers in a solution of toluene and the other was a blend with EVA-*g*-PMMA polymer in a toluene solution, produced *via in situ* free radical polymerization using redox system initiators. A scanning electron microscopy (SEM) revealed the improved microstructure that was achieved blending with graft polymer that could also be seen by FTIR spectral analysis. Optical fibers were glued together using both of the solutions and subjected to a micro mechanical testing machine. The adhesion test showed that the graft copolymer had enhanced mechanical properties as an adhesive than the physical polymer blend. Optical microscopy of the samples after the adhesion test enabled the determination of the type of adhesive failure. Image analysis of the SEM micrographs was used to determine an area of the contact surface, and characterization microstructure. These results were then implemented in a numerical simulation that demonstrated the influence of microstructure on the adhesive properties showing the stress distribution for both samples. The main aim of obtaining an adhesive with uniform structure, great miscibility of the polymers, and good mechanical and adhesive properties was achieved and a numerical model was established that can be used in selecting the adhesive.

1. Introduction

When operating with optical fibers, it is necessary not only to ensure that the fibers maintain their position on the spool during handling and storage, but also to ensure that the unwinding of the fiber from the bobbin occurs without damage to the fibers and to maintain their functionality.

Adhesive groups that are commonly used in optical assemblies are: epoxy system, UV curable acrylics, elastomers, cyanoacylates, anaerobics, and structural adhesives. Each offer a unique combination of performance and processing benefits and the choice of the adhesive is of great importance for assembly functionality. Among the most commonly used adhesives are acrylates and epoxides. Some of commercially available adhesives are from companies: Dynax (acrylated urethane adhesives), MasterBond (epoxy system of adhesives,.), Norland Optical Adhesives (mercapto esters of acrylates,.).

Microbending is an important phenomenon which must be taken into account. Microbending is an increase of attenuation caused by bends < 1 mm from a straight axis of the optical fiber. There is some unavoidable bending during transport, storage, manufacturing, installation, etc [1]. When a load is applied to the optical fibers, the attenuation increased significantly for the smaller coating diameter in a range 250–900 μm [2]. Using an apparatus that will ensure constant diameter and maintaining symmetry of the optical fiber coated with adhesive can minimize microbending effect.

Signal transmission trough the optical fiber can also be disturbed by changing the geometry of protective polymer layer, or by chemical erosion. For this reason, the polymer coating has to be monitored after the adhesive deposition to avoid any changes.

Pure PMMA adhesive was not considered because it causes fiber breakage. PMMA is one of the most available polymers with good wettability, which is important for adhesives that impart adhesive properties [3]. The brittleness of PMMA could be reduced by the addition of some rubbery materials. Copolymers based on EVA have a

Abbreviations: EVA, poly(ethylene-co-vinyl acetate); PMMA, poly(methyl methacrylate); MMA, methyl methacrylate; MEKP, methyl ethyl ketone peroxide; FTIR, Fourier transform of infrared light; DSC, differential scanning calorimetry; FEM, finite element modeling; SEM, scanning electron microscopy

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long life span and the ability to withstand large temperature ranges [4–9]. EVA copolymers are widespread adhesives that are frequently used in the science of polymeric materials, usually in a form of hot melts for different types of substrates (metals, wood, paper, leather and plastics). Pure EVA adhesive, known as a rubbery material, is a good adhesive for optical fibers depending on the vinyl acetate (VA) content [10], which has inferior thermal stability due to the presence of acetate groups compared to PMMA [6,11].

Therefore, adhesive coatings for optical fibers based on polymer blends of poly (ethylene–co-vinyl acetate) (EVA) and poly (methyl methacrylate) (PMMA), which served to fix fibers to the spool and to balance the unwinding fiber from the bobbin, were applied in this study. Since both of components are already in use for optical fibers assembly, this blend can be of great interest for optical adhesives industry. By making polymer blends of these two polymers, the production price was lowered and higher thermal stability was attained [12]. Grafting PMMA on EVA is of great interest for developing materials with improved mechanical characteristics and impact resistance [13–15].

The effect of changing the structure by adding one polymer to another was observed and examined to determine the dependence between the microstructure and adhesive properties. It was necessary to determine the optimal bond ing strength between the fiber coating and the adhesives. In order to achieve good chemical compatibility, the chemical composition of the final polymer layer of the optical fiber was determined. The obtained mechanical and geometric properties were implemented in a numerical model to gain insight into the stress distribution in the adhesive layer that exhibits porosity and to study the dependence on the pore diameters.

2. Experimental

2.1. Materials

The optical fibers were produced by Draka Cableteq, USA. The EVA (Elvax 410, 18% VA, DuPont, USA), MMA (M55909, Sigma Aldrich) and PMMA (Acryrex® CM-205, M_w=90,400 g/mol Chi Mei Corporation, Taiwan) used in this work as an adhesive were commercial products. The toluene (Lachema a.s, Czech Republic, min. 99.0%) and acetone (Zorka Pharma Šabac, min 99.5%) were used as a solvents, and MEKP (Boyteroks A, Boytek Recine Boya ve Kimya San Tic, As, Catalists & Initiators), potassium persulfate and sodium metabisulfite (Sigma-Aldrich Chemie GmbH, Steinheim, puriss. p.a), were used as initiators.

2.1.1. The preparation of EVA/PMMA copolymer blends

The first adhesive was obtained by mechanical mixing 5.00 g of PMMA and 5.00 g of EVA in 46.2 cm^3 in toluene for 8 h at 60 °C.

The second one was produced in an *in situ* graft polymerization by dissolving 10.02 g EVA (25.40 wt%) in 30.7 cm^3 MMA (71.75 wt %), then 0.14 g potassium persulfate (0.37 wt%), 0.12 g sodium metabisulfite (0.29 wt%) and 0.92 cm^3 MEKP (2.68 wt%) were added to the mixture and was heated for 3 h at 70 °C in a sealed container under atmospheric conditions. The obtained graft copolymer was dissolved in toluene to obtain a 20 wt% solution for use as an adhesive.

2.2. Adhesive layer application on the surface of the fiber

The optical fibers were coated using 20 wt% solutions of the prepared adhesives in a specially designed apparatus at 60 °C, Fig. 1. The nozzle could be easily changed in order to control the thickness of the coating. The thickness could also be controlled by optimizing the speed of fiber drawing. The radial velocity of rotation of the spinner was 300 rpm and the axial speed of the spinner was 100 rpm [10,16].

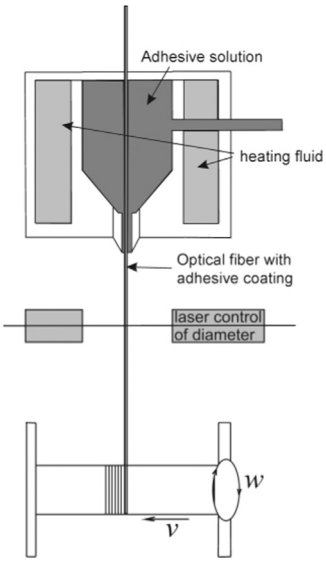

Fig. 1. Apparatus for coating optical fibers with an adhesive.

2.3. Methods

2.3.1. Image analysis for the determination of the geometrical parameters of fiber and adhesives

Using software Image-Pro Plus allows the diameter of a bare fiber without coating and one coated to be measured. In addition, image analysis of the adhesives enables statistical data of the diameters, spacing and pores of the obtained adhesives to be determined. After testing of the mechanical properties of the adhesive, image analysis of the optical micrographs showed the type of the loosening of the adhesive.

In order to determine the geometrical characteristics of the optical fiber and the coating, it was necessary to design a simple and reliable method for the determination of the diameter. An optical fiber consists of quartz glass that has a high melting point and this is the basic element of the system for signal transmission. A coating composed of a polymer is applied to the fiber by drawing. According to the nature of the layers, it is possible to predict that it would be possible to remove the coating by burning without damage to the fibers located in the center. By observing parts of the fiber with a coating and without, it is possible to determine the diameter of a telecommunications fiber and the thickness of the coating [17].

2.3.2. Optical microscopy

Optical microscopy was used to capture images that were analyzed by Image ProPlus to determine the geometric characteristics of optical fiber and to determine the type of adhesive failure. A Carl Zeiss – Jena, NU2 optical microscope was used in this study.

2.3.3. Determination of chemical composition by FTIR analysis

Infrared spectroscopy enables the determination of the composition of the final coating of optical fibers, which it is of great importance for establishing a compatible adhesive. When the adhesive has a similar chemical composition to that of the final coating of the optical fibers, it could be a suitable choice.

Structural analysis for existing optical fiber coating was performed by single-beam Fourier-Transfer Infrared Spectroscopy (FTIR) using a Nicolet 6700 spectrometer (Thermo Scientific) in the attenuated total reflectance (ATR) mode with a single bounce 45 °F Golden Gate ATR accessory with a diamond crystal, and an electronically cooled DTGS detector. The spectra were the co-addition of 64 scans at 4 cm^{-1} spectral resolution, and were ATR corrected. The Nicolet 6700 FT-IR spectrometer was equipped with OMNIC software and recorded the