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Modification in polyethylene–iron oxide joints induced by laser irradiation

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ABSTRACT

The laser welding is a thermal bonding technique with high localized heating that produces a strong adhesion between polymers. The plastic is able to absorb the laser energy with the use of embedded fillers in itself. In this work we studied blends made by polyethylene and different amount of fillers (iron oxide, red color). Mechanical and physical analyses were performed with the aim to study the effect of filler percentage on the welding features.

Experimental results showed that the filler amount influences the optical properties of the polyethylene since it regulates the amount of adsorbed energy and the depth of the material layers involved in the welding. A high concentration of filler improves the absorption laser energy, decreasing the layers involved in the welding and worsening the mechanical strength of the joint. Vice versa, a low filler amount involves deeper layers of the material so that the sealing action improves. The efficiency of the joint can be regulated by the filler concentration. Some applications of thermoplastic polymeric joints with different geometries are proposed and discussed.

1. Introduction

Thermoplastic polymers are replacing progressively metals in the automotive, aero spatial and biomaterial industries over the past few decades. A reduction in the cost of strategic manufacture materials is based on the following factors: weight saving, flexibility and thermal insulation properties, mechanical and chemical resistance. New design opportunities are also introduced by special polymers. The polymer joints, for example, permit to realize special devices and composed materials with peculiar properties useful for many applications [1].

For this reasons a lot of new techniques are developing during the last years. One of these is the "visible through transmission laser welding" (TTVLW) that involves the coupling of two polymeric sheets, one laser absorbent and the other laser transparent [2]. In particular the absorption occurs at the interface between the two sheets and the deposited energy causes melting and welding of the two parts. Physical tests highlighted that not only a thermal effect happens during the laser irradiation, but the plasma gas, generated by the high-power laser pulse, also accelerates ion species (protons and carbon ions). The ion implantation and sputtering effects, occurring at the interface between the two polymeric sheets, promote their adhesion [3]. The plastic is able to absorb the laser energy embedding in itself different fillers which have high absorption power at the used laser wavelength [4]. In a previous paper we highlighted that the TTVLW is an effective technique to obtain high resistant joints: we studied in particular ultra high molecular weight polyethylene polymer (UHMWPE) filled with different concentrations of carbon nanotubes (CNTs) [5]. Experimental measurements confirmed that the CNTs as filler are capable of absorbing efficiently the laser light. The laser source modifies the polymer in the contact area. It generates an energetic process that develops heat, ions, radical species and new chemical rearrangements. These processes promote the adhesion of the polymeric chains. The conditions to have the best adhesion between the two polymeric sheets are obtained at low CNT concentrations (around 0.1 wt%). In these conditions, the mechanical strength of an UH/UH–CNT joint is about 115 N, a value comparable with the pristine UHMWPE material on which the investigated welding is based [6].

In this work we have prepared polymeric joints based on pristine UHMWPE and UHMWPE filled with different percentages of iron oxide. With this latter the polymer is able to absorb the Nd:Yag laser. We have studied how the laser source modifies the layers of materials involved in the welding at the interface by measuring physical and mechanical features. In particular, we have valuated

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the possibility to regulate the thickness involved in a polymeric joint by controlling the filler amount. This aspect could be very interesting in view of the several possible applications of the laser welding in different geometries, like those proposed and discussed in this paper.

2. Materials and methods

The materials used were: ultra high molecular weight polyethylene (UHMWPE resin, Ticona-GUR 1020 $d = 930\ kg/m^3$, appearance: semitransparent, $M_w \approx 3 \times 10^6$ g/mol) and iron oxide, employed as filler, supplied by Across Organics with a purity level of 99.999%, particle size of ten nano meter order. Nanocomposites were made by the UHMWPE mixed with different weight percentages of Fe_2O_3, within the range 0.1–10%, with final density values within the range of 935–1018 kg/m^3 and appearance red. They were codified as "UH–Fe_2O_3" with a number that specifies the filler percentage. The pure UHMWPE and the UH–Fe_2O_3 nanocomposites were moulded in a hot press at 200 °C for 20 min at pressure of 20 MPa, obtaining sheets 60 mm × 60 mm and 1 mm of thickness.

The polymeric sheets were irradiated in air at room temperature (25 °C) for 2 min by a 3 ns Nd:Yag laser operating at 532 nm (second harmonic) in single pulse or at 10 Hz repetition rate, with an intensity of $1.8 \times 10^8\ W\,cm^{-2}$, a maximum pulse energy of 150 mJ and a laser spot of ~28 mm^2 (no focusing lens were employed). The laser pulse energy had a Gaussian shape with a diametric FWHM of about 3 mm. The incident angle of the laser beam was 0°. The welded joints were obtained by coupling two rectangular polymer sheets, partially overlapped for a length of 15 mm. In particular, the UHMWPE sheet, highly visible transparent, was placed on the top (irradiated face) while the highly visible absorption sheet (made by UH–Fe_2O_3) was placed on the bottom (un-irradiated face) and pressed between each other with pressure of about 200 kPa.

The absorption coefficient (μ) of the laser light transmission was measured through thin films (90 μm thickness) with 532 nm wavelength and 20 mJ pulse energy. It was calculated by the following formula:

$$\mu = -\frac{1}{x}\ \ln\frac{E}{E_0} \quad (1)$$

where x = thickness, E_0 = laser energy of 20 mJ and E = energy transmitted through the material.

Changes in crystallinity amount and in melting temperature were assessed by heating samples in a differential scanning calorimeter (Q100 TA Instruments) from 30 °C to +180 °C, with a heating rate of 10 °C/min. Samples crystallinity was determined integrating the enthalpy peak and normalizing it with the enthalpy of melting of 100% crystalline polyethylene, corresponding to 291 J/g.

The mass quadrupole spectrometry (Balzer MQS 300) operating between 1 and 300 amu range with sensitivity higher than 1 ppm was employed. The MQS measurements were obtained by irradiating the different polymers in high vacuum conditions (10^{-6} mbar).

The shear test was carried out on the joints at 25 °C by a LLOYD LR 10 K universal testing machine with a crosshead speed of 10 mm/min. The specimens had a rectangular geometry, 20 mm × 30 mm and 1 mm of thickness (Fig. 1a). For each irradiation dose 10 specimens were tested in order to give the average value.

The morphological observations were performed by a scanning electron microscopy (SEM) with a JEOL JSM 5600 LU microscope. The investigations concern mainly the welded polymer zones after the detachment caused by the shear test. For the SEM investigations the polymer samples were coated in vacuum with a thin gold film to make them electrically conductive. Generally the electron

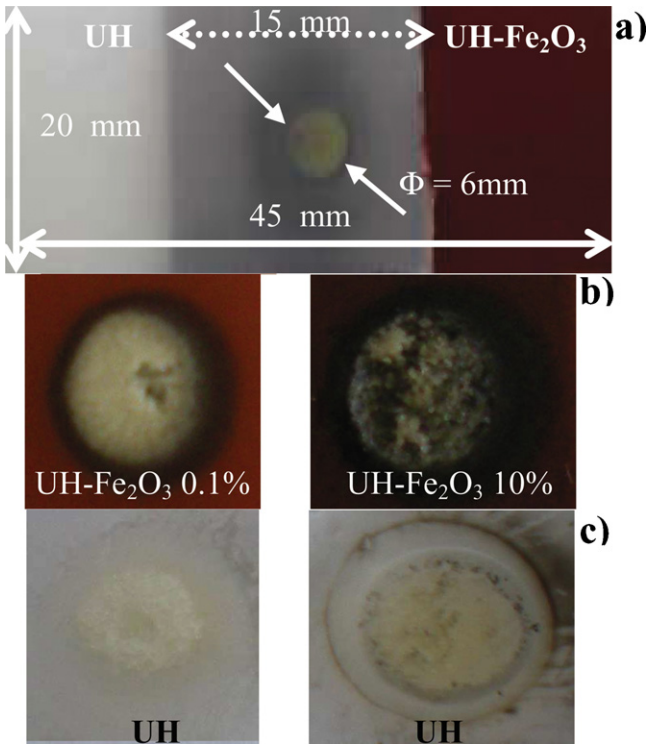

Fig. 1. Geometry details of a laser joint before (a) and after the detachment (b) in the two component sheets (UH and UH–Fe_2O_3, with 0.1% and 10% of filler). (For interpretation of the references to color in the text, the reader is referred to the web version of the article.)

acceleration voltage was 5 kV and the photo magnification was performed at low (25×) and high magnifications (100× and 400×).

The sample roughness was determined by a surface profiler (Tencor-P10) that moves a micrometric tip along a single direction; the scansion is assisted "on line" by an optical microscope. Generally, the measurements were performed by using a force of 1 mg, a scansion length of 1 mm, a scan speed of 100 μm/s and a depth resolution of 1 nm. Measurements were performed in different zones of the same surface and the average values were calculated.

3. Results and discussion

The absorption coefficient measurements of laser light were performed both on pure UH and on filled UH–Fe_2O_3. The value of pure UHMWPE was about 25 cm^{-1} while the measurements of the UH–Fe_2O_3 films, at different weight percentage of filler (0.1–1.0%), showed a linear increase of the absorption coefficient with the increment of the filler concentration up to a value of about 200 cm^{-1}. At higher filler percentages (for example 5 and 10 wt%), the adsorption coefficient reaches values higher than 500 cm^{-1}. These results confirmed that the UHMWPE polymer is transparent to the laser light while the filled polymer is capable to absorb the laser light so that the material becomes progressively less transparent with increasing the filler content.

In Fig. 1b and c are shown the photographs of the two joints (UH/UH–$Fe_2O_3$0.1 and UH/UH–$Fe_2O_3$10) detached by mechanical test after their welding. Observing the laser spot in the red sheets, we can see a whitening effect (Fig. 1b) while a yellow area surrounded by circular rings appears in the corresponding UH sheets (Fig. 1c). This means that the laser energy push out the nanoparticles of iron oxide from the matrix. The adsorbed laser energy produces a spot geometry that increases with high filler percentage. In particular, at the highest amount of filler (10 wt%) the adsorbed energy is spread around the polymeric surface. The photos suggest

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