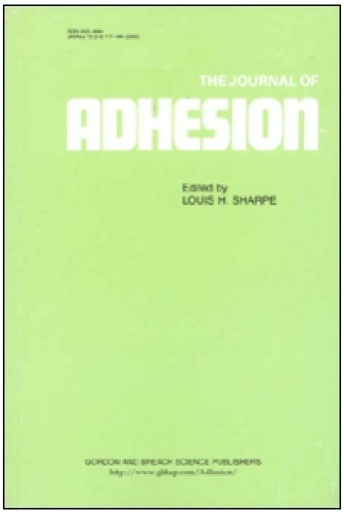

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Physicochemical and Adhesion Characteristics of High-Density Polyethylene when Treated in a Low-Pressure Plasma under Different Electrodes

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Physicochemical and Adhesion Characteristics of High-Density Polyethylene when Treated in a Low-Pressure Plasma under Different Electrodes

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The present investigation studys the effects of different electrodes such as copper, nickel, and stainless steel under low-pressure plasma on physicochemical and adhesion characteristics of high-density polyethylene (HDPE). To estimate the extent of surface modification, the surface energies of the polymer surfaces exposed to low-pressure plasmas have been determined by measuring contact angles using two standard test liquids of known surface energies. It is observed that the surface energy and its polar component increase with increasing exposure time, attain a maximum, and then decrease. The increase in surface energy and its polar component is relatively more important when the polymer is exposed under a stainless-steel electrode followed by a nickel and then a copper electrode. The dispersion component of surface energy remains almost unaffected. The surfaces have also been studied by optical microscopy and electron spectroscopy for chemical analysis (ESCA). It is observed that when the HDPE is exposed under these electrodes, single crystals of shish kebab structure form, and the extent of formation of crystals is higher under a stainless-steel electrode followed by nickel and then copper electrodes. Exposure of the polymer under low-pressure plasma has essentially incorporated oxygen functionalities on the polymer surface as detected by ESCA. Furthermore the ESCA studies strongly emphasize that higher incorporation of oxygen functionalities are obtained when the polymer is exposed to low-pressure plasma under a stainless-steel electrode followed by nickel and then copper electrodes. These oxygen functionalities have been transformed into various polar functional groups, which have been attributed to increases in the polar component of

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surface energy as well as the total surface energy of the polymer. Therefore, the maximum increase in surface energy results in stronger adhesion of the polymer when the polymer is exposed under a stainless-steel electrode rather than nickel and copper electrodes.

Keywords: Adhesive joint; ESCA; High-density polyethylene; Lap-shear strength; Plasma treatment; Surface energy; Surface modification; XPS

1. INTRODUCTION

Polymeric materials such as polypropylene (PP), polyethylene (PE), polycarbonate (PC), and so forth are progressively replacing the traditional engineering materials such as steel and aluminium in the fabrication of secondary structures of aircraft, automobiles, and railway coaches as well as in many civil engineering applications because of their superior properties such as better corrosion resistance, high strength-to-weight ratio, relatively low cost, and easy recycling [1, 2]. Unfortunately, the surfaces of these polymers are hydrophobic in nature and exhibit low surface energy and, therefore, represent challenges for adhesive bonding. Hence, surface modifications of polymers are often carried out to enhance their surface energy to overcome technological challenges.

Several surface-modification methods are employed to modify the polymer surfaces, such as chemical, thermal, mechanical, and electrical treatments under atmospheric pressure plasma (corona discharge) and low-pressure plasma (glow discharge). Glow discharge under low-pressure plasma is a popular technique that results in better uniformity in the surface modification of the polymers [3–6]. Moreover, it is a dry treatment method, better suited for industrial applications. It is now well established that the glow-discharge treatment creates physical and chemical changes such as cross linking, degradation, formation of free radicals, and oxygen functionalization [7–11]. The gas temperature in a glow discharge generally remains low, and the plasma plays a predominant role in the surface modification of polymers.

In view of these observations, the present study investigates the surface modification of high-density polyethylene (HDPE) under DC glow discharge across copper, nickel, and stainless-steel electrodes at a power level of 13 W [4–6] for different exposure times. The polymer surface has been characterized by estimating the surface energy of the polymer followed by studies under an optical microscope using quantitative microscopy and electron spectroscopy for chemical analysis

TABLE 1 Polar, Dispersion, and Total Surface Tension of the Test Liquids

Liquid	γ_{LV}^{P} (mN/m)	γ_{LV}^{D} (mN/m)	γ_{LV} (mN/m)
Deionized water	50.2	22.0	72.2
Formamide	18.6	39.6	58.2

(ESCA). Finally, at a given parameter of DC glow discharge, the results of surface modification of HDPE using these different electrodes have been compared in terms of wetting, physicochemical, and adhesion characteristics.

2. EXPERIMENTAL

2.1. Materials

In this investigation, commercial HDPE sheets are used-, manufactured by Caprihens Indian Ltd, and the HDPE sheet was filled with silicate, mild steel sheets, and an epoxy adhesive (Resin Araldite AY 105 and Hardener HY 140) manufactured by Hindustan Ciba-Geigy. The mixing ratio of resin to hardener, curing temperature, and time of this adhesive are 1:1, 25°C at 24 h respectively. The HDPE sheets of thicknesses 0.05 mm and 8.0 mm were used, respectively, for ESCA and contact-angle measurement. Two test liquids, such as deionised water and formamide of known polar and dispersion components of surface tension, were used to evaluate the polar and dispersion components of surface energies of HDPE. This was done by measurement of their contact angles by the sessile drop method [10–12]. The temperature during the measurements was 25°C. The known components of surface tension of liquids are given in Table 1.

2.2. DC Glow Discharge Setup

A diagram of the DC glow discharge setup used for surface modification of HDPE sheets is shown in Figure 1. The set up consists of a 175-mm-high and 150-mm-diameter closed glass chamber. Through an inlet, air or other desired gases are introduced into the glass chamber. A pair of 10-mm-thick copper plates of 80 mm in diameter are used as anode and cathode inside the glass chamber. In the setup, the anode is fixed and the cathode is adjustable for the desired spacing between the two electrodes. The HDPE sheet, cleaned by wiping with acetone, is kept on the cathode. Inside the glass chamber, a vacuum is created with the help of a rotary oil pump having a pumping capacity of

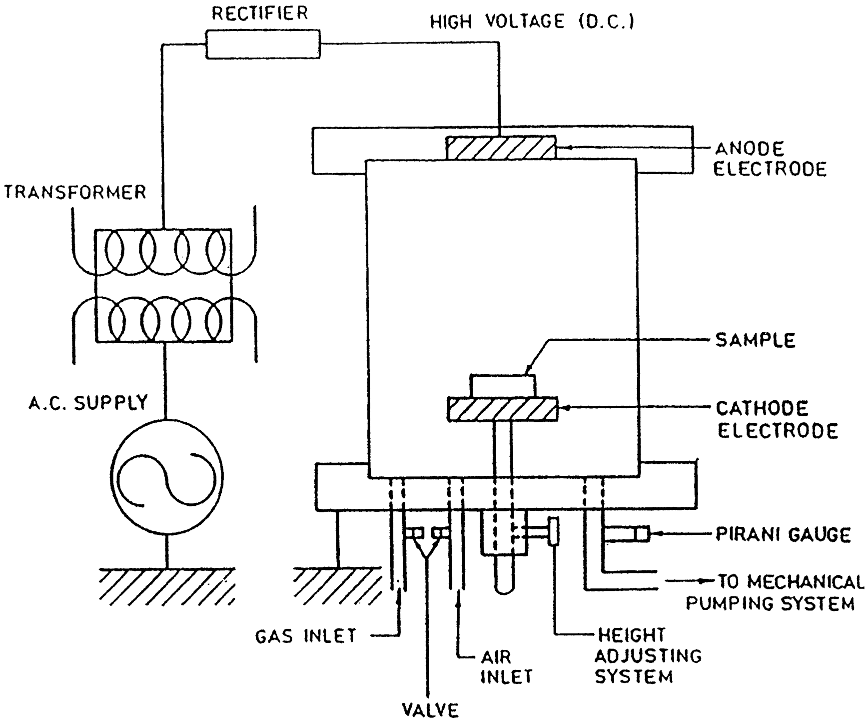


FIGURE 1 Schematic diagram of the DC glow discharge system.

$12\,m^3/h$. A pressure of 0.5 Torr (65.8 Pa) measured by a Pirani gauge is maintained inside the glass chamber. At this low air pressure, DC voltage exceeding 500 V is applied between the two electrodes to ignite the glow discharge.

In this investigation, the surface of the polymer was modified by DC glow discharge through air across copper, nickel, and stainless-steel electrodes with a fixed spacing of 110 mm for different times of exposure and at a 13-W power level.

2.3. Estimation of the Surface Energy

Contact angles of deionised water and formamide (grade AR, manufactured by Fischer Chemic Ltd, Chennai, India) on the unexposed HDPE sheets and those surfaces modified by exposure under DC glow discharge were measured by the sessile drop technique. The geometry of the sessile drop was studied at a magnification of ×12.8 under an optical stereo zoom microscope with a crosshead glass graticule fitted with an eyepiece. Vertical and horizontal reference lines of the graticule were positioned at the corner of the drop and rotated to make the vertical line tangent to the drop. The extent of the rotation angle was measured using a goniometer of $\pm 1^\circ$ accuracy. The surface energy was estimated by using the following equation [12],

$$(1 + \cos\theta)\gamma_{LV} = 2(\gamma_S^D \gamma_{LV}^D)^{1/2} + 2(\gamma_S^P \gamma_{LV}^P)^{1/2}, \tag{1}$$

on the unmodified HDPE sheet and those HDPE sheets exposed to the DC glow discharge. First, the contact angle of deionised water, θ, was measured on the HDPE surface. In this equation, θ is the measured contact angle of deionised water where the surface tension of deionised water, γ_{LV}, and its two components, the polar, γ_{LV}^P, and the dispersion, γ_{LV}^D, are known and the two unknowns are γ_S^P and γ_S^D for the solid HDPE surface. Second, the contact angle of formamide, θ, was measured on the HDPE surface and, consequently, θ is the measured contact angle of formamide where surface tension of formamide, γ_{LV}, and its two components, the polar, γ_{LV}^P, and the dispersion, γ_{LV}^D, are known and the two unknowns are γ_S^P and γ_S^D. By solving these two equations, the unknowns γ_S^P and γ_S^D of the HDPE were calculated. Finally, the total surface energy, γ_S, was estimated by adding γ_S^P and γ_S^D as given in Equation (2):

$$\gamma_S = \gamma_S^D + \gamma_S^P \tag{2}$$

2.4. Optical Microscopy Studies

Microstructural features of the polymer surfaces, both unmodified and modified under DC glow discharge across copper, nickel, and stainless-steel electrodes at 13 W power for 120 s, have been studied under a Leitz Wetzler MM6 optical microscope manufactured by Leco Corporation, Michigan, USA. To ensure a scratch-free polymer surface, before exposure under DC glow discharge, polymeric samples were polished with up to 1000 grade emery paper (i.e., grit size up to 1 micron), and then a final polishing was carried out using a polishing cloth. The samples were then cleaned with acetone prior to exposure under the DC glow discharge. The microstructural changes taking place in the polymer surfaces during exposure under the DC glow discharge were examined and photographed.

Quantitative measurements have been carried out to determine the area fraction of crystallization. A point-counting technique measured the area fraction. A grid containing 100 points was inserted into the eyepiece of the optical microscope. The magnification of the microscope was adjusted in such a way that maximum resolution was attained. The grid was located randomly, and the number of grid points falling on the crystalline area was counted. Twenty such observations were made for each sample. The area fraction, A_m, was then calculated from the relationship,

$$A_m = \frac{\sum_{i=1}^{20} N_i}{20 \times 100} \tag{3}$$

where N_i is the number of grid points falling in each area fraction of crystallisation in the ith observation.

2.5. Electron Spectroscopy for Chemical Analysis (ESCA)

ESCA of the polymer surface was carried out on the unmodified polymer as well as on the exposed polymer surfaces. The measurements were made with an SSLSSX-100 X-ray photoelectron spectrometer at the Institute for Plasma Research, India, by using monochromatised AlK$_\alpha$ X-ray source operated at a power of 200 W. The binding energy reference for the carbons of the C–C and C–H peak was set at 285.0 eV. The base pressure in the sample chamber during analysis was 2×10^{-10} Pa. Survey spectra were obtained using a pass energy of 58.7 eV and, during the analysis, the electron take-off angle was set at 45° and the acquisition time was 6 min.

2.6. Adhesive Joint Preparation

Prior to the preparation of an adhesive joint, degassing of the adhesive was carried out under a pressure of 1 Pa for 10 min. An 8-mm-thick polymer sheet was used for the preparation of the polymer-to-steel adhesive joint to ensure a very small possibility of failure of the lap joint in either the polymer or the mild steel. The failure might take place either within the adhesive or at its interfaces with the polymer and the mild steel. Thus, this provided an opportunity to study the characteristics of the adhesive joint. The lap-shear tensile specimens were prepared using the strips of mild steel and polymer sheets of dimensions $110 \times 25 \times 1\,\text{mm}^3$ and $110 \times 25 \times 8\,\text{mm}^3$, respectively, by applying epoxy adhesive at an overlap length of 12.5 mm, according to the DIN 23281 standard (schematically shown in Figure 2). Any excess adhesive present at the interface was expelled by rolling the joint at a load of 2 kg, which resulted in a joint having adhesive about

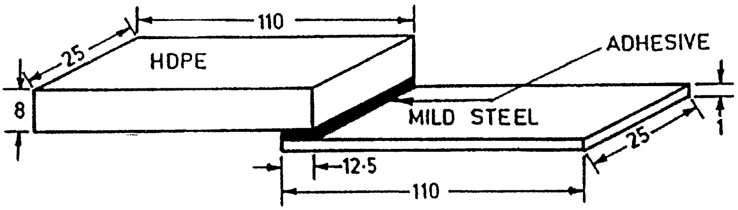


FIGURE 2 Schematic diagram of the adhesive joint for lap-shear tensile test (dimensions in mm).

0.2 mm thick. The shear tensile test was performed according to the DIN 53283 standard, using a universal testing machine, manufactured by Mohr and Federhaff AG, Germany, at a test speed of 5 mm/min. At each parameter ten joints were prepared and the mean value is reported in the result.

3. RESULTS

3.1. Surface Modification Under Different Electrodes of DC Glow Discharge and Wettability

The influence of different electrodes on the polar component of the surface energy of HDPE obtained after surface modification by DC glow discharge at a power level of 13 W is shown in Figure 3. It is observed that, for all three electrodes, the polar component of the surface energy of the HDPE increases significantly with increasing time of exposure, reaches a maximum and then decreases with further increase in time of exposure. The use of stainless-steel electrodes results in a relatively larger increase in the polar component of surface energy of the polymer, followed by those obtained using the nickel and the copper electrodes as evident from Figure 3.

The variations of the dispersion components of surface energy of HDPE with exposure time under different electrodes are relatively much less than for the polar component of surface energy under similar conditions, as shown in Figure 4. The dispersion component of surface energy of HDPE increases with increasing time of exposure, reaches a maximum, and then decreases with further increase in time of exposure. It is interesting to note that the maximum dispersion component of surface energy was observed for the copper electrode, followed by those obtained using nickel and stainless-steel electrodes.

The variation of the total surface energy of the HDPE with time of exposure to the DC glow discharge is shown for the different electrodes

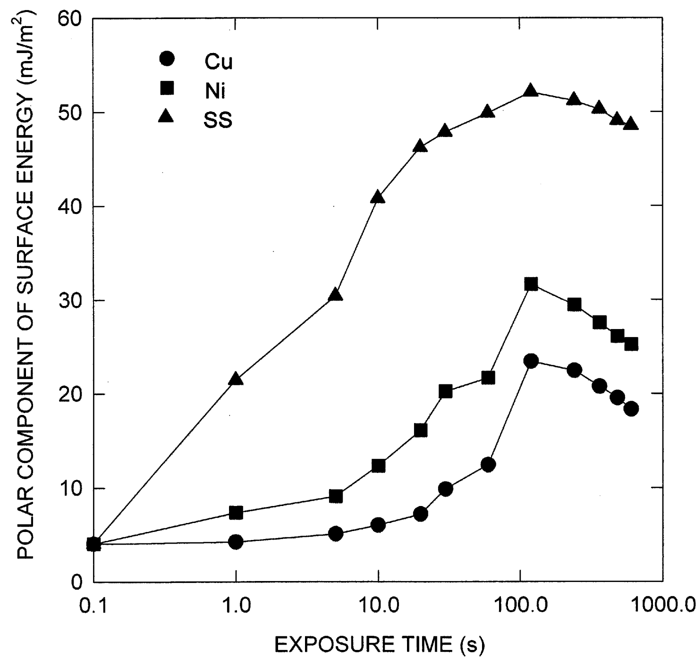


FIGURE 3 Variation in the polar component of surface energy of HDPE surface exposed to DC glow discharge through air using copper, nickel, and stainless-steel electrodes at a power of 13 W.

in Figure 5. Under these electrodes, the total surface energy of the HDPE increases significantly with increasing time of exposure, reaches a maximum, and then decreases with the increase in time of exposure. It was observed that the use of stainless-steel electrodes for the glow discharge results in a relatively larger increase in the total surface energy of the polymer followed by those obtained using nickel and the copper electrodes, as shown in Figure 5. The polar surface-energy component dominates and overcomes the different trend observed in the case of the dispersion component of surface energy.

3.2. Microscopy of Modified Polymer Surfaces

Polished surfaces of HDPE sheets, both unexposed and exposed to DC glow discharge across copper, nickel, and stainless-steel electrodes, were examined under optical microscopy. Figure 6a shows an optical

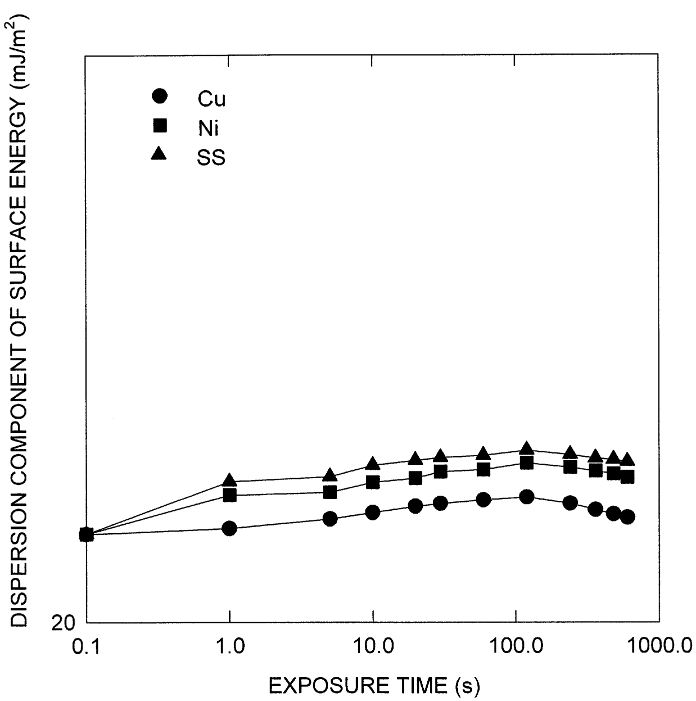


FIGURE 4 Variation in the dispersion component of surface energy of HDPE surface exposed to DC glow discharge through air using copper, nickel, and stainless-steel electrodes at a power of 13 W.

micrograph of an unexposed HDPE surface. It is observed that only a few silicate particles are present on the unexposed HDPE surface. An optical micrograph of an HDPE surface exposed to the DC glow discharge at 13 W for 120 s across a copper electrode is shown in Figure 6b. This micrograph of the HDPE surface exposed to the DC glow discharge is significantly different from that of the unexposed HDPE surface. When an HDPE surface is exposed to a DC glow discharge, single crystals with extended chain-like structures appear in the shish kebab form as shown in Figure 6b.

Figures 6c and 6d show optical micrographs of HDPE surfaces exposed to the DC glow discharge across nickel and stainless-steel electrodes at 13 W for 120 s. One can see that, when the HDPE is exposed under these electrodes, shish kebab structures of crystalline regions form to a greater extent than for those observed under exposure to DC glow discharge across the copper electrode (although the power and exposure time to DC glow discharge are the same).

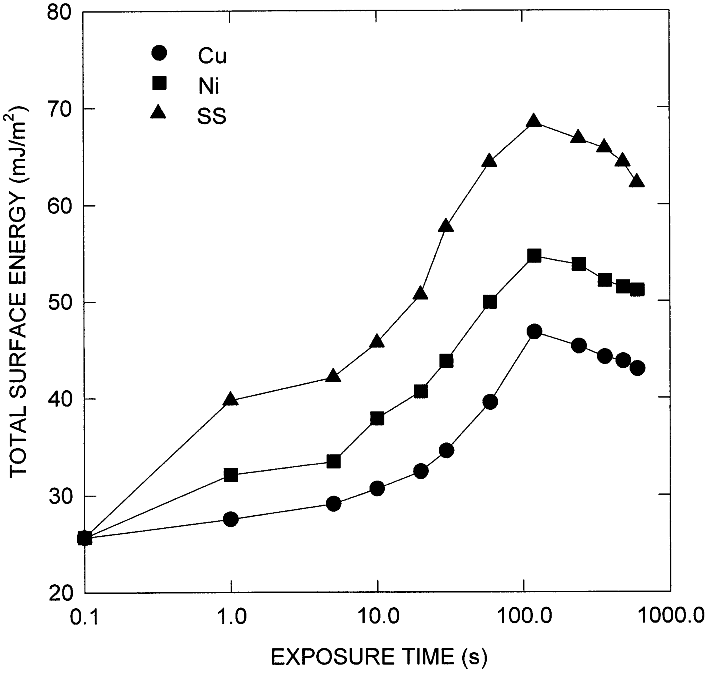
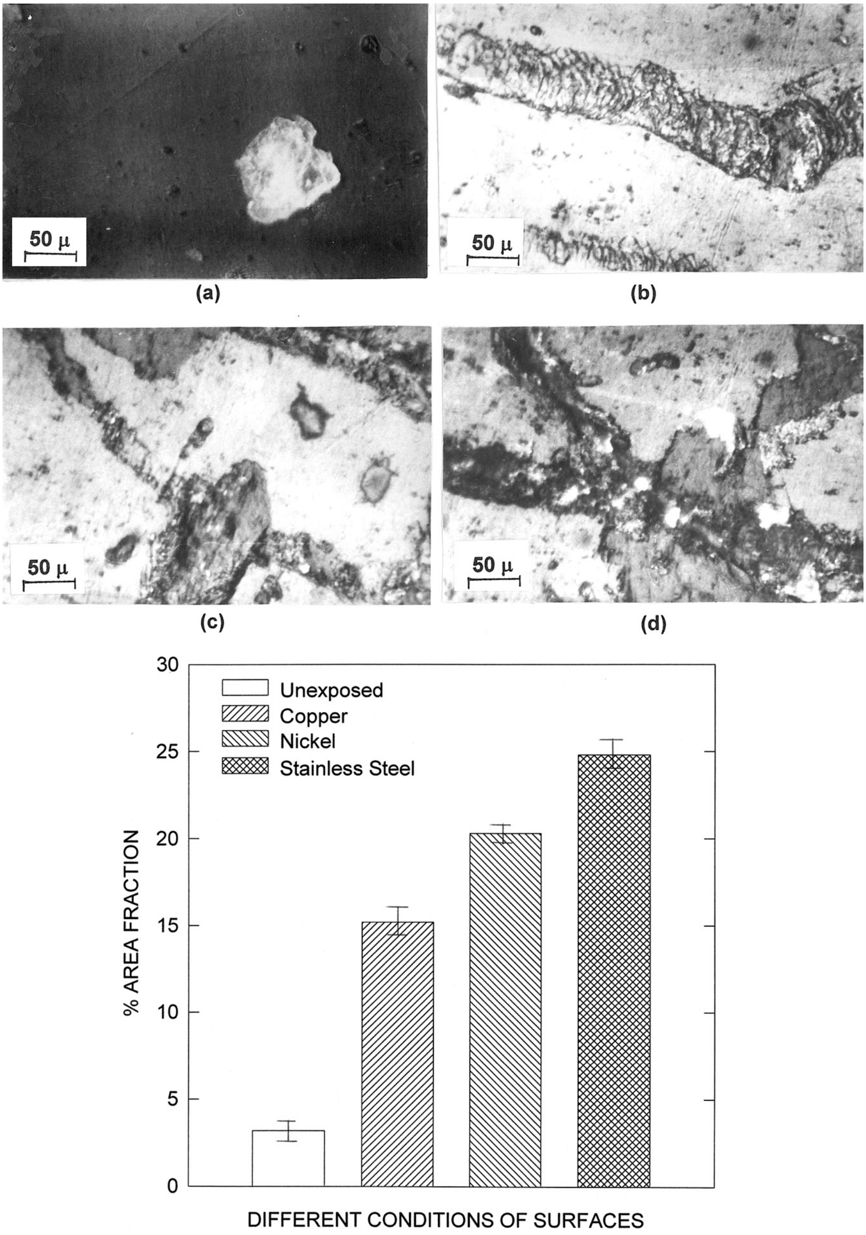


FIGURE 5 Variation in total surface energy of HDPE surface exposed to DC glow discharge through air using copper, nickel, and stainless-steel electrodes at a power of 13 W.

The percentage increase in area fraction of the crystalline region for different electrodes is shown in Figure 6e.

3.3. ESCA Studies on Modified Polymer Surface

ESCA studies of HDPE were carried out for the unexposed surface and for those exposed to DC glow discharge for 120 s and at a 13-W power level under copper, nickel, and stainless-steel electrodes. A widescan spectrum of the unexposed HDPE shows the C 1s peak and a negligible peak of oxygen 1s (Figure 7). However, the HDPE surfaces exposed to DC glow discharge show significant incorporation of oxygen as evident from the corresponding heights of oxygen peaks shown in Figures 8, 9, and 10 across copper, nickel, and stainless-steel electrodes, respectively, resulting in an increasing oxygen/carbon atomic ratio (O/C) on the HDPE surface. Furthermore, it is observed that inorganic species, particularly copper, silicon, and magnesium, are



(a) (b) (c) (d) (e)

FIGURE 6 Optical micrograph of HDPE surface (a) unexposed, (b) exposed to DC glow discharge at 13 W for 120 s under copper electrode, (c) exposed to DC glow discharge at 13 W for 120 s under nickel electrode, (d) exposed to DC glow discharge at 13 W for 120 s under stainless-steel electrode, and (e) crystallization of HDPE under exposure to DC glow discharge across different electrodes.

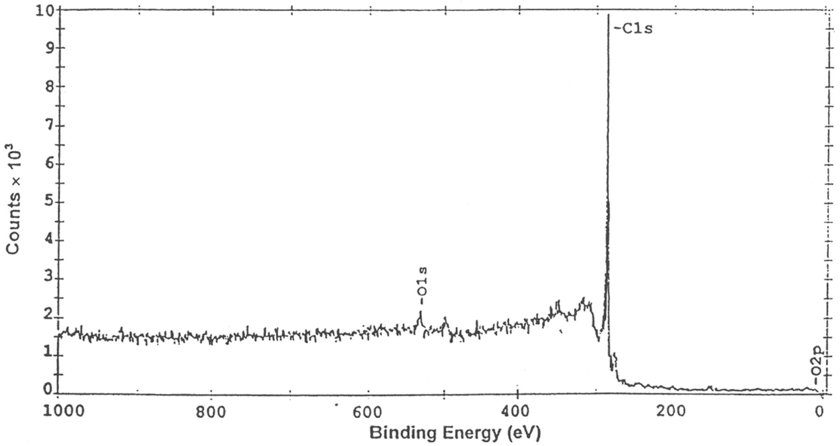


FIGURE 7 ESCA widescan spectrum of unexposed HDPE surface.

sputtered on the polymer surface under these three electrodes, respectively.

3.4. Influence of Different Electrodes on Lap-shear Properties of the Polymer–Metal Adhesive Joint

The influence of different electrodes of DC glow discharge such as copper, nickel, and stainless-steel, resulting in improvement of

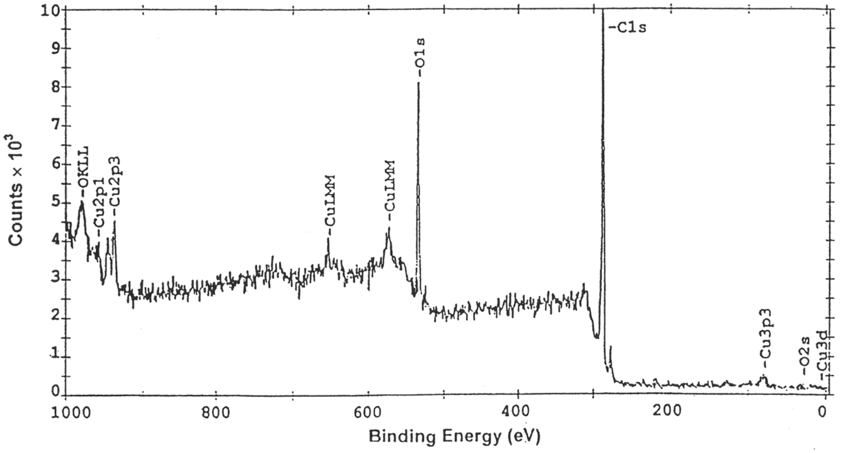


FIGURE 8 ESCA widescan spectrum of HDPE surface modified by DC glow discharge at 13 W power for 120 s under copper electrode.

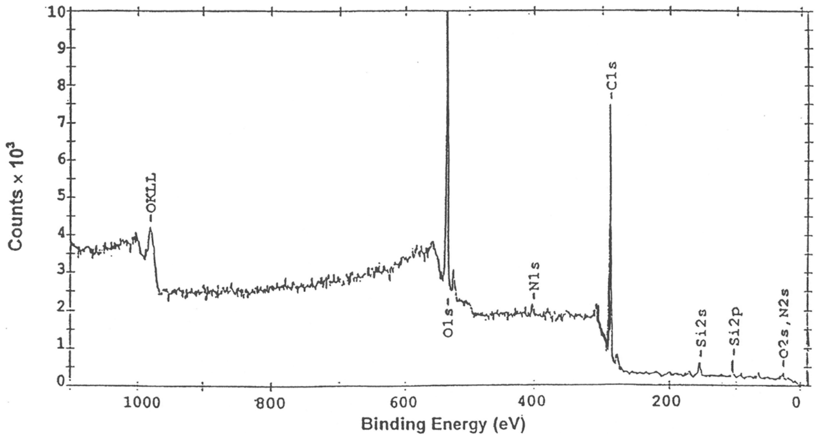


FIGURE 9 ESCA widescan spectrum of HDPE surface modified by DC glow discharge at 13 W power for 120 s under nickel electrode.

adhesion characteristics of polymer with the mild steel, is shown in Figure 11. The exposure of the polymer under the stainless-steel electrode yielded the highest joint strength of the polymer to mild steel followed by those obtained for exposure under nickel and copper electrodes as shown in Figure 11. The joint with the unexposed polymer sheet failed at the interface between the adhesive and polymer as

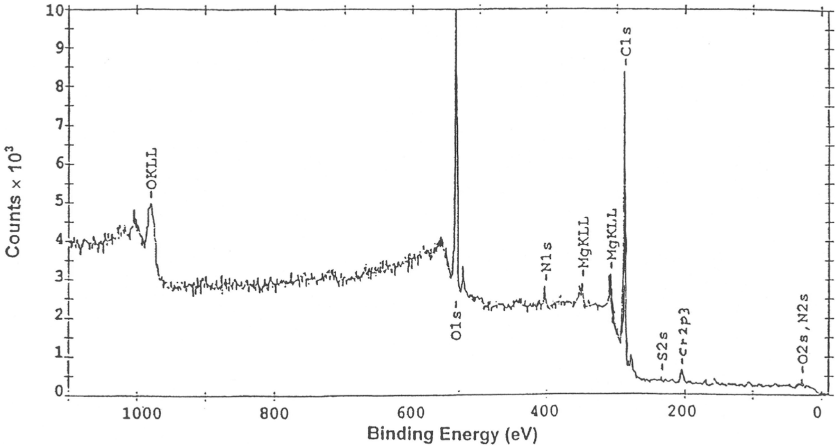


FIGURE 10 ESCA widescan spectrum of HDPE surface modified by DC glow discharge at 13 W power for 120 s under stainless-steel electrode.

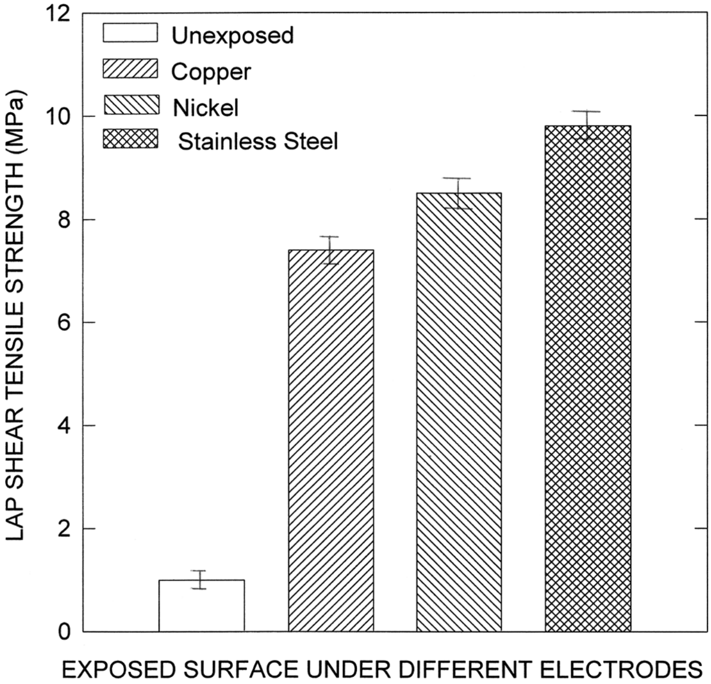
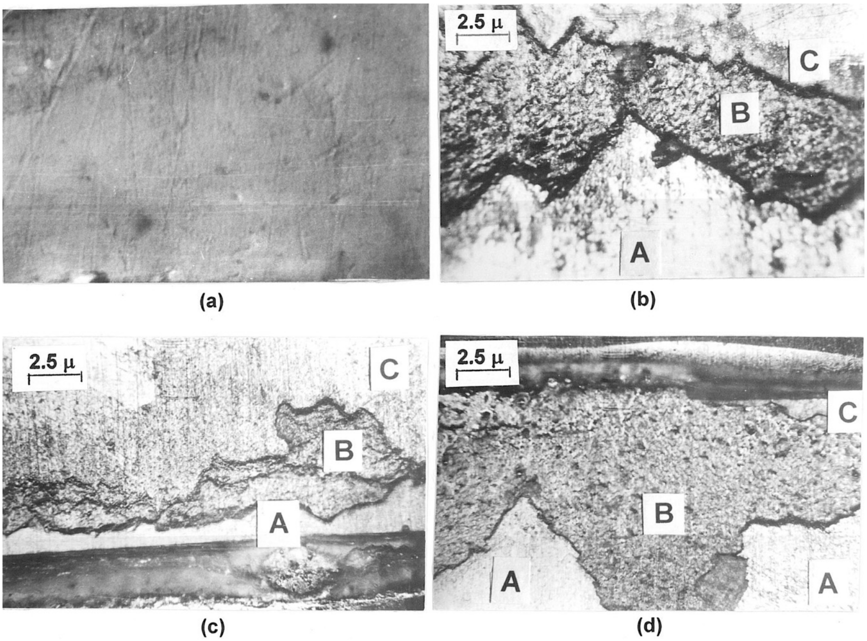


FIGURE 11 Lap-shear strength of adhesive joint of HDPE to steel when HDPE surface is exposed to DC glow discharge at 13 W power for 120 s under copper, nickel, and stainless-steel electrodes.

shown in Figure 12a, resulting in significantly low joint strength. The mode of failure becomes complex when the polymer is exposed to the glow discharge, leading to a significant increase in the joint strength. The failure is primarily confined within the adhesive and, at times, through the polymer–adhesive and mild steel–adhesive interface. This is shown in the optical micrograph where A, B, and C areas correspond to the adhesive failure at the HDPE–adhesive interface, cohesive failure of the adhesive and adhesive failure at the mild steel–adhesive interface, as shown in Figures 12a–d.

4. DISCUSSION

Surface modification of a polymer by exposure to a DC glow discharge is primarily governed by the collision rate of the ionized gas. The energy of the ion colliding with the polymer surface must be sufficient to break the chain and the number of collisions decides the extent of



(a) (b) (c) (d)

FIGURE 12 Optical micrograph of HDPE fracture surfaces of HDPE–steel adhesive joints (a) showing the polymer-adhesive interface failure (A) in unexposed polymer joined to mild steel, and (b), (c) and (d) showing complex mode of failure through the polymer-adhesive interface (A), adhesive cohesively (B), and the adhesive–steel interface (C) in DC glow discharge exposed at 13 W power for 120 s across copper, nickel, and stainless-steel, electrodes, respectively.

surface modification. Carlsson and Johansson [13] have revealed that the increase in surface energy is primarily due to an increase in the polar component of surface energy and the contribution of the dispersion component of surface energy is insignificant. This could be because with the increase in duration of exposure to the glow discharge, interaction of plasma to polymer is longer- and, consequently, more polar groups form on the polymer surface, resulting in an increase in surface energy. Beyond a certain exposure time, there is saturation of polarity with prolonged exposure time. Guezenoc *et al.* [14] have also argued that further collisions at longer exposure times take place dominantly with already formed oxygen functionalities, destroying some of these polar groups. Belkacemi and Coelho [15] have demonstrated that the surface of polypropylene film is affected during corona treatment. It has been reported that as soon as the polypropylene surface is exposed to AC corona, nodules corresponding to the agglomeration of oxidized oligomers start to accumulate on the polymer surface. Furthermore, these particles grow up to a critical size of about 10 microns with increasing time of exposure of about 7 min until they coalesce to form crystals because, under this condition, the temperature could have exceeded the crystallization temperature of the polymer. One can refer to the detailed investigation carried out by Tager [16] on the formation of crystals on the polymer surface.

When the high-density polyethylene sheet is exposed to the plasma under a glow discharge, ions can break C–C or C–H bonds, and, therefore, the produced free radicals could transform into various polar groups. Surface studies by ESCA on the polymer surface exposed under glow discharge indicates that polar functional groups result on the treated surfaces [17]. ESCA spectra of exposed polypropylene [18] clearly reveals the new peaks resulting from plasma oxidation, namely C2 at 286.5 eV due to C–O groups (hydroxyl, ether, or epoxide), C3 at 288.0 eV due to C=O or O–C–O (carbonyl or double ether), and C3 at 289.4 eV due to O=C–OH or O=C–O–C. ESCA widescan spectra of unexposed polyethylene surface and that treated under RF glow discharge at 13 MHz in air and nitrogen atmospheres reveals a small O 1s peak for unexposed polymer, but a significant O 1s peak when exposed to air plasma and a significant N 1s peak when exposed to nitrogen plasma [19].

Therefore, when copper, nickel, and stainless-steel electrodes were employed for surface modification of HDPE at 13 W, the polar components of surface energy increased with increasing exposure time, reached a maximum, and then decreased as shown in Figure 3. The use of a stainless-steel electrode resulted in a larger increase in the polar component of surface energy, followed by the nickel and copper electrodes. The dispersion component of surface energy was more or less unaffected as shown in Figure 4. Thus, it becomes apparent that with the stainless-steel electrode there are more chain-breaking collisions and oxidation of the HDPE surface. It could be possible that under the stainless-steel electrode, there is an increase in temperature of the plasma that could exceed the crystallization temperature of HDPE and, therefore, oxidized oligomers could accumulate on the polymer surface as shown in Figure 6d. Therefore, it can be concluded that different electrodes have different ionic potentials leading to a temperature rise of the polymer surface and resulting in different levels of oxidation of the polymer surface; however, the contributions of inorganic species sputtered under these electrodes are not yet known.

The ESCA studies also indicate a higher oxygen peak of the polymer surfaces exposed with the stainless-steel electrode, as shown in Figure 10. The oxygen peak for exposure to the nickel/is lower than that for stainless steel but higher than that for copper, as shown in Figure 9 for the nickel electrode and Figure 8 for the copper electrode. The optical microscopy results also confirm a higher level of crystallization and debris formation for the stainless-steel electrode as shown in Figure 6d, in comparison with those observed for the nickel electrode in Figure 6c and for the copper electrode in Figure 6b. Therefore, the total surface energy is higher for a given exposure time for the stainless-steel, electrode, as shown in Figure 5. This increase in surface energy has essentially helped to increase the lap-shear properties of the polymer in the mild steel adhesive joint. Therefore, the lap shear the strength of the joint with mild steel is highest when the polymer is exposed under the stainless-steel electrode, followed by exposure under the nickel and copper electrodes, as shown in Figure 11.

5. CONCLUSIONS

The present investigation has led to the following conclusions:

(i) When the HDPE sheet is exposed to a DC glow discharge at a given power level of 13 W across different electrodes of copper, nickel, and stainless steel for similar times of exposure (120 s), the use of the stainless-steel electrode results in a relatively larger increase in the polar component of the surface energy of the polymer, followed by those obtained using the nickel and the copper electrodes.

(ii) The increase in the dispersion component of surface energy of treated HDPE is clearly less than that of the polar component of surface energy.

(iii) When the HDPE sheet is exposed to a DC glow discharge at a given power level of 13 W using different electrodes of copper, nickel, and stainless steel, there is a larger increase in total surface energy of the polymer with the stainless-steel electrode for similar exposure times, followed by those obtained using nickel and copper electrodes.

(iv) When HDPE sheet is exposed to a DC glow discharge, crystallization occurs in certain regions on the polymer surface, which can be observed under an optical microscope. The extent of crystallization is greater under the stainless-steel electrode, for similar exposure times, followed by those obtained using nickel and copper electrodes.

(v) Studies under ESCA confirm that the extent of oxidation of the HDPE surface, as given by the O/C ratio, is relatively larger for the DC glow discharge using a stainless-steel electrode followed by those obtained using nickel and copper electrodes at a given power and exposure time. This confirms the total surface energy of the HDPE findings in (iii).

(vi) When the HDPE sheet is exposed to DC glow discharge at a given power level of 13 W at 120 s using different electrodes of copper, nickel, and stainless steel, there is larger increase in the lap-shear strength of the polymer to mild steel joint with the stainless-steel electrode, followed by those obtained using nickel and copper electrodes.

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