International Journal of Adhesion & Adhesives 40 (2013) 145–148

Contents lists available at SciVerse ScienceDirect

International Journal of Adhesion & Adhesives

journal homepage: www.elsevier.com/locate/ijadhadh

Properties of Fe_2O_3 coating on polymer substrate by ion beam mixing

Sunmog Yeo *

PEFP, Korea Atomic Energy Research Institute, 150 Dukjin-Dong, Yuseong-Gu, Daejeon, Republic of Korea

ARTICLE INFO

Article history:
Accepted 6 September 2012
Available online 17 September 2012

Keywords:
Ion beam mixing (ion assisted deposition)
Adhesion
Polymer modification

ABSTRACT

A polyphenylene sulfide (PPS) substrate is coated with Fe_2O_3 by ion beam mixing, and the properties of the Fe_2O_3 coating are investigated. The adhesion of the Fe_2O_3 coating to the polymer substrate is sufficiently strong, and therefore, the coating is not delaminated even at a high frequency of 20,000 Hz. The adhesion strength is evaluated by vibrating the polymer substrate at high frequencies. X-ray diffraction experiments reveal that the Fe_2O_3 coating is amorphous. In addition, a cross-sectional scanning electron microscope image shows that the Fe_2O_3 coating has a columnar structure.

1. Introduction

Various types of polymers have been intensively studied because they are light-weight, easy to manufacture, and allow mass production. Although polymers are themselves versatile materials, their versatility has been extended by coating them with other materials. Two additional desirable properties can coexist by coating polymers with different materials and such materials are used in printed circuit boards, anti-sticking coating for kitchen utensils, adhesive tapes and materials for use in the biomedical industry [1,2]. Flat panel displays require polymer substrates coated with an inorganic material because polymers are much lighter and less fragile than glass [3,4]. However, when an inorganic material is deposited on a polymer surface, achieving suitable adhesion between surfaces is a challenge in practical applications [5–8] because the surface properties of polymers are completely different from those of inorganic materials [9]. To realize a strong and durable adhesive coating on polymers, it is necessary to treat the polymer surfaces by methods such as corona discharge, plasma treatment, and ion beam mixing (IBM) [10–15]. Among these methods, IBM is environmentally friendly and one of the most useful methods to fabricate an adhesive coating on polymers [1]. IBM has been reported to enhance adhesion between two layers, but there have been few studies on the enhancement of adhesion between oxide materials and polymers.

Adhesion to a substrate is an important property of a coating. However, it is difficult to precisely measure the adhesion of a coating. Many different adhesion tests have been proposed, such as scratch test, scotch tape test, acceleration test, and pull adhesion test [16–18]. However, almost each of these tests has its drawbacks. For example, the scratch test, which is the most widely used commercial test, is sensitive to variations in the friction between the indenter and the coating [16]. In this letter, we show that a typical oxide, Fe_2O_3, and typical metal, Cu, can be adhesively coated on polymer substrates by IBM. In addition, we propose a method to measure the adhesion strength of a coating on polymer substrates on the basis of the sound pressure level (SPL).

2. Experimental details

2.1. Ion beam mixing

Fe_2O_3 and Cu coatings were prepared by IBM using an electron beam evaporator. In this experiment, commercial polyphenylene sulfide (PPS) diaphragms (thickness of 20 μm) were used as the substrate materials. The polymer diaphragms were carefully mounted on a rotatable sample stage, and ion beam bombardment and deposition were performed in the same chamber at a pressure of less than $\sim 2\times10^{-5}$ Torr. Prior to deposition, the polymer diaphragm surface was activated by ion beam bombardments, and then, about 60–70 nm of Fe_2O_3 or Cu was deposited. The thickness of the coating layer was measured using a thickness monitor manufactured by Sycon Instruments Inc. (model STM-100/MF). After the initial deposition, nitrogen ions were irradiated with an ion dose of $\sim 5\times10^{17}$ particles/cm^2 by regulating the irradiation time. Additional deposition was then performed to obtain a certain coating thickness. The initial deposition thickness was calculated using the stopping and range of ions in matter (SRIM) code; it was the thickness at which the irradiated ion beam activated the boundary between the deposited material and the polymer diaphragm most effectively. In our experiment, nitrogen ions were accelerated at an irradiation energy of 70 keV and were incident between the Fe_2O_3 (or Cu) coating

* Tel.: +82 42 868 4670; fax: +82 42 868 8131.
E-mail address: sunmog@gmail.com

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http://dx.doi.org/10.1016/j.ijadhadh.2012.09.006

and the polymer diaphragm at room temperature. Nitrogen ions from a DuoPIGatron ion source were extracted through an extraction unit and accelerated via an acceleration unit. The DuoPIGatron ion source produced N^+ and N_2^+ which were at a ratio of 2 to 8. An X-Y recorder in conjunction with the Faraday cup was used to obtain an accurate ion dose.

2.2. X-ray diffraction and scanning electron microscope

To observe the Fe_2O_3 (or Cu) coating and polymer diaphragms, θ–2θ scans were performed using a Rigaku X-ray diffractometer. The voltage and current of the Cu X-ray radiation source (wavelength of 0.15406 nm) were 40 kV and 20 mA, respectively. Surface morphology was evaluated by obtaining images with a scanning electron microscope (SEM; JSM 5200, JEOL) in a high vacuum state. Because polymer diaphragms are insulators, all coatings in SEM experiments are coated with metallic materials to avoid charging effect. In the experiments described here, a gold coating was deposited by sputtering (Fine Coat Ion Sputter, JFC-1000) in a vacuum. The diaphragms were cooled in liquid nitrogen and cut with scissor to observe the cross-sectional images.

2.3. Sound press level test

The SPL was measured in an anechoic room with micro-speakers fabricated using the diaphragms. The setup for the SPL measurement system is shown in Fig. 2(a). A micro-speaker was placed 0.1 m from a micro-phone in the anechoic room. A signal in the range of 20 Hz to 20 kHz was produced by an AC generator and amplified by a power amplifier that was connected to the micro-speaker. The sound signal from the micro-phone was then analyzed with a B&K 2012 audio analyzer. The measurement details of SPL have been published elsewhere [19].

3. Results and discussion

One of the simplest adhesion tests involves the use of an adhesive tape. Fig. 1(a) shows the adhesion test results for the Fe_2O_3 coating deposited on a polymer diaphragm without IBM, which indicate that the adhesion of the Fe_2O_3 coating is so weak that even a minor impact destroys the coating. However, the Fe_2O_3 coating deposited on a polymer diaphragm by IBM shows strong adhesion. Fig. 1(b) displays a photograph taken after the adhesion test conducted using the adhesive tape for the Fe_2O_3 coating deposited on a polymer diaphragm by IBM. Fig. 1(c) shows a photograph of the Cu coating deposited on a polymer diaphragm by IBM. Fig. 1(b) and (c) shows that adhesion between the Fe_2O_3 (or Cu) coating and the polymer diaphragm is strong and thus, the adhesive tape does not detach from the coating.

Fig. 2(b) shows SPL as a function of frequency for three different diaphragms: Fe_2O_3 coated diaphragm, Cu coated diaphragm, and as received PPS diaphragm. The coating thicknesses of the Fe_2O_3 coated diaphragm and Cu coated diaphragm were 1 μm and 0.4 μm, respectively. All the coatings in this experiment were fabricated by IBM. Interestingly, the thicker coating displays the lower first resonant frequency at around 1000 Hz. This is expected because a large thickness usually implies a large mass, which is inversely proportional to the first resonant frequency. There are no indications that the coating materials delaminate from the substrate after repeated experiments at a high frequency of 20,000 Hz.

The adhesion test with an adhesive tape does not yield a quantitative result. However, when the polymer substrate is vibrated at a high frequency, the adhesion strength can be estimated

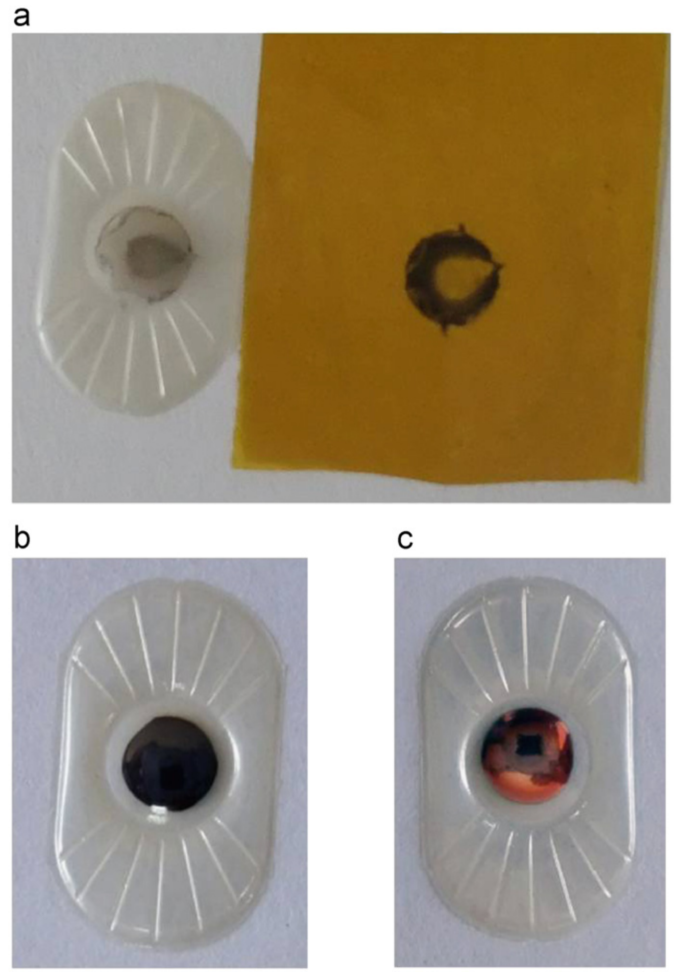

Fig. 1. Adhesion test with adhesive tape for (a) Fe_2O_3 coating deposited without IBM, (b) Fe_2O_3 coating deposited by IBM, and (c) Cu coating deposited by IBM.

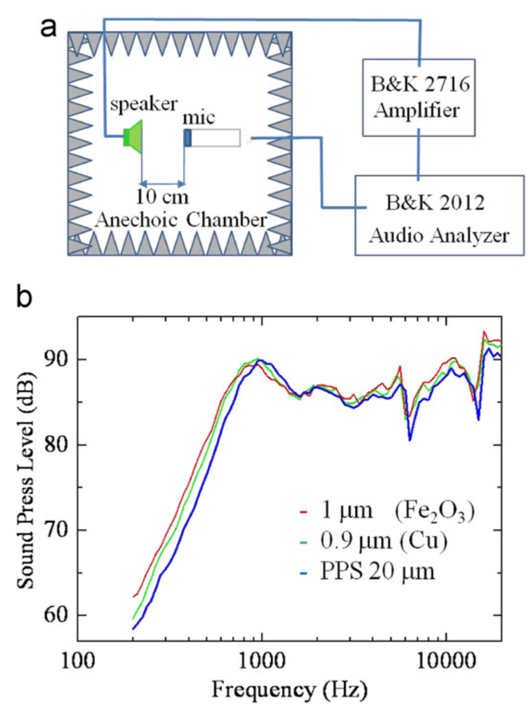

Fig. 2. (a) Schematic of measurement of SPL. (b) SPL for as received PPS substrate, Fe_2O_3 coating, and Cu coating.