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REPORT

Specimen Preparation Technique for Polymeric Materials

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ABSTRACT A simple and cost-effective specimen preparation technique, using two chrome washers to sandwich the specimen has been adopted to assess polymeric materials under the Scanning Electron Microscope (SEM). Areas of interest on both sides of the same specimen can be examined by simply flipping over this specimen assembly to the other side after the initial surface has been viewed. In the conventional specimen preparation mounting on conductive carbon tape, different specimens are required if both surfaces need to be studied. This new technique has greatly helped in investigations of the failure modes on polymeric films and in the understanding of the degradative impact of chemical or mechanical agents on both sides of the polymeric films. In addition, this simple specimen preparation technique also provides a solution for viewing thin and soft polymeric materials such as condoms in which the suspension of the condom between the chrome washers has been shown to largely eliminate artifacts as reported by Rosenzweig et al.

ABSTRAK Teknik penyediaan spesimen yang ringkas dan berkos-efektif dengan menggunakan dua kepingan "chrome washer" telah digunakan untuk menilai bahan polimer di bawah Mikroskop Elektron Imbasan (SEM). Kedua-dua permukaan pada spesimen yang sama boleh dicerap dengan hanya memutar balik set pemasangan spesimen ke arah sebelah yang lain selepas pencerapan pada permukaan yang sebelumnya. Dalam penggunaan teknik penyediaan yang lama dengan tape karbon yang konduktif, dua kepingan spesimen yang berlainan diperlukan jika kedua-dua permukaan dikehendaki untuk pencerapan. Teknik penyediaan baru ini telah memudahkan siasatan terhadap analisis jenis kegagalan pada bahan polimer dan pemahaman terhadap impak dari kesan bahan kimia dan mekanikal terhadap bahan polimer. Di samping itu, teknik penyediaan baru ini telah berjaya menyelesaikan masalah pencerapan terhadap bahan polimer yang nipis, sebagai contoh, kondom di mana pengapungan spesimen kondom di antara dua kepingan "chrome washer" telah berjaya menyingkirkan kesan sampingan yang dilaporkan oleh Rosenzweig et al.

(Simple and cost-effective specimen preparation technique, chrome washers, SEM, polymeric films)

INTRODUCTION

Conventional specimen mounting on a conductive carbon tape only yields details of a surface that is exposed to the electron beam [1] while the underside of the specimen which is attached to the conductive carbon tape no longer serves any purpose. Thus, no proper understanding of the failure modes and degradation by chemical or mechanical agents can be made on a polymeric film which has been prepared using the specimen mounting on the conductive carbon tape.

Moreover, this specimen mounting technique for thin and soft polymeric materials such as condoms revealed a variety of artifacts as reported by Rosenzweig et al [2]. The artifacts were classified as ridging [3], cracking and melting.

The purpose of this article is to introduce a simple and cost-effective specimen preparation technique which uses two chrome washers to sandwich the polymeric materials to be assessed under the SEM prior to mounting on the conductive carbon tape. This simple specimen

mounting technique allows areas of interest on both sides of the same specimen to be viewed as well as making the viewing of condoms under the SEM possible without interference from surface artifacts [4].

MATERIALS AND METHODS

Preparation for Polymeric Film

A chrome washer which was pre-adhered with a double-sided conductive carbon tape was attached onto the area of interest of the polymeric film. Subsequently, the other side of the polymeric film was attached to the other chrome washer which had also been pre-adhered with a double-sided conductive carbon tape.

Preparation for Condom Film [4]

Initially, a circular test specimen was cut out from a circular die from each condom sample. The circular condom specimens were then immersed in isopropanol at room temperature for 10 seconds with gentle rubbing before drying in an oven at 40°C for half an hour.

The washed condom specimens were prepared and analyzed under these conditions:

a) specimen preparation technique as reported by Rosenzweig et al [2] and
b) new specimen preparation technique as described in this study.

The washed condom specimen was mounted without stretching on a chrome washer which was pre-adhered with a double-sided conductive carbon tape. The other side of the washed condom specimen was attached to the other chrome washer which had been pre-adhered with a double-sided conductive carbon tape.

The assemblies of the polymeric films and condom films were sputtered for 2 minutes at 25 mA with gold-palladium. The polymeric films and condom films which need to be viewed on both sides were coated on each side in order to study the film property, failure mode and degradation impact on the films.

All coatings were conducted on an Emitech K550X Sputter Coater. The coated assembly was then mounted on an aluminium stub as shown in Figure 1. SEM analysis was performed at 15kV accelerating voltage in a Hitachi S-3000N SEM.

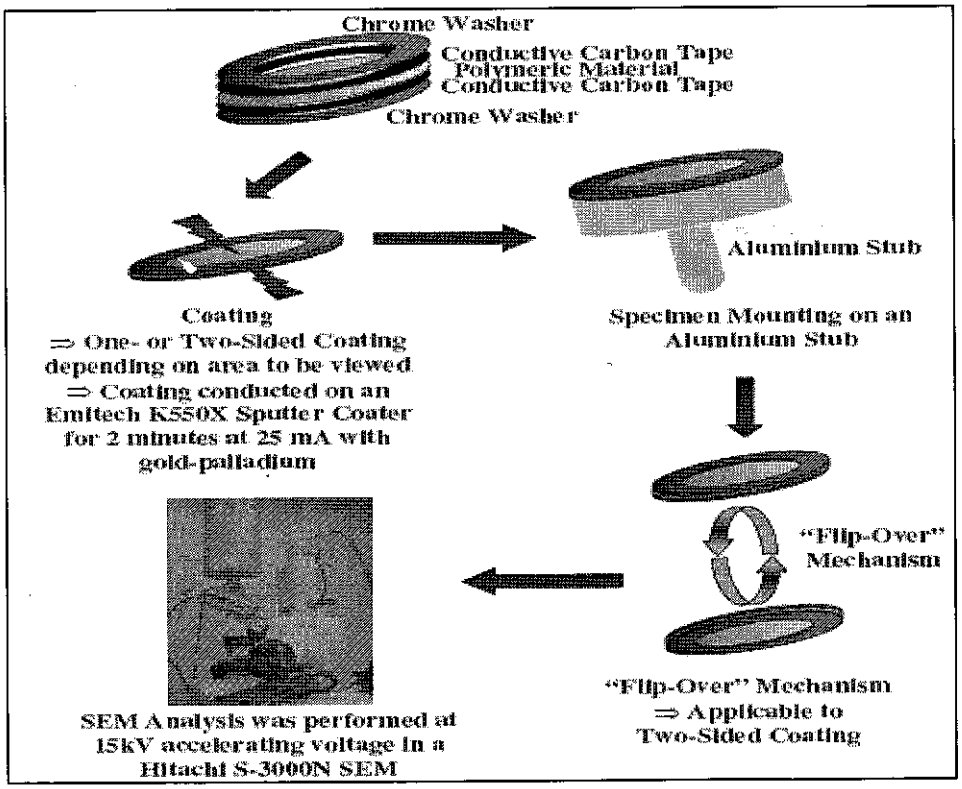


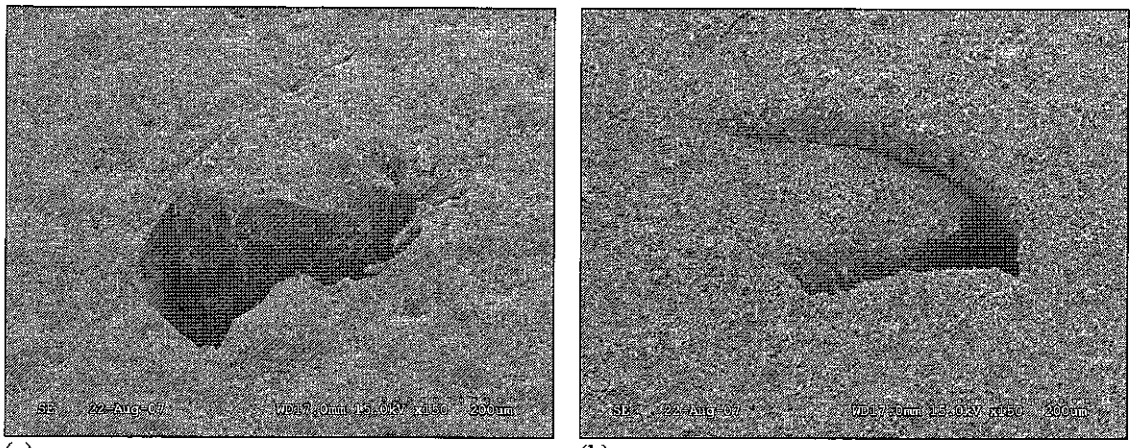
Figure 1. Flow chart of new specimen preparation technique

RESULTS

Defective polymeric and condom films were examined. Figure 2 shows the tilted SEM imaging for defective polymeric films A viewed on both sides of the film at the same location. The failure mode was seen to be a puncture caused by mechanical impact which had penetrated through to the other surface of the defective polymeric film. Figure 3 and Figure 4 show the SEM imaging for defective polymeric film B and condom film C viewed on both sides of the films at the same location respectively. In fact, SEM imaging on the same location on both defective polymeric film B and condom film C films has provided a better understanding of the failure modes.

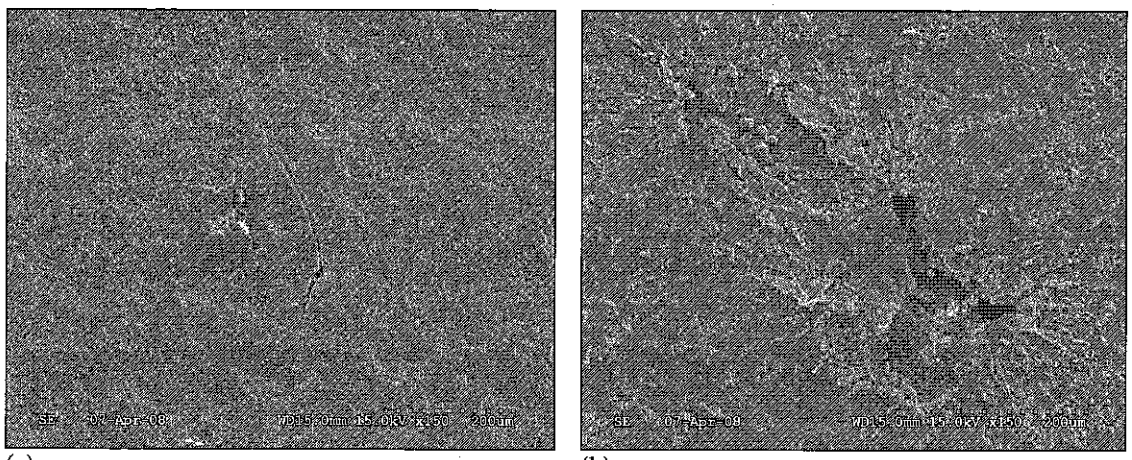
Figure 5 shows the observed surface artifacts in the condom film D prepared using the specimen preparation technique reported by Rosenzweig et al [2].

Figure 6 shows the surface morphology of the condom film E (Figure 6a) and condom films F (Figure 6b) with no surface artifacts. No surface artifacts [4] were observed on all the condom films prepared from the new technique. Figure 7 shows the tilted SEM imaging for condom film G without surface artifacts viewed on the exterior and interior.



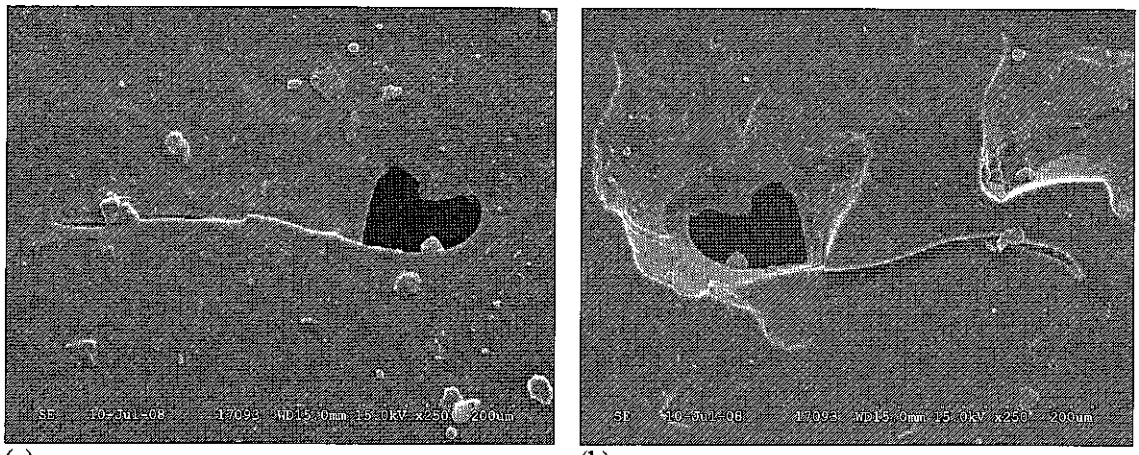
(a) (b)

Figure 2. Tilted SEM imaging of defective polymeric film A with puncture impact viewed on the (a) exterior and (b) interior



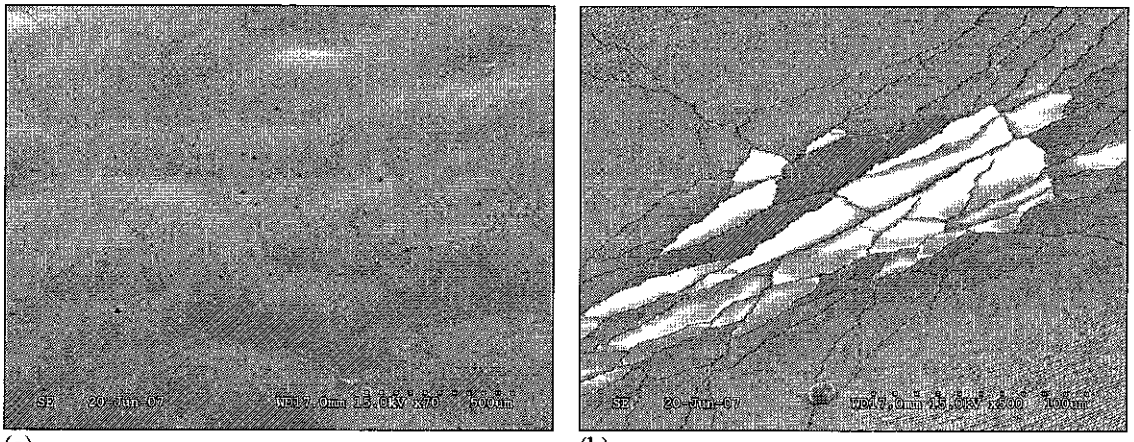
(a) (b)

Figure 3. SEM imaging of defective polymeric film B viewed on the (a) exterior and (b) interior



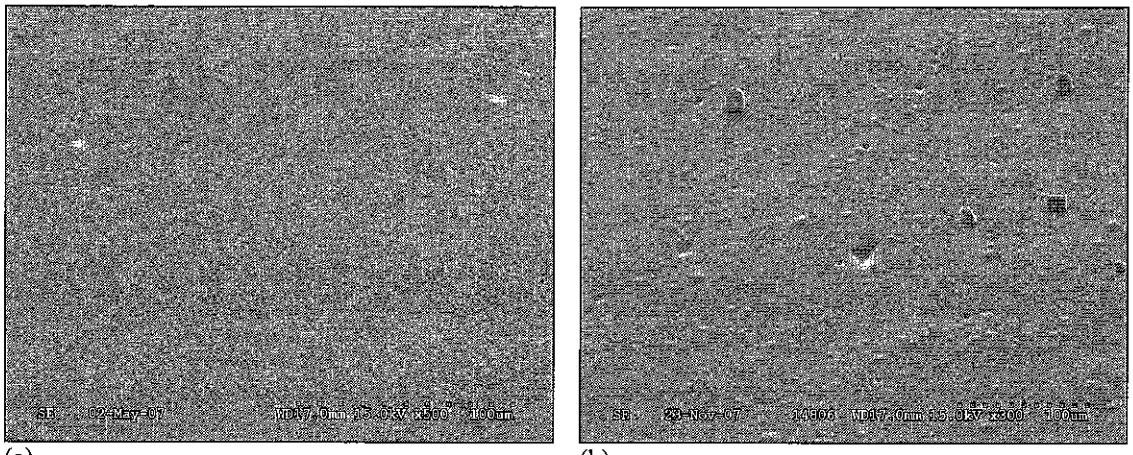
(a) (b)

Figure 4. SEM imaging of defective condom film C viewed on the (a) exterior and (b) interior



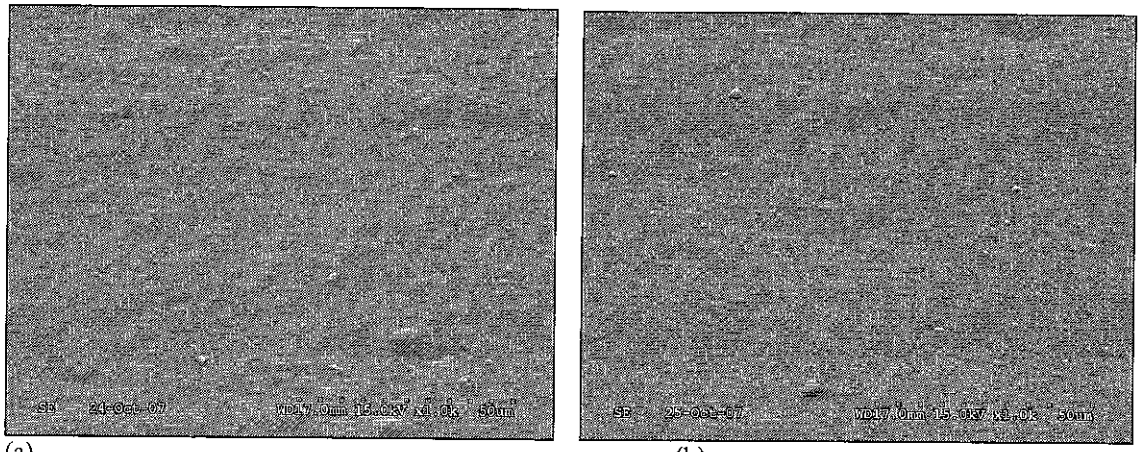
(a) (b)

Figure 5. Tilted SEM imaging of condom film D showing (a) surface artifacts and (b) melting



(a) (b)

Figure 6. Tilted SEM imaging of (a) condom film E and (b) condom film F showing no surface artifacts



(a) (b)

Figure 7. Tilted SEM imaging of condom film G showing no surface artifacts viewed on the (a) exterior and (b) interior

DISCUSSION

The new specimen preparation technique which allows for opposite sides of the polymeric film to be examined on the same location has made the use of SEM more flexible and exciting especially in the investigation of film failures. A better understanding of the degradative impacts on the polymeric films caused by chemical or mechanical agents could be derived from the assessments of both sides of the material.

Furthermore, the new specimen preparation technique which suspends the specimen between the chrome washers has allowed more accurate information to be obtained from the SEM images of thin and soft polymeric films such as condoms through the elimination of artifacts [2] caused by the conventional mounting process.

Consequently, the use of the new specimen preparation technique in the enhanced evaluation of thin and soft polymeric materials surface characteristics by SEM has helped to advance research and developmental work in this area of endeavour.

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