A MORPHOLOGICAL INVESTIGATION OF A CONDUCTIVE POLYMER COMPOSITE RESETTABLE FUSE USING VOLTAGE CONTRAST SEM

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INTRODUCTION

By loading an insulating polymer matrix with an electrically conductive filler and cross-linking the material, it is possible to fabricate devices that are both reliable and relatively inexpensive [1]. One example is a polymeric resettable fuse in which the conductive pathway through the matrix is provided by carbon black[2]. Such devices are widely used in automotive applications[3].

When an abnormally high current passes through such a device, the matrix expands and forces much of the conductive network to break[4]. This raises the resistance by many orders of magnitude and protects any following circuits or devices from the high current. The device is said to be in a 'tripped state'. When the fault passes, the matrix cools and the fuse returns to its conductive state. Such materials are classed as having a Positive Temperature Coefficient of Resistance (PTCR) [1, 4, 5, 6].

The location at which the conductive network breaks is governed by thermodynamic processes, and is found to be along a plane from which rate of heat loss is at its lowest compared to rest of the bulk. This region is termed the 'hot-line'.

Along the 'hot-line' of a tripped device, the potential will drop from the applied voltage to very close to zero. This is due to the high resistance from the broken network. Hence it is possible to exploit this by using Low Voltage Scanning Electron Microscopy (LVSEM) to view the voltage contrast [7] in this region. This would enable active devices to be examined in the microscope and the switching characteristics and any associated morphological changes to be investigated[8].

EXPERIMENTAL

An SEM sample holder was designed to enable a powered, and hence tripped, device to be accommodated in both a Philips 501 SEM and a Cambridge Instruments Stereoscan 250 Mk3 SEM.

A PolySwitch* resettable fuse (type RXE050, obtained from Raychem, Ltd.) was prepared for examination by slicing a section of the device with a sharp razor blade. This was angled to optimise electron collection at the detector and to reveal a large flat area of polymer composite.

The SEM was operated at 1.8 kV and 5.0 kV using the secondary electron detector biased at +250 V. The trip current for the device was 1 A at 12 V, and was provided by an external power supply.

The device was tripped a number of times in succession and the 'hot-line' position recorded on 35 mm photographic film. Because the SEM builds up an image by simultaneously rastering over the specimen and the monitor, it was also possible to slow down the scan speed and trip the device several times during a single photographic exposure.

Recently a new 'flower-press' device holder has been developed to enable the 'device-material' to be analysed in the microscope prior to actual device fabrication. This was used to investigate 1 cm^2 wafers of the composite, in which electrical contact was achieved by sputtering a gold film onto the surface in place of the electrodes, and gripping the device in the holder.

RESULTS AND DISCUSSION

Voltage contrast was obtained on both the PolySwitch device and the 'device-material'. In each case the 'hot-line' was seen to be located close to the centre of the polymer composite material, and was stable over successive trip cycles. Figure 1a illustrates the voltage contrast of such a device, the dark side of the line was at +12 V and so attracted the secondary electrons back in to the material. Hence only those on the ground side reached the detector.

Figure 2b shows rapid tripping while the image was being scanned. The 'hot-line' is seen to be stable on each cycle, and runs diagonally across the photograph. The narrow region of intermediate contrast that separates the un-tripped and tripped states is when the device is powered but not-tripped. This is due to a minimal and gradual voltage drop across the device, and is somewhere between 0 and 12 V. The finite time taken to trip can thus be deduced from the scan speed and it is reduced with successive cycles. This is primarily because the device is already warmer from the previous trip. The switching time is enhanced and the reset time extended because of the poor thermal conductivity of the vacuum in the SEM.

Figure 2c is a digitally frame-grabbed image of the voltage contrast seen on the 'device-material' at high magnification. It reveals features in the vicinity of the 'hot-line' that can be interpreted as carbon black aggregates. This illustrates the potential that this technique has for understanding the morphological differences that arise at the active region of a device.

CONCLUSION

The results have shown that both a device and the composite material can be investigated using LVSEM. Because of the stability of the device, it is anticipated that by identifying the 'hot-line' in this way, and then probing the region at higher magnification, differences in the composite morphology will be highlighted, which can be directly correlated to the tripping mechanism. Some progress towards this goal has already been made, and a variety of sample preparation techniques are being explored.

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*PolySwitch is a trademark of Raychem Corporation.

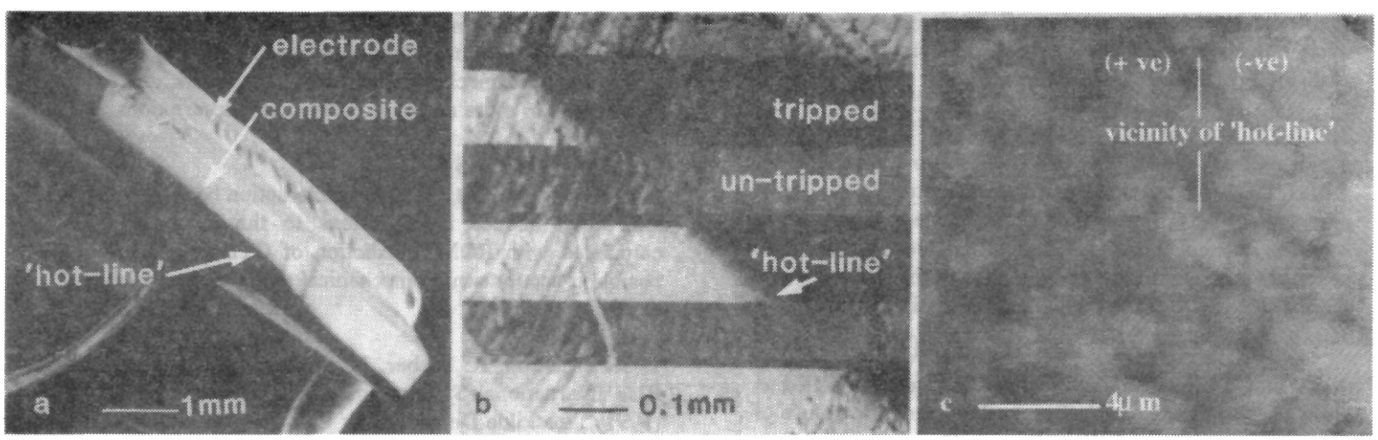

Figure 1: a The voltage contrast seen at low magnification on a tripped PolySwitch device. b: Successive trip cycles during a single scan indicates the stability of the 'hot-line'. The SEM scans horizontally, and the 'hot-line' runs diagonally from left to right in the regions imaged when the device was tripped c: Higher magnification digital image of carbon black aggregates in the matrix near the 'hot-line'.