Use of gold/palladium surface replicas for high kV (30), SEM examination of low Z materials

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Abstract. A procedure for making high fidelity gold/palladium surface replicas of various low atomic number (Z) materials for SEM examination has been developed. The procedure facilitates simultaneous replication of a number of SEM specimens. These replicas provide improved image quality, elimination of charging and beam penetration artifacts and microtopographical information, often not available by direct SEM examination of low Z materials at 30 kV.

Keywords. Scanning electron microscopy; low Z specimens; resolution; replica; perspex; gold; palladium.

1. Introduction

Quite often, an actual specimen to be studied is not suitable for direct emissive mode examination in the SEM particularly at high kV and high magnification. For example, fragile materials such as cotton fabric and unstained or unfixed biological materials may suffer severe thermal damage due to the high energy of the beam. Besides, siliceous, amorphous and microporous materials such as diatomaceous earths, zeolites, catalysts, etc. exhibit random charging due to adsorbed water. Specimens such as skin, teeth and machine parts cannot be moved to the specimen chamber of the SEM. In such cases surface replicas of the specimens have to be made and examined in place of the actual specimens. For a given accelerating voltage, beam damage and charging are proportional to the extent to which the beam can penetrate into the bulk of the specimen. Since this range of penetration (Bethe range) varies inversely as the atomic number (Z) (Wells 1974) of the material of the specimen, the damage and charging artifacts are particularly severe with materials such as above. Besides these artifacts, the other effects of beam penetration namely obfuscation (Koch 1975) of the surface topography (i.e. the appearance of overlapping images of underlying structures of the specimen) and flaring of small structures (due to the signal emanating from their entire surface), further deteriorate the image. Although efficient coating techniques can often effectively eliminate the damage and charging artifacts, loss of resolution of fine details of low Z materials during high kV examination imposes severe restrictions on the useful magnification of the image. Although lower kV (5-10kV) reduces beam penetration, the expected benefit is often nullified at high magnifications, due to increased noise levels and electron optical aberrations associated with low kV beams.
Generally materials like ‘Silflo’, acetate tape, poly-ethylene, etc have been used for making the negative replica and araldite for positive transfer. However, for obtaining an impression of high Z replication material for improved image quality a novel procedure was employed using ‘Perspex’ for making negative imprint and Au/Pd reinforced with copper for making the positive replica. The results obtained with various materials examined directly in the SEM or after using this new replica technique are described in this paper.

2. Materials and methods

2.1 Materials

The following materials and their Au/Pd replicas were examined in the emissive mode at 30 kV in the Cambridge Stereoscan S4-10: (a) cotton fibre (b) diatomaceous earths (Kieselguhr) (c) gypsum (d) zeolite (e) nickel catalysts.

2.2 Replica making technique

One inch square sheet of 3 mm thick perspex was placed on a large SEM stub. The stub was placed over chloroform in a closed petridish which was kept warm (50°C) over a hot plate. About 1 mm thick surface layer of the perspex sheet got sufficiently softened over the chloroform vapour in an hr. The softened plastic was pressed onto the specimen mounted on a glass slide. The whole assembly was then held in a clamp which was gently tightened and kept overnight in an oven at a temperature of 60 to 70°C. When the solvent evaporated and the plastic hardened, the assembly was cooled to room temperature and the glass slide detached from the plastic negative replica. The replica was cleared of the specimen material by various processes depending upon the nature of the material. For example, (i) Cotton fabric could be pulled off with a pair of fine forceps. The remaining fibres were dissolved in cupriethylenediamine hydroxide (Cuene). (ii) Silicious materials such as Kieselguhr, rice husk silica, zeolites and catalysts were treated in hydrochloric acid and then dissolved in hydrofluoric acid. After clearing the material, the replica was treated in a detergent solution, washed with distilled water and dried in a dessicator. (Sonication for a few seconds during cleaning also helps in dislodging small particles). The positive replica was made by plasmaglo cleaning of the negative, followed by vacuum deposition of about 2 inch length of 0.020 inch/0.5 mm diameter gold/palladium wire (60:40) onto the cleared plastic negative replica using a tungsten basket heater. The thin gold/palladium film was reinforced by vacuum deposition of copper through a molybdenum boat, followed by a sufficiently thick layer of electrodeposited copper. The copper plating bath consisted of a saturated solution of copper sulphate containing a few drops of concentrated sulphuric acid. The electrodes were made of heavy gauge copper strips. The coated negative replica was attached to the cathode with silver dag. Plating was done initially at a current of 100 mA for 30 min followed by 500 mA for 3-4 hr. By the end of this period the copper deposit was sufficiently thick to impart rigidity to the gold/palladium film.

The composite replica was detached from the electrode carefully, washed thoroughly and dried under vacuum. The plastic was dissolved in chloroform on a hot plate at