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PA32) A Comparative Study on Collecting Substrates in Single Particle Analysis

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1. Introduction

A recently developed single particle analytical technique, named low-Z particle electron probe X-ray microanalysis(EPMA), has been successfully applied to characterize various types of individual aerosol particles(Hwang and Ro, 2006). For the low-Z particle EPMA, aerosol particles are collected on a substrate, typically Ag foil, by using a cascade impactor. In several studies(Choel et al., 2005; Laskin et al., 2003; Szaloki et al., 2001), different collecting substrates were examined to evaluate their feasibility in single particle analysis. In this study, the characteristics of three collecting substrates(i.e. Al foil, Ag foil, and copper TEM grid) are investigated to determine which collecting substrate is the best for single particle analysis.

2. Sampling and method

Atmospheric aerosols were collected at an underground shopping area(Dongdaemun subway station, Seoul) on 11 January 2007. Particles were sampled on Al foil, Ag foil, and copper TEM grid using a three stage cascade impactor(Dekati PM-10) having aerodynamic cut-off diameters of 10, 2.5, and 1μm for stage 1, 2, and 3, respectively, at 10L/min flow rate. For this study, only stage 2 sample was analyzed. About 70 particles each for three substrate samples were analyzed manually. The measurements were carried out on a Hitachi S3500N scanning electron microscope(SEM) equipped with an Oxford Link SATW ultra-thin window EDX detector.

3. Results and discussions

3.1 Morphologies of particles on Al, Ag, and TEM grid substrates

Typical secondary electron images of particles with similar chemical compositions collected on Al, Ag, and TEM grid substrates are shown in Fig. 1. The particles collected on Al and Ag foils(see Fig. 1a and 1b) exhibit core-shell structures; white in core regions and dark in shell regions.

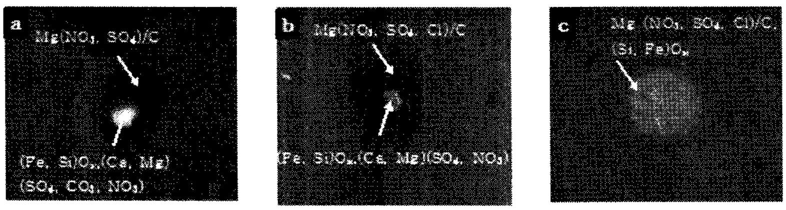

Fig. 1. Secondary electron images of particles with similar chemical compositions on (a) Al foil, (b) Ag foil, and (c) TEM grid.

From the secondary electron images of particles on Al and Ag foils(Fig. 1a and 1b), shell regions (i.e dark areas with low yields of secondary electrons) are probably composed of deliquescent reacted species. Core regions(i.e. bright areas on secondary electron images) presumably consist of solid unreacted species. X-ray spectra obtained from the core regions indicate iron and silicon oxides(i.e. (FeO_x, SiO_x)) and sulfates, carbonates, and nitrates of calcium and magnesium(i.e. (Ca, Mg)(SO_4, CO_3, NO_3)). Shell regions are mainly composed of magnesium nitrate and sulfate with carbonaceous species(i.e. (Mg)(SO_4, NO_3)). Fig. 1c shows a particle on TEM grid with a chemical composition similar to that of particles in Fig. 1a and 1b. However its morphology is different compared to the other two particles; it looks round in shape and white as a whole. Its X-ray spectrum shows a mixture of unreacted mineral and reacted chemical species. The different morphologies observed for particles with similar chemical compositions on Al foil, Ag foil, and TEM grid are thought to be due to the difference in hydrophilicity of the three substrates.

3.2 X-ray spectra of particles on Al, Ag, and TEM grid substrates

The peak-to-background ratio is used here for identifying as well as quantifying the elemental compositions of individual particles measured by EPMA. An increase in peak-to-background ratio allows an analyst to measure a smaller elemental mass fraction in the specimens(Goldstein et al., 2003). X-ray spectra of particles with similar chemical compositions and sizes deposited on Al foil, Ag foil, and TEM grid are presented in Fig. 2. Due to the high background level of Ag substrate, net intensities of elements present in the particle become low. TEM grid has lower background level as compared to the other two substrates. Thus the X-ray intensities measured for the particle sitting on the TEM grid are found to be stronger than those on the other two substrates.

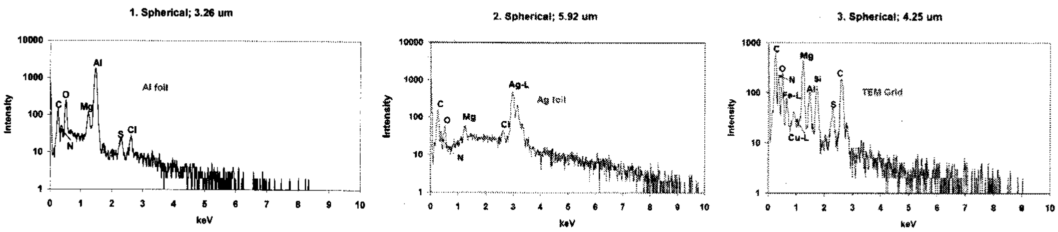

Fig. 2. X-ray spectra of particles with similar chemical compositions on Al foil, Ag foil, and TEM grid.

Furthermore, AgL-lines can hinder the determination of Cl and K concentrations and AgM-lines can overlap with K_α-line of C. The determination of C and O in particles can be impaired by C and O X-rays from the TEM grid itself. This substrate would be adapted for low-Z particle EPMA after C, O and Cu interferences are corrected. A correction procedure is currently under investigation.

References

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