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Organic SIMS with single massive gold projectile: Ion yield enhancement by silver metallization

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Abstract

The yield of CN from glycine rises from one CN per projectile impact to two when the sample is covered with a nominally 1 nm thick layer of silver. These yields were obtained for bombardment with Au_{400}^{4+} projectile with 136 keV impact energy. A multitude of CN-based clusters, e.g. $Ag_xO_y(CN)_z$ are produced concurrently. As a result, a total of three CN-based secondary ions are generated per projectile impact. The exceptionally high yield for CN is of interest for biological SIMS.
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Keywords: Yield enhancement; Silver deposition; Massive gold projectile; Silver adduct ions

1. Introduction

Significant advances in detection sensitivity have been achieved in SIMS with the introduction of polyatomic and, most recently, massive projectiles [1,2]. The purpose of this study was to test whether silver metallization could still further enhance the secondary ion yields and hence detection sensitivity. Metallization has been shown to be an efficient method for increasing molecular ion yields from organic targets in SIMS using atomic and SF_5^+ polyatomic projectiles [3–5]. We present here the first report on secondary ion yields when combining massive gold cluster bombardment with target metallization. As a test case we examined the response obtained from silver metallized glycine targets. An account of the nature and abundance of the secondary ions is presented below including the yield of the deprotonated molecular ion and CN fragment ion. The latter is of practical interest in biological SIMS since a molecule can be labeled with ^{13}C and/or ^{15}N and thus be detected with high accuracy via the labeled CN.

2. Experimental and sample preparation

The experimental procedure used is that of SIMS with key differences: the nature of the projectile, the bombardment with individual projectiles each resolved in time and space and the corresponding event-by-event detection of the secondary ions, SIs.

A description of the experimental set-up including the Wien filter for projectile selection, pulsing for single impact experiments, ToF mass spectrometer, data acquisition and analysis software for SI identification is provided elsewhere [6]. Details on the operational conditions of the LMIS to produce massive gold clusters such as "Au_{400}^{4+}" are available in the article by Bouneau et al. [7]. In the present study, Au_{400}^{4+} projectiles with 136 keV total energy were sent on a target biased at −9 kV. Samples were prepared by vapor deposition of glycine powder, purchased from Aldrich, on a silicon wafer. The thickness of glycine after vapor deposition was ~5 μm. Silver deposition was carried out with a sputter coater (208HR Ted Pella, Inc.) using argon as operation gas and equipped with a film thickness controller. Samples were analyzed immediately following the silver coating.

3. Results and discussion

The mass spectra of a silver coated glycine (nominally 1 nm thickness) sample and the uncoated equivalent are shown in Fig. 1a and b. Peak intensities were normalized to the total number of projectiles used to generate the spectra. The differences in the spectrum shown in Fig. 1b versus that in Fig. 1a are due to metallization under practical reproducible

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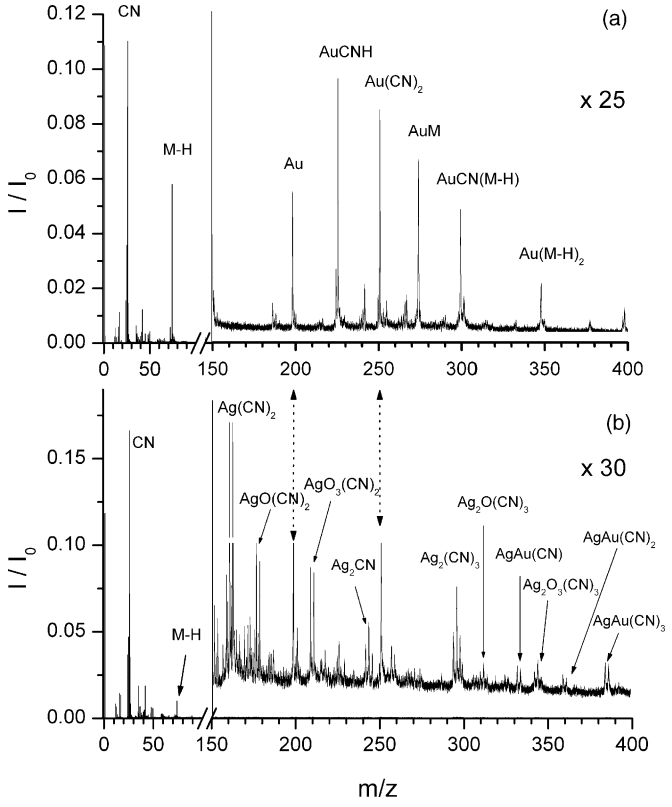

Fig. 1. Mass spectra obtained from: (a) an uncoated and (b) silver coated (nominally 1 nm thick layer) glycine target under Au_{400}^{4+} bombardment at 136 keV total energy. The peak intensities (I) are normalized to the total number of projectiles (I_0) used to generate the spectra.

conditions resulting in a nominal 1 nm thick silver cover. One concern is the quality, i.e. the completeness, of the silver coating. It has been shown on other surfaces [5,8] that the silver layer is most probably composed of nanoparticles organized in islands with silver free zones in-between. In our case an indication of the area covered by silver versus that left uncovered may be inferred from the intensity of the glycine molecular ion peak in Fig. 1b. This intensity is six-times smaller when glycine is coated with a nominally 1 nm thick cover of silver. However, the disappearance of the glycine signal could also be attributed to the sputter coating method which may be destructive for the sample surface [8]. A remarkable feature observed in Fig. 1b is the variety and abundance of silver adduct ions. In contrast, the adducts in Fig. 1a are, as expected, only those formed with gold [9]. We note in Fig. 1b, the silver adduct ions, $AuAg(CN)_z$ and $Ag_xO_y(CN)_z$, with x, y and z up to three. Some recombination chemistry with gold atoms still occurs when glycine is covered with a silver layer as evidenced with the AuCNH and $Au(CN)_2$ ions. The disappearance of the glycine molecular ion with gold as an adduct from the mass spectrum (Fig. 1b) relates to its mechanism of formation which apparently requires the presence of glycine molecules at the surface of the target.

The dependence of the ionization enhancement for analytically significant ions was investigated for various silver thicknesses. Mass spectra obtained with nominally 1, 3 and 5 nm layer thicknesses are shown in Fig. 2a–c, respectively. The peaks intensities are normalized to the total number of events. The CN peak intensity remains constant with increasing layer thickness. This observation suggests that CN ions can be emitted from up to 5 nm below the surface. This observation is in agreement with a molecular dynamic (MD) simulation model [10] using Au_{400}^{4+} projectiles of 40 keV energy impacting various targets. Also noteworthy is that silver and gold ions are clearly visible in all three mass spectra. The only significant change concerns the gold and silver CN adducts which decrease with increasing layer thickness. Those ions are barely detected when glycine is covered with the 5 nm layer. In this sample we observe

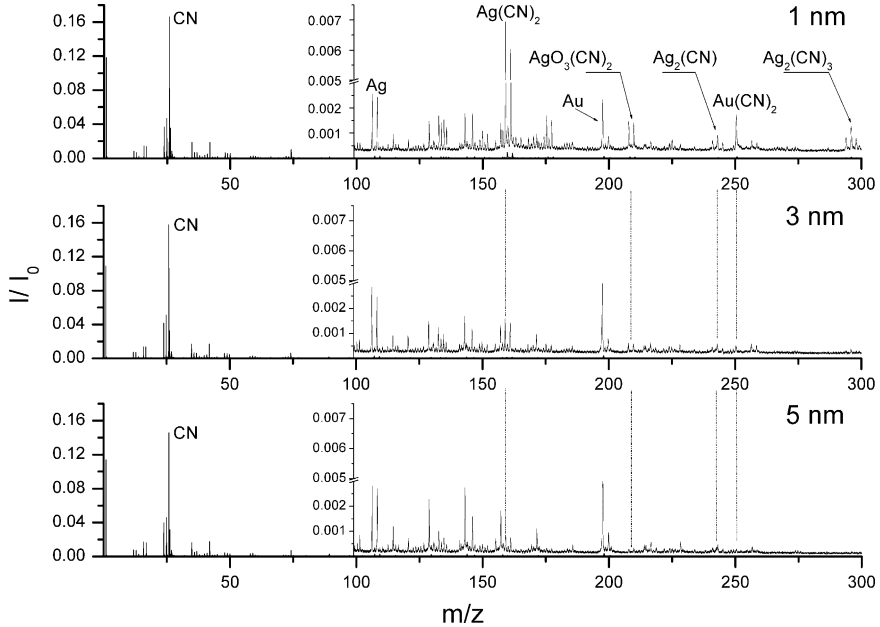

Fig. 2. Mass spectra obtained from a silver coated glycine target under Au_{400}^{4+} bombardment at 136 keV energy for 1, 3 and 5 nm nominally silver thicknesses. The peak intensities (I) are normalized to the total number of projectiles (I_0) used to generate the spectra.

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