

Low-energy electron irradiation of a thin sheet of a potential copper precursor

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Taylor A. Low-energy electron irradiation of a thin sheet of a potential copper precursor. *J Nanosci Nanomed.* 2022;6(2):1-2.

ABSTRACT

Copper's strong electrical conductivity makes it a preferred material for the construction of nanoscale electronic devices. In such applications, the deposit's conductivity and thermal behaviour are highly dependent on the purity achieved, which can still be increased using traditional procedures. Nonstandard techniques, such as FEBID in aqueous solution or ion beam aided deposition with plasma treatments, have yielded pure deposits. The use of precursors with multiple copper ions can help increase the quality of copper deposits.

This class of metallic complex precursors has been proposed for applications requiring tight composition control, such as alloy deposition. The co-authors from Nicolaus Copernicus University in Toru, Poland, developed a new group of copper precursors $[\text{Cu}_2(\text{R}'\text{NH}_2)_2(\text{O}_2\text{CR})_4]$ for chemical vapour deposition (CVD) using two copper(II) cations.

Key Words: *Electrical conductivity; Precursors*

OPINION

The present paper will focus on two different compounds, $[\text{Cu}_2(\text{EtNH}_2)_2(\text{O}_2\text{CC}_3\text{F}_7)_4]$ and $[\text{Cu}_2(\text{EtNH}_2)_2(\text{O}_2\text{CC}_2\text{F}_5)_4]$, which will be referred to as compound A and compound B, respectively. The length of the four perfluorinated carboxylate ligands is the only difference. The evaporation of the precursor in FEBID investigations is a delicate process. Not only must the precursor be in the gaseous state, but it must also be deposited on the surface where the electron beam is focused. The major goal of this research is to learn more about how this precursor responds to electron irradiation in vacuum under FEBID circumstances. In a method comparable to successful investigations of various precursors placed on surfaces and irradiated by 500 eV electrons, we separate the deposition and electron irradiation stages here.

When using a set precursor quantity, there are two things to keep in mind. To begin with, the recorded signals' temporal evolution is mostly attributable to changes in the composition or quantity of the accessible precursor in the irradiation area. Second, when the precursor quantity exposed to the electron beam is not limited by adsorption and diffusion, irradiation is done at the start of the experiment in the electron-limited phase. This regime is recognised to produce the greatest results in terms of spatial resolution, therefore it will be useful in FEBID in the future.

In Poland, the IR spectra of the virgin chemicals A and B were recorded. The vibrational fingerprints of compounds A and B are quite similar.

The hallmarks of the $\nu(\text{CH})$ stretching band, positioned in the 360–380 meV range due to the coordination by the nitrogen group, and the (NH) stretching band, positioned in the 380–400 meV range, are visible for ethylamine apical ligands. At 100 meV and as a shoulder at 120 meV, there are tiny signatures of NH_2 scissoring at 190 meV and (CC) in C–CN chains. With the (COO) stretching at 206 meV and the strong bands of mixed CC and CF_3 stretching modes from 111 to 165 meV, the four perfluorocarboxylate ligands are apparent. The biggest differences between the two compounds may be found in this spectral band. By monitoring their IR spectra again during the transfer from Poland to France under ambient conditions, the compound's stability was confirmed. The sample used to record spectrum e was placed under vacuum for the Electron-Stimulated Desorption (ESD) studies detailed in the following section, however HREELS analysis of these samples was not possible due to the mixture of native oxide and compound layers on it. With fact, probing a poorly conductive material in HREELS is a difficult task. Always a challenge. However, a portion of the brain could be probed a silicon surface in a corner that hasn't had any material put on it. The THF, the solvent used in the dip-coating procedure, had a minor signature in the spectrum.

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Received: 05-Mar-2022, Manuscript No. PULJNN-22-4527(M); Editor assigned: 07-Mar-2022, PreQC No. PULJNN-22-4527(P); Reviewed: 21-Mar-2022, QC No. PULJNN-22-4527(Q); Revised: 21-Mar-2022, Manuscript No. PULJNN-22-4527(R); Published: 28-Mar-2022, DOI:

10.37532/puljnn.22.6(2).01-02



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As a result, by depositing the undiluted chemical directly into HREELS, the THF signal in HREELS was avoided. A thin layer of compound B was placed on a bare gold surface for the vacuum vibrational investigation of the complex. The spectrum was captured at 30 K to improve spectral resolution, despite the fact that it was still significantly wider than with IR (approx. 7.6 meV with HREELS, 0.2 meV with IR). The spectrum displays broad and overlapping fingerprints that closely resemble those seen with IR in air, with the caveat that relative intensities can differ from IR to HREELS.

Nonetheless, there are a few key factors to keep in mind. When compared to the typical signature of hydrocarbon layers in HREELS, the broad peak in the 350–400 meV range is modest. This indicates that a significant number of amine ligands are lost, which is consistent with the absence of $\nu(\text{NH}_2)$ stretching, which is supposed to be a weak and broad signal at 380–400 meV. Despite the fact that NH stretching modes are notoriously difficult to observe in HREELS Other spectral fingerprints can be used to deduce the loss of amine ligands. The other expected contribution, the (NH) angular deformation, which is normally more intense, cannot be detected at 192 meV, nor can the $\nu(\text{C}-\text{CN})$ stretching at 115–120 meV. Because fluorocarbon ligands exhibit signatures in the same range as amine ligands, the signature of (CC) stretching at 130 meV cannot be attributed to them definitively. Only the shoulder at 180 meV, attributable to (CH₂) and (CH₃), is unique to amine ligands in this study. In conclusion, the ethylamine ligands' fingerprints are all quite tiny in the spectrum. Heating was used to further investigate the compound's potential to remove its amine ligands. Figure 2 shows the spectrum g, which was collected

under the identical conditions as spectrum f but after a slight annealing at ambient temperature. ESD studies on dip-coated samples of compound A introduced under vacuum at ambient temperature were used to further investigate the precursor decomposition.

A Quadrupole Mass Spectrometer(QMS) is used to detect neutral fragments generated during irradiation. Different processes can emit neutral fragments following irradiation. Above the ionisation threshold, Dissociative Ionisation (DI) is the most efficient mechanism at high energy. It generates a neutral fragment and a cation that can be neutralised later. The most efficient mechanism at lower energy and above the electronic excitation threshold is Neutral Dissociation (ND), which creates two neutral fragments. It competes with Dipolar Dissociation (DD), which creates two oppositely charged ions that can be neutralised further. The only efficient mechanism in the low-energy region, below the two previously described thresholds, is Dissociative Electron Attachment (DEA), which creates neutral species and a neutralizable anion.

Because DI is the most efficient technique, ESD efficiency is higher at high energy. In FEBID experiments, however, the distribution of secondary electrons sharply peaks in the 1–5 eV range, significantly increasing the likelihood of DEA. Furthermore, the ESD tests given here were carried out at 300 K, which makes them more vulnerable to light species detection. At this temperature, heavier pieces can be held on the surface via physisorption, while lighter neutral species, with the exception of those with substantial dipolar momentum, will desorb. Furthermore, the detection yield of QMS is higher for light species.