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Intelligent Data Analysis System (IDAS) tool for Oxidation State Analysis of Copper Leadframes using Auger Electron Microprobe

Santillan, Abegail I.[1], Bauca, Marianne Therese A. [1], Castro, Lynne Jerisa A. [1], Martin, Zamantha Nadir Z.[1] and Monsada, Araceli M.[1]

[1] *Industrial Technology Development Institute, ADMATEL Bldg., DOST Compound, Bicutan Taguig Metro Manila Philippines, aisantillan.itdi@gmail.com*

***Abstract* – The Semiconductor and Electronics (S&E) sector is vital to the Philippine economy, with copper (Cu) extensively used in semiconductor packaging. However, Cu's susceptibility to oxidation can impair leadframe reliability. This study establishes an Auger electron spectroscopy (AES) spectral profiling database to identify oxidation states in Cu alloy-based leadframes, using the Intelligent Data Analysis System (IDAS) developed by DOST-ITDI and ASTI. Oxidation was simulated via controlled heat treatment, with surface changes analyzed through optical microscopy, FE-SEM, EDS, and AES. Optimized AES parameters (10.0 kV, 10.0 nA) were validated using Cu and Cu_2O references. Oxidation resulted in color changes, increased surface texture, and chemical modification, with AES confirming the conversion of Cu to Cu_2O. Principal Component Analysis (PCA) and *k*-means clustering using IDAS effectively classified oxidation states, with PC1, PC2, and PC3 accounting for 42.22%, 25.06%, and 6.94% of the variation, respectively. This demonstrates IDAS as a reliable tool for monitoring copper degradation in semiconductor packaging.**

I. INTRODUCTION

The Semiconductor and Electronics (S&E) sector is a major contributor to the Philippine economy, accounting for nearly 60% of the country's exports and significantly influencing its GDP [1][2]. Copper (Cu) is widely used in semiconductor packaging, especially in leadframes and wire bonding, due to its excellent electrical and thermal conductivity, mechanical stability, and lower cost compared to alternatives like gold, aluminum, and nickel [3][4][5][6][7].

However, copper's high affinity for oxygen makes it highly susceptible to oxidation, especially under elevated temperatures. This oxidation leads to reliability issues such as delamination between the copper leadframe and epoxy molding compound (EMC) and failures like non-stick on lead (NSOL) during wire bonding [8][9][10]. Such failures can compromise package integrity, ultimately reducing product yield and reliability.

To mitigate oxidation, various surface treatments such as plating with nickel, gold, or tin have been adopted [11]. However, these treatments add to production costs and complexity. Alternatives like phosphor bronze and other Cu alloys are also used for better corrosion resistance [12][13]. Despite these approaches, oxidation of copper leadframes remains a persistent challenge in the microelectronics industry.

Advanced analytical techniques like Auger Electron Spectroscopy (AES) are crucial in understanding oxidation at the surface level. AES provides high-resolution surface chemical analysis and is capable of distinguishing between elemental Cu and its oxides [14][15]. Multivariate analysis (MVA) tools such as Principal Component Analysis (PCA) and k-means clustering can further enhance the interpretation of spectral data, facilitating effective classification of oxidation states [2][16].

This study presents an integrated approach combining AES and an Intelligent Data Analysis System (IDAS) developed by the Industrial Technology Development Institute (ITDI) and Advanced Science and Technology Institute (ASTI) of the Department of Science and Technology (DOST-Philippines). The goal is to establish a spectral profiling database for Cu oxidation states and validate IDAS's effectiveness in classifying these states using MVA tools. The research ultimately aims to support more reliable and cost-effective quality control processes in semiconductor manufacturing.

II. MATERIALS AND METHOD

A. Sample preparation

A total of 40 leadframe (LF) units were utilized, subdivided into fresh and oxidized groups. Fresh samples

were analyzed as-received, while oxidation was simulated for the remaining samples via heat treatment in an oven at 250 °C for 5.5 hours, replicating conditions similar to those encountered in die attach and wire-bonding steps of semiconductor packaging lines.

B. Surface characterization

To validate oxidation, both control and treated samples underwent surface analysis. Visual and morphological changes due to oxidation were examined using optical microscopy (Carl Zeiss compound microscope), field emission scanning electron microscopy (FE-SEM; FEI Helios Nanolab 600i, 5 kV, 86 pA), and energy dispersive x-ray spectroscopy (EDS; Oxford X-max, 15 kV, 0.69 nA). These techniques provided qualitative and semi-quantitative information on surface texture, color, and elemental composition.

C. AES analysis and parameters optimization

Auger electron spectroscopy (AES) was conducted using a JEOL JAMP-9500F Auger microprobe. Prior to analysis, parameters such as accelerating voltage, beam current, and scan modes were optimized using a bare Cu leadframe. Four parameter sets were evaluated (Table 1) and optimal parameters were selected based on signal-to-noise ratio and clarity of Cu peaks.

D. Method Validation Using Reference Materials

Reference standards of elemental Cu and Cu_2O from Geller Microanalytical Laboratory were used for method validation. Thirty datasets were acquired from multiple analysts with the standards analyzed at five (5) locations using the optimized AES settings. These were used to confirm the system's ability to detect and differentiate oxidation states using AES and to validate IDAS classification outputs.

E. Data processing and IDAS analysis

A total of 200 AES-derived datasets (100 fresh, 100 oxidized) were processed and labeled using a coding system. All the prepared datasets were subjected to multivariate analysis (MVA) using the IDAS software version 1.0 user interface developed by ITDI and ASTI. Data were compiled and uploaded to the IDAS tool. These data were subjected to principal component analysis (PCA) and *k*-means clustering. PCA is a means of data compression by reducing high-dimensional data into low-dimensional component variables [16], enabling visualization of the dataset into two or three dimensions. Meanwhile, the *k*-means clustering algorithm is used in grouping the data points into clusters [17]. MVA and cluster analysis results produced from the system were then collected and evaluated.

III. RESULTS AND DISCUSSION

A. AES parameter optimization

Among the tested settings (Table 1), the combination of 10.0 kV and 10.0 nA provided superior spectral resolution and image quality. As shown in Table 2, this setting enabled clearer elemental peaks, reduced background noise, and well-defined surface morphology under FE-SEM. Validation using fresh and heat-treated samples confirmed its robustness, with Cu-only peaks for the fresh sample and additional O, C, and N peaks for the oxidized counterpart, an indicative of Cu oxidation (see Fig. 1). The optimal settings chosen for all analyses were 10.0 kV, 10.0 nA with 30° tilt, M5 wide scan (1.0 step, 20 ms, 10 sweeps), and M2 split scan (0.5 step, 20 ms, 10 sweeps).

Table 1. Parameters used for AES analysis for spectral acquisition and chemical state analysis

SETTINGS	Parameter 1	Parameter 2	Parameter 3	Parameter 4
FE-SEM magnification	3,000x	3,000x	3,000x	3,000x
Accelerating voltage (kV)	5.0 kV	5.0 kV	10.0 kV	10.0 kV
Beam current (nA)	5.0 nA	10.0 nA	5.0 nA	10.0 nA
Tilting angle (deg.)	30°	30°	30°	30°

B. Visual and morphological changes

Oxidation simulation induced observable color shifts from bright copper to dull brown or blackish tones, consistent with the formation of copper oxides (Fig. 2). Optical microscopy (OM) revealed uniform surfaces in fresh samples, contrasting with discolored, uneven textures in oxidized ones (Fig. 3). FE-SEM images confirmed this, showing blister-like textures and increased surface roughness post-treatment. These morphological features were characteristic of oxide layer growth on metallic surfaces (Fig. 4).

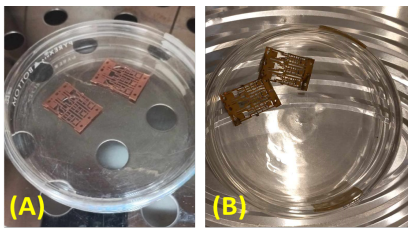
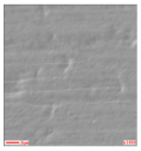
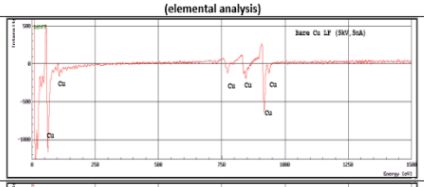
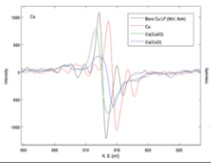
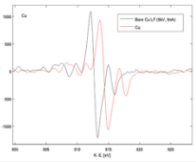
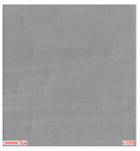
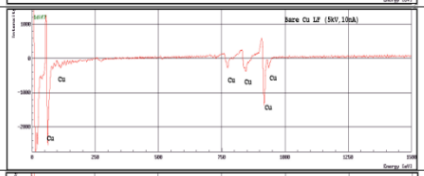
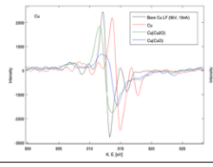
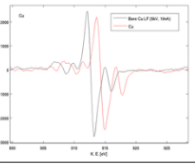
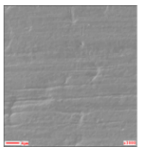
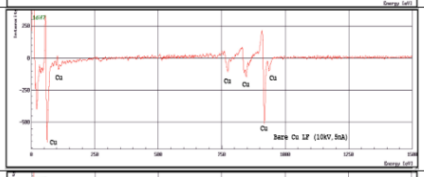
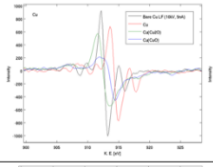
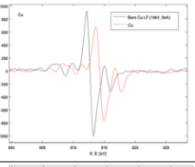
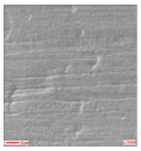
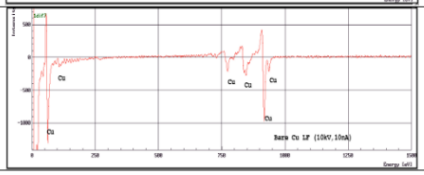
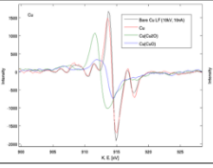
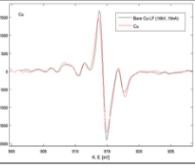


Fig. 2. Cu LF units placed in a glass petri dish: (A) before and (B) after heating in oven (250 °C, 5.5 hours)

The change in color during heat treatment could be due to changes in surface roughness, thickness, and possibly stoichiometry of copper oxide [18].

Table 2. Summary of results of the optimization of AES parameters

ANALYSIS	SEM imaging	Wide scan mode (elemental analysis)	Split scan mode (chemical state analysis)	Split scan mode (elemental Cu peak)
Parameter 1 (5.0 kV, 5.0 nA)				
Parameter 2 (5.0 kV, 10.0 nA)				
Parameter 3 (10.0 kV, 5.0 nA)				
Parameter 4 (10.0 kV, 10.0 nA)				

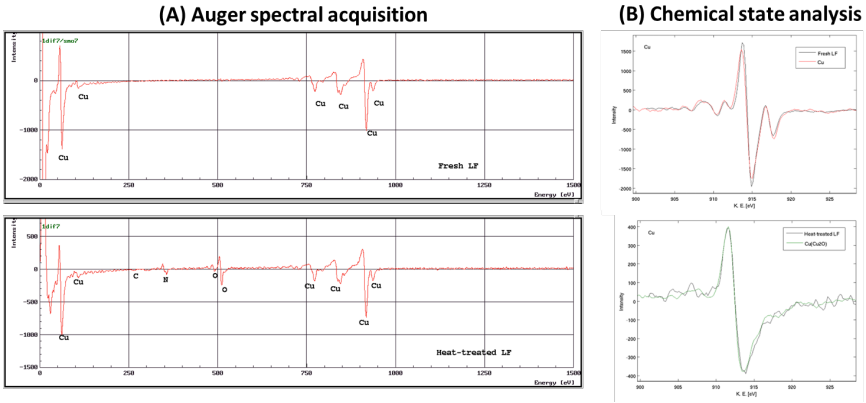


Fig. 1. Results of validation of parameters used for both fresh and heat-treated leadframes: (A) Auger spectral acquisition and (B) Chemical state analysis

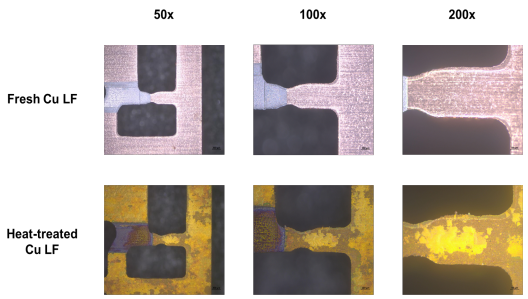


Fig. 3. Cu LF units (A) before and (B) after heating (250 °C, 5.5 hours)

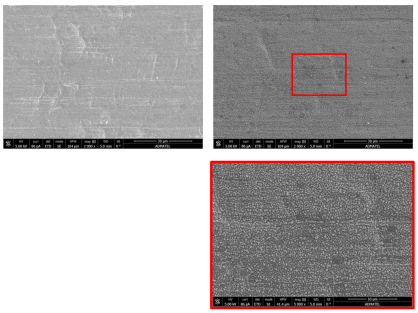


Fig. 4. FE-SEM images of the Cu leadframes before (left) and after (right) oxidation taken at 2,000x and 5,000x magnification

C. Elemental and chemical state analysis

EDS analysis (Figure 5) confirmed increased oxygen content in oxidized samples, alongside minor signals for sulfur, silicon, and chlorine – suggesting incidental surface contamination while on Figure 6, AES provided more insights by identifying specific chemical states. The chemical state analysis showed that Cu in heat-treated samples was primarily in the +1 oxidation state (Cu_2O), while fresh samples retained elemental Cu signatures.

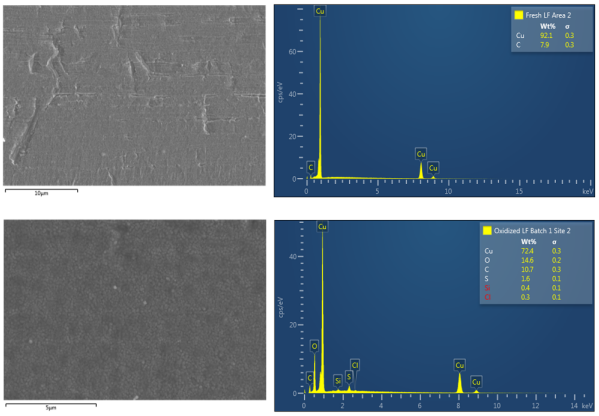


Fig. 5. EDS spectra obtained from the fresh (top) and oxidized (bottom) leadframes at 15 kV accelerating voltage and 0.69 nA beam current

Heat treatment simulation was also performed in one previous study [19] using a simulated aging method exposing leadframes to different temperatures (150 °C to 300 °C) for various time durations to accelerate the aging process. Results showed FE-SEM images of oxidized leadframes possessed visible roughness; more lumps with tiny grains were formed and spread over the whole surface. FTIR spectroscopy was performed to determine the chemical composition of the oxidized leadframes, which revealed distinct cuprous oxide and cupric oxide peaks compared to fresh leadframes with no distinct oxide signals observed [19].

D. Method validation with Cu and Cu_2O standards

Reference spectra for elemental Cu and Cu_2O were used to benchmark IDAS classification performance. PCA of the validation dataset revealed three distinct clusters, with Cu and Cu_2O forming separate groups. The third cluster, consisting of spectra from instrument library, highlighted variations possibly arising from acquisition settings.

Specifically, the three principal components (PC1, PC2 and PC3) accounted for 48.51% of the total variance from the data obtained in the groups. Three clusters (0, 1 and 2) were produced as evaluated through the average silhouette method. In cluster 0 and 1, oxidation state Cu_2O and elemental state Cu have the most influence on variation, with the majority of the Cu_2O spectra in cluster 0 and elemental Cu in cluster 1. On the other hand, only the elemental state Cu belonged to cluster 2. Its distinction may be caused by the nature of this dataset, in which Cu in cluster 2 is an extracted dataset from the library of the equipment. These results show that the IDAS software is able to group the datasets based on their chemical states (Cu, Cu_2O) and origin (equipment) of the data.

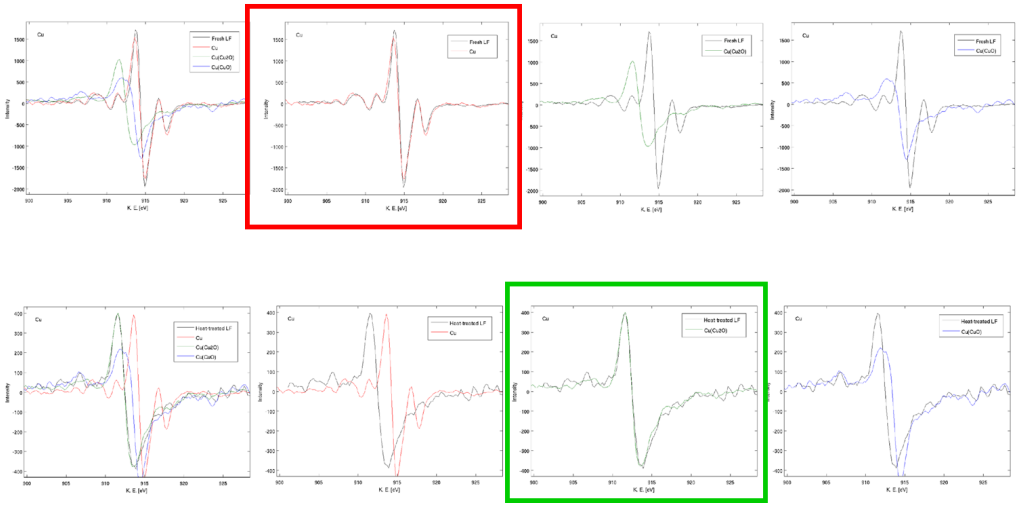


Fig. 6. Chemical state analysis (Cu) of fresh LF unit and heat-treated LF unit; best peak fit for fresh LF – elemental Cu (red box), best peak fit for heat-treated LF – oxidized Cu+ (Cu_2O) (green box)

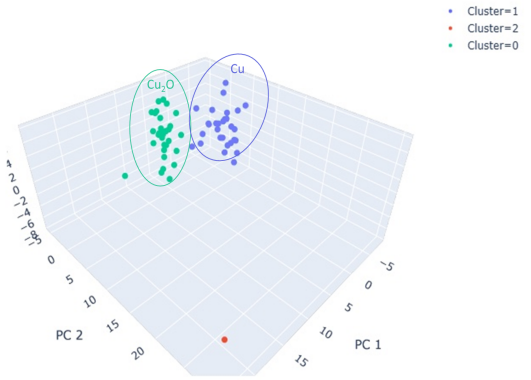


Fig. 7. 3D plot of the method validation data

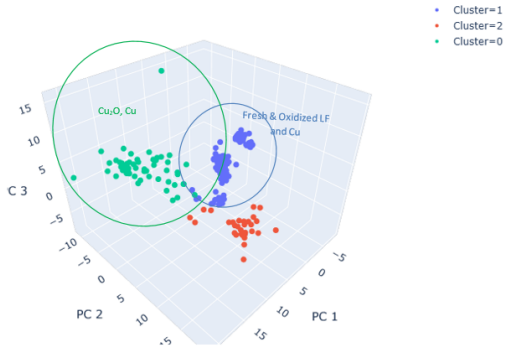


Fig. 8. 3D plot of the obtained data from actual leadframe strips

E. Multivariate Analysis Using IDAS

Based on the PCA result (Figure 8), PC1 (42.22%) captured the most variation while PC2 (25.06%) and PC3 (6.94%) captured the second most variations. Three clusters were obtained from the PC plot.

For cluster 0, Cu_2O from the reference material has the most influence on variation. All data generated from the Cu_2O reference material are found in this cluster. Interestingly, there are no spectra from oxidized LFs in cluster 0.

On one note, both fresh and oxidized Cu LF datasets have the most influence on variation in cluster 1. Most of the spectra from oxidized LFs are found in this cluster. The two overlapping groups in the PC plot (Cluster 0 and 1) may be linked by the presence of elemental state Cu in both clusters. The presence of Cu_2O was evidently observed in oxidized LFs, while some fresh LF samples manifested its presence. The Cu_2O detected in oxidized LFs clearly confirmed the oxidation of the samples during simulation. For the fresh LFs, one possible reason why Cu_2O did manifest in their respective spectra may be attributed to the nature of the samples – these dummy samples are not newly acquired and had long been stored in the laboratory. Leadframe surface oxidation at ambient temperature may have occurred across a period [20].

It is also notable that the extracted datasets of Cu and Cu_2O from the library of the equipment are mostly evident in Cluster 2. Again, these results manifest that the chemical states (Cu, Cu_2O) and origin (equipment) of the data largely contribute to the distinction of the samples.

IV. CONCLUSION

This study demonstrated that the IDAS tool can reliably classify oxidation states in copper leadframes. The use of PCA and clustering methods enabled robust differentiation between elemental and oxidized states, validating the effectiveness of AES-IDAS integration for real-time monitoring in semiconductor applications.

The integration of AES with IDAS has proven to be a powerful approach for identifying and classifying oxidation states in copper leadframes. Optimized AES settings provided high-quality data that enabled PCA and k-means clustering to distinguish between elemental Cu and Cu_2O with high accuracy. These findings support the use of this methodology for improving quality assurance in semiconductor manufacturing, especially in addressing failure modes such as NSOL and delamination due to oxidation.

Future work should include the use of a broader range of Cu alloy types and sources to expand the robustness of the IDAS classification library. Depth profiling using AES and time-resolved oxidation studies could further enhance understanding of oxidation kinetics. Integrating machine learning techniques into IDAS may improve classification accuracy. Real-time oxidation monitoring in production settings and linking oxidation states with reliability test data such as thermal cycling would strengthen the practical implications of this method.

V. ACKNOWLEDGMENT

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