

INTRODUCTION

Metallized film capacitors are used in a wide variety of applications such as telecommunications, industrial electronics or automotive, among others. They are composed of a micrometric polymer film (usually polypropylene) and a nanometric metallized layer. Metallization layers are typically composed of aluminium, with a thickness ranging between 5 and 100 nm. Despite their relatively easy manufacture and performance, some failures might occur during the production, testing or service life of these capacitors. These failures are related to the variations of the chemical composition within the metallization layer, moisture ingress, inhomogeneous metallic thicknesses or demetallization processes.

The combination of dynamic SIMS depth profiling and kinetic energy distribution analysis has allowed to analyze this sort of metallized film capacitors (d-SIMS) and the excellent repeatability and sensibility of the technique allows studying the degradative process in the capacitor films and the accurate location of the metal-polymer interface. These factors affect drastically to the capacitor performance and they will determine the potential failures that could be developed during de capacitor life cycle.

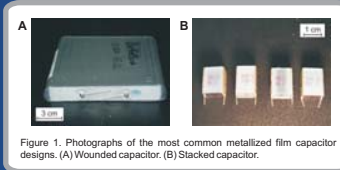


Figure 1. Photographs of the most common metallized film capacitor designs. (A) Wounded capacitor. (B) Stacked capacitor.

ENERGY-RESOLVED ANALYSIS

In Al-metallized polymer films, several isobaric interferences occurred for ions coming from the different layers involved (e.g. $^{27}\text{Al}^+$ or $^{43}\text{AlO}^+$ from the metallized layer and $^{27}\text{C}_2\text{H}_5^+$ or $^{43}\text{C}_2\text{H}_5^+$ from

Based on the different kinetic energy distributions (KEDs) of the isobaric ions (Figure 3) an energy-resolved depth profiling analysis was performed in order to discriminate the metal-polymer interface. The critical importance of the KEDs in depth-resolved analysis is well-illustrated in Figure 4.

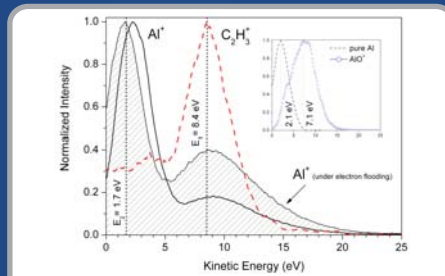


Figure 3. Kinetic Energy Distributions (KEDs) of isobaric species at m/z 27. Solid line: $^{27}\text{Al}^+$ in the metallic layer; Hatched area: $^{27}\text{Al}^+$ in the metallic layer (under electron flooding); Dashed line: $^{27}\text{C}_2\text{H}_5^+$ in the polymer film (under electron flooding). Inset: Solid line: $^{27}\text{Al}^+$ in a pure aluminium standard; Circles: $^{43}\text{AlO}^+$ in the metallic layer

When the KE of the ions coming into the quadrupole is set to 1.7 eV, the metal-polymer interface is resolved. The analysis performed at 8.4 eV shows a non-resolved depth profile, with contributions of the $^{27}\text{Al}^+$ ions from the metallic layer and $^{27}\text{C}_2\text{H}_5^+$ ions from the polymer film. The unresolved depth profile corresponds to oxidized Al regions within the metallized layer. This oxidized layers are formed in the presence of oxygen and moisture. The profile obtained for m/z 43 -corresponding to $^{43}\text{AlO}^+$ ions from the metal layer- confirms the double-oxidized metallic layer structure (Figure 5).

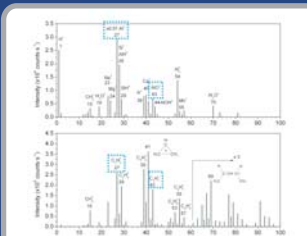


Figure 2. Positive ion mass spectra of the Al layer (top) and the polypropylene film (bottom).

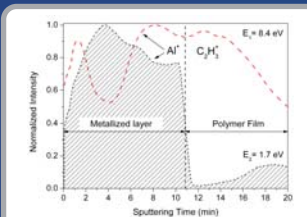


Figure 4. Positive ion depth profiles of a 100-nm Al-metallized film capacitor for m/z 27 ions with a kinetic energy of 8.4 eV and 1.7 eV.

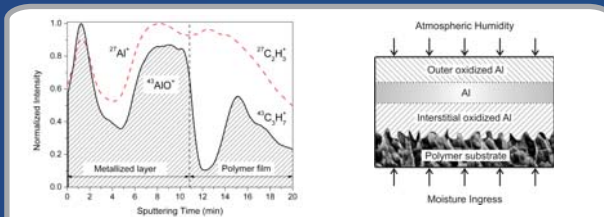


Figure 5. Depth profiles obtained with a kinetic energy filtering of 8.4 eV for $^{27}\text{Al}^+$, $^{43}\text{AlO}^+$, and $^{43}\text{C}_2\text{H}_5^+$ ions. The metallized film exhibits two oxidized region in direct contact with moisture and oxygen, as shown in the diagram.

EXPERIMENTAL CONDITIONS

Positive secondary ion depth profiling was performed using a 5 keV Ar^+ primary ion beam with a current of 100 nA. Despite its lower ion yield, Ar^+ has been used as primary ion keeping unaltered the oxidation state of the surface, in order to monitor possible oxidation processes along the sample thickness. The sample was sputtered at a 45° incidence angle and the emitted secondary ions were analyzed by means of a triple stage quadrupole analyzer, situated at the surface normal.

A 45° electrostatic energy analyzer (EEA) placed prior to the mass filter allows energy-resolved selection of the secondary ions coming into the quadrupole. The primary ion was digitally rastered over an area of $650 \times 790 \text{ m}^2$, collecting the signal from the 20% central region in order to prevent crater edge effects. Under these conditions, the sample is experiencing an ion dose of 4×10^{13} ions/cm².

Surface charging effects appear as the polymer film is being exposed by the primary beam sputtering. In order to minimize those effects, the sample was grounded to the sample holder, assuring direct contact with the conductive metallized (Figure 7). Additionally, the sample surface is flooded with low energy electrons (500 eV) to compensate the accumulated charge. The synergistic effect of electron flooding and the metallization layer improves the sensitivity of the analysis while reducing the thermal-induced sample degassing (Figure 8).



Figure 6. The Hidden SIMS Workstation

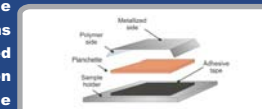


Figure 7. Schematic diagram of the sample assembly assuring grounded conditions

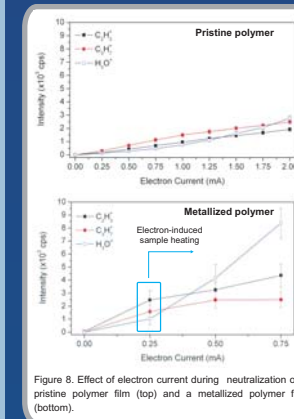


Figure 8. Effect of electron current during neutralization of a pristine polymer film (top) and a metallized polymer film (bottom).

METHOD REPEATABILITY

In order to apply the analysis method as a quality control tool in the capacitor industry, it must produce reliable and reproducible results, as they will be related to thickness differences between the nanometric metallized layers for each capacitor and/or to demetallization processes.

Two different repeatability evaluations were performed to determine the origin of the analysis variability. Firstly, the method variability was studied by analyzing nearby profiles, where the composition and thickness of the metallic layer is quite homogeneous. Secondly, the variability between far profiles - within an area of 2 cm² - was measured to evaluate the specimen heterogeneity.

Excellent results were obtained for the sputtering times, related to the metal-polymer interface location, with a relative standard deviation (RSD) better than 1.50 % for nearby regions. The comparison between remote profiles indicates that the sample is quite homogeneous, without significant difference in the metallic layer thickness across the film surface.

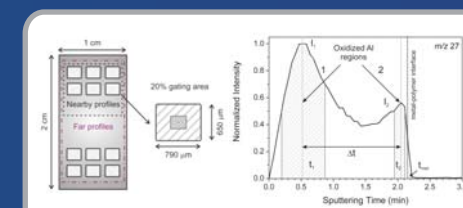


Figure 9. Typical $^{27}\text{Al}^+$ depth profile in metallized film capacitors shows two oxidized regions: (1) uppermost oxidized region (2) interstitial oxidized region

Nearby profiles				Far profiles			
Time variables (min)							
Mean	SD	RSD (%)	Mean	SD	RSD (%)	Mean	RSD (%)
t_1	0.526	0.056	10.75	0.514	0.047	9.19	
t_2	2.070	0.033	1.58	2.074	0.031	1.49	
Δt	1.545	0.065	4.23	1.560	0.064	4.13	
t_{tot}	2.240	0.0326	1.46	2.221	0.040	1.80	
Intensity variables ($\times 10^9$ cps)							
Mean	SD	RSD (%)	Mean	SD	RSD (%)	Mean	RSD (%)
I_1	12.28	1.030	8.39	14.12	2.688	19.04	
I_2	6.906	0.756	10.81	8.105	1.613	19.90	

The parameters for repeatability assessment are defined in terms of intensity variables (I_1 , I_2) and sputtering time variables (t_1 , t_2)

ACKNOWLEDGEMENTS

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REFERENCES

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