EAG Evans Analytical Group

# AN 447

## Cause and Location of Delamination of Multi-layer Metal Foil by XPS

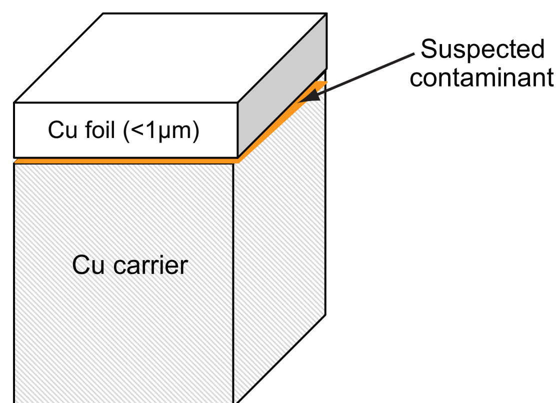
May 19, 2008 (Version 1.0)

### Discussion

Determining the locus of failure in multilayer laminates is often the first step in understanding the cause of failure. In many cases a fraction of a monolayer of contamination is enough to weaken a bond to the point of failure. In this example we examine mating faces from a failed Cu-laminate (see schematic below). X-ray photoelectron spectroscopy (XPS/ESCA) is used because of its surface sensitivity and ability to quantitatively identify not only the elements present but also determine their chemical or oxidation state.

As expected, copper was detected on both sides of the failed interface. Significant quantities of Ni°, $Ni^{2+}$, Mo°, $Mo^{4+}$, $Mo^{6+}$ and $PO_4^{3-}$ were detected on both sides of the failed interface by XPS. Carbon was also detected, but likely came from adsorption of organic species during the brief air exposure.

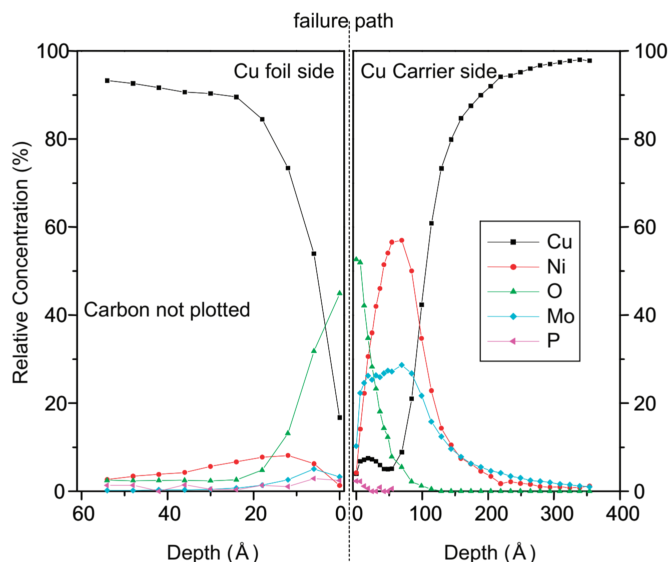
To probe the thickness of the Ni, Mo and P containing layers, the surfaces were depth profiled using a focused beam of argon ions. The resulting profiles reveal a roughly 10 Å phosphate layer on both sides of the failed interface. The P concentration is relatively low (2.5%); however, it is determined to be present as $PO_4^{3-}$, meaning the total amount of phosphate at the interface is >5X the P concentration. The cation associated with the phosphate is likely either $Cu^{+}$, $Mo^{x+}$ or possibly $H^{+}$ (hydrogen is not detected by XPS). Beneath the phosphate layer on the Carrier side there is ~100Å $Ni_2Mo$ layer. A small amount of Ni was detected on the mating foil surface.

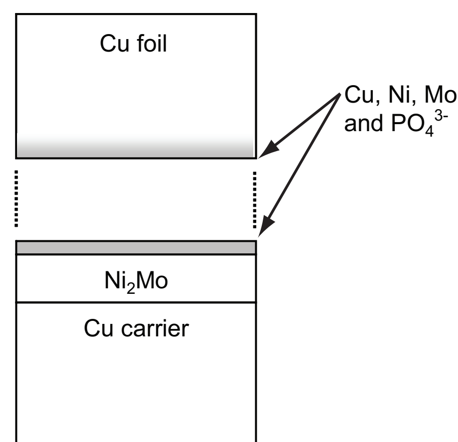



XPS was used to determine that adhesive failure occurred at a $Ni_2Mo$-Cu interface as a result of a high concentration of phosphate (see schematic in lower right).

Table 1. Concentration of Elements Detected on Failed Surfaces (in atom%)

| Sample | C | O | P | S | Cl | Ni | Cu | Mo |
|---|---|---|---|---|---|---|---|---|
| Cu thin foil side | 28.8 | 34.5 | 2.5 | 0.5 | 0.9 | 8.9 | 20.2 | 3.7 |
| Cu carrier side | 29.4 | 42.7 | 2.6 | 0.8 | 0.1 | 10.5 | 3.1 | 10.9 |

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