Technical Note

Gold Star Mothers Stamp
Analysis Using Scanning Electron Microscopy and Mass Spectrometry

Introduction:

Since 1949, the JEOL legacy has been one of outstanding innovation in developing instruments used to advance scientific research and technology. JEOL has over 60 years of expertise in the field of electron microscopy, more than 50 years in mass spectrometry.

In this applications note, we performed an analysis of a *Gold Star Mothers Postage Stamp* by using three JEOL instruments. We used the JSM-IT300LV which the latest addition to JEOL's popular series of analytical low vacuum SEM, , the JMS-T100LP "AccuTOF- DART" the first commercially available ambient ionization mass spectrometer, and the JMS-S3000 "SpiralTOF" which has highest mass-resolution and mass accuracy of all commercially available MALDI-TOFMS systems. We can therefore correlate analyses from various analytical techniques on the same sample.

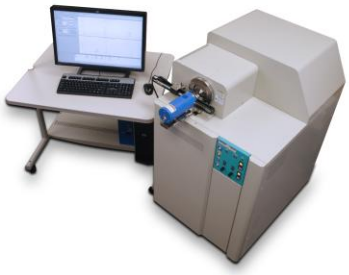
Figure 1. A Gold Star Mothers Stamp from 1948. It is almost the same age as JEOL

Experimental:

A *Gold Star Mothers Stamp* from 1948 has a lead chromate pigment that we analyzed via: the JSM-IT300LV with an EDS spectrometer, the JMS-T100LP "AccuTOF DART" and the JMS-S3000 "SpiralTOF".

Filament	Pre-centered W hairpin filament (with continuous auto bias)
Resolution	High Vacuum mode: 3.0 nm (30kV), 8nm (3kV), 15nm (1kV) Low Vacuum mode: 4.0 nm (30kV)
Accelerating Voltage	300 V to 30 kV
Magnification	x5 to 300,000 (printed as a 128mm x 96mm micrograph)
LV Detector	Multi-segment BSED (std.) LV-SED (option)
LV Pressure	10 to 650 Pa
Max. Specimen Size	Observation: 178mm diameter, Maximum loadable: 300mm, Height: 80mm
Specimen Stage	5 axis motor control with asynchronous movement Eucentric goniometer X=125mm, Y=100mm, Z=5 to 80mm T= -10 to 90°, R=360° (endless)
Stage Navigation System	Embedded Color CCD Camera for Sample Navigation
Frame Store	Up to 5120×3840 pixels
PC	Desktop, Windows 7
Vacuum mode changeover	Automatic (PC interface controlled)

Figure 2. JSM-IT300LV SEM with basic specifications



Mass resolution	R>6,000 (m/z 609)
Mass accuracy	3 ppm (r.m.s.)
Sensitivity	S/N > 10 (Reserpine 10pg, ESI+ mode)
Mass range	Up to *m/z* 10,000
Ionization mode	*DART, ESI, APCI, Paper spray*

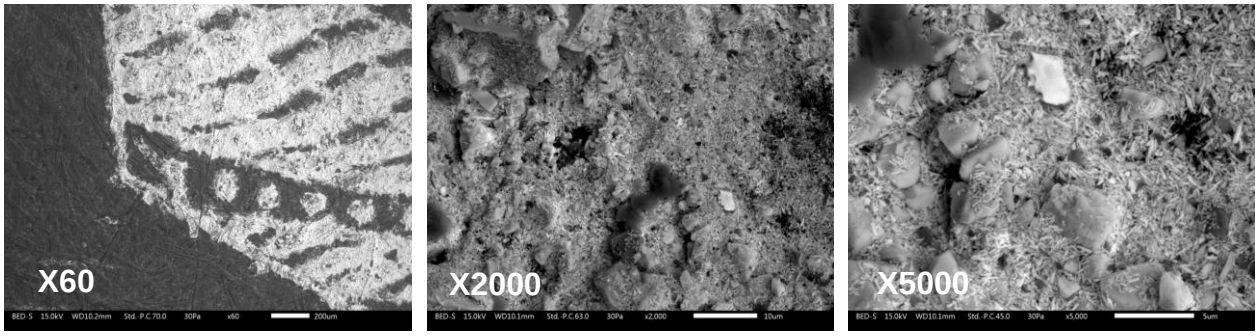
Figure 3. AccuTOF-DART Mass Spec with basic specifications

Mass Resolution	R>75,000 (*m/z* 2465.2)
Mass accuracy (internal calib.)	1 ppm (average error)
Mass accuracy (external calib.)	10 ppm (average error)
Sensitivity	S/N > 50 (AgiontensilII 500 amol)
Mass range	Up to *m/z* 30,000 (Spiral TOF mode) Up to *m/z* 500,000 (Linear TOF mode)
Ionization mode	*MALDI, LDI*

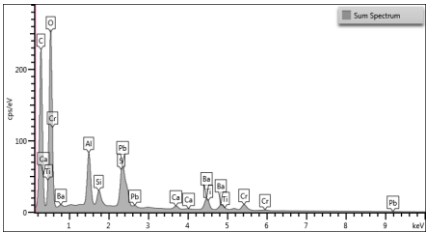
Figure 4. SpiralTOF Mass Spec with basic specifications

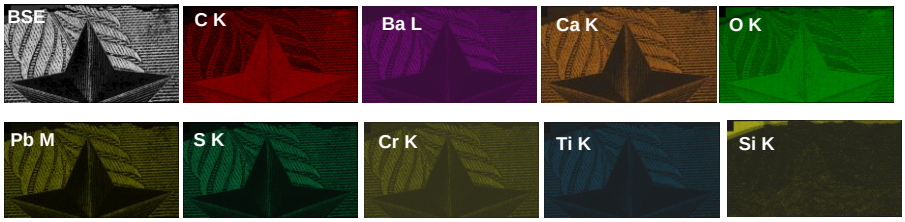
Scanning Electron Microscopy Data:

Backscattered Electron Images at 15kV in low vacuum mode, with no destructive sample prep required. In BSE the contrast is created from a difference in mean atomic number, with high Z number materials bright and low atomic number materials dark.



Energy Dispersive X-ray Spectrum at 15kV in low vacuum mode showing all elements present and elemental X-ray Maps showing the "positions" of each element.

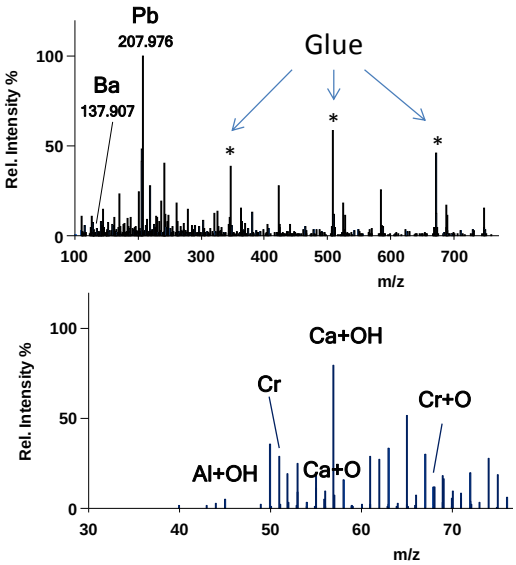




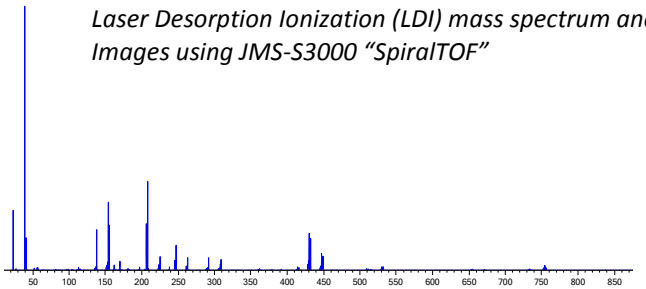
Mass Spectrometry Data:

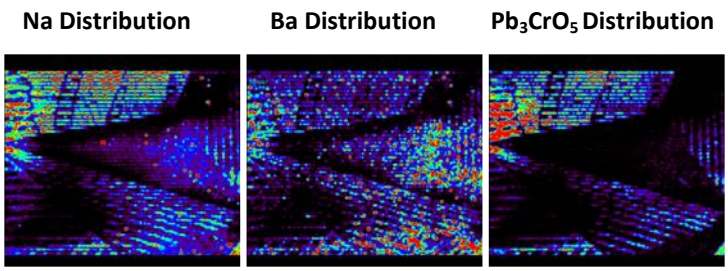
A paper triangle cut from the stamp was wetted with dilute HNO_3 and biased to 3000 V to induce paper spray. LDI imaging with the SpiralTOF showed the distribution of both elemental species and characteristic cluster ions.

Paper Spray Ionization [1] mass spectrum using JMS-T100LP "AccuTOF DART"



Laser Desorption Ionization (LDI) mass spectrum and LDI Images using JMS-S3000 "SpiralTOF"





[1] R. B. Cody and A. J. Dane,
Rapid Communications in Mass Spectrometry, *2014, in press*