# TN 102
# EAGLABS℠ Glow Discharge Mass Spectrometry (GDMS) Services

## Technique Note

October 13, 2008 (Version 1.0)

*GDMS provides excellent detection limits for bulk trace level impurities in solid samples*

## Introduction

Glow-Discharge Mass Spectrometry (GDMS) is a technique that is capable of providing trace-level elemental quantification for a wide range of solid and powder materials. Applications include high purity materials, sputter targets, aerospace materials, refractory metals and alloys, rare earth metals and oxides, precious metals, and solar cell materials. Excellent detection limits (ppm to ppb), nearly matrix- independent quantification, and a wide dynamic range make GDMS a technique of choice for direct analysis of solid materials. GDMS can also be utilized for sputter depth profiling to characterize thick layers and coatings (0.5 - 50µm). The technique can accommodate conducting, semi-conducting and insulating samples.

## Principles

In Glow-Discharge Mass Spectrometry (GDMS), the sample to be analyzed forms the cathode in a gas discharge or plasma. Argon is typically used as the discharge gas. Argon positive ions are accelerated towards the cathode (sample) surface with energies from hundreds to thousands of eV resulting in erosion and atomization of the upper atom layers of the sample. The sputtered species leave the cathode surface and are transported into the plasma where they are ionized. The atomization and ionization processes are separated in space and time, which is a keystone for simplified calibration and nearly matrix-independent quantification. The ions are then extracted into a mass spectrometer where they are separated according to their mass to charge ratio.

## Common Applications

The ability to provide elemental composition from matrix levels down to ultra-trace levels (sub-ppb) directly from the solid state without the need for dissolution makes GDMS an ideal technique for the following types of applications:

- Quantitative measurement of trace and ultra-trace contaminants in bulk
  - high purity metals
  - alloys
  - manufactured carbons and graphites
  - semiconductor materials
  - oxides, carbides and ceramics
- Depth profiling for major, minor and trace elements
- Identification of unknown species from a minute amount of material
- Full elemental surveys of powders or particulate materials

## Strengths

- Survey technique
- ppm to sub-ppb detection limits
- Not sensitive to matrix, allowing for easy quantification
- Can depth profile through thick layers
- Can analyze conductors and insulators

## Limitations

- ~1 µm depth resolution during depth profiling
- Samples must be vacuum compatible
- Smallest analytical area ~1 cm in diameter

## Technique Comparisons

GDMS provides trace-level quantitative surveys of bulk samples and thick films. Other tools with similar or complementary analytical capabilities include Inductively Coupled Plasma Optical Emission (ICP-OES) or Mass Spectrometry (ICP-MS), X-Ray Fluorescence (XRF), Instrumental Gas Analysis (IGA) and Secondary Ion Mass Spectrometry (SIMS). The ICP techniques are solution based. GDMS has a much larger dynamic range and better detection limits than the ICP techniques and does not require sample dissolution. XRF can also be used as a survey technique and offers the advantages of high precision, higher spatial resolution (down to 100µm), and being nondestructive. However, XRF has much poorer detection limits (~50ppm) and it can suffer from spectral interferences that limit the elements that can be analyzed. SIMS is also a technique that can achieve ppm to ppb detection limits. It is complementary to GDMS in that it can achieve excellent detection limits for atmospheric species (C, O, and H) for which GDMS has higher background signal limitations. GDMS has greatly reduced matrix affects, however, SIMS has much better depth resolution. IGA is complementary to GDMS for atmospheric species (H, C, N, O, S).

## GDMS at Evans Analytical Group

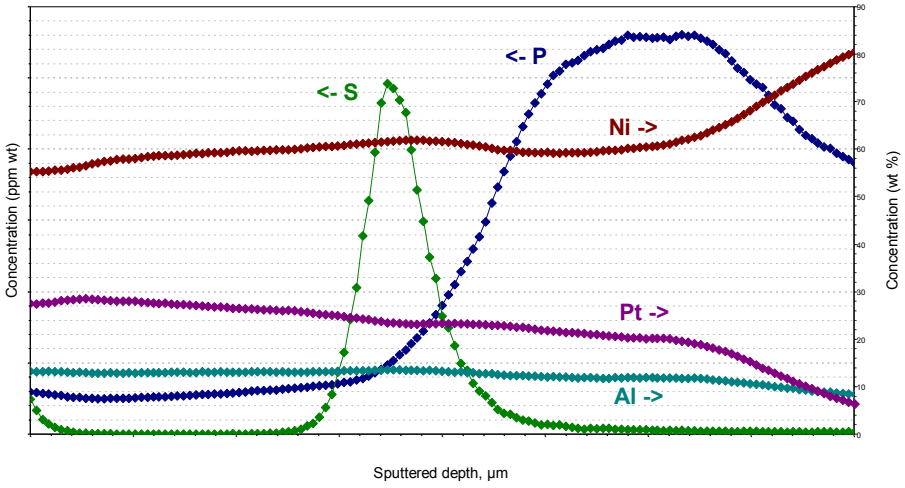
Evans Analytical Group® (EAG) offers the best detection sensitivity along with accurate mass fraction determinations in bulk solids and thick coatings. We have sixteen (16) GDMS instruments worldwide and highly qualified scientists familiar with a wide range of applications from production, quality assurance, failure analysis, and R&D environments. EAG's GDMS instruments consist of a balanced mix of low pressure type and fast flow type instruments, allowing us to cover a wide range of applications.

## Typical Data

Upgraded Metallurgical grade Silicon for Photovotaics

| Element | ppm wt | Element | ppm wt | Element | ppm wt | Element | ppm wt |
|---|---|---|---|---|---|---|---|
| Li | < 0.001 | Co | < 0.005 | Cd | < 0.05 | Tm | < 0.005 |
| Be | < 0.001 | Ni | < 0.01 | In | < 0.01 | Yb | < 0.005 |
| B | 1.9 | Cu | < 0.01 | Sn | < 0.01 | Lu | < 0.005 |
| F | < 1 | Zn | < 0.05 | Sb | < 0.01 | Hf | < 0.01 |
| Na | < 0.01 | Ga | < 0.05 | Te | < 0.01 | Ta | Source |
| Mg | < 0.005 | Ge | 2.4 | I | < 0.01 | W | < 0.05 |
| Al | < 0.01 | As | < 0.05 | Cs | < 0.001 | Re | < 0.01 |
| Si | Matrix | Se | < 0.01 | Ba | < 0.01 | Os | < 0.01 |
| P | 11 | Br | < 0.01 | La | < 0.01 | Ir | < 0.01 |
| S | < 0.1 | Rb | < 0.01 | Ce | < 0.005 | Pt | < 0.01 |
| Cl | < 0.05 | Sr | < 0.01 | Pr | < 0.005 | Au | < 0.1 |
| K | < 0.05 | Y | < 0.01 | Nd | < 0.005 | Hg | < 0.01 |
| Ca | < 0.05 | Zr | < 0.01 | Sm | < 0.005 | Tl | < 0.01 |
| Sc | < 0.001 | Nb | < 0.01 | Eu | < 0.005 | Pb | < 0.01 |
| Ti | < 0.005 | Mo | < 0.05 | Gd | < 0.005 | Bi | < 0.01 |
| V | < 0.005 | Ru | < 0.01 | Tb | < 0.005 | Th | < 0.005 |
| Cr | < 0.01 | Rh | < 0.01 | Dy | < 0.005 | U | < 0.005 |
| Mn | < 0.005 | Pd | < 0.01 | Ho | < 0.005 | | |
| Fe | < 0.05 | Ag | < 0.01 | Er | < 0.005 | | |

GDMS depth profile of a Ni-Pt-Al coating
on a Ni super-alloy substrate




GDMS sputter depth profiles
provide concentration and impurity information with depth