



GELLER MICROANALYTICAL LABORATORY, Inc.

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Our 25th Year!

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Do it right... the first time!

GELLER MICROANALYTICAL LABORATORY

Geller MicroAnalytical Laboratory, Inc. is certified to ANSI Z540A, ISO-9001 and 17025 providing analytical services and design and manufacturing of a limited number of unique products that are directly related to microanalysis. Some of these products, such as our magnification reference standard and ion sputter standards are NIST and NPL (National Physical Laboratory in the U.K.) traceable.

Our staff takes pride in performing state-of-the-art analyses on difficult specimens. As our satisfied repeat clients and publications reveal, we have developed several techniques for solving problems.

Rest assured your confidentiality will be maintained with the utmost care as our small company concentrates on satisfying your needs. We are only a phone call away. Remember, delaying an analysis is usually more costly than waiting for a problem to go away. History does have a way of repeating itself.

COMPANY BACKGROUND:

Geller MicroAnalytical Laboratory was started in 1985 by Joe Geller. He has been working with electron beam instrumentation since 1969 when he originated one of the first SEM services in the United States. In 1973 he joined JEOL (USA), inc., being responsible for demonstrations and technical sales support for that company's SEMs and electron microprobes. In 1979 he was promoted to computer systems group and surface analysis instrumentation manager. His responsibilities included demonstrations and technical sales. Mr. Geller (B.S. and M.B.A.) has made hundreds of presentations, has approximately 35 technical papers in various refereed journals (including MAS, SEM Inc., Journal of Vacuum Science & Tech., Surface & Interface

Analysis, Journal of Applied Physics, and the Journal of Applied Optics), authored a chapter in "Surface analysis by Auger and X-ray Photoelectron Spectroscopy", holds three patents, and is an active member of several technical societies. He is the U.S. Delegation Leader for ISO-TC201 (surface analysis) and an expert for ISO-TC202 on microbeam analysis. He is a Fellow of the American Vacuum Society, the 2nd Vice-Chairman of the E-42 Surface Analysis committee and was the chairman (2000) of the ASSD Division of the American Vacuum Society.

Paul Engle, formerly of Digital Equipment Company, our QC manager also manages our computer development and metallographic laboratory support areas. His attention to detail and creativity in preparing difficult specimens often make the difference in successful sample characterization.

Dr. Menghua (Marty) Liu, Research Scientist, operates the electron probe (EPMA) as a tool for research. He was an adjunct professor and the former director of the EPMA lab at the U. Western Ontario. He has expertise in synthesizing alloys, sulfides and silicates. With experience in mineral, mineral deposits, mineral processing, platinum group element geochemistry and meteorite research (during a Postdoctoral fellowship at the U. Hawaii) he adds yet another dimension to our capabilities and your analyses.

Charles (Chuck) Herrington continues as a consultant. He is further developing our EPMA software and provides customer support and installations.

PRODUCTS

Many companies, such as ours, are facing requirements from their QC departments to calibrate analytical instruments to satisfy the terms of QS and ISO-9000 and ISO-17025 (which replaced Guide 25). Before these requirements came into being we were developing standards for our own needs to give our customers the most accurate analyses possible. We now offer these unique items to the scientific community.

ANALYTICAL STANDARDS FOR MICRO-ANALYSIS:

Calibration standards suitable for energy and wavelength dispersive x-ray analysis, Auger electron and XPS (small beam) spectroscopy, and other techniques. Choose from over 280 high purity elements, compounds, minerals, glasses and alloys.

These ultra high vacuum compatible standards are all 3mm in diameter, are individually polished to prevent cross-contamination, and can be mounted either in a laser engraved SS304 holder or left unmounted. Assortments of standards are offered in

6, 9, 12, 15, 18, and 37 element sizes. We can also prepare your own materials to fit into the holders. Our long list of standards has been developed by listening to our customer's needs- as well as our own requirements in the service laboratory

VACU-STORR:

A series of high vacuum desiccators that pump down readily to the 10^{-5} torr range. These were originally developed by us to protect the Geller MicroAnalytical Laboratory standards from oxidation and contamination. We offer a range of sizes in glass from 2" to 4" O.D. Stainless steel models are available in larger sizes. All Vacu-Storr's have holding times of approximately 5 years.

MAGNIFICATION REFERENCE STANDARDS:

The **MRS-3, 4 and 5** magnification reference standards are a NIST/NPL traceable device which allows for the accurate calibration of optical (transmission and reflected light), scanning electron microscopes (in the secondary and backscattered electron imaging modes), and scanning probe instruments such as Scanning Tunneling and Atomic Force microscopes. The magnification range is from ~10X to 1,000,000X. The copyrighted pattern consists of a series of nested squares with pitches of 0.08, 0.1, 0.5, 1, 2, 50, and 500 μ m. The uncertainties of the finer pitches are 0.002 μ m. A 1 μ m X 6mm micro ruler is also included in the MRS-4. Pattern height is a NIST traceable 0.1 μ m. Squares are used such that magnification can be calibrated in two directions. Pincushioning, barreling, skewness, and the accuracy of tilt angles can also be measured. The non-charging, yet optically transparent pattern offers high contrast and resistance to electron beam damage. New patterns also confirm particle size counting system accuracy. The **MRS-5** has pitch patterns from 2 μ m to 80nm with an uncertainty less than 3nm. Recently available is the NPL traceable **Micro-Ruler MR-1**, which has a 150mm scale with 0.01mm increments.

METALLOGRAPHIC EQUIPMENT:

The **Counter-Rota-Cutter** (US patent #4,949, 605), a metallographic saw attachment, attaches to the Buehler ISOMET, LECO VC-50 or the Struer's

Minitom. Again, a result of our analytical needs to section materials faster (up to 10X), smoother ($\frac{1}{4}$ the surface roughness), and thinner (6 mils over 1" dia.) than available technology offers. A new hollow chuck for accommodating specimens of any length is now available for any of the above saws.

COMPUTER CONTROL SYSTEMS:

dPict7 (Windows) is a PC based digital imaging system for analog SEMs, electron probe microanalyzers (EPMA) and Auger electron (AES) spectrometers. **dQuant7** is a replacement system for the Tracor Northern (NORAN) 5500, DEC (Digital Equipment Co.) based energy-dispersive/ (wavelength dispersive spectrometer) control system. **dSpec7** is a "PAC", "Sesame", and "Lima" system for control the spectrometer and stage motors and crystal flipping control. **AugerII** is a replacement automation system for the DEC based JEOL/KeveX Jamp-10(S) and 30 or other manufacturer's AES automation systems. Both programs offer a state-of-the-art, user-friendly approach to the complicated task of data collection and reduction for EPMA and AES data. The Auger program reads the international standard VAMAS data.

ION SPUTTERING STANDARDS:

For calibrating ion sputtering systems on surface analysis equipment we offer a range of thickness of thin films of SiO₂ and Si₃N₄ on Si and Ta₂O₅ on Ta. The NIST traceable **NiCr-3** thin film stack of alternating layers of Ni and Cr (~500 \AA X 12 layers) provides the analysts with an excellent specimen to test depth resolution and procedures.

SPECIMEN HOLDERS:

For JEOL[©] scanning electron microscopes we offer top referencing specimens holders that accommodate multiple 1/4" specimens.

LABORATORY EQUIPMENT

AUGER MICROPROBE:

JEOL Jamp-7800, fully automated

- SEM resolution: 80 \AA , Auger spot size: 300 \AA
- 0.1% atomic sensitivity, Vacuum: 10^{-10} torr
- Digital imaging: SEI, BEI, and Auger

- X-ray analysis: integral Si(Li) detector, low Z
- Depth profiling; stationary and rotational
- Integral residual gas analyzer

ELECTRON MICROPROBE:

JEOL Superprobe 8600, fully automated

- 1 energy + 4 wavelength dispersive x-ray spectrometers.

- Quantitative analysis Sensitivity to 10ppm
- Digital image analysis & particle size counting

**SCANNING ELECTRON MICROSCOPE:
JEOL JSM-6300 & JSM-6300F (FEG)**

- SEM Resolution: very high
- Low Z energy dispersive x-ray spectrometer
- Digital imaging

OTHER EQUIPMENT:

- Metallography laboratory/mounting & polishing
- Leitz Ergolux and several stereo microscopes
- Mounting & polishing, many unique capabilities
- Microhardness- Zeiss light load, μm sized areas
- Profilometer- Dektak 3030, 1nm sensitivity
- Vacuum deposition, plasma cleaners, residual gas analysis & machine shop

ANALYTICAL APPLICATIONS

SURFACE ANALYSIS OR X-RAY ANALYSIS?

Electron beam excited x-ray analysis has a sampling depth is generally greater than $\frac{1}{2}\mu\text{m}$. The EDS detector collects a spectrum quickly in micrometer-sized areas but is limited in both its sensitivity (0.1 wt%) and its elemental detectability range. The WDS detectors have the same depth and area sensitivity as EDS but have an extended sensitivity range ($Z>4$), much better detection limits (to 10ppm) and far superior wavelength/energy resolution.

The Auger electron microprobe is useful for analyzing thin films and determining surface composition. When combined with an ion-sputtering source we can "depth profile". This allows us to study the sub-surface chemistry. Detectability is for elements $Z>2$ and with sensitivity to 0.1 % atomic. Complementing Auger spectroscopy is a integral Si(Li) x-ray detector ($Z>4$). It is our "crystal ball" looking 1000X farther into the specimen. The instrument operates in ultra high vacuum (10^{-10} torr). With its pre-pump chamber specimens are taken from atmosphere to 10^{-9} torr in 5 minutes!

A small list of materials analysis investigations we have expertise follows:

SEMICONDUCTORS:

- Bond pad analysis to determine oxide thickness and cause of discoloration and poor bondability
- Bond wire analysis to determine presence of inclusions
- Pinhole, void and defect analysis
- IC metallization scheme determinations
- Ceramic package analysis to determine contamination in Pb glass
- Determination of electrical leakage
- Quantitative analysis of seal and package glasses
- Substrate material characterization: BeO, AlN, Al_2O_3 , BaTi_4O_9 , etc.
- Adhesion and contamination analysis
- Thin film composition & thickness
- Thick " " "
- Diffusion into substrate
- Solder composition analysis, eutectic characterization, oxide thickness, homogeneity
- High Tc quantitative analysis
- Magnetic coating compositions & thicknesses
- TAB interconnect characterization
- Bump characterization, BGA array analysis

- Polished cross and taper sections to determine metal or composite/ceramic bonding failure causes
- Diffusion of coatings into ceramics, sub-micrometer scale, for nitrogen, etc.
- Quantitative line scans & maps.
- Determination of stoichiometry of carbides, nitrides, etc.
- Analysis of loose powders for size, composition and coating
- Combined with microhardness study fracture length and extinction in micron sized areas
- Flame sprayed coating analysis, embedded abrasive identification and particle size distribution

METALS:

- Quantitative compositional analysis & alloy identification- in particles down to $1\mu\text{m}$
- Inclusion analysis
- Phase identification & size distributions
- Optical microscopy & image analysis
- Stain characterization, including oxide and carbonaceous species
- Diffusion analysis
- Fractographic analysis, failure modes and origins
- Corrosion identification
- Measurement of thin (or thick) film coatings for composition, thickness, purity and diffusion. Performed using a combination of

FIBER OPTICS, CERAMICS, & GLASSES:

- Fiber optics –quantitative doping profiles for boron and most other elements
- Analyze coatings placed on ceramic (SiC , Si_3N_4) whiskers and CVD deposited materials for composition and thickness

metallography, SEM analysis, X-ray analysis and Auger electron spectroscopy. Coating thickness minimum- 0.04 micro-inches (1nm).

- Implant analysis; carbon, nitrogen, oxygen, etc. Composition and depth distribution
- Surface roughness- flat or curved surfaces

MINERALS:

- Quantitative analysis of phases
- Optical microscopy- cross nichols polarization analysis

- Mapping of phases & speciation
- Optical & SEM image analysis

POWDERS:

- Photo microscopy of powder ranging from less than 1 μ m, size distributions
- Quantitative analysis- speciation
- Analysis of coatings on powders; oxides, nitrides, carbides, etc.

SPECIALIZED APPLICATIONS

Geller MicroAnalytical Laboratory has performed analysis in the semiconductor, hybrid device, metallurgical, medical and other industries since 1985. We have found that our experience from analyzing materials used in one industry often directly relates to the current state-of-the-art in others. Listed below are just some of the areas in which we have contributed. Let our broad experience in related fields help you.

ELECTROPOLISHED SURFACES

The treatment of stainless steel and other metal surfaces is crucial to corrosion protection. Electro-polishing of SS *should* enhance Cr at the surface. If done improperly, Cr can be depleted. We have developed a testing procedure (Bulk Analysis Ratio technique) using Auger and XPS spectroscopy that is now being evaluated in ASTM round robin. Applications are in the water treatment industry, semiconductor gas distribution systems, and the medical industry.

DENTAL IMPLANT INDUSTRY

Titanium and titanium alloy implants are used in the dental industry for affixing dentures to jaw bone. The implant surfaces must have not much more than a native oxide and also be free from machining oils and other contaminants. Auger spectroscopy in combination with SEM and EDS X-ray is an excellent technique for characterizing implants. Results from our testing procedure are reproducible year after year.

MEDICAL APPARATUS INDUSTRY

Our investigations on behalf of medical industry companies have led us on fascinating paths.

- Developing green cleaning procedures
- Endoscope failures and found root causes of corrosion leading to control cable failures
- Analyzed ¼" steel (Harrington type used for scoliosis patients) rod fractures that were traced to poor annealing procedures
- Auger microprobe has been used to identify metallic source particles in cosmetics and corrosive stains
- Surface roughness of galled parts
- Cardiac stent analysis for structure, composition, corrosion and fatigue
- Foreign particle analysis
- Reverse engineering
- Critical dimension measurements

MICRO PARTICLE ANALYSIS

Fine sub-millimeter particles originating from lubricating oils, blocked orifices or ground weld fragments can be mounted and polished then

analyzed for composition using the EPMA to determine alloy types and concentration levels of

suspected contaminants down to 10 ppm. Several customers send us samples to test on a regular basis.

CARBON AND BORON GRADIENT ANALYSIS

We can accurately quantify the elemental composition and distribution of carbon and boron in steels. The electron probe Micro-analyzer (EPMA) uses a finely focused electron beam to excite characteristic x-rays in areas as small as 1µm which are measured by wavelength dispersive spectrometers. Being a micro-analytical technique, a series of analyses can accurately show carbon or boron distributions and concentrations before heats, and after heats to show gradients for case depth analysis- even on irregularly shaped specimens.

SEMICONDUCTOR MATERIAL ANALYSIS

Our customers have had us characterize bond pads for oxide thickness and surface contaminants. We also depth profile (using sample rotation for high resolution) to study film diffusion and composition. We do this quantitatively using the proper standards. If no standard is available we can often use the EPMA for determining chemistry using low voltage electron beam excitation. Auger sensitivity facts are then determined and applied.

NICKEL AND PLATINUM ALUMINIDE COATING ANALYSIS

The definitive technique for determining the nickel aluminum concentrations in new and refurbished turbine coatings is by using wavelength dispersive x-ray analysis in the electron probe microanalyzer. Several re-work shops send us samples for analysis on a regular basis.

WHY USE GELLER MICROANALYTICAL?

We are always available, without obligation, for telephone consultation. Rush jobs can often be accomplished with overnight or next day service. All of our reports can be emailed- at no extra cost. How can we do this? Being a small company, we do not have the formalities, and we don't stand on ceremony. Our personalized service means you can discuss data directly with the analyst- even after the

work is complete. Let us be your competitive advantage!