ISO 9001-2015 Certified   Analytical Services to the Mining Industry

==

Surface Science Western (SSW) is a consulting and research laboratory at The University of Western Ontario, providing a broad range of analytical services to the industry. Established in 1981, SSW is one of the most comprehensively equipped surface analytical laboratories in Canada.

SSW offers a variety of analytical services to the mining industry related to the discovery, characterization and processing of value minerals. Some of the areas SSW has become established as a "go to place" for analytical services within the mineral processing sector are:

- Determining the concentration and distribution of precious metals at low concentration levels (ppm-ppb range) in various mineral phases
- Surface chemistry in relation to flotation selectivity: flotation scheme optimization and process control
- Extraction technology: issues involved with sulphide oxidation in autoclaves and gold losses during leaching in response to preg-robbing by *in situ* organic material

Our strength is based not only on our expertise and state-of-the-art analytical equipment and techniques, but also on our commitment to and relationship with our clients. We engage in direct, person to person interactions with our clients and we strive to provide them with "one stop" solutions to their problems. We have established a partnership network with some of the best research facilities in the industry to provide comprehensive, in-depth services to our clients.

We are committed to quality (SSW is ISO 9001-2015 certified) and guarantee confidentiality.

As a consulting and research laboratory, we are also actively involved in research and development for the mining industry. Part of our mission is to work together with our research partners from the mining industry to develop new applications and commercialize them as routine diagnostics tools.

This brochure provides details on the various analytical techniques and instrumentation in direct relation to the mineral processing industry and the cost breakdown by services offered by SSW.

Our goal is to meet your needs quickly, completely and confidentially.

CONTENTS

ANALYTICAL SERVICES

The laboratory at SSW is well equipped to analyze the surface chemistry of mineral phases in flotation streams and as well as to determine precious elements concentration and partitioning within mineral phases from variety of metallurgical processes.

Low level concentration and distribution of precious metals (ppm and ppb range) in mineral phases are routinely analysed by Dynamic Secondary Ion Mass Spectrometry (D-SIMS). Quantitative analyses can be performed for a whole range of elements (for example Au, Ag and PGMs) in value minerals as well products from other metallurgical applications. This type of information is vital for establishing deportments of precious metals in mineral samples and process development and optimization.

The relative distribution of adsorbed species on mineral grains is routinely analysed by Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS). This technique provides information on the surface chemistry of minerals and can identify potential problems with lower recovery and grades related to a variety of mineral processes:

<u>Factors Influencing Flotation</u>
● activators   ● depressants   ● surface oxidation   ● collector loadings

<u>Mineral Chemistry and Leach Products</u>
●preg-robbing   ●surface coatings: Ag, Hg, Cu.. ●refractory gold minerals

<u>Roasting/AC POX/CIL circuits</u>
● refractory gold locking   ● preg-robbing ● encapsulation

Examples of experimental studies involving trace element analysis and mineral surface chemistry are presented in the Application Notes.

Upon request, we will recommend to our clients specific, optimised study proposals that would best address their needs. In addition, we offer a variety of standard service packages for the most common applications of micro-beam analytical techniques in the mineral processing industry.

ANALYTICAL TECHNIQUES AND INSTRUMENTATION

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)

Technique:

Time-of-flight secondary ion mass spectrometry (ToF-SIMS) employs nano-second pulses of energetic primary ions to sputter material from the sample surface. In the static SIMS mode, only the outermost monolayer (a few nm) of the sample is analyzed. To ensure this "static" condition, a primary ion dosage of less than 10^{13} ions/cm^2 is employed, which also minimizes molecular fragmentation. The secondary ions produced are extracted from the sample surface and mass analyzed in a time-of-flight mass spectrometer. By scanning the primary ion beam over the sample it is possible to generate maps showing the surface concentration distribution of those secondary ions with a high spatial resolution. Recent development and introduction of a new generation of cluster liquid metal ion sources ($Bi3^+$ and $Au3^+$) into the ToF-SIMS instrumentation lead to a dramatic improvement in the detection sensitivities and ability to detect complex compounds with minimum fragmentation.

Instrument:
ION TOF-SIMS IV equipped with Bi^+, Cs^+, O^+ and $C60^+$ ion sources

System capabilities:
- Non-destructive, comprehensive inorganic and organic surface analysis
- Minimum detection limits in the low ppm and sub-ppm concentration range
- Imaging capabilities with spatial resolution down to 0.3µm
- Depth profiling

Selected applications in the mining industry:
- Identification of collectors on individual mineral particles from plant samples. The identification is based on detection of unfragmented molecular ions
- Measurements of collector loadings on mineral grains from flotation circuits at plant concentration levels. Practical detection limits less than 1g/t
- Identification of activators and depressants on mineral particles. Comparative analysis on the degree of activation/depression of mineral phases from flotation products
- Characterization and mapping of surface coatings on gold grains from cyanidation leach residues
- Characterization of gold in carbonaceous matter from AC/CIL residue products

Dynamic Secondary Ion Mass Spectrometry (D-SIMS)

Technique:

Secondary ion mass spectrometry (SIMS) provides elemental and isotopic analysis of very small volumes situated on the surface of solid samples. Operated in dynamic SIMS mode, an energetic primary ion beam sputters particles from the sample surface. Some of the sputtered material is ejected as either positive or negative secondary ions. Therefore, each point of the surface becomes a source of secondary ions that are characteristic of the elements or isotopes found in the near surface region. These secondary ions are further mass analyzed in a magnetic sector mass spectrometer. By rastering the primary ion beam a distribution map of elements and isotopes from the analyzed region of the sample can be obtained.

Instruments: Two Cameca IMS-3f SIMS instruments equipped with Cs^+ and O^+ ion sources

System capabilities:

- Elemental analysis covering the entire periodic table
- Quantitative microanalysis with detection limits of 200-300ppb. It effectively addresses the analytical gap between the electron microprobe (MDL≈ 500ppm) and bulk analytical techniques (MDL≈ low ppb)
- Elemental depth profiling
- Imaging of the elemental distribution

Selected applications in the mining industry:

- Quantitative analysis of precious metals (Au, Ag, Pd, Pt, Rh…) in feed samples and/or process stream products
- Quantitative analysis of other trace elements in minerals: Ni, Co, As, Pb, Hg, Sb, Se…
- Mapping trace element distributions in minerals
- Quantitative analysis of sub-microscopic gold (solid solution and colloidal-type) in sulphide minerals
- Characterization of the carriers and forms of gold: quantitative analysis and distribution
- Identification of specific causes for gold losses in the process stream products from roaster and autoclave pressure oxidation AC POX/CIL circuits

Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDX)

Technique:

With the scanning electron microscopy (SEM) technique, a primary electron beam with energy up to 30keV is focused and rastered on the sample surface. These primary electrons can generate secondary electrons or X-rays that are characteristic of different elements. High resolution images of the sample surface can be generated by detecting either secondary electrons or backscattered primary electrons (BSEs) in the SEM mode. The BSE images are especially valuable for mineralogical applications. The brightness of the BSE image increases with the atomic number of the element and it can characterize areas with different elemental composition, which is particularly useful for mineral phase determination. Energy dispersive X-ray (EDX)

spectroscopy is a technique used to identify the elemental composition of the sample. It is applied in conjunction with the SEM technique. The information about the elemental composition is derived by detection of the characteristic X-rays generated by the primary electron beam of the SEM instrument.

Instruments: LEO 440 SEM/Quartz Xone EDX system
Hitachi   S-4500 FESEM/EDAX^{TM} EDX system

System capabilities:
- Imaging with spatial resolution down to 2-5nm
- Secondary and backscattered electron imaging. The backscattered electron imaging is especially useful in differentiation of mineral phases
- Quantitative compositional analysis with detection sensitivity down to 0.5-1wt%.
- Elemental mapping

Selected applications in the mining industry:
- Mineral composition of an ore sample
- Mineral liberation analysis
- Identification and characterization of surface coatings on mineral particles from process stream samples

X-Ray Photoelectron Spectroscopy (XPS)

Technique:
When the surface of a sample is excited with soft X-rays, high-resolution energy analysis of photoelectrons emitted from atoms near the surface can be used to characterize a variety of inorganic and organic materials. The binding energies of the detected series of photoelectron peaks are characteristic for different species. The peak areas can be used to determine the composition of the sample surface. The shape of the photoelectron peaks and their respective binding energies are affected by the chemical state of the emitting atoms. Hence, chemical bonding information may be determined from the chemical shift of the atomic transitions.

Instruments: Kratos AXIS ULTRA
Kratos AXIS NOVA

System capabilities:
- Very surface sensitive. Only the uppermost 5-10 nm of solid surfaces is analyzed
- Elemental identification and quantification
- Minimum detection limits in the range of 0.2-0.5 wt%
- Chemical speciation
- Depth profiling
- Imaging capabilities

Selected applications in the mining industry:
- Characterization of surface coatings on mineral grains
- Imaging for mineral phase selection
- Chemical state information of surface coatings

Auger Electron Spectroscopy (AES)

Technique:

The Auger Electron Spectroscopy technique uses a principle similar to that of the XPS technique, which detects the so called "Auger" electrons. These Auger electrons are characteristic of the elemental surface composition of the sample. The use of an electron beam as the excitation source provides a much smaller analytical spot (x100nm) compared to the XPS (10μm) and allows for high spatial resolution analysis of small sample features. Composition depth profiles can be generated by utilizing an additional ion sputter source.

Instrument: PHI 660 Scanning Auger Microprobe (SAM)

System capabilities:

- Surface sensitive; the analytical depth is only 3-5 nm
- Identification of surface species with minimum detection limits in the range of 0.5 wt%
- Spatially resolved compositional depth profiles measured to depths of several microns

Selected applications in the mining industry:

- Compositional depth profiles of surface coatings on mineral grains from down stream products in pressure oxidation (POX), cyanide leaching or flotation circuits
- Characterization of oxidation/passivation layers on mineral particles

SERVICE PACKAGES

I. ABBREVIATED METALLURGICAL STUDY

Deliverables
1. Basic SEM/EDX spectroscopy screening of the sample composition.
2. Comparative ToF-SIMS surface analysis of feed, con and tail samples to determine statistically significant differences in the surface composition related to potential activators and depressants. Twenty grains per sample will be analyzed.
3. Written report issued within 15 business days of sample receipt.

Cost: $3,500

II. FEED ORE CHARACTERIZATION: ORE CHEMICAL ACTIVITY TESTING BY TOF-SIMS

Deliverables
1. Basic SEM/EDX spectroscopy screening of the sample composition.
2. Estimate of the ore capacity to undergo "inadvertent activation" during the grinding process by ToF-SIMS surface analysis.
3. Data comparison with ore sample library, which reflects the mineralogical variability of ore types from different deposits. The data covers a variety of poly-metallic ore deposits (for example skarn, hypogene and supergene) with large dynamic range of chemical activity.
4. Written report issued within 15 business days of sample receipt.

Cost: $1,000 per ore sample

III. ABBREVIATED QUANTITATIVE SCREENING FOR CARRIERS OF PRECIOUS METALS IN ORE SAMPLES

Deliverables
1. Quantitative D-SIMS analysis of 35 mineral grains selected by the client.
2. Written report issued within 15 business days of sample receipt.

Cost: $2,500

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519-661-2173 · FAX: 519-661-3709 · WWW.SURFACESCIENCEWESTERN.COM
8

IV. QAUNTITATIVE D-SIMS ANALYSIS OF SUB-MICROSCOPIC GOLD IN SULPHIDE MINERALS

Deliverables
1. Recommended 30 analyses per mineral. The study includes the following:
   - Classification of microscopic gold content (solid solution and colloidal-size) per morphological type
   - Simultaneous quantification of gold and arsenic content
   - Mapping of the gold and arsenic distribution within the mineral grains (optional)
   - Establishing a relationship (if any) between the gold and arsenic content and distribution
2. Written report issued within 15 business days of sample receipt

Cost:
- $3,360 for 55 SIMS analyses
- $3,360 for 10 map plates (optional). Each plate includes images of Au, As, on matrix element and an optical microphotograph of the analyzed grain

V. CHARACTERIZATION OF DISCHARGE SAMPLES FROM AC/CIL CIRCUITS BY SEM/EDX AND AES

Deliverables
1. SEM/EDX and AES study:
   - Compositional analysis of surface coatings on mineral grains
   - Evaluation of the thickness and nature of surface coating on discharge products
   - Characterization of oxidation products of mineral particles
2. Written report

Cost:
- $3,500/sample AES surface analysis and depth profiling
- $580/sample SEM/EDX analysis

VI. DEPORTMENT OF GOLD IN DISCHARGE SAMPLES FROM AC/CIL CIRCUITS

Deliverables
1. Evaluation of refractory gold in residual (unoxidized) sulphides using quantitative D-SIMS analysis:
   - Quantification of sub-microscopic gold content in sulphides of different morphological types
   - Quantification of the arsenic content
   - Mapping of the gold and arsenic distribution (optional)
2. Evaluation of gold trapped in primary FeO_x and oxidation products by D-SIMS:
   - Quantification of sub-microscopic gold content in the iron oxides
   - Quantification of the arsenic content
   - Mapping of the gold and arsenic distribution (optional)

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519-661-2173 · FAX: 519-661-3709 · WWW.SURFACESCIENCEWESTERN.COM
9

3. ToF-SIMS analysis of exposed gold with impermeable coatings: Hg, Ag, Cu, phosphates, arsenides, carbonates, *et cetera*.
4. Written report

Cost:
- ◆ $3,360 for 55 SIMS analyses
- ◆ $3,360 for 10 map plates (optional)
- ◆ $2,000 for ToF-SIMS surface analysis of 20 gold grains

VII. DETERMINATION OF SURFACE GOLD PREG-ROBBED ON CARBONACEOUS MATTER FROM AC POX STREAM PRODUCTS

Deliverables

1. Characterization of different types of carbonaceous matter present in the sample:
   - Composition established by SEM/EDX (scanning electron microscopy coupled with energy dispersive Xray) spectroscopy
   - Structure (maturity) of the carbonaceous matter determined by laser Raman spectroscopy
   - Preg-robbing capacity of each type of carbonaceous matter evaluated by ToF-SIMS
2. Speciation and quantification of surface gold preg-robbed on carbonaceous matter by ToF-SIMS:
   - Detection ("speciation") of different forms of gold preg-robbed on carbonaceous matter: metallic gold Au^o and gold compounds ($Au(CN)_2^-$, $AuCl_2^-$, $AuS(CN)_2^-$ …)
   - Quantification of the established forms of preg-robbed gold based on compound-specific Au standards in activated carbon
   - Independent evaluation of the total amount of surface gold preg-robbed on carbonaceous matter
3. Written report

Cost:
- ◆ $4,000 for 40 ToF-SIMS analyses
- ◆ $1,000 for assay and surface area analysis

Notes: **1. All abovementioned service packages can be tailored according to the needs and the requests of the client.**

2. With the exception of the abbreviated studies, there will be an additional charge for reporting. This reporting charge will depend on the volume of analytical work covered by the study.

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519-661-2173 · FAX: 519-661-3709 · WWW.SURFACESCIENCEWESTERN.COM
10

Application note 1

QUANTITATIVE D-SIMS ANALYSIS OF SUB-MICROSCOPIC GOLD IN SULPHIDE MINERALS

Request for analysis:

A refractory feed ore sample containing pyrite and arsenopyrite mineral phases was to be analysed for the presence of "invisible" sub-microscopic gold.

Objectives:

1. To quantify the sub-microscopic gold content in pyrite and arsenopyrite mineral phases by dynamic SIMS.
2. To map the distribution of sub-microscopic gold in pyrite and arsenopyrite mineral phases by dynamic SIMS

Methodology:

Polished sections from specific size fractions of the ore sample were prepared and grains representing both mineral phases were selected and marked for analysis. These mineral particles were analysed using the Cameca IMS-3f SIMS instrument and concentration depth profiles for Au, As, S and Fe were produced. The quantification of sub-microscopic gold and arsenic in pyrite and gold in arsenopyrite was established using internal mineral specific standards.

Results of the study:

Three different morphological types of pyrite (coarse, porous and fine) and arsenopyrite (coarse, porous and fine crystalline) were studied. The quantified sub-microscopic gold and arsenic (for pyrite only) concentrations in arsenopyrite and pyrite are listed in Tables 1 and 2. Concentrations measured in each grain and their average values per mineral type with corresponding 95% confidence intervals are included in the tables.

Major findings:

  i. The gold in both the arsenopyrite and pyrite mineral phases occurs exclusively in sub-microscopic form.

 ii. The arsenopyrite is the principal gold carrier in the ore sample**.** All morphological types carry high gold content: coarse (86.39ppm), porous (100.98ppm) and fine (55.5ppm) arsenopyrite, Table1. All SIMS concentration depth profiles show the presence of solid solution gold, Figure 3a.

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519-661-2173 · FAX: 519-661-3709 · WWW.SURFACESCIENCEWESTERN.COM
11

iii. The determined gold concentration in the second mineral carrier, pyrite, is in the low ppm range. Different morphological types carry increasing amounts of gold in the following order: coarse (0.35ppm), porous (1.39ppm) and fine crystalline (6.03ppm) pyrite, Table 2. Statistically, 85% of the SIMS concentration depth profiles in pyrite show the presence of colloidal-size gold, Figure 3b.

iv. Comparison by mineral type between measured mean values of sub-microscopic gold concentration is shown on Figure 1.

v. There is a positive correlation between the measured gold concentration and arsenic content in pyrite, Figure 2.

vi. The mapped distribution of sub-microscopic gold in a pyrite grain is shown on Figure 4.

Table 1 - Measured concentrations of sub-microscopic gold in arsenopyrite

Arsenopyrite					
Coarse		Porous		Fine	
Grain I.D.	Au ppm	Grain I.D.	Au ppm	Grain I.D.	Au ppm
m2apc01	111.57	m2app10	191.92	m1apf01	16.10
m2apc02	83.16	m2app18	18.32	m1apf05	75.74
m2apc03	123.47	m2app23	488.92	m3apf11	121.95
m2apc07	158.72	m2app34	89.64	m3apf12	48.87
m2apc12	159.07	m2app35	2.26	m3apf13	68.96
m2apc13	68.18	m2app37	27.82	m3apf16	1.40
m2apc14	36.75	m2app38	9.51		
m2apc15	61.10	m3app07	44.34		
m2apc17	5.07	m3app08	36.12		
m2apc19	27.56				
m2apc20	88.53				
m2apc22	96.83				
m2apc33	103.03				
Average	86.39		100.98		55.50
±λ	26.03		104.51		35.65

±λ: 95 % confidence interval
λ= 2 σ/√n; σ is the standard deviation; n is the number of analyses

Table 2 - Measured concentrations of sub-microscopic gold and arsenic in pyrite

Pyrite								
Coarse			**Porous**			**Fine crystalline**		
Grain I.D.	**Au ppm**	**As ppm**	**Grain I.D.**	**Au ppm**	**As ppm**	**Grain I.D.**	**Au ppm**	**As ppm**
m1pyc02	0.37	28.70	m1pyp04	10.12	88368.73	m1pyfc08	1.19	21328.63
m1pyc12	0.25	17.84	m1pyp05	0.69	37.30	m1pyfc09	0.43	3383.18
m2pyc02	0.14	1037.54	m1pyp06	3.41	2709.37	m1pyfc13	9.13	42495.30
m2pyc05	0.06	35.99	m1pyp10	1.91	899.18	m1pyfc15	1.01	21154.18
m2pyc08	0.25	1758.93	m1pyp11	0.14	1158.28	m1pyfc21	0.64	5360.73
m2pyc15	0.43	2.90	m1pyp17	0.38	155.96	m2pyfc01	5.02	30009.33
m2pyc18	0.29	1114.73	m1pyp19	0.35	41.97	m2pyfc13	13.99	53410.98
m2pyc25	1.18	324.42	m1pyp20	0.44	182.67	m2pyfc22	1.52	72375.41
m2pyc34	0.15	16.10	m2pyp04	0.23	1875.09	m2pyfc24	13.12	160804.61
			m2pyp20	0.16	170.59	m2pyfc33	0.25	5013.66
			m2pyp21	0.23	187.20	m2pyfc39	29.88	32522.38
			m2pyp26	0.94	1726.63	m2pyfc45	1.27	24265.77
			m2pyp27	0.11	25.81	m2pyfc48	0.95	25466.99
			m2pyp29	0.35	28.11			
			m2pyp31	0.26	253.02			
			m2pyp37	0.44	127.84			
			m2pyp38	3.51	53919.80			
Average	**0.35**	**481.91**		**1.39**	**8933.39**		**6.03**	**38276.20**
±λ	**0.22**	**436.58**		**1.21**	**11742.26**		**4.82**	**23134.16**

±λ: 95 % confidence interval

λ= 2 σ/√n; σ is the standard deviation; n is the number of analyses
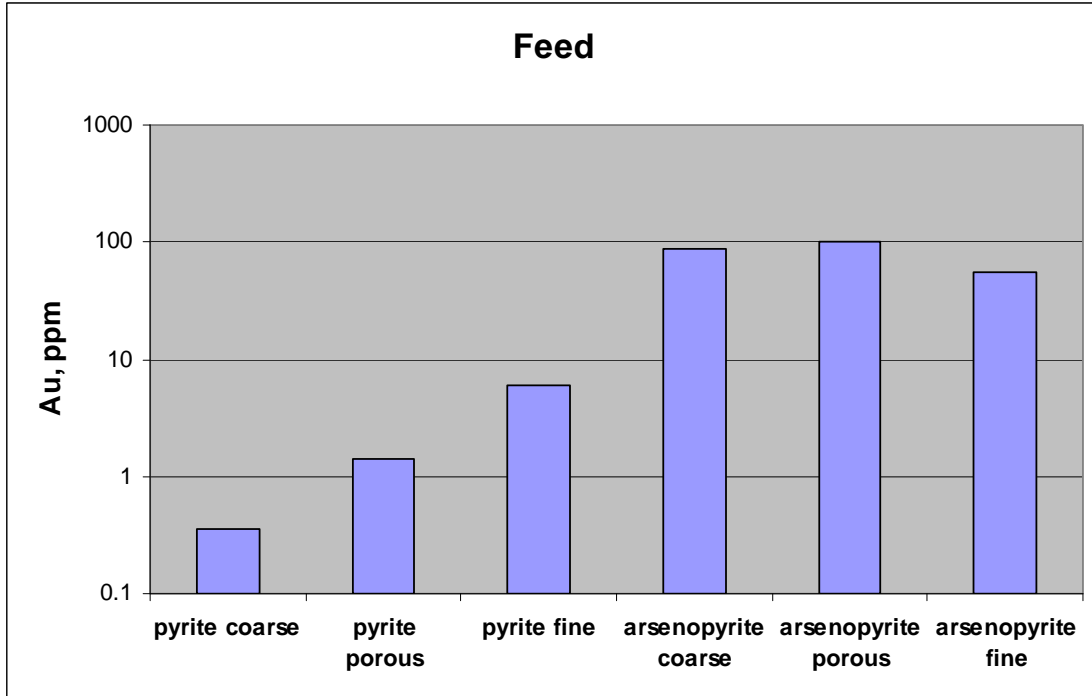
Figure 1. Comparison by mineral type of the measured mean values of sub-microscopic gold concentration.
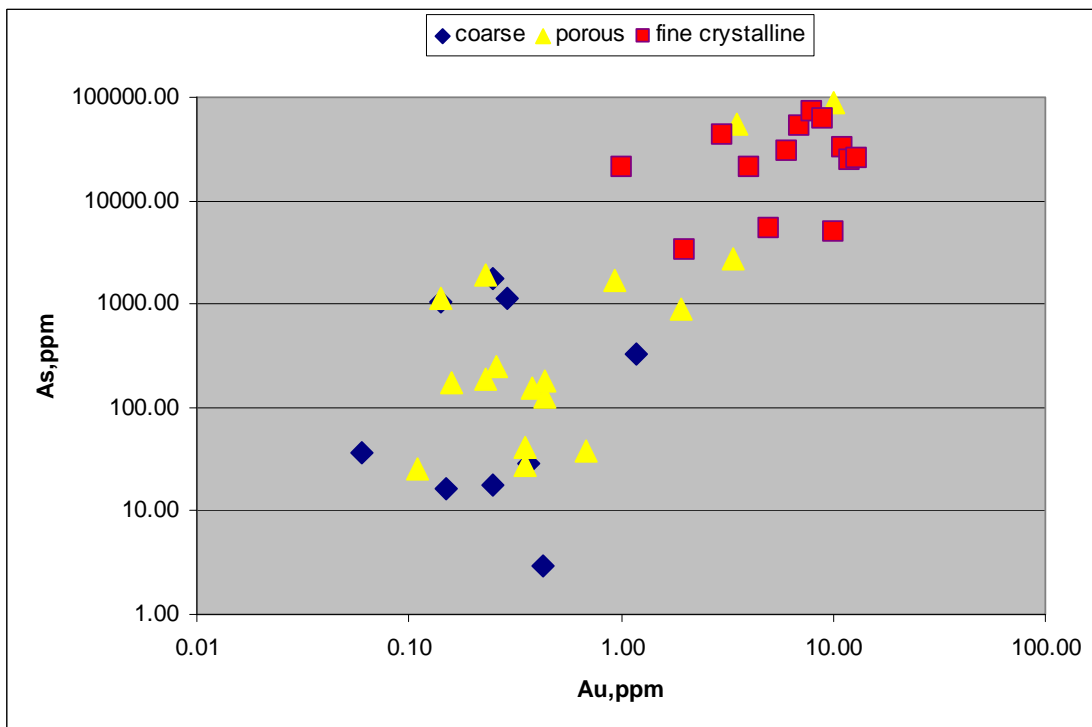

Figure 2. Scatter plot of gold and arsenic concentration in different morphological types of pyrite grains.
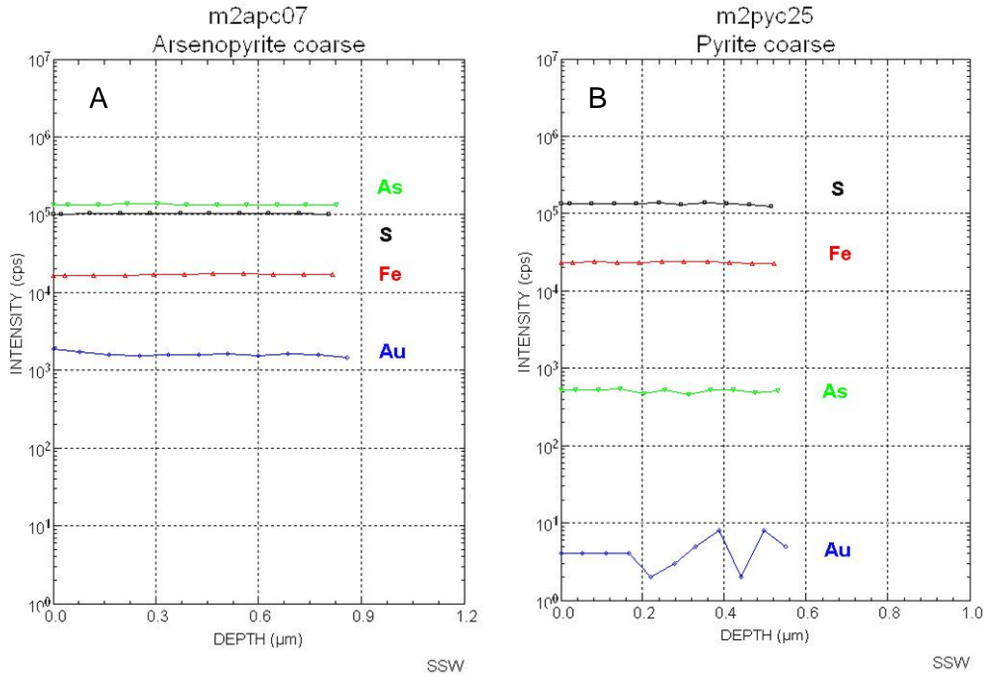
Figure 3. Concentration depth profiles of sub-microscopic gold.
A) coarse arsenopyrite grain containing solid solution gold: Au=158.72ppm
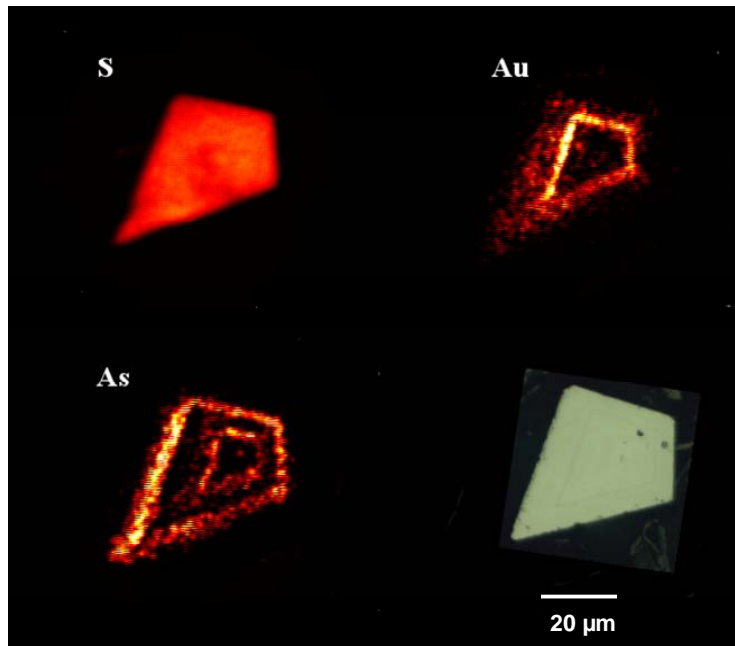**B) coarse pyrite grain containing colloidal gold: Au=1.18ppm**

Figure 4. Sub-microscopic gold and arsenic distribution in a coarse pyrite grain.

Application note 2

DETERMINATION OF SURFACE GOLD PREG-ROBBED ON CARBONACEOUS MATTER FROM AUTOCLAVE PRESSURE OXIDATION (AC POX)/CARBON IN LEACH (CIL) STREAM PRODUCTS

Request for analysis:

A refractory sulphide ore containing carbonaceous matter has been processed in an AC POX/CIL test plant. The gold recovery after cyanidation leaching is low. The CIL residue was analysed for possible losses due to preg-robbing of gold from the cyanide solution onto carbonaceous material present in the sample.

Objectives:

1. Detection ("speciation") of different forms of surface gold preg-robbed on carbonaceous matter: metallic gold Au^o and gold compounds ($Au(CN)_2^-$, $AuS(CN)_2^-$ …) by ToF-SIMS.
2. Quantification of the established forms of preg-robbed surface gold by ToF-SIMS based on compound specific Au standards in activated carbon.

Methodology:

1. Various types of carbonaceous matter present in the sample are characterized with regard to their preg-robbing capacity using a set of several complementary techniques:

   ■ Composition established by SEM/EDX (scanning electron microscopy coupled with energy dispersive x-ray spectroscopy)

   ■ Structure (maturity) of the carbonaceous matter determined by laser Raman spectroscopy

   ■ Preg-robbing capacity of each type of carbonaceous matter evaluated by ToF-SIMS

2. A large number of individual carbonaceous particles were analysed by ToF-SIMS for the presence of surface gold. This technique provides non-destructive elemental and molecular surface analysis and allows for simultaneous detection of metallic gold and gold compounds. Due to the low molecular fragmentation during the ToF-SIMS analysis, it is possible to detect ("speciate") simultaneously the presence of Au in both elemental (Au^0) and compound forms such as $Au(CN)_2$, $AuCl_2$ or $Au(SCN)_2$. The quantification of the ToF-SIMS data is based on element and compound specific standards with established detection limits for surface metallic and compound gold in the low ppm range.

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519–661–2173 · FAX: 519–661–3709 · WWW.SURFACESCIENCEWESTERN.COM

16

Results of the study:

Separate carbonaceous particles from the CIL residue sample were selected under the optical stereoscope and mounted on a copper substrate for analysis.

Major findings:

i. The SEM/EDX analysis identified two different groups of carbon-containing particles: total carbonaceous matter particles (TCM) containing almost 100% carbon (Figure 1) and carbonaceous particles (quartz particles with variable amounts of finely disseminated carbonaceous matter, Figure 2).

ii. A comparison between the laser Raman spectra of carbon standards and the carbonaceous material from the CIL residue samples shows that the TCM structure is similar to that of graphitic carbon while the Disseminated TCM particles carry the characteristics of activated carbon, Figure 3.

iii. ToF-SIMS surface spectra and images of the surface Au distribution were established for each particle. The speciation of the gold preg-robbed on carbonaceous matter from the CIL tail sample showed the presence of both metallic gold and $Au(CN)_2$ compound, Figure 4.

iv. The quantified metallic gold and $Au(CN)_2$ gold preg-robbed on carbonaceous matter is listed in Table 1. Concentrations measured for each grain and their average values per mineral type with corresponding 95% confidence intervals are included in the table.
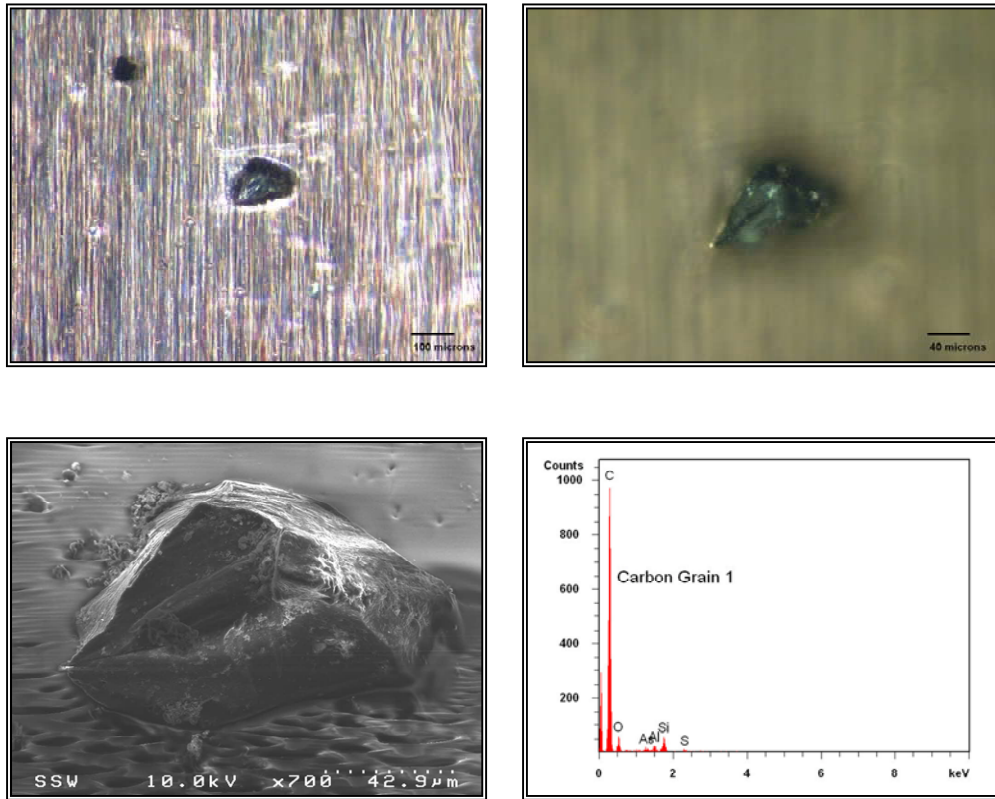
Elemental analyses of large area on Carbon grain 1. All data in wt. %

	C	O	Al	Si	S
carbon grain 1	84.3	13.3	0.4	1.3	0.2

Figure 1. Optical microscope and SEM images along with EDX spectra and semi-quantitative elemental analyses of total carbonaceous matter (TCM) grain.
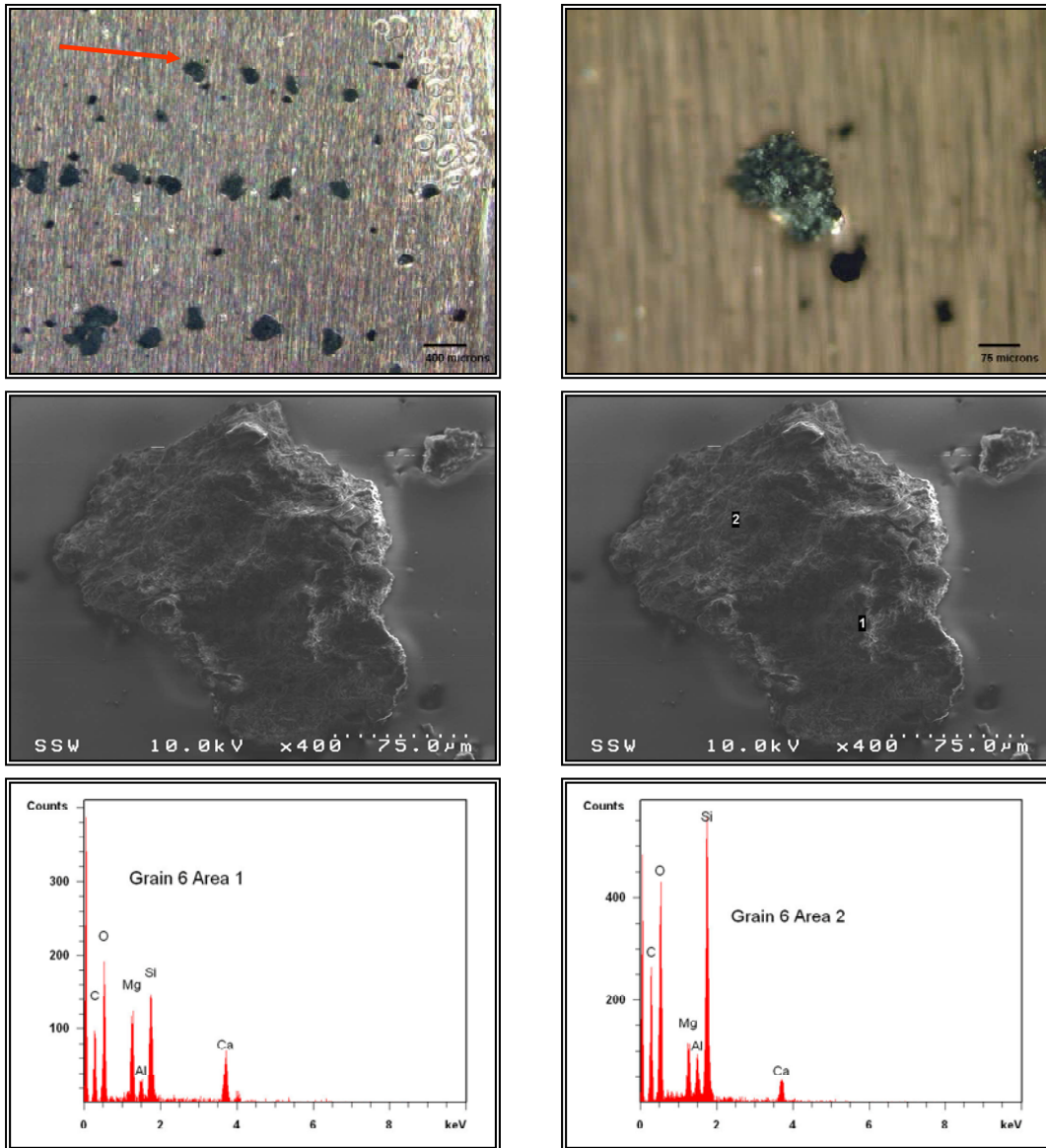
Elemental analyses of 2 areas on grain 6. All data in wt. %

	C	O	Mg	Al	Si	Ca
grain 6 area 1	26.1	50.1	5.2	1.0	7.6	10.0
grain 6 area 2	31.5	50.2	2.1	1.5	11.6	3.1

Figure 2. Optical microscope and SEM images along with EDX spectra and semi-quantitative elemental analyses of two areas on a grain with disseminated carbonaceous matter. The arrow indicates the grain analyses in the context image.
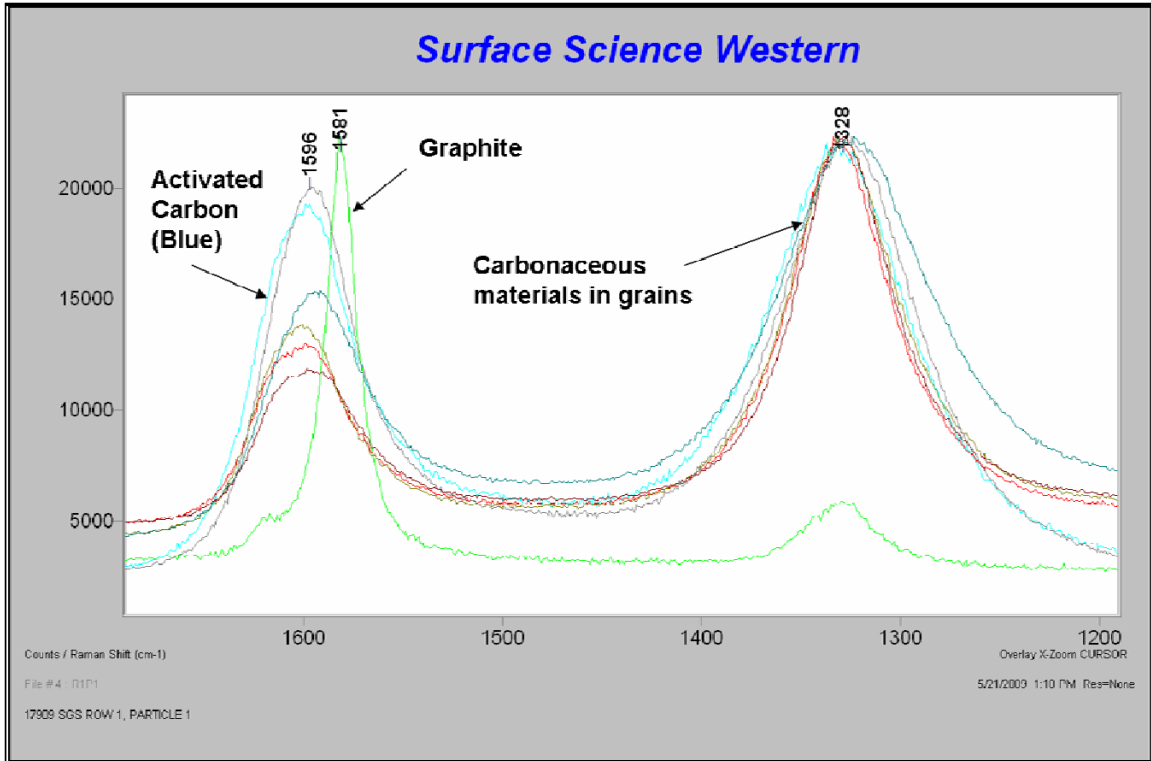
Figure 3. Overlain Raman spectra from carbonaceous areas in several carbonaceous particles along with reference graphite and activated carbon spectra

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519-661-2173 · FAX: 519-661-3709 · WWW.SURFACESCIENCEWESTERN.COM
20
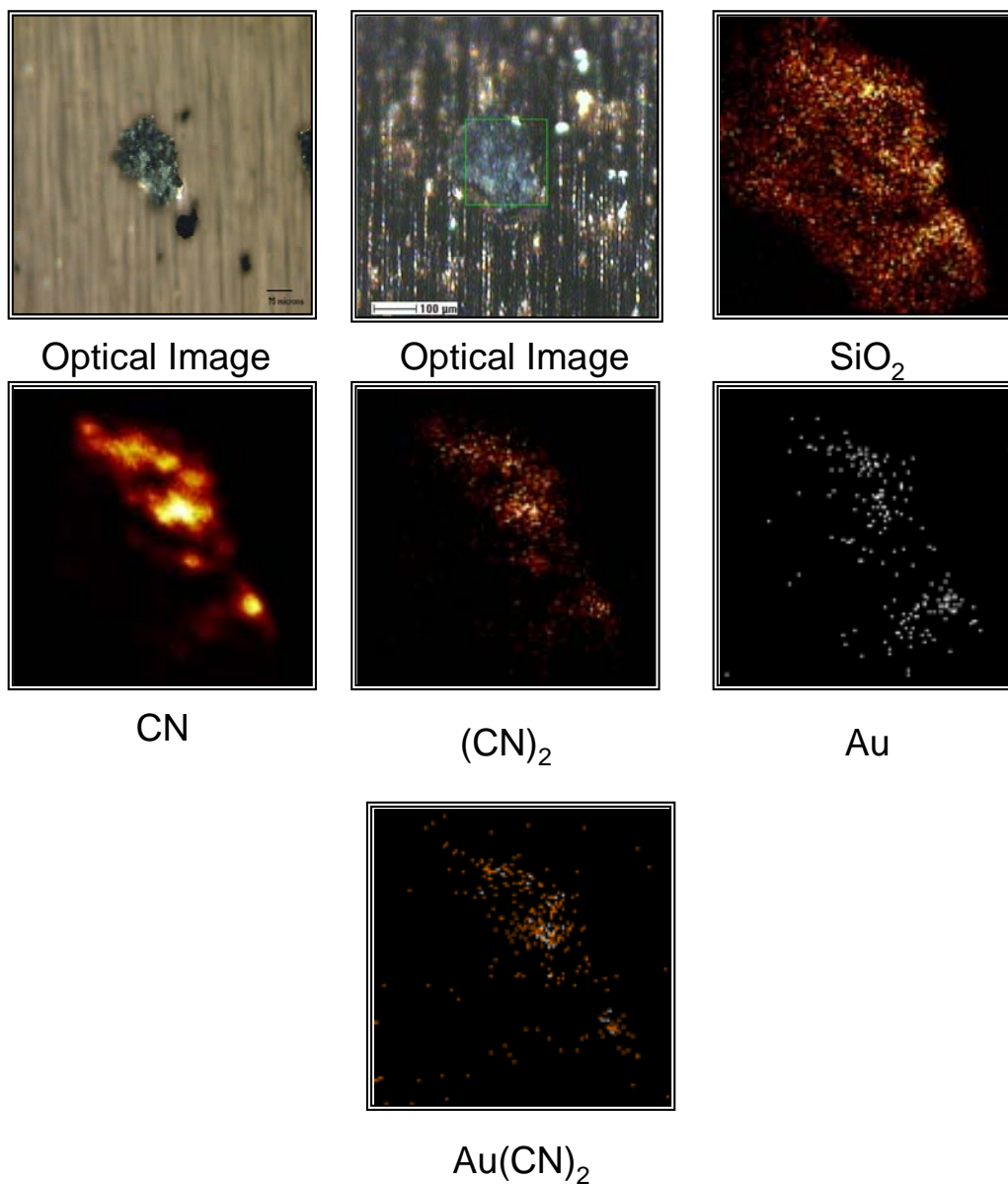
Figure 4. Optical microscope images and ToF-SIMS elemental and compositional maps for a selected carbonaceous particle. The quantified amount of metallic gold on this particle was 4.5ppm, while the amount of Au as Au(CN)$_2$ compound gold was 38.1ppm.

Table 1 - Measured concentrations of surface gold on C-matter from CIL tails

CIL Leach Residue							
Disseminated carbonaceous particles				**TCM Particles**			
Grain I.D.	**Metallic Au, ppm**	**Au(CN)$_2$ ppm**	**Total surface gold, ppm**	**Grain I.D.**	**Metallic Au, ppm**	**Au(CN)$_2$ ppm**	**Total surface gold, ppm**
Cbm01	15.2	22.1	37.3	graph01	3.2	18.7	21.9
Cbm02	3.8	30.2	34.0	graph02	9.0	37.5	46.5
Cbm03	7.2	55.2	62.4	graph03	4.1	21.5	25.6
Cbm04	5.5	42.4	47.9	graph04	5.7	43.1	48.8
Cbm05	8.0	52.0	60.0				
Cbm06	4.9	38.1	43.0				
Cbm07	4.1	35.3	39.4				
Cbm08	5.2	46.3	51.5				
Cbm09	20.1	81.3	101.4				
Cbm10	8.4	53.2	61.6				
Cbm11	4.2	36.2	40.4				
Cbm12	3.0	35.1	38.1				
Cbm13	7.2	52.4	59.6				
Cbm14	4.5	38.3	42.8				
Cbm15	5.1	43.4	48.5				
Cbm16	7.7	52.5	60.2				
Cbm17	7.7	48.1	55.8				
Cbm18	7.3	52.3	59.6				
Cbm19	3.2	34.8	38.0				
average	**7.0**	**44.7**	**51.7**		**5.5**	**30.2**	**35.7**
±λ	**1.9**	**5.8**	**7.1**		**2.5**	**11.9**	**13.9**

±λ: 95 % confidence interval

λ= 2 σ/√n; σ is the standard deviation; n is the number of analyses

DEPORTMENT OF GOLD IN AN AUTOCLAVE PRESSURE OXIDATION (AC POX)/CARBON IN LEACH (CIL) RESIDUE

Request for analysis:

A carbonaceous refractory sulphide ore is processed in an autoclave pressure oxidation plant and subsequently subjected to a cyanide leaching. An initial mineralogical study of the feed ore by quantitative dynamic SIMS analysis has shown that 95% of the gold is contained as sub-microscopic gold in pyrite with the remainder contained in primary Fe-oxide minerals. In addition, the feed ore contains a substantial fraction of carbonaceous material. The gold recovery is low and a gold deportment study of the CIL residue was requested in order to establish the gold distribution in the residue sample towards identifying the reasons for low recovery.

Objectives:

1. To quantify the sub-microscopic gold associated with residual unoxidized pyrite.
2. To map the distribution of gold in partially oxidized pyrite mineral grains.
3. To quantify the sub-microscopic gold content in the primary FeOx.
4. To quantify the surface gold preg-robbed on carbonaceous matter.
5. To determine the fraction of gold associated with silicates.
6. Based on tasks 1-5 to determine the deportment of unrecovered gold in the CIL residue sample
7. Based on the gold deportment to identify cause(s) for experienced poor gold recovery.

Methodology:

A standard procedure for gold deportment study in AC CIL residue is applied. It involves an independent quantification of sub-microscopic gold in different mineral carriers by dynamic SIMS and determination and speciation of surface gold preg-robbed on the carbonaceous matter present in the CIL residue by ToF-SIMS (For more details on these two techniques see the previous Application notes). The CIL residue was assayed for Au, As, $S^=$ and total carbonaceous matter (TCM) in triplicate, then sized by screening and gravity separated. The sized gravity tails was further processed to obtain a clean silicate fraction which was assayed for Au and $S^=$ content.

Results of the study:

The assayed values for sulphide sulphur, $S^=$, indicated the presence of unoxidized pyrite in the CIL residue samples, which was confirmed by optical microscopy of polished sections. Different morphological types of pyrite grains were analyzed by dynamic SIMS and the amount of sub-microscopic gold contained in the unoxidized pyrite was determined using mineral-specific gold standards. Similarly, the sub-microscopic gold content in primary iron oxide minerals (hematite and goethite) was established. The ToF-SIMS analysis of carbonaceous matter present in the residue determined the presence of preg-robbed surface gold in two different forms: metallic gold and $Au(CN)_2$ compound.

The established deportment of gold in the CIL residue sample is shown in Figure 1. Figures 2-4 show D-SIMS distribution maps for Au, As, Fe and S in partially oxidized pyrite grains.

Major findings:

Two major causes for poor gold recovery were identified:

i.   The analyses identified a substantial proportion of unoxidized pyrite in the CIL residue samples. This indicates the need for optimization of the oxidation process in the autoclave facility. The gold deportment data show that 37.5% of the gold losses in the CIL residue are related to sub-microscopic gold contained in the unoxidized pyrite mineral phase (Figure 1).

ii. The carbonaceous matter present in the ore exhibits strong preg-robbing capacity and contributes to 44.5% of the gold losses in the form of surface gold (Figure 1).
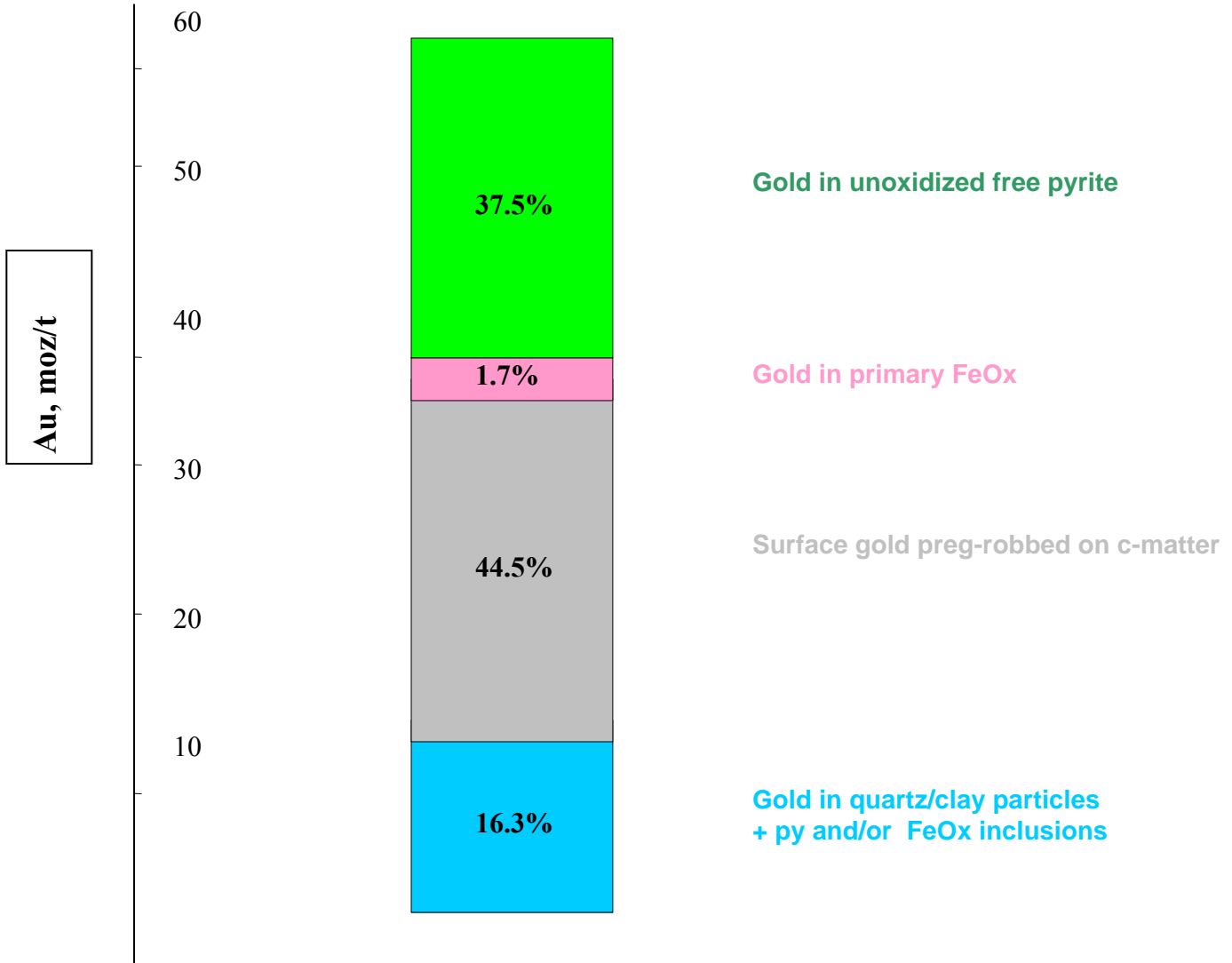
ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519-661-2173 · FAX: 519-661-3709 · WWW.SURFACESCIENCEWESTERN.COM

24

Figure 1. Gold deportment diagram for the CIL residue. The total assayed gold in the sample was 57 moz/t. The contribution of different gold carriers towards the gold losses is shown as a percentage of the total assayed gold value in the CIL residue sample.
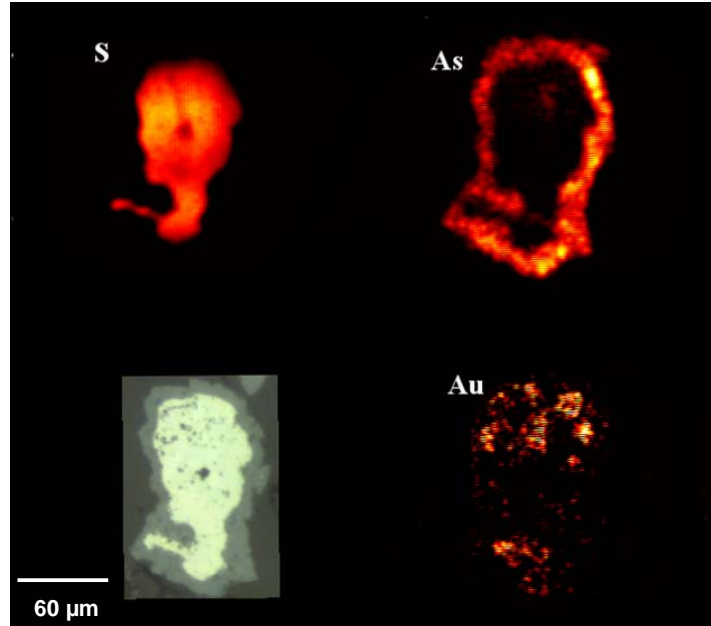
Figure 2. Gold distribution in a rimmed, partially oxidized pyrite grain from AC POX/CIL residue. Note: Brighter colors correspond to higher concentration of the imaged element.
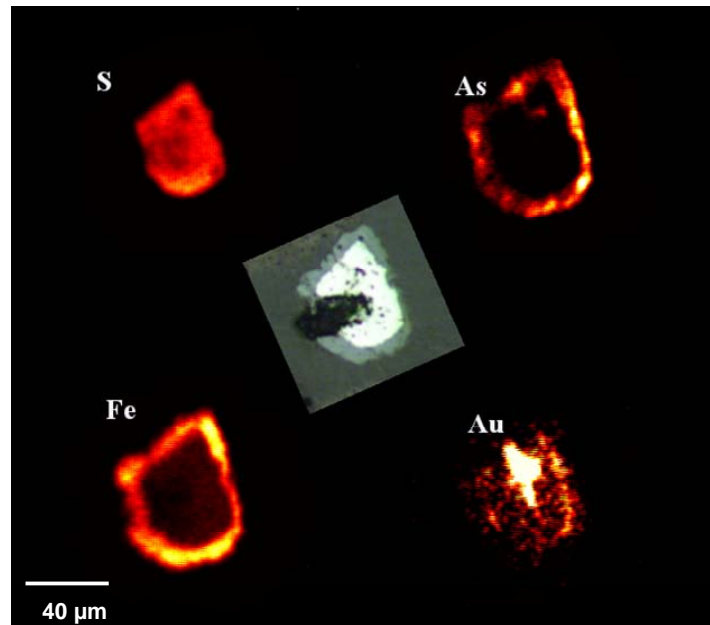

Figure 3. Gold distribution in a rimmed, partially oxidized pyrite grain from AC POX/CIL residue. Note: Optical microscope image taken after the SIMS depth profile analysis. The burned areas are the locations of the SIMS analysis at the oxide rim and the sulphide core.
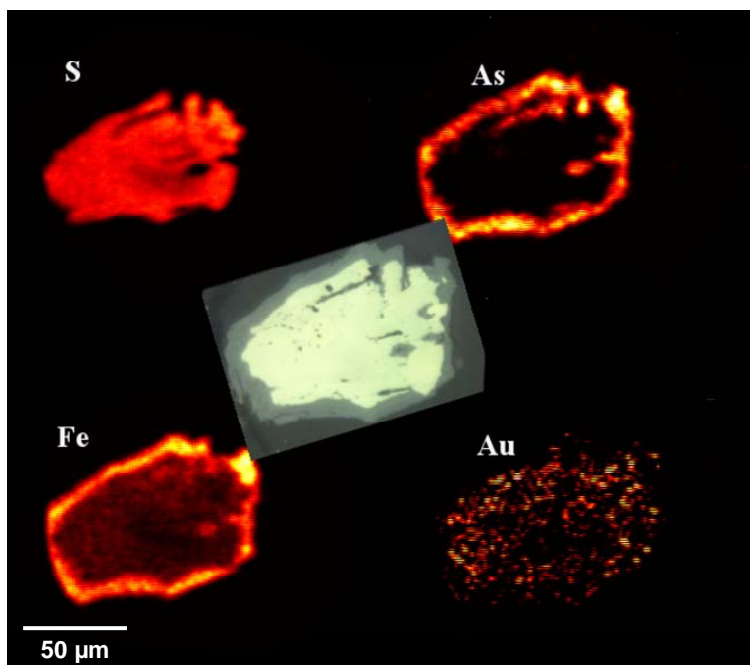
Figure 4. Gold distribution in a rimmed, partially oxidized pyrite grain from AC POX/CIL residue.

Application note 4

SURFACE CHEMISTRY IN RELATION TO FLOTATION SELECTIVITY: TOF-SIMS SURFACE ANALYSIS OF MINERAL PHASES FROM METALLURGICAL TESTS IN FLOTATION CIRCUITS

Request for analysis:

Metallurgical testing on a poly metallic Cu/Pb/Zn ore indicated either copper or lead activation of sphalerite in a Cu/Pb flotation test.

Objective:

To establish and rank the factors controlling sphalerite flotation.

Methodology:

The surface composition of individual sulphide grains was examined by ToF-SIMS and comparative analyses of the surface chemistries on sphalerite grains from the feed, concentrate (fast floating particles) and tails (rejected particles) were performed.

Results of the study:

Both Cu and Pb were identified on the surface of the sphalerite grains examined. Figure 1 shows a Zn map (secondary ion image of Zn in the concentrate sample) along with an example of the positive ion spectra from the surface of sphalerite grains from the concentrate and tail samples in the mass region of Pb (Pb isotopes 206, 207 and 208 are shown). The spectra clearly illustrate peak positions and windows identified for peak (mass) intensity measurements.

Activators species on surface of sphalerite:

Plots of normalized counts for Pb and Cu on the surface of the sphalerite grains from the feed, concentrate and tails samples (Figure 2) reveal that:

- The Pb content is not significantly different on the surface of the sphalerite grains in the concentrate relative to the feed.
- The Pb content on the surface of the sphalerite grains in the tail is less than that in the feed and concentrate.
- Cu is more abundant on the surface of the sphalerite grains in the concentrate relative to the feed.
- The range of Cu content in the tail samples is dispersed over the entire range defined by the feed and concentrate.

Depressant species on surface of sphalerite:

Potential depressant species: Ca does not appear to be favoured on either the concentrate or tails sphalerites; Mg appears to be slightly more abundant on the sphalerites in the tail samples (Figure 3).

Statistical comparison of sphalerite surface composition between concentrate and tails:

The data in the scatter plots is emphasized in the T-test comparison analyses (Figure 4). T-test values for mass species that exhibit statistically significant differences at 95% confidence level will be presented in a green colour on the T-test graph, while species with t-values that are not as statistically significant will be presented in a grey colour.

- The data show a strong statistically significant presence of Pb on the surface of the sphalerite from the concentrate relative to the tails.
- Cu does occur on the surface of the sphalerite, however the strength of the presence is markedly less than that for Pb.

Relevant mechanisms based on surface analyses:

- Analyses indicate that Cu and Pb are involved in the activation of the sphalerite grains.
- The greater discrimination in the Pb signal between the concentrate and tail samples suggests that Pb may be dominant in the inadvertent activation of sphalerite. Moreover, the distribution of Pb on sphalerite surfaces in the concentrate is limited in range, relative to Cu, suggesting it may be a more consistent activator.
- Depressant species (for example Ca and Mg) are not strongly discriminatory between concentrate and tail samples. This indicates that depressant species are not likely controlling the flotation characteristics of the sphalerite. The slight enrichment of Mg in the tail samples suggests that the sphalerite surfaces may contain a greater proportion of adsorbed gangue material, thus inhibiting activation by Cu or Pb.

Cu *versus* Pb activation:

- Pb activation; given the similar proportion of Pb on sphalerites in the feed and concentrate suggests that adsorption likely occurred during grinding.
- Cu activation; given the significant difference between the proportion in the feed and concentrate, adsorption likely occurred during conditioning and flotation.

Major findings:

- Pb is the predominant activating agent.
- Activation of sphalerite by Pb occurs during grinding.
- Cu is mostly a secondary activation agent.
- Activation by Cu likely occurs during conditioning and flotation.
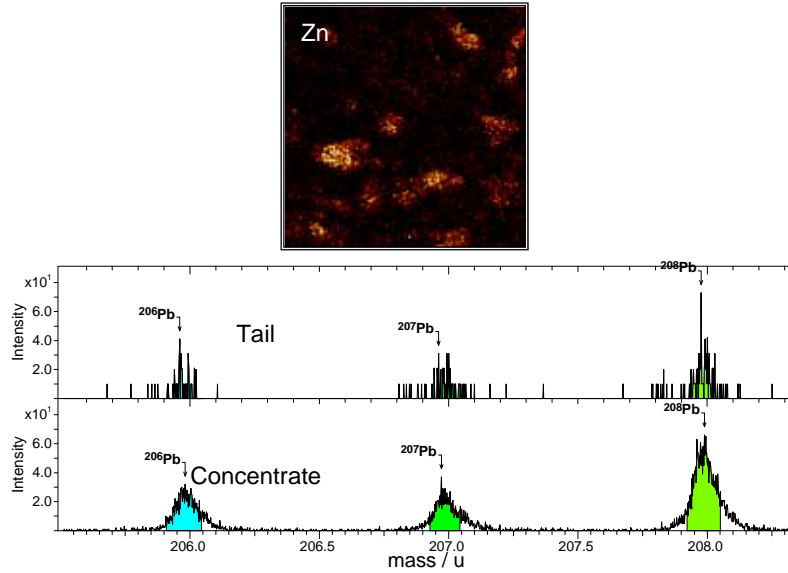- No significant role of common depressants (Ca, Mg) observed.

Figure 1. Secondary ion image (map) for Zn in the concentrate. Area in the image is 300x300μm. Positive ion spectra from the surface of sphalerite in the concentrate and tails in the mass region for Pb (205.5 to 208.5 amu). The data in the spectra are normalized by total ion intensity.
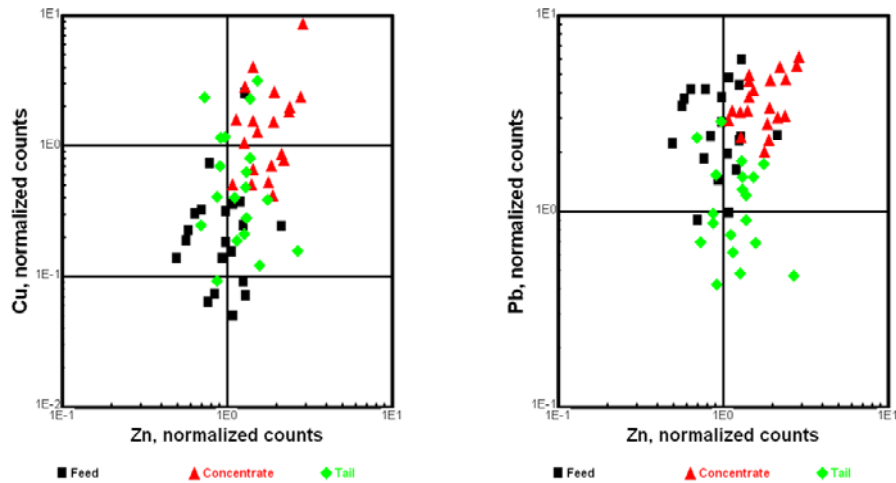

Figure 2. Scatter plots showing the distribution for Pb and Cu *versus* Zn on sphalerite surfaces in the feed, concentrate and tail. The data are normalized by total ion intensity and area.
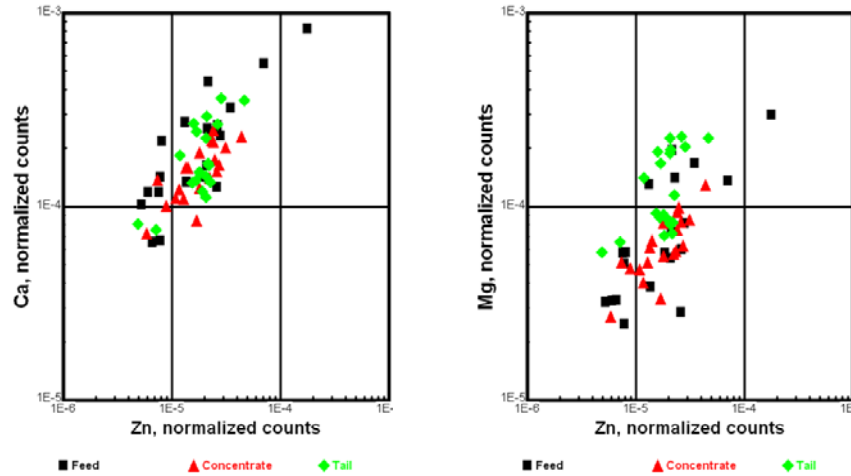
ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519-661-2173 · FAX: 519-661-3709 · WWW.SURFACESCIENCEWESTERN.COM
30

Figure 3. Scatter plots showing the distribution for Ca and Mg *versus* Zn on sphalerite surfaces in the feed, concentrate and tail. The data are normalized by total ion intensity
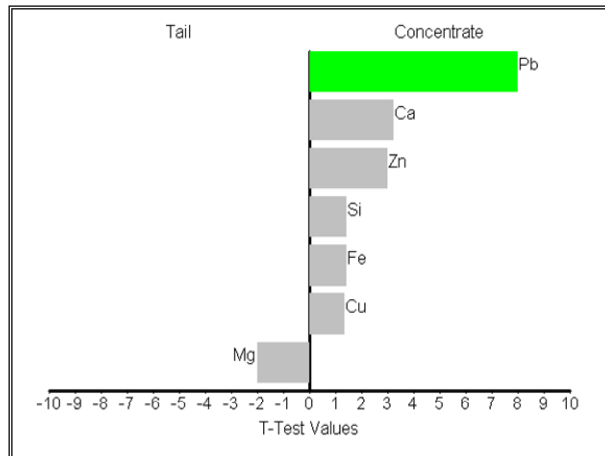

Figure 4. T-test statistical analyses for data from sphalerite grain surfaces from concentrate/tail flotation tests. All data in the tests were normalized by the total ion intensity and area. The surface components in bright green are considered to be statistically different between the concentrate and tail samples

Application note 5

CHARACTERIZATION OF PASSIVATING COATINGS ON GOLD GRAINS FROM CARBON IN LEACH (CIL) RESIDUE SAMPLE BY TOF-SIMS

Request for analysis:

Recovery efficiency during the leaching process of an ore sample containing free gold is low. Examine the surface of unleached free gold grains found in the CIL residue tails for the presence of passivating coatings.

Objectives:

1. To establish the presence of passivating coatings on the surface of free gold grains from CIL residue that could impede the process of cyanide leaching.
2. To gain insight on the origin of coatings.

Methodology:

Individual free gold grains were selected under the optical stereo microscope from both feed and CIL residue samples and mounted on an indium substrate. Surface spectra and images of selected surface species present on these gold grains were collected using an ION-TOF ToF-SIMS IV instrument. Depth profiles through the surface coating on selected gold grains were produced using a combination of two separate ion sources: a Cs^+ sputter ion gun and $Bi3^+$ ion cluster analytical source.

Results of the study:

Comparative ToF-SIMS surface analysis between gold grains from the feed and cyanide leach residue samples showed significant differences in the amount of surface silver (Figure 1).

Examples of free gold grains from the CIL residue sample are presented in Figure 2. Images of surface species on the gold grains before (as received) and after sputtering of their surfaces with the Cs^+ sputter ion gun are presented on Figures 3 and 5. The relative change of surface Au and Ag on these grains is presented on Figures 4 and 6.

Major findings:

 i. The unleached free and exposed gold grains from the CIL residue samples exhibit passivation surface coatings of Ag that slow down and inhibit further leaching of the grains in a cyanide solution.
 ii. These coatings are related to the extraction process and not to the ore mineralogy of the feed sample. Comparison between the surface silver present on gold grains from feed and residue samples indicates that the silver passivation layer is developed on the surface of the gold grains during the leaching process.
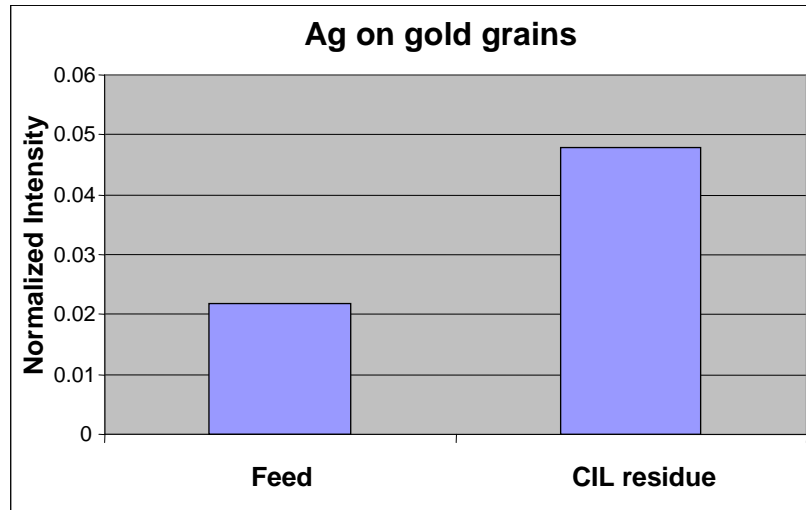

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519–661–2173 · FAX: 519–661–3709 · WWW.SURFACESCIENCEWESTERN.COM
32

Figure 1. Surface Ag measured on free gold grains from a feed and CIL residue by ToF-SIMS
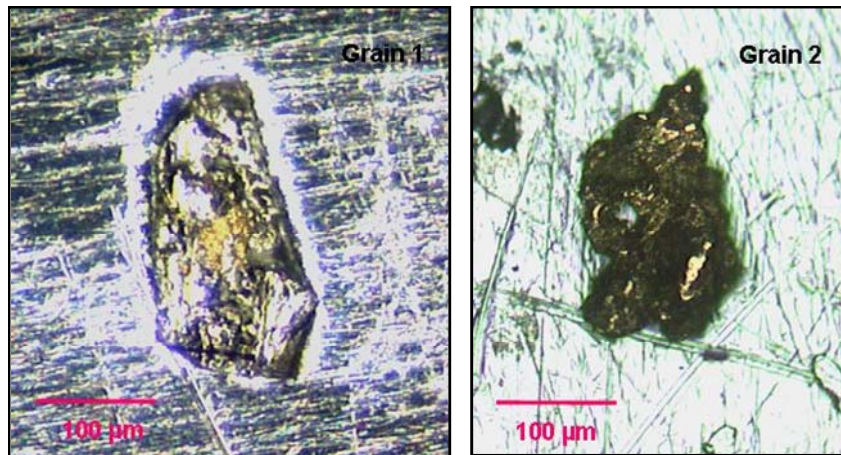

Figure 2. Optical microscope images of gold grains from CIL residue examined by ToF-SIMS. Grain #1 is partially locked in a sulphide grain.
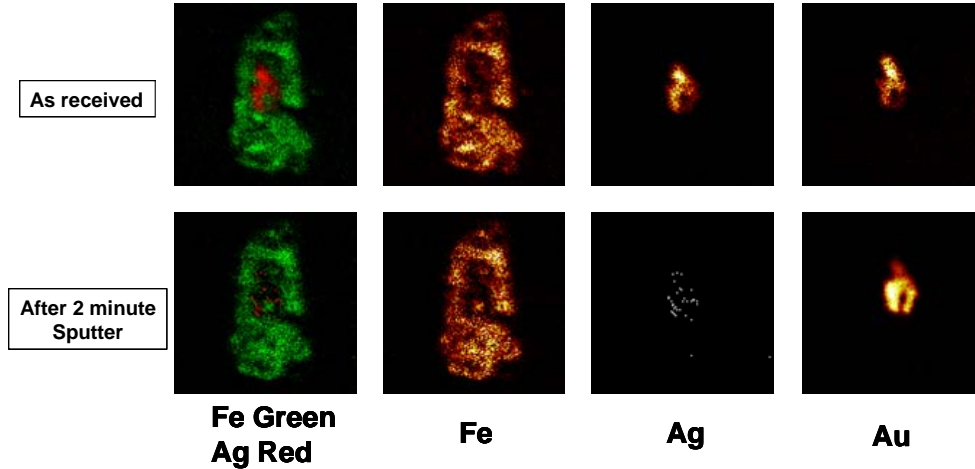
Passivation of Au Gold grain surface

As received

After 2 minute Sputter

Fe Green
Ag Red
Fe
Ag
Au

Figure 3. ToF-SIMS ion maps for selected surface species detected on gold grain #1 before (original "as received" surface) and after removing the upper surface layer by sputtering with a Cs$^+$ ion source. This gold grain was attached to a pyrite grain. The ion maps show that the surface Ag coating is present on the gold grain, but not on the pyrite mineral phase.
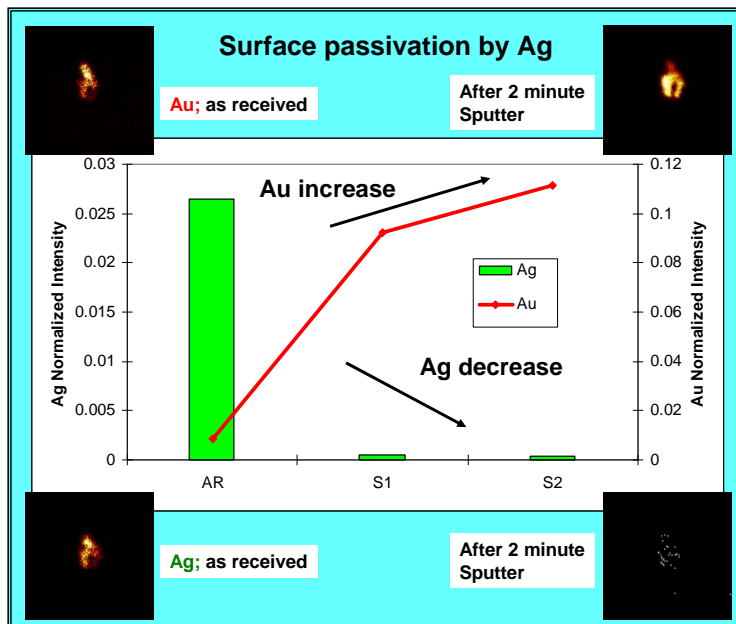

Figure 4. Relative change of surface Au and Ag measured by TOF-SIMS on gold grain #1 before and after sputtering with Cs ion source

Passivation of gold grain surface

Silver on Au grain
As received

Silver on Au grain
After 90 Cs Sputter

Silver on Au grain
After 180 Cs Sputter

Gold on Au grain
As received

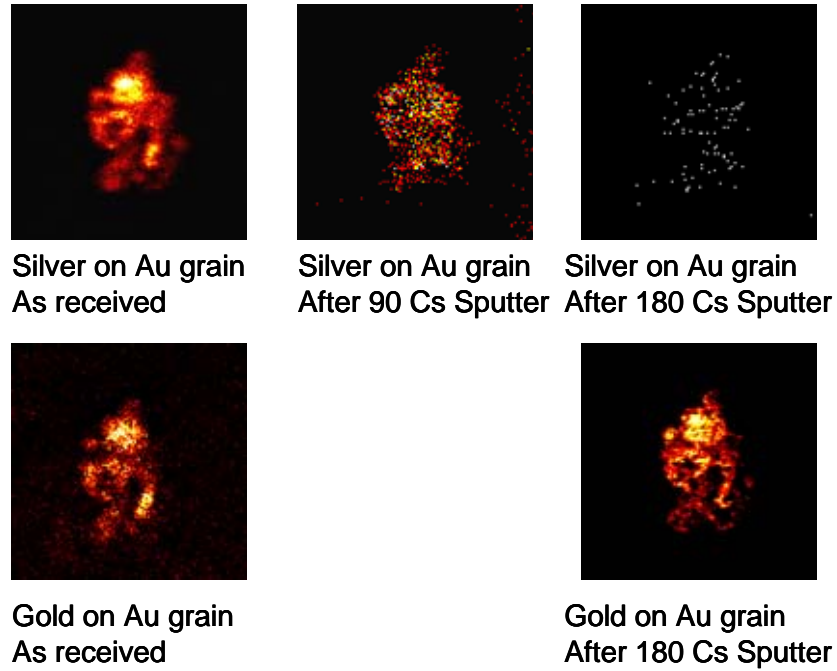Gold on Au grain
After 180 Cs Sputter

Figure 5. ToF-SIMS ion maps for selected surface species detected on gold grain #2 before (original "as received" surface) and after removing the upper surface layer in two consecutive sputter steps with a Cs$^+$ ion source.
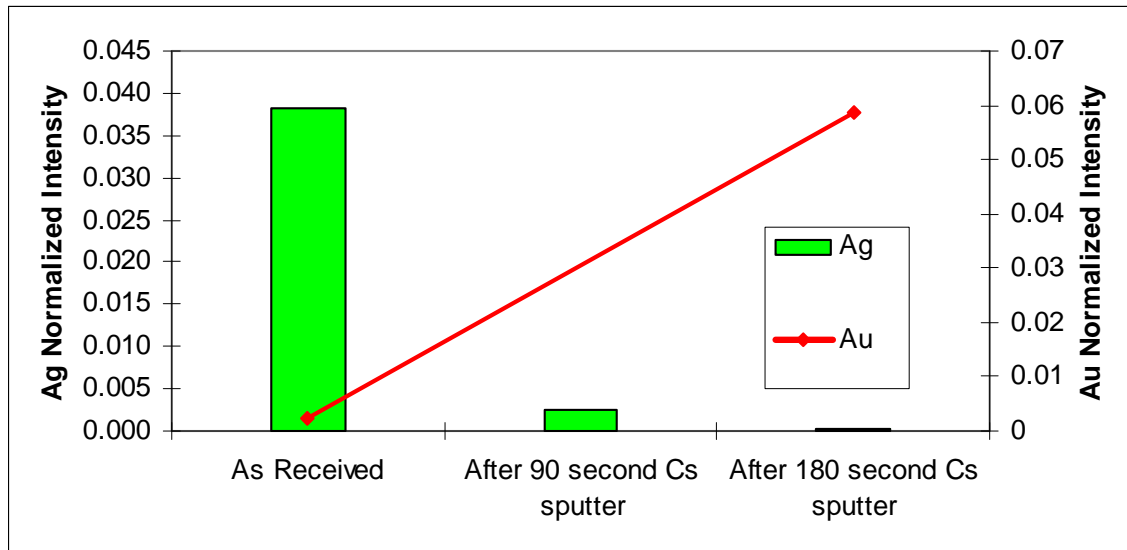

Figure 6. Relative change of surface Au and Ag measured by ToF-SIMS on gold grain #2 before and after sputtering with Cs ion source

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519–661–2173 · FAX: 519–661–3709 · WWW.SURFACESCIENCEWESTERN.COM
35

Application note 6

FEED ORE CHARACTERIZATION: ORE CHEMICAL ACTIVITY TESTING BY ToF-SIMS

Request for analysis:

Many ore deposits have varying degrees of primary and/or secondary copper minerals, the presence of which can strongly affect the flotation behaviour of the ore. The ToF-SIMS technology will be used as a predictive tool in providing an estimate of the ore capacity to undergo "inadvertent Cu activation" during the grinding process. This information would complement that gained through automated mineralogy and help complete the information package needed to better guide the metallurgist in the pre-selection of a suitable flotation process.

Objectives:

1.   To establish the degree of "chemical activity" of the ore during the grinding process which is determined by chemical transfer of a specific ion (Cu^+) between mineral phases present in the ore.
 2.   To provide a data comparison with the ore sample library which reflects the mineralogical variability of ore types from different deposits. The data base covers a variety of poly-metallic ore deposits (for example skarn, hypogene, supergene) with a large dynamic range of chemical activity.

Methodology:

To establish the database of surface chemical signatures for different minerals and ore deposits, a standard protocol for sample preparation and subsequent surface analysis was developed. The test protocol includes grinding of a small amount of a feed ore sample under standardized conditions. A two-chamber ball mill was specifically designed, commissioned and tested for determining surface reactivity of ore minerals under specified conditions. The surface modification (Cu activation) of specific mineral phases placed in a separate chamber in the ball mill (where they interact with the pulp only) is characterized by ToF-SIMS surface analysis. The development of a data base (library) with Cu bearing poly-metallic ores using this test has shown the measured concentration of Cu transfer on mineral surfaces extends over a several orders of magnitude.

Results of the study:

The test data in Figure 1 represent a summary of chemical reactivity tests from a number of ore samples with varying mineralogies in order to investigate the relative degree of Cu transfer from the ore minerals to the specimen pyrite surface during the grinding process. The established chemical reactivity for ore samples #1 and #2 is compared with library data set (ores #3-#13). For comparative analyses a baseline of Cu transfer was established by performing a number of tests under standard operating conditions (SOP) with pyrite and sand.

The variability of Cu on pyrite for the entire ore data set illustrates that the Cu content on mineral surfaces extends over a several orders of magnitude dynamic range. It is to be noted that some of the highest Cu loadings occurred when "non-Cu" bearing ores were milled. For example, ore samples #1 and #2 are from the same mine but exhibit significantly different flotation behaviour (see discussion below).
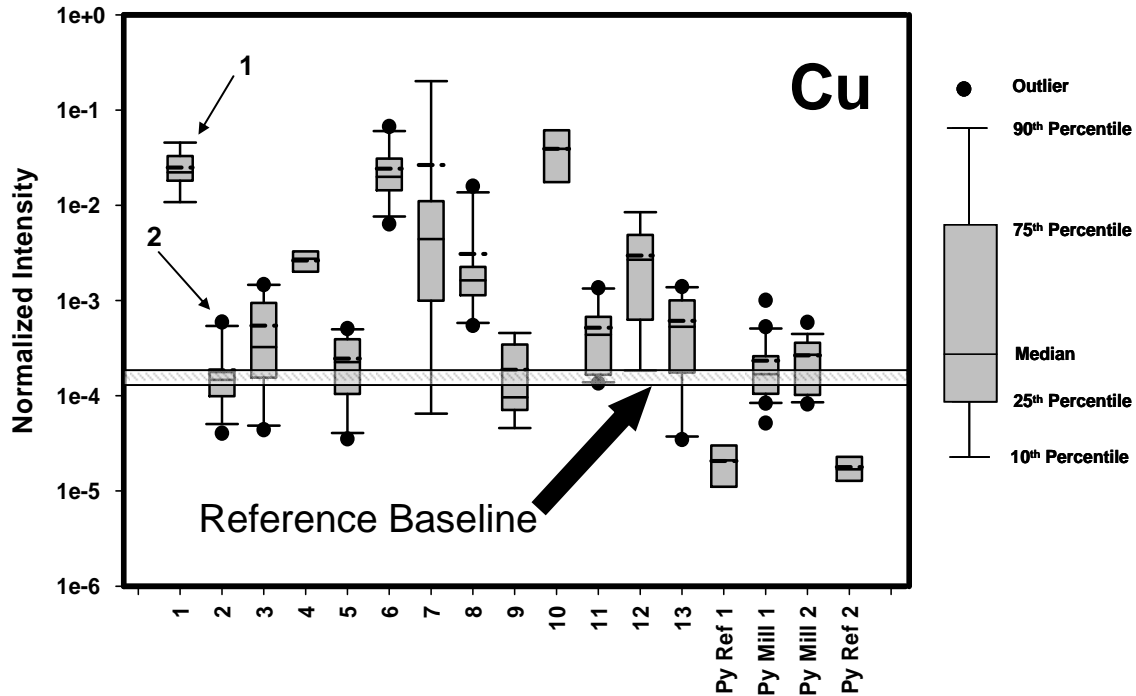

Figure 1: Vertical box plots showing ToF-SIMS data for Cu as measured on the surface of pyrite specimen grains from the chemical reactivity test for 13 different ore specimens The data for the Py Ref represents the normalized Cu intensity on the as received pyrite reference samples and for the Py mill samples for Cu on the surface of the pyrite after milling with sand. The horizontal line through the plot is the estimated baseline for the testing program. Ore numbers 1 and 2 are indicated by the arrows.

The following is a discussion regarding observations made regarding Cu transfer in relation to the mineralogical make-up of the tested ore samples. Figure 2 shows the variability of Cu surface loading on pyrite for various mineralogical zones in a single deposit. The samples show a distinct dependence of Cu loading with respect to ore type; increasing Cu content on pyrite surfaces in the following sample sequence: skarn, hypogene, supergene. This trend correlates quite well with the increase in secondary minerals in the hypogene and supergene sample (Figure 2 inset) whose solubility is greater than the primary minerals found in the skarn. The result is an increase in solution Cu during the grind resulting in a transfer to the surface of the pyrite. The difference between the copper loading from the skarn and hypogene samples may also be related to different pH levels developed in the mill in response to the different mineralogy of the samples.

ISO 9001:2015 REGISTERED

THE UNIVERSITY OF WESTERN ONTARIO · WESTERN SCIENCE CENTRE · LONDON, ONTARIO N6A 5B7 CANADA
PHONE: 519–661–2173 · FAX: 519–661–3709 · WWW.SURFACESCIENCEWESTERN.COM
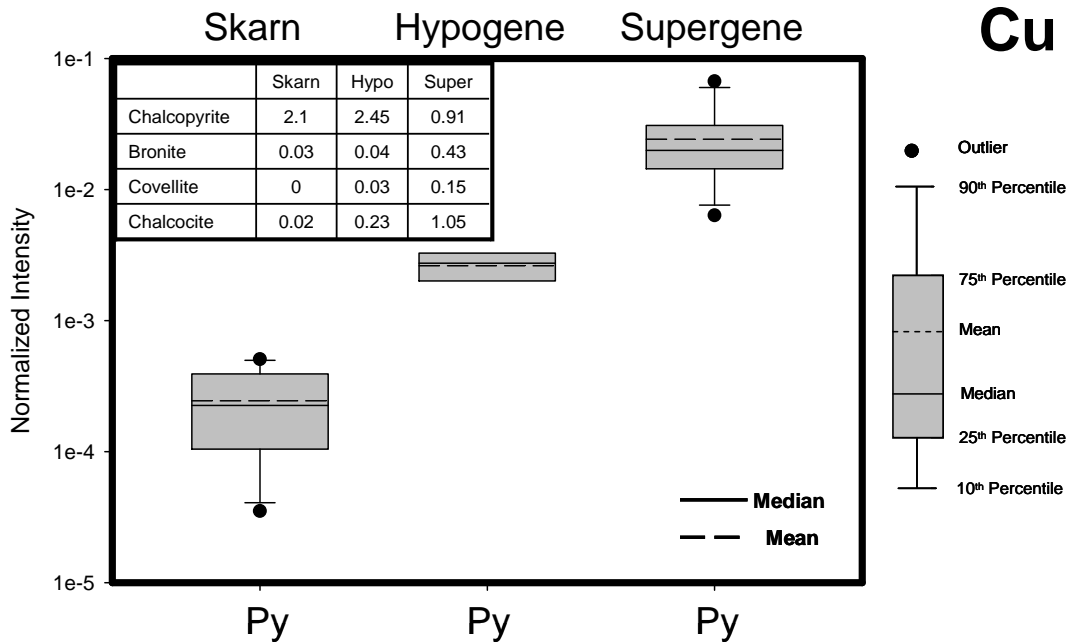37

Figure 2: Skarn, Hypogene, Supergene ore types (ores #5, #4 and #6 in Figure 1): Vertical box plots showing ToF-SIMS data for Cu as measured on the surface of pyrite (Py) specimen grains from the chemical reactivity test.

Major findings:

As noted above, there is a significant difference in Cu loading on the pyrite surfaces between ore samples #1 and #2 (Figure 1 and 3). Ore #1 (Figure 3) contains minimal copper mineralization (~0.05%Cu) however, in practice this ore exhibited strong inadvertent sphalerite flotation and metallurgical testwork elsewhere had indicated that copper activation was indeed playing a role. By comparison, Ore #2 from the same deposit with similar copper mineralization (0.05% Cu) yielded excellent flotation separation; no inadvertent sphalerite activation. The flotation behaviour of Ore #1 suggests that the presence of minute amounts of highly soluble copper minerals (insufficient, perhaps, to be detected in the QEMSCAN analysis) can have a substantial effect on flotation chemistry. The observed flotation response would not have been predicted from mineralogical analysis alone, and demonstrates the value of conducting this test in parallel with automated mineralogy as a preliminary ore assessment tool ahead of flotation testing.
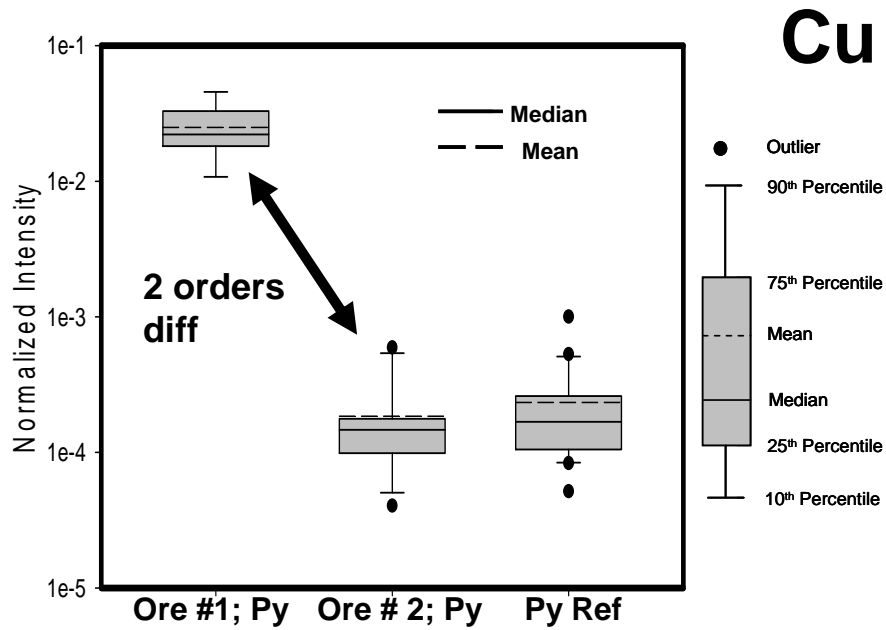
Figure 3: Ore numbers 1 and 2 from Figure 1: Vertical box plots showing ToF-SIMS data for Cu as measured on the surface of pyrite (Py) specimen grains from the chemical reactivity test. Also included is the pyrite reference sample.

CONTACT INFORMATION

	Dr. Brian Hart	Dr. Stamen Dimov
E-mail	bhart@uwo.ca	sdimov@uwo.ca

The University of Western Ontario
Surface Science Western
London, On N6A 5B7 CANADA
Tel: 519-661-2173
Fax: 519-661-3709