

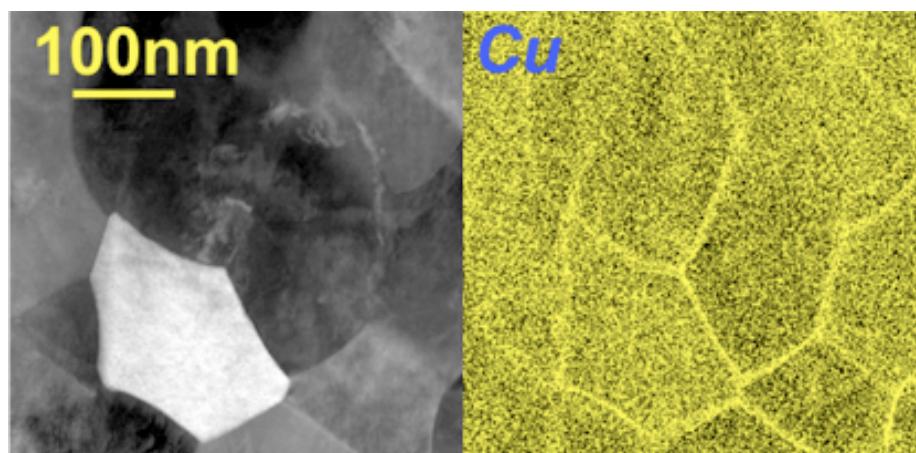
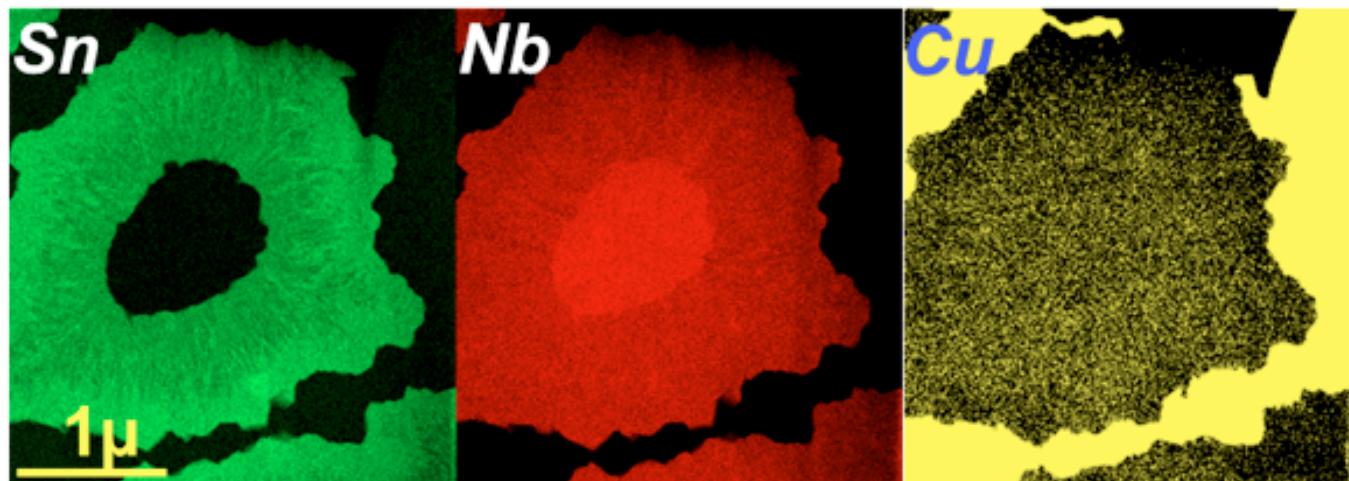
# SSOM

Swiss Society for Optics and Microscopy

Société Suisse pour l'Optique et la Microscopie

Schweizerische Gesellschaft für Optik und Mikroskopie

Mitteilungsblatt / Bulletin d'information 1+2 / 2013



**ChemiSTEM element maps**

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### Courses and Conferences 2013

*[www.ssom.ch](http://www.ssom.ch)  
[www.bmpn.ch/activities.php](http://www.bmpn.ch/activities.php)  
[www.opteth.ethz.ch/news/index](http://www.opteth.ethz.ch/news/index)  
<http://photonics.epfl.ch/>  
[www.swisslaser.net/](http://www.swisslaser.net/)  
[www.myeos.org/events](http://www.myeos.org/events)*

### SSOM Anmeldeformular / Demande d'adhésion

## *From the President*

Dear members,

this issue is governed by the priming for the General Assembly that will take place on 15 November 2013 at Biketec AG in Hüttenwil. Behind Biketec is a product called Flyer. Flyer is an e-bike. The history, the philosophy and the marketing of selling this product might be an example, how modern industry could get in accord with environmental aspects.

Our assembly will lead us through the financial topic to the three scientific prizes. If you know anybody in your surroundings that would qualify for one of the prizes, please encourage the person to apply for it.

Methodologies are advancing all the time. Why the combination of a Scanning Transmission Electron Microscope (STEM) with a new generation of x-ray detector can be a fruitful pairing is explained in our centerfold and can be seen on the cover of this issue.

I wish you a pleasant lecture.

A handwritten signature in blue ink, appearing to read "M. Dürrenberger".

Markus Dürrenberger  
President SSOM

# SSOM financials 2012

## Kassabuch Abschluss nach Kostenart per 31.12.2012

		<b>Belastung</b>	<b>Gutschrift</b>	<b>Saldo</b>
Ausgaben und Einnahmen für	Adm-Sekret	20'453.94	9'974.54	-10'479.40
Ausgaben und Einnahmen für	Beitr. Org.	9'323.22		-9'323.22
Ausgaben und Einnahmen für	Bulletin	6'429.60	4'000.00	-2'429.60
Ausgaben und Einnahmen für	Delegation	7'681.00	6'457.60	-1'223.40
Ausgaben und Einnahmen für	Fonds	84'388.00	88'050.00	3'662.00
Ausgaben und Einnahmen für	Mitglieder		18'036.74	18'036.74
Ausgaben und Einnahmen für	Nachwuchsf.	2'500.00	5'200.00	2'700.00
Ausgaben und Einnahmen für	Sponsoring	6'511.20	3'000.00	-3'511.20
Ausgaben und Einnahmen für	Zinsen		1'139.60	1'139.60
Total		137'286.96	135'858.48	-1'428.48
		-	137'286.96	
		<b>Verlust 2012</b>	<b>-1'428.48</b>	

Basel, 18.Januar 2013

Der Kassier Gianni Morson

Der Präsident Markus Dürrenberger




## Bilanz per 31.12.2012

<b>AKTIVEN</b>	Engelbergkonto UBS	12'089.34	
	Sparkonto ZKB	28'114.20	
	Kontokorrent CS	23'630.84	
	PC SSOM	1'921.56	
	Verrechnungssteuerguthaben 2012	375.20	
	Fonds Ausgewogen ZKB	88'050.00	
<b>PASSIVEN</b>	Eigenkapital 1.1.2012	0.00	155'609.62
	Verlust Jahresrechnung 2012	0.00	- 1428.48
		<b>154'181.14</b>	<b>154'181.14</b>

Der Kassier

Der Präsident

## Revisorenbericht

Die Rechnung der SSOM für das Jahr 2012 wurde von den Revisoren geprüft und in allen Belangen als richtig befunden. Die sorgfältige und korrekte Arbeit des Kassiers wird bestens verdankt. Die Revisoren empfehlen der Mitgliederversammlung die Rechnung zu genehmigen und dem Kassier Entlastung zu erteilen.

Basel, 18. Januar 2013

sig. Marcel Düggelin

sig. Victor Colombo

## Mitgliederzahlen per 31.12.2012

Einzelmitglieder	inkl. 10 Freimitglieder	313
Delegierte von Total 60 Kollektivmitgliedern		232
<b>Total</b>		<b>545</b>

# *Recent EDS instrumental advances in STEM: from principles to applications*

## **Introduction**

The electron microscopy community makes a wide use of Energy Dispersive X-ray Spectrometry (EDS) for elemental mapping in STEM. This technique was however long reckoned slow, dozens of minutes if not an hour, to collect enough information for one map.

Recent instrumental advances have dramatically changed the situation, reducing the acquisition time to minutes, improving the maps quality and boosting their spatial resolution beyond the nanometer scale or even down to the single atom column in crystalline materials. This opens the way to true composition mapping – no longer raw counts mapping –, interface quantitative analysis and soon EDS tomography to extract chemical composition of buried nano-objects.

## **The major progress**

### **Step 1: Improving the X-Ray production with better electron source**

A higher electron probe current or a thicker sample is required to produce more X-rays. However, increasing thickness rapidly degrade the spatial resolution due to beam spreading through the sample. Even the effort is rather opposite to produce thinner samples (a very few tens of nm) for imaging atom columns or interfaces seen edge-on. The first key point to get intense electron probes is a high gun brightness  $\beta$  – not the total emitted electron current – that links the current  $i$  emitted by a source of diameter  $d$  within a solid angle corresponding to a cone of semi-angle  $\alpha$ : 
$$\beta = 4i/\pi^2 d^2 \alpha^2$$

From optics laws, this quantity is constant throughout the whole path of electrons as long as no absorbing media is met (lenses, apertures do not change it!). So the same equation holds when  $i$ ,  $d$  and  $\alpha$  are considered at any level between the gun and the impact point on the sample. If the operator wishes to increase the probe current  $i$  and decrease its diameter  $d$ , it leads to an increase the convergence angle  $\alpha$ ... that rapidly blurs the probe due to spherical aberration.

Since the eighties, analytical TEM/STEMs use mainly "field emission" Schottky guns (however not really *field emission!*) which bring  $\sim 4 \cdot 10^8 \text{ A/cm}^2\text{sr}$  at 200kV. FEI made a step forward four years ago by redesigning its Schottky gun and boosting the brightness of the X-FEG that fits the Osiris and Titan microscopes line ( $2 \cdot 10^9 \text{ A/cm}^2\text{sr}$  at 300 kV). At the same time, JEOL chose to develop a more stable cold field emission gun for its ARM200F analytical microscope.

One should note that while gun brightness, spatial resolution and possibly irradiation damage increases with accelerating voltage, the ionization cross-section decreases. Thus, one should seek the optimum accelerating voltage for each material and sample thickness.

## **Step 2: Improving the X-Ray production with better probe forming optics**

A spherical aberration corrector for illumination is also present on the Titan, ARM200 and H-2700. It helps keeping thin probes while the probe current, and thus the convergence angle  $\alpha$ , is increased. Nowadays HRSTEM resolution is better than 0.1nm at some tens of pA probe current and 0.2nm at 2nA ( $0.1\text{MW}/\text{cm}^2$ !). The chromatic aberration and often the sample irradiation damage are now the limiting factors to get still higher beam current in thin probes.

## **Step 3: improving the X-ray collection**

The maximum counting rate of EDS systems has increased during the last decade by a factor of at least 20 times by replacing the Si(Li) detectors (Silicon Lithium drifted) by SDD detectors (Silicon Drift Detectors). The Si(Li) uses a thick (~3.5mm) Si crystal doped under its entrance window by Li diffusion. The anode collecting the electrons resulting from the conversion of X-ray energy in electron-holes pairs has the same area as the crystal active area. That leads to a high electrical capacitance and limits the counting rate to some 3000-8000 photons/s depending on the expected energy resolution. The SDD is built on a thinner Si crystal (~0.5mm) and electrons are driven toward a small anode by a set of electrodes resulting in a much smaller capacitance that boost the maximum counting rate to at least 50'000 or even more than 100'000 counts/s. However the highest count rates are never attained – even with the brightest electron guns – with probes used to tackle the nm and very thin samples used for atom column EDS mapping. Moreover, the SDD detector becomes partly transparent to X-rays above 10 keV and its detection efficiency drops by 50% at 20 keV compared to the Si(Li) for instance.

The main weakness of the EDS system remains the poor collection efficiency of X-ray photons. Emission of characteristic X-rays by ionized atoms is isotropic but the active head of the detector has to be small to fit the narrow space close to the objective lens pole-pieces.

EDS suppliers did efforts to increase the collection solid angle by using detectors of larger area at the expense of a slight loss of energy resolution. Efficient in the SEM, this solution has only marginal benefits in the TEM, the larger the diode the farther its position. For instance, FEI uses in Osiris and Titan the Super-Twin pole pieces (pole-piece gap 5.4mm, Cs coefficient of 1.2mm) to keep a 0.23nm HRTEM Scherzer resolution at 300kV (without image corrector) and 0.07 nm in STEM mode with a probe Cs-corrector while retaining 40° sample tilt capability. According to FEI data, the Titan can fit lateral detectors with a collecting angle of 0.13sr, though Virginia Tech claims 0.3sr/EDAX.

In 2009, FEI launched the *Super-X* concept with four built-in windowless diodes, 30mm<sup>2</sup> each, arranged around the pole-pieces and protected by shutters [1]. The total collection and take-off angles comes to 0.9sr/22° and 0.7sr/18° on Osiris and Titan respectively. The diode window removal increases that 7 or 5 times geometrical gain by another 2 times for light elements. This better efficiency improves the elemental maps quality (better statistical relevancy) at constant acquisition time or reduces the acquisition time and the irradiation dose at constant map quality.

More recently Bruker developed the *QUANTAX 400-STEM* for Libra Zeiss microscopes that gather four windowless 30mm<sup>2</sup> diodes on one side of the goniometer, two being

above the sample and the two others below. They cover together 1sr at the large 25° take-off angle. Unfortunately, this detector requires a larger EDS port than usual and cannot fit into other microscopes without special order. JEOL developed the lateral *Centurio* detector given as a 100mm<sup>2</sup> windowless (single?) diode reaching 0.98sr on the ARM200F (no details available).

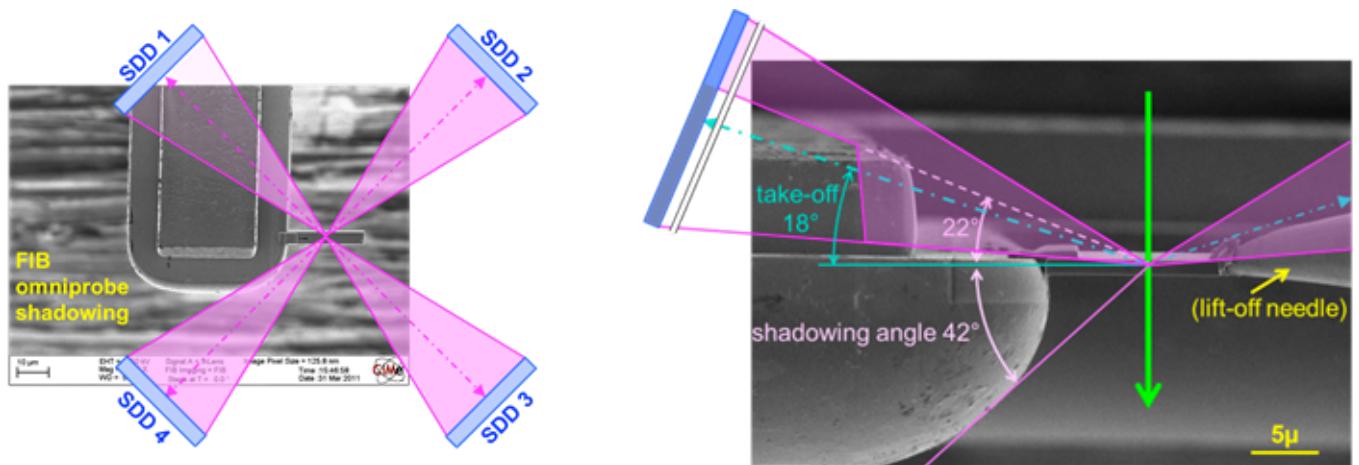
The symmetrical distribution of the Super-X diodes around the microscope column axis allows quite large sample tilt (~20°...30°) without losing too much EDS signal. This situation is favorable to tomography as well as interface studies in materials where they lay normal to the thin foil plane. However looking in more detail, small sample tilt or bending may bring some diodes in (partial) shadow (fig.1). If this effect is partly offset by the better exposition of the other diodes and if it sounds negligible for element distribution maps, it may nevertheless bias quantitative analysis owing to the energy dependent absorption of X-rays at the edge of the shadowing feature (roughness or bending of the sample, edge of the sample holder).

### **Example 1: Improving the map quality (M. Cantoni, EPF-Lausanne)**

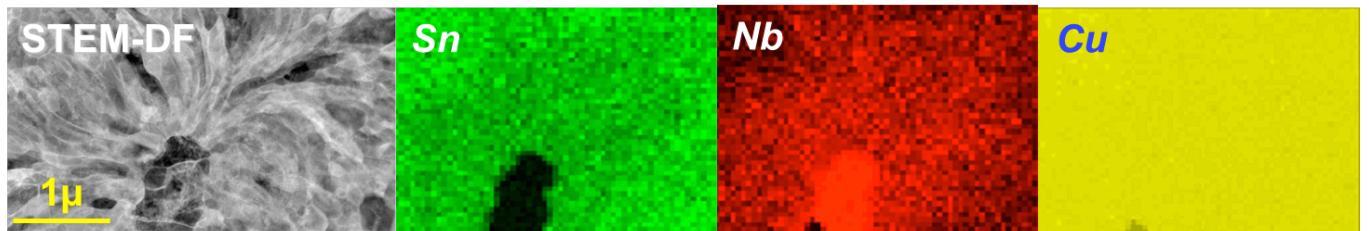
Marco Cantoni did a comparison of Nb<sub>3</sub>Sn superconductor maps obtained on a CM300 fitted with 10mm<sup>2</sup> Si(Li) detector (fig. 2) and ChemiSTEM (fig. 3). The material is polycrystalline and contains Cu that is expected to segregate to grain boundaries [2].

### **Example 2a: Improving quantitative analysis (P.A. Buffat, IC-EM AGH, Krakow)**

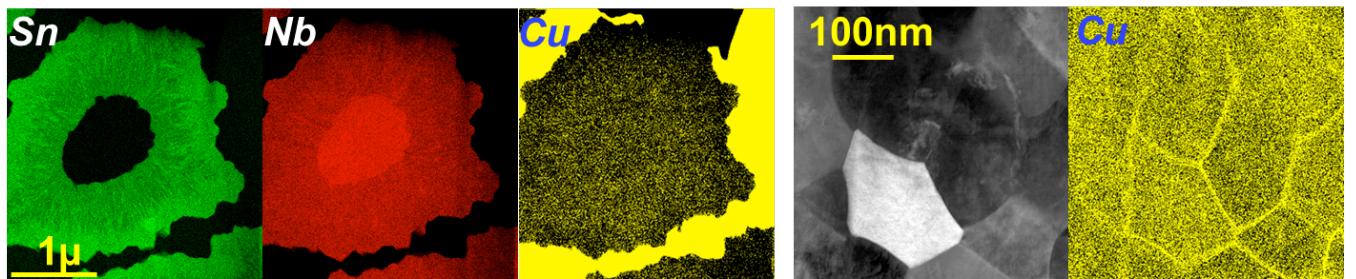
The mechanical properties of nickel-base superalloys for high temperature and extreme environment are improved by the dispersion of nanoprecipitates  $\gamma'$  (type Ni<sub>3</sub>(Al,Ti) fcc ordered) and  $\gamma''$  (type Ni<sub>3</sub>Nb tetragonal bc ordered) in the matrix  $\gamma$  (solid solution Ni-Cr-Fe fcc disordered). TEM dark field and HAADF/STEM give ambiguous images and only EDS maps bring the phase distribution using Al and Nb as selective elements (fig. 4). The nanoprecipitates are buried in the matrix. However several arguments (shape of profile, atom resolution EDS maps of different thicknesses samples) show that Fe and Cr are absent from the nanoprecipitates. This led to write a short routine to remove the contribution of the surrounding matrix and to extract the true  $\gamma'$  and  $\gamma''$  composition [3].



**Figure 1:** At least one diode is in the shade of the Omniprobe grid when the FIB lamella is mounted at the mechanically safe position. Left: view from top. Right: lateral view.



**Figure 2:** DF/STEM and element maps from CM300 (300kV, 1nA, 128ms dwell time, full map 128x98 pixels, 1 hour). The Cu map does not exhibit any structure.

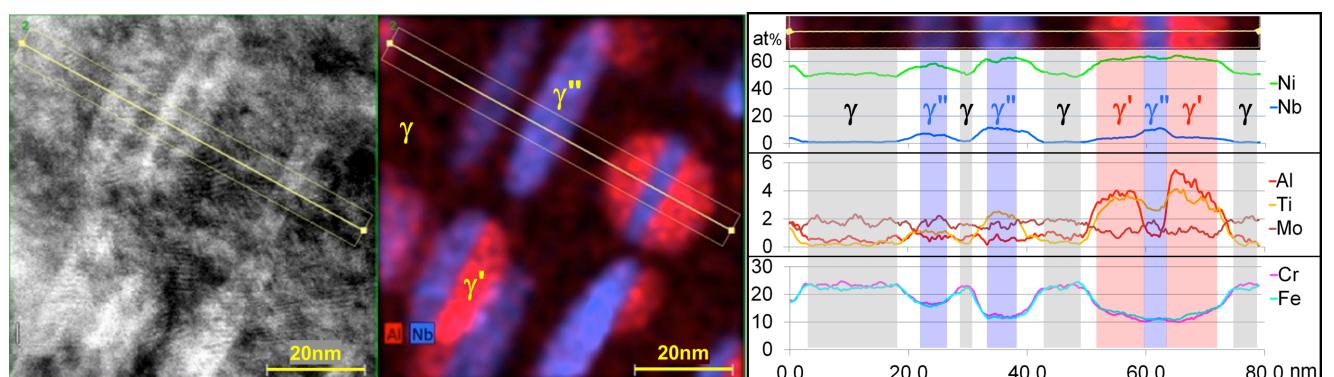


**Figure 3:** ChemiSTEM element maps from Osiris (200kV, 2.5nA, 4ms dwell time, 400x400 pixels, 10min). Left: Overview of a wire. Right: Cu segregation at grain boundaries.

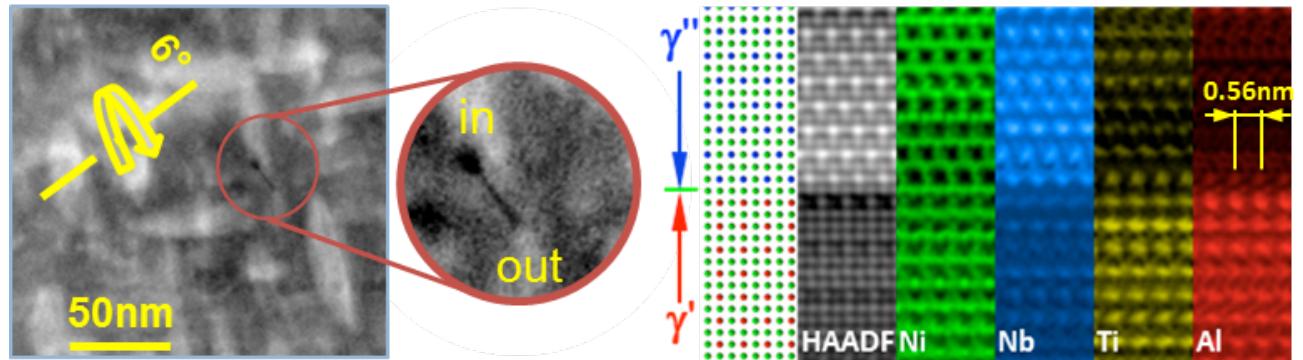
### Example 2b: Interface structure using maps at atom column resolution

EDS maps showing distinct atom columns of alloys appear significantly more delicate to obtain than for ceramics (often perovskites) or semiconductors. On the hand sample preparation is more tricky to get foils only a few tens of nanometers thick and reasonably flat. On the other hand, alloys seem more prone to electron beam damaging (fig. 5).

The original EDS map of the  $\gamma'/\gamma''[010]$  interface (fig. 6) was averaged over the equivalent vertical rows to filter noise while retaining the composition change across that interface. The interface plane is a pure Ni plane. The  $\gamma'$  Al/Ti columns are facing the closest  $\gamma''$  Nb columns across the interface. The first plane in  $\gamma'$  along the interface exhibits a dark HAADF contrast. It corresponds to a slight reinforcement of the Al atom columns contrast suggesting that Al segregation occurs. Geng has observed a similar effect using atom probe tomography [4].



**Figure 4:** Left: HAADF/STEM. Centre: phase distribution Al (red) and Nb (blue) raw counts maps differentiating the 3 phases. For the particular heat treatment used here,  $\gamma'$  and  $\gamma''$  join on a  $(001)_{\gamma/\gamma''}$  plane to form so called "compact morphology particles". Right: the linescan shows that unexpectedly Nb and Ti are present in  $\gamma'$  and  $\gamma''$  respectively.(200kV, 430pA, 10mrad, 17min)



**Figure 5:** Hole drilled by a 0.1nm probe standing ~2s (300kV, 120pA, 25mrad,).

**Figure 6:** Model, HAADF and element maps of a  $\gamma'/\gamma''$  interface (200kV, 220pA, 16mrad, 330s).

## Conclusion

The main limits of EDS using ChemiSTEM or equivalent system is nowadays the resistance of the sample to electron irradiation and the need for better quantification software, in particular for standardless analysis (better Cliff-Lorimer factors or moving to the zeta method [5]).

Nowadays, the EDS mapping competes or even supersedes the EFTEM and ELSI mapping for elemental analysis with exception of the ultra-light elements (Li, Be, B). Moreover it does not come up against unfavourable or delayed edges (Al in the example above for instance) and is significantly more accessible to a wide range of users.

## References

- [1] P. Schlossmacher et al., Microscopy and Analysis 24 7 (2010) S5-S8 (EU)
- [2] V. Abächerli et al. IEEE Trans. Appl. Superconductivity 15 2 (2005) 3482-3485
- [3] P.A. Buffat et al., Scripta Mater. (submitted)
- [4] W.T. Geng, Phys. Rev. B76 (2007) 224102
- [5] Watanabe M. and Williams D., J. Microscopy, 221 89–109 (2006)

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# *SSOM General Assembly*

## **Date**

15 November 2013

## **Location :**

Biketec AG  
Schwende 1  
CH - 4950 Huttwil / BE  
<http://www.biketec.ch>

## **Provisional Program :**

- 09.30 Kaffee und Gipfeli
- 10.00 Referat Kurt Schär (Verfügbarkeit muss noch abgeklärt werden)
- 10.30 Werksbesichtigung mit anschliessendem Probefahren auf dem Werksareal
- 12.00 Mittagessen mit Catering (kein Tellerservice -> betreutes Buffet) bei der Biketec AG
- 14.00 Vereinsversammlung / Preisverleihung im Schulungsraum
- 16.00 Apéro

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# *Call for 2013 SSOM Prizes*

## **Ausschreibung**

Die SSOM vergibt zum achten Mal in zweijähriger Folge

### **Forschungspreise für den wissenschaftlichen Nachwuchs.**

Es gibt drei Preise, die traditionell an der Jahrestagung der SSOM im Herbst verliehen werden, dieses Jahr am 15. November in Huttwil ([www.ssom.ch](http://www.ssom.ch)). Die drei Preise werden diesmal von den Firmen Heptagon, Zeiss und FEI gestiftet.

#### **Der Heptagon-Preis (CHF 5000.-)**

wird für hervorragende Arbeiten auf dem Gebiet der Entwicklung von Optik für intelligente Mikrosysteme vergeben.

#### **Der Zeiss (Schweiz) und Gloor-Preis (CHF 2500.-)**

wird für hervorragende Arbeiten auf dem Gebiet der Mikroskopie vergeben.

#### **Der FEI-Preis (CHF 2500.-)**

wird für hervorragende Arbeiten in der Anwendung von Elektronen- und Ionenstrahlen vergeben.

Für die Durchführung des Wettbewerbes ist eine von der SSOM eingesetzte Jury aus Hochschul- und Industrievertretern zuständig. Die Zusammensetzung der Jury wird an der Preisverleihung bekannt gegeben.

Bei den eingereichten Arbeiten muss es sich um einen Originalbeitrag aus der Grundlagenforschung, der angewandten Forschung oder der ingeniermäßig betriebenen Entwicklung handeln. Die Arbeit kann an Hochschulen, Fachhochschulen oder in der Industrie geleistet worden sein. Auch Beiträge von Teams sind willkommen. Teilnahmeberechtigt sind Arbeiten, die vorwiegend in der Schweiz ausgeführt wurden, und deren Bearbeiter nicht älter als 35 Jahre am Tag der Abgabe sind.

Die Vorschläge oder Eigenbewerbungen müssen bis spätestens

**1. August 2013**

beim Sekretariat der SSOM (Adresse siehe unten) eingegangen sein. Neben einer ausführlichen Beschreibung (Bericht, Publikation, Diplomarbeit, Dissertation, etc.) soll auf maximal zwei A4-Seiten die Besonderheit der Arbeit hervorgehoben werden. Außerdem ist ein ‚Curriculum vitae‘ der Bewerbung beizulegen. Die Dokumente sind im elektronischen Format einzureichen.

Gewinner sollten zusätzlich zu ihrer eingereichten Arbeit einen Artikel für das SSOM-Bulletin schreiben und ein Poster an der oben genannten Jahrestagung und Mitgliederversammlung aufstellen.

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