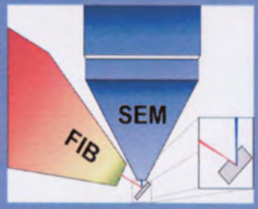

Thin Films Thickness Measurement by Secondary Electrons Contrast

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Goal:

To develop a method for thickness evaluation of thin films using Secondary Electron (SE) contrast in Dual Beam FIB/SEM systems. This method may be useful in MEMS, Nano-Fabrication, and for TEM samples preparation.

Background:

In Dual Beam FIB/SEM systems, the sample thinning by ion milling is typically being continuously monitored by SEM imaging. It was experimentally observed that the SE contrast of SEM image, measured at certain Primary Electron (PE) beam energy by using the Everhart Thornley Detector (ETD) located at sample side, changes as the sample becomes thinner. At first, the SE contrast increases, possibly because the sample becomes transparent to the PE beam and SE are generated at both, front and back sides, thus adding to a total SE signal (Fig.1). During further thinning, the SE contrast reaches maximum and than decreases, possibly because a large portion of the PE beam is transmitted without interacting with the sample and does not contribute to SE generation.

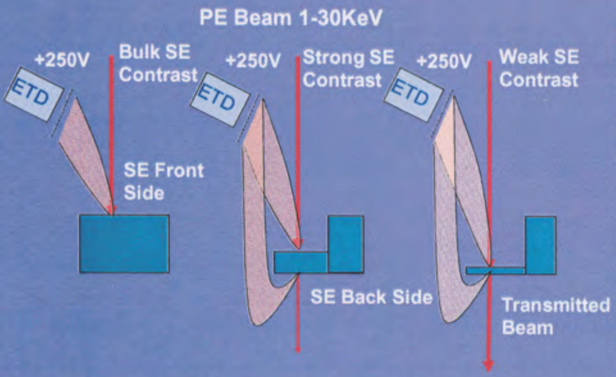


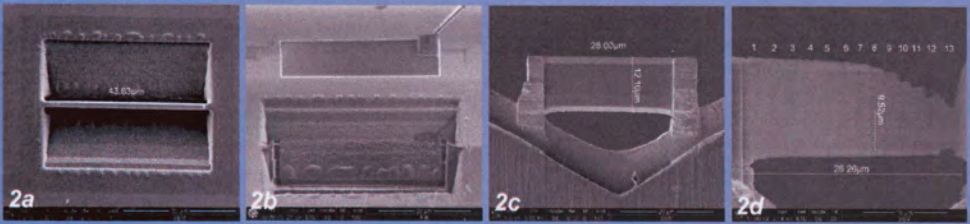
Fig.1. Schematics of SE yield for SE emitted from front and back sides for different sample thickness and collected by Everhart Thornley Detector (ETD)

Experimental:

A FEI Company Strata 400 Dual Beam FIB/STEM System was used for GaAs and Si samples preparation and SE contrast measurements. The cross-sections (~1 micron thick) were milled by FIB (Fig.2a), than lifted out using Omniprobe micromanipulator (Fig.2b), and attached to the TEM grid on the Flip Stage (Fig.2c). The samples were further thinned on the Flip Stage (Fig.2d). One side of the samples was cut flat. The other side was cut in the form of segments of different thickness. The thickness of each segment was measured in top view using High Resolution SEM (Fig.2e).

For inspection, the GaAs and Si samples were oriented normally to PE beam. The ETD detector with bias +250V was used for collecting SE images at PE accelerating voltage in the range of 1 - 30KeV (Fig.3 a-d). The SE values at each segment were digitized and plotted graphically as a function of beam voltage and segment thickness. The values of PE energy at which SE contrast starts to increase were identified as the most useful parameter for specimen thickness evaluation. These values, are shown as a function of sample thickness (Fig.4).

The experimental results were compared with Monte-Carlo simulations of electron trajectories using the MC–SET program (Fig. 5,6). (http://www.mc-set.com, free download).



GaAs Segment #	Thickness (nm)
1	640
2	535
3	450
4	410
5	375
6	315
7	260
8	200
9	150
10	115
11	90
12	70
13	50

Fig.2. (a) GaAs FIB Cross-section normal to the sample surface. (b) Lift out using Omniprobe micromanipulator. (c) Sample attached to the TEM grid on the Flip Stage. (d) Sample thinned on the Flip Stage. One side cut flat, the other side was cut in the form of segments of different thickness. (e) The thickness of each segment was measured in top view using High Resolution SEM.

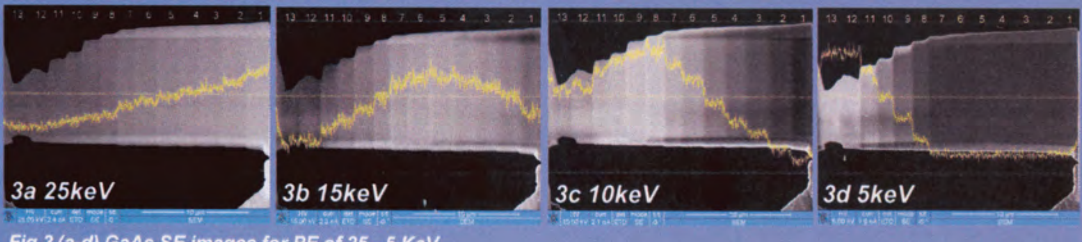


Fig.3 (a-d) GaAs SE images for PE of 25 - 5 KeV.

Most useful parameter – SE Contrast increase during milling

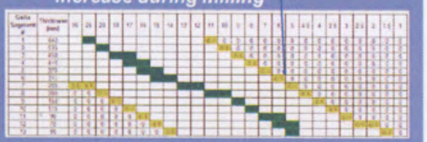
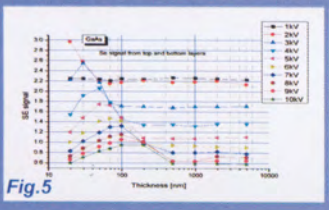
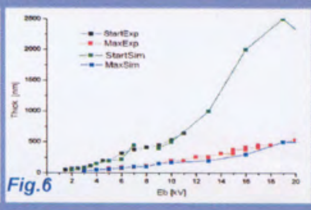


Fig.4. GaAs relative SE Contrast values: 0 - Bulk or Weak Contrast ; 0.1 ~10% Contrast Increase; 1 - Max Contrast.





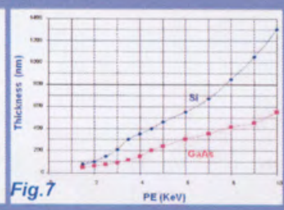


Fig.5. SE Monte Carlo Simulation for GaAs for PE 1-10 KeV (www.mc-set.com).
Fig.6. Comparison of Experimental Results for GaAs in Fig.4 and Simulation data in Fig.5.
Fig.7. Correlation between GaAs and Si sample thickness and the start of SE Contrast Increase at certain PE energy. The SE contrast was measured at normal PE beam incidence. For tilted specimens the appropriate thickness correction should be done.

Results and Conclusions:

Experimental results and Monte Carlo simulations (Fig.4-6) allowed to identify singular points in the SE yield curves, where PE beam starts exiting the specimen from its bottom surface, thus increasing the obtained SE contrast. The correlation between the PE energy at which SE contrast starts to increase and the thickness of GaAs and Si samples is shown in Fig.7.

Thus, Monte Carlo simulation can be used to produce sets of data for different materials, to allow thickness evaluation, based on the change of the observed secondary electron contrast .

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M. Bar-Sadan[1] , L. Houben[2] , A.N. Enyashin[3] , S. Gemming[3] , G. Seifert[3] , Y. Prior[1] , K. Urban[2] , R. Tenne[1] , [1] *Weizmann Institute of Science,* [2] *Ernst Ruska Centre for Microscopy and Spectroscopy with Electrons, Research Centre Jülich, Germany,* [3] *Institute of Physical Chemistry and Electrochemistry, Technische Universität Dresden, Dresden, Germany.*

Tomography of Inorganic Fullerene-Like WS_2 Nanoparticles
M. Bar-Sadan[1] , S. G. Wolf[2] , R. Tenne[1] , [1] *Materials and Interfaces department, Weizmann Institute of Science,* [2] *Electron Microscopy Unit, Weizmann Institute of Science.*

Short-Range Order in Relaxor $Pb(In_{1/2} Nb_{1/2})O_3 :Pb(Mg_{1/3} Nb_{2/3})O_3$
Cheuk W. Tai, *Faculty of Engineering, Tel Aviv University, Ramat Aviv, Tel Aviv 69978, Israel.*

Evolution of Dislocation Patterns in Deformed Aluminum, Nickel and Copper
P. Landau[1] , R.Z. Shneck[1] , G. Makov[2] and A. Venkert[2] , [1] *Department of Materials Engineering, Ben-Gurion University, P.O.Box 653, Beer- Sheva, 84105, Israel,* [2] *NRCN, P.O.Box 9001, Beer- Sheva, 84190, Israel.*

MoS_2 Fullerene-like Nanoparticles and Nanotubes using gas-phase reaction with $MoCl_5$
F. L. Deepak[1] , Alexander Margolin[1] , Inna Wiesel[1] , Maya Bar-Sadan[1] , Ronit Popovitz-Biro[2] , Sidney Cohen[3] , Hagai Cohen[3] and Reshef Tenne[1] , [1] *Department of Materials and Interfaces,* [2] *Electron Microscopy Unit,* [3] *Chemical Research Support, Weizmann Institute of Science, Rehovot, Israel.*

Fabrication of Self-Lubricating Cobalt Coatings on Metal Surfaces
Hilla Friedman[1] , Orly Eidelman[1] , Yishay Feldman[2] , Alexey Moshkovich[3] , Vladislav Perfiliev[3] , Lev Rapoport[3] , Hagai Cohen[2] , Alexander Yoffe[2] and Reshef Tenne[1] , [1] *Department of Materials and Interfaces, Weizmann Institute, Rehovot 76100, Israel,* [2] *Chemical Research Support Unit, Weizmann Institute, Rehovot 76100, Israel,* [3] *Department of Science, Holon Institute of Technology, Golomb St. 52, P.O.B 305, Holon 58102, Israel.*

The Effect of Sintering on Ni-$Al_2 O_3$ Nanocomposites Microstructure
Gali Gluzer and Wayne D. Kaplan, *Department of Materials Engineering, Technion, Haifa, Israel.*

Spark Plasma Sintering (SPS) of $Pb_{1-x} Ge_x Te$ Compounds
Y. Gelbstein[1] , M. Gelbstein[1] , O. Ben-Yehuda[2] , Z. Dashevsky[1] , Y. George[1] and M.P. Dariel[1] , [1] *Department of Materials Engineering, Ben-Gurion University, Beer-Sheva 84105, Israel,* [2] *Department of Physics, Ben-Gurion University, Beer-Sheva 84105, Israel.*

TEM Study of Allotropic Phase Transformation Ceγ↔Ceβ in Ce-Nd Alloy
V. Hanin [1] , P. Landau [1] , A. Venkert [2] , M.P. Dariel [1] , [1] *Department of Materials Engineering, Ben-Gurion University of the Negev, Beer-Sheva, Israel,* [2] *Nuclear Research Centre-Negev, P.O. Box 9100, Beer-Sheva, Israel.*

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Spark Plasma Sintering of Nanocrystalline Yttrium Oxide Powder
Amit Shlayer[1] , Rachman Chaim[1] and Claude Estournes[2] , [1] *Department of Materials Engineering, Technion, Haifa, Israel,* [2] *CIRIMAT et Plateforme Nationale CNRS de Frittage Flash University of Paul Sabatier, 33062 Toulouse, France.*

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I. Kaplan-Ashiri[1] , S.R. Cohen[2] , K. Gartsman[2] , V. Ivanovskaya[3] , T. Heine[3] , G. Seifert[3] , I. Wiesel[1] , H.D. Wagner[1] and R. Tenne[1] , [1] *Department of Materials and Interfaces, Weizmann Institute of Science,* [2] *Chemical Research Support, Weizmann Institute of Science,* [3] *Institut für Physikalische Chemie, Technische Universität Dresden, D-01062, Germany.*

Intercalation of the WS_2 Nanotubes and Inorganic Fullerene-Like Particles with Alkali Metals
F. Kopnov[1] ,R. Popovitz-Biro[2] , A. Zak[3] , R. Tenne[1] , [1] *Department of Materials and Interfaces, Weizmann Institute, Rehovot 76100, Israel,* [2] *Chemical Research Support Unit, Weizmann Institute, Rehovot 76100, Israel,* [3] *"NanoMaterials, Ltd.", Einstein 18, Building 18, P.O. Box 4088, Nes Ziona 74140.*

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