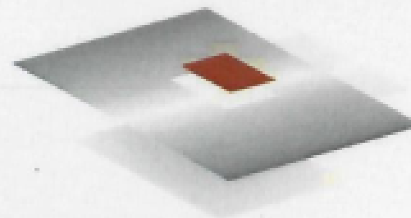
A10561

ToF-SIMS Analysis Particles on a Cu Detection Screen

Intellect Technologies
Dirk Avau



tascon GmbH
Analytical Services & Consulting

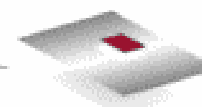
Materialcharakterisierung
Materials Characterisation

DIRK AVAU
BEUKENLAAN 38
B-1653 DWORP
02/380 91 41

Heisenbergstraße 15
48149 Münster | Germany
www.tascon-gmbh.de

Document of 44 pages

ToF-SIMS Analysis of Particles



TASCON Report A10561, June 2007

Customer: Intellect Technologies
Prepared for: Dirk Avau

Analysis: Elke Tallarek
Author: Birgit Hagenhoff
Tel.: 0251-1622-420
Email: birgit.hagenhoff@tascon-gmbh.de

QM Release: Elke Tallarek

________________________ ________________________

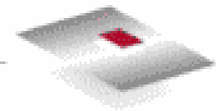
(date and signature author)* (date and signature release)*

*: see also disclaimer section at the end of this report

DAP-PL-4137.00
DIN EN ISO/IEC 17025:2005

DIN EN ISO 9001:2000

ToF-SIMS Analysis of Particles

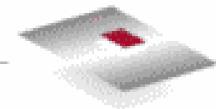


Sample Description and Analytical Task

- On a Cu detection screen splash lines which origin from the explosion of an anode are observed.
- Additionally, particles can be found close to the splash lines. The chemical composition (including isotopic ratios) is of interest in order to identify the origin of the particles.
- By means of SEM/EDX and Auger analysis one particle was found to contain Pb, Fe, Si, O, C, Al and possibly Au. The anode mainly contains Cu.
- Now, the particle should again be analyed using Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) because of the higher sensitivity and the possibility to obtain information in the isotopic patterns of the elements found.

tascon GmbH · Münster, Germany

ToF-SIMS Analysis of Particles

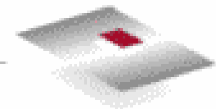


Sample Description and Measurements Performed

- Two particles of sample 232 were analysed:
  - the particle which was analysed previously by SEM/EDX and Auger spectroscopy („particle 1“)
  - a particle close to a distinct splash line which was not analysed before („particle 2“)
- First the surface composition of both particles was analysed by means of ToF-SIMS imaging. For particle 1 additionally the chemical composition in deeper layers was probed. Ion sputtering was used to get access to the deeper layers.

tascon GmbH · Münster, Germany

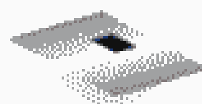
ToF-SIMS Fundamentals



- Characteristics of ToF-SIMS:
  - The chemical composition of surfaces can be probed by means of Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS). The technique provides information on the atomic and molecular composition of the uppermost 1 - 3 monolayers with sensitivities at ppm level and lateral resolutions down to 100 nm.
    ToF-SIMS is not an inherently quantitative technique because the detected intensities depend on the chemical composition of the ambient material ("matrix effect"). Semi-quantitative information can be obtained if the chemical environment of the samples to be compared is similar.

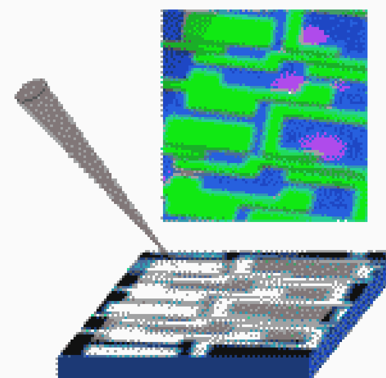
tascon GmbH · Münster, Germany

ToF-SIMS: Imaging



Explanation

- Imaging:
  - For acquisition of mass resolved secondary ion images (imaging, chemical mapping) a usually focused primary ion beam is used to probe the surface of interest. For each pixel addressed a complete spectrum is recorded. The intensities of secondary ion signals of interest are colour coded resulting in a mass resolved intensity map of the lateral distribution of secondary ion emission positions.

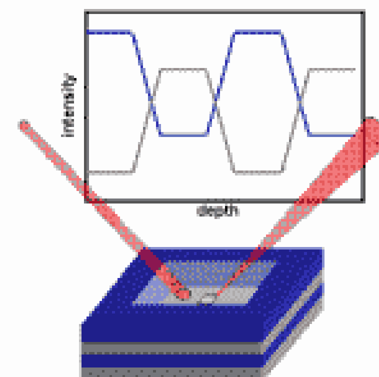


  - Fields of view of up to 500x500 µm² can be analysed by rastering of the primary ion beam. Larger areas (500x500 µm² ... 9x9 cm²) can be analysed by an additional movement of the sample stage ("stage raster").
  - The lateral resolution is 3-5 µm for routine analysis at full mass resolution and 300 - 500 nm at nominal mass resolution.

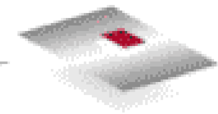
ToF-SIMS: Depth Profiling

Explanation

- Depth Profiling:
  - Depth profiles are acquired in order to investigate the chemical composition of a solid as a function of depth. In ToF-SIMS two different ion beams are used for data acquisition. A so-called sputter beam (e.g. O_2^+ or Cs^+) is applied to erode the sample while a 2nd ion beam (analysis beam e.g. Au^+) is used for a chemical characterisation of the resulting crater bottom. In most cases depth profiles only show the distribution of elements because the massive sample erosion causes a destruction of molecular structures. In selected cases also organic information can be gained. Although the resulting intensities are not inherently quantitative a comparing semi-quantitative analysis of chemically similar samples is possible after a suited normalisation.



Particle 1 - without Sputtering

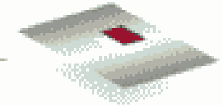


Measurement Conditions (Surface Imaging)

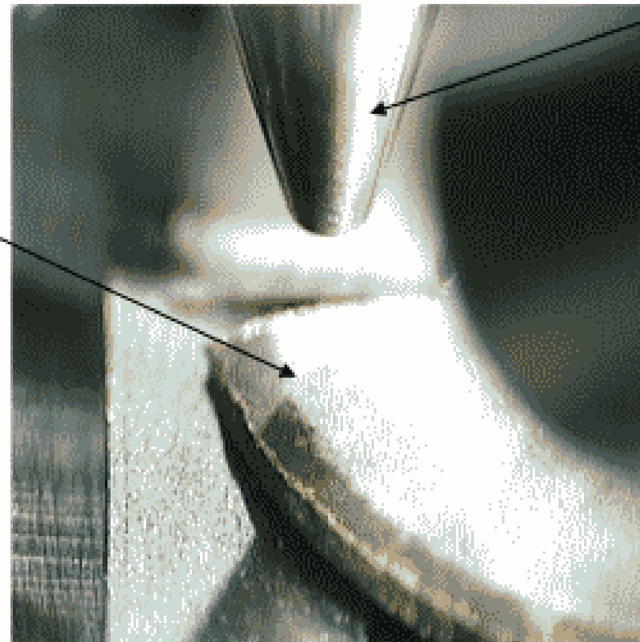
- Instrumentation:
  - IONTOF „TOF.SIMS 5“
- Analytical Conditions:
  - Ion / Energy: Bi_1^+, 25 keV
  - Mode: bunched mode (high mass resolution, focus approx. 3 µm)
  - Analysis Current: 0.2 pA
  - Area: 156 x 156 μm^2

tascon GmbH · Münster, Germany

ToF-SIMS Analysis of Particles

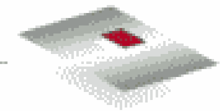


Optical Image of Sample 232 (Overview)



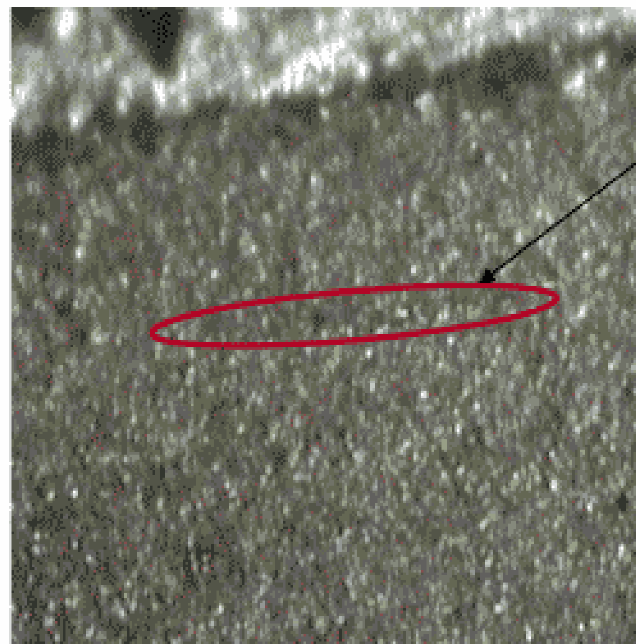
X08512.bmp

ToF-SIMS Analysis of Particles



Optical Image of Sample 232 (Detail View, Particle 1)

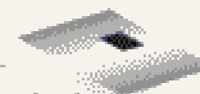
Field of View approx. 700 x 850 µm²



X08513.bmp

tascon GmbH · Münster, Germany

Particle 1 - without Sputtering

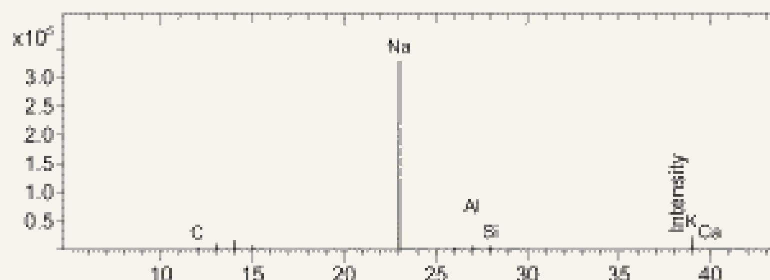


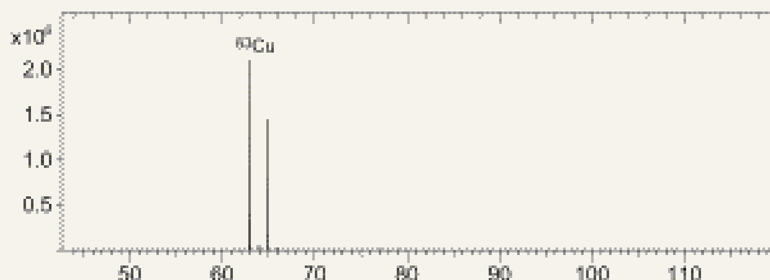
Reconstructed Spectrum from 156 x 156 µm² (Incl. Particle)

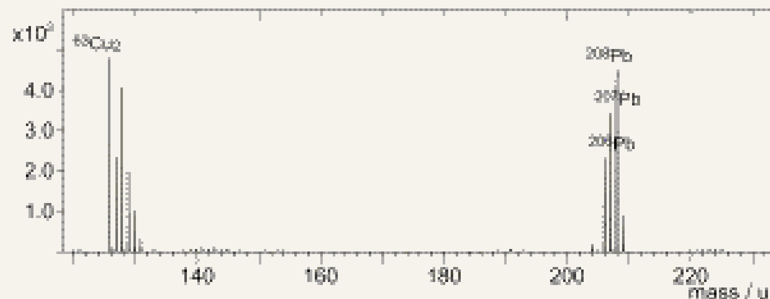
Sample: **Particle 1**
Comment: without pre-sputtering
Origin: Int.Techn.

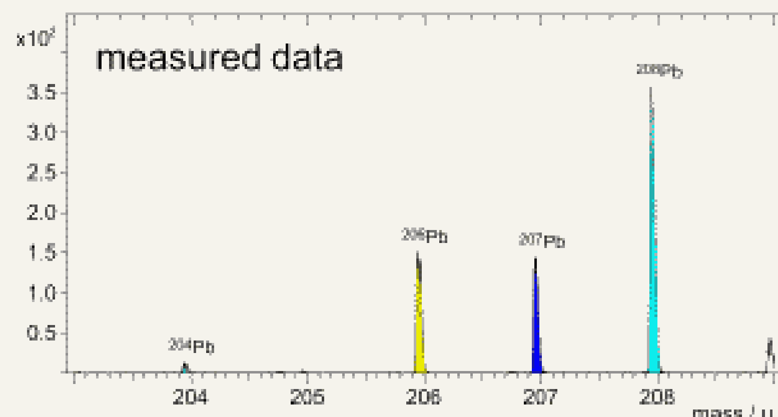
Polarity: positive
PI: Bi1+ (PID: 9.08E+008)
Area: 156x156 µm²

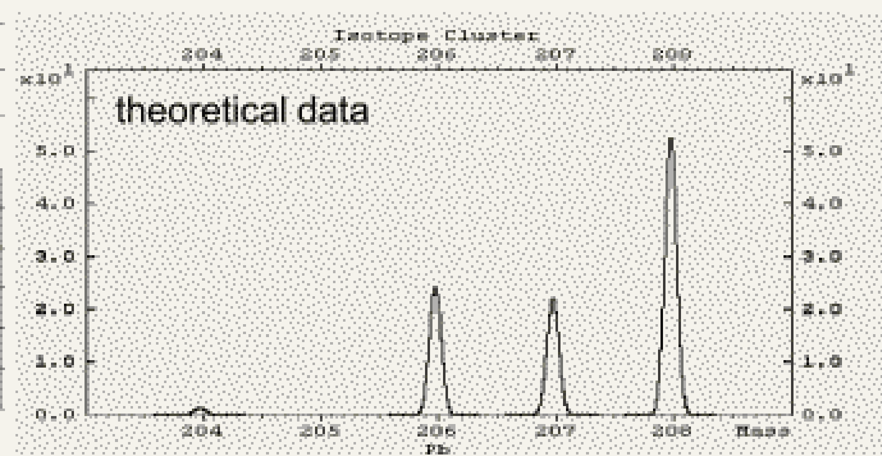
File: RX08514.DAT
Date: Wed Jun 06 2007



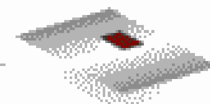








Particle 1 - without Sputtering



Explanation of Image Plots

colour scale used to represent image intensities

^{208}Pb ← ion identity

mc:47 tc:8.398e+3

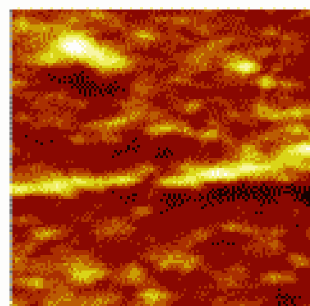
intensity in the brightest pixel

total number of counts in the image

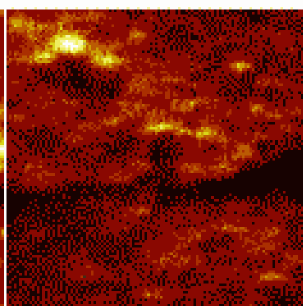
Particle 1 - without Sputtering

Image, Positive Secondary Ion Polarity

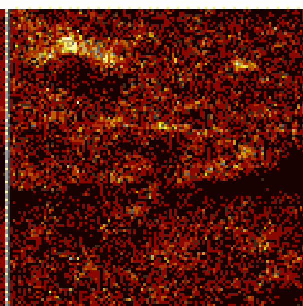
Field of view: 156.0 x 156.0 µm²; Substance: Particle 1 Surface; Polarity: positive; File: RX08514A.MIF



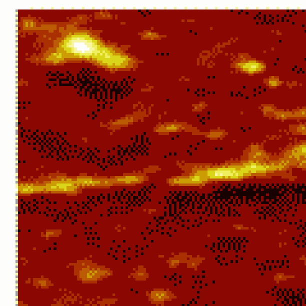
^{63}Cu
mc:403 tc:2.759e+6



Na
mc:77 tc:3.286e+5

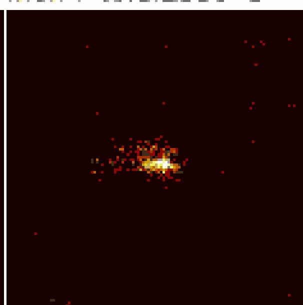


K
mc:9 tc:2.951e+4

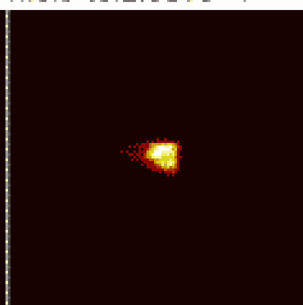


total ion
mc:922 tc:4.890e+6

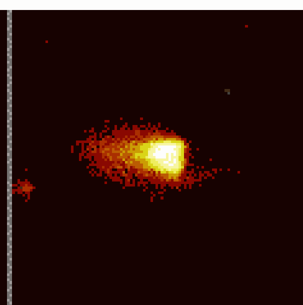
Al (lin scale)
mc:7 tc:6.090e+2



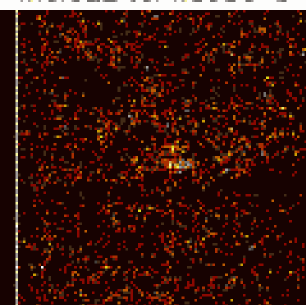
Al (lg scale)
mc:7 tc:6.090e+2



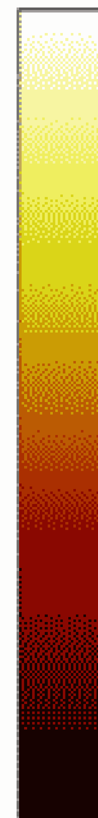
Pb (lin scale)
mc:86 tc:1.575e+4



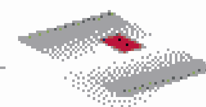
Pb (log scale)
mc:86 tc:1.575e+4



Si
mc:4 tc:6.429e+3



Particle 1 -without Sputtering



Correlation Analysis: 3 Colour Overlay

field of view: 156 x 156 µm²

Pb

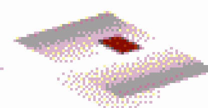
Al

Cu

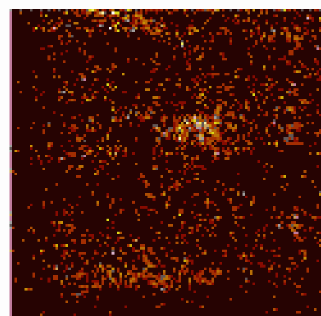
data taken from RX08514a.MIF

Particle 1 -without Sputtering

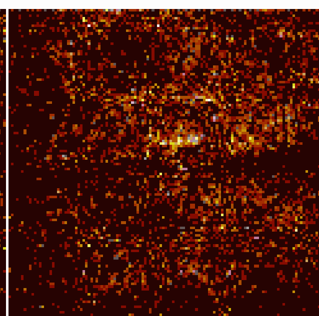
Image, Negative Secondary Ion Polarity



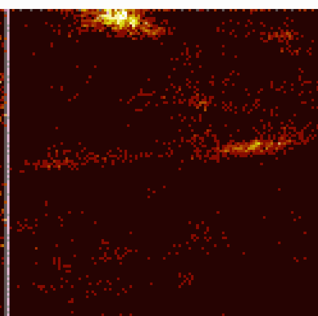
FoV: 156 x 156 µm²; Particle 1 Surface; Pol.: neg.; RX08516A.MIF



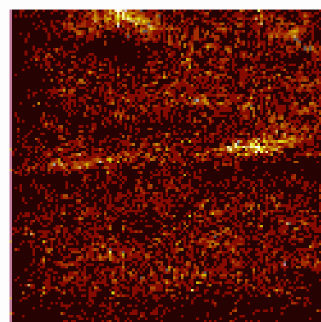
F
mc:4 tc:6.931e+3



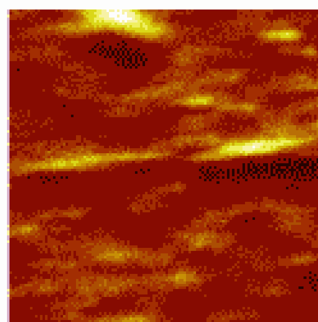
Cl
mc:5 tc:1.218e+4



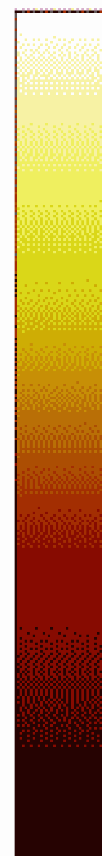
C_2H
mc:17 tc:2.627e+4



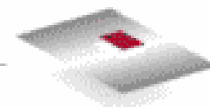
^{63}Cu
mc:10 tc:3.149e+4



total ion
mc:487 tc:3.109e+6



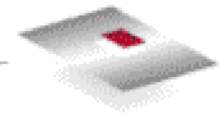
Particle 1 - without Sputtering



Results of Surface Imaging (Bunched Mode)

- The following elements were detected in the analysed area of 156 x 156 µm²:
  H, Li, B, C, O, Na, Mg, Al, Si, K, Ca, Ti, Fe, Ni, Cu, Zn, Ag, Pb
- Within the particle mainly Al, Si and Pb are found. Additionally, F and Cl can be detected.
- The lateral distribution of Al and Pb show a halo in splash direction.
- The splash line mainly consists of Cu. Na and K are not found in the splash line. However, they can be detected on the Cu surface of the detection screen. Additionally, organic material is detected.
- The isotopic distribution of the elements is in good agreement with the natural occuring isotopic ratios. However, the intensity of most elements is too weak to show deviations of less than 20%.

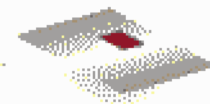
Particle 1 - without Sputtering



Measurement Conditions (Surface Imaging)

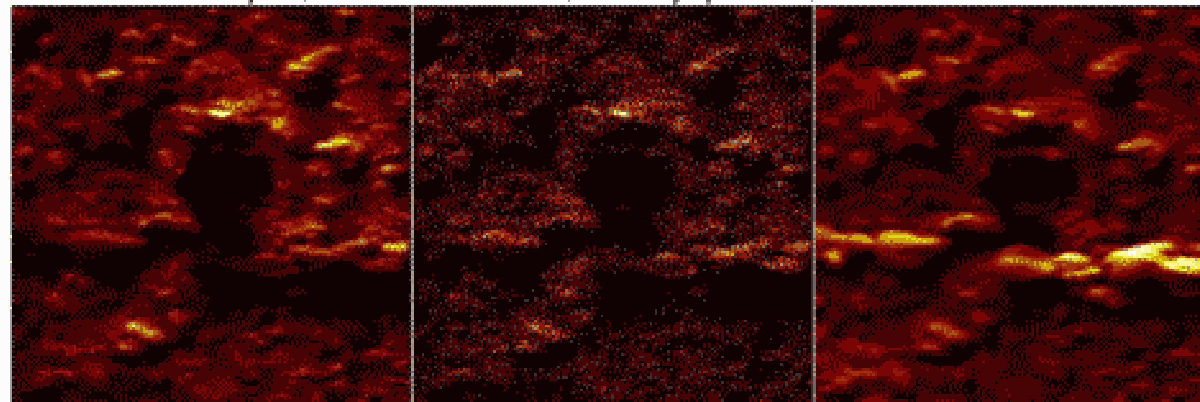
- Instrumentation:
  - IONTOF „TOF.SIMS 5“
- Analytical Conditions:
  - Ion / Energy: Bi_1^+, 25 keV
  - Mode: burst alignment mode (nominal mass resolution, focus approx. 300 nm)
  - Analysis Current: 0.2 pA
  - Area: 72 x 72 μm^2

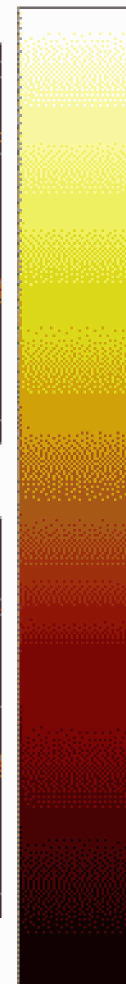
Particle 1 - without Sputtering



Image, Positive Secondary Ion Polarity

FoV: 72.3 x 72.3 µm²; Particle 1 Surface; Polarity: positive; File: R54319A.MIF





Na
mc:67 tc:6.275e+5

K
mc:10 tc:6.412e+4

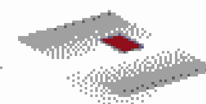
Cu
mc:390 tc:4.018e+6

Al
mc:15 tc:6.091e+3

Pb
mc:70 tc:4.672e+4

total ion
mc:477 tc:4.979e+6

Particle 1 - without Sputtering



Correlation Analysis: 3 Colour Overlay

field of view: 72 x 72 µm²

Pb

Al

Cu

data taken from R54319a.MIF

Particle 1 - after 2 min Sputtering

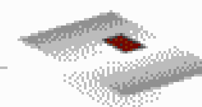
Measurement Conditions (Surface Imaging)

- Instrumentation:
  - IONTOF „TOF.SIMS 5“
- Analytical Conditions:
  - Pre-Sputtering: O_2^+, 2 keV; 560 nA on 500 x 500 µm²
  - Sputter Time: 2 minutes

  - Ion / Energy: Bi_1^+, 25 keV
  - Mode: burst alignment mode (nominal mass resolution, focus approx. 300 nm)
  - Analysis Current: 0.2 pA
  - Area: 72 x 72 µm²

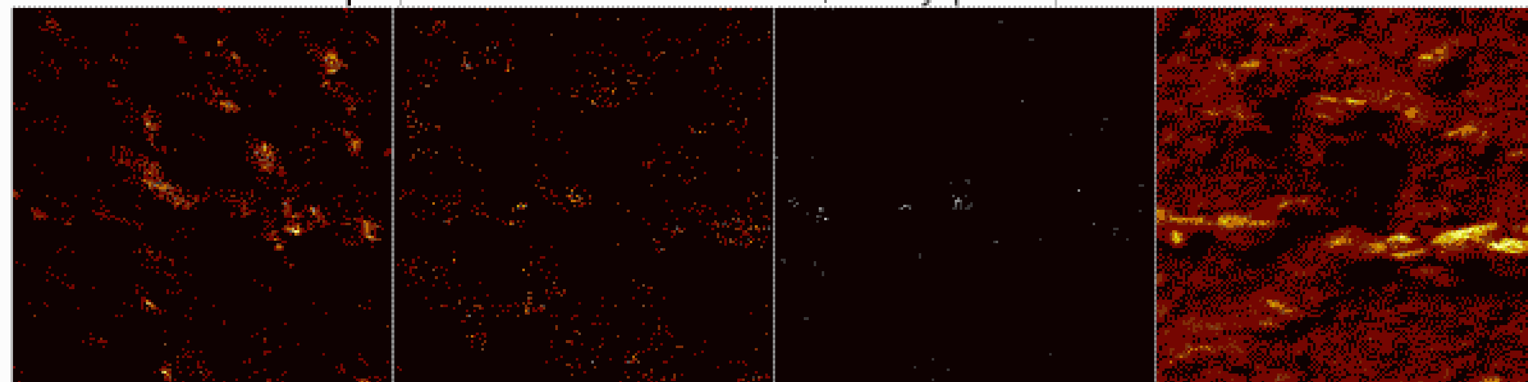
tascon GmbH · Münster, Germany

Particle 1 - after 2 min Sputtering



Image, Positive Secondary Ion Polarity

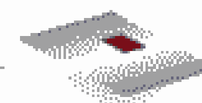
Field of view: 72.3 x 72.3 µm²; Substance: Particle 1 O2 2 min; Polarity: positive; File: R54321A.MIF



Na	Ca	Ti	Cu
mc:7 tc:1.828e+4	mc:3 tc:4.620e+3	mc:1 tc:7.700e+1	mc:35 tc:4.096e+5

Al	Si	Fe	Pb
mc:19 tc:5.328e+3	mc:2 tc:4.780e+2	mc:1 tc:3.380e+2	mc:7 tc:7.693e+3

Particle 1 - after 2 min Sputtering



Correlation Analysis: 3 Colour Overlay

field of view: 72 x 72 µm²

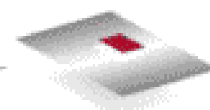
Pb

Al

Cu

data taken from R54321a.MIF

Particle 1 - after 6 min Sputtering



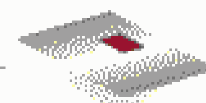
Measurement Conditions (Surface Imaging)

- Instrumentation:
  - IONTOF „TOF.SIMS 5“
- Analytical Conditions:
  - Pre-Sputtering: O_2^+, 2 keV; 560 nA on 300 x 300 µm²
  - Sputter Time: 4 minutes (total: 6 minutes)

  - Ion / Energy: Bi_1^+, 25 keV
  - Mode: burst alignment mode (nominal mass resolution, focus approx. 300 nm)
  - Analysis Current: 0.2 pA
  - Area: 72 x 72 µm²

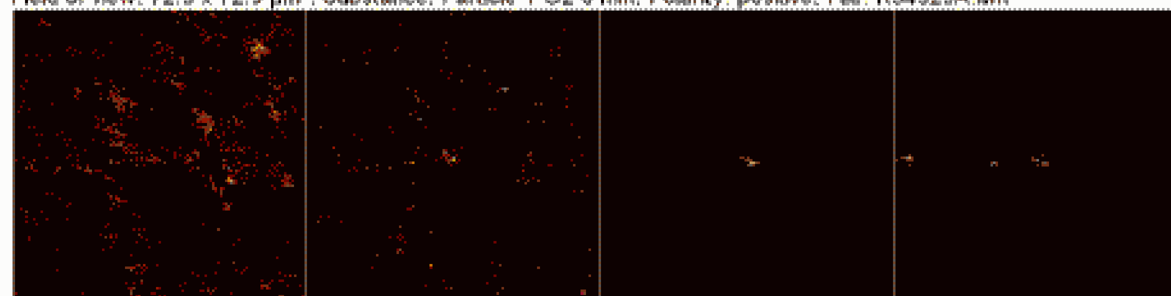
tascon GmbH · Münster, Germany

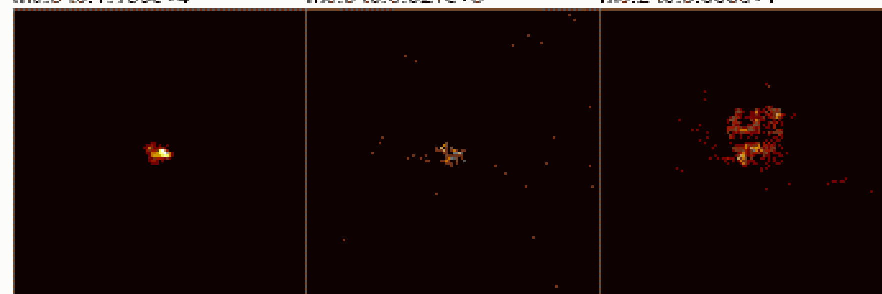
Particle 1 - after 6 min Sputtering

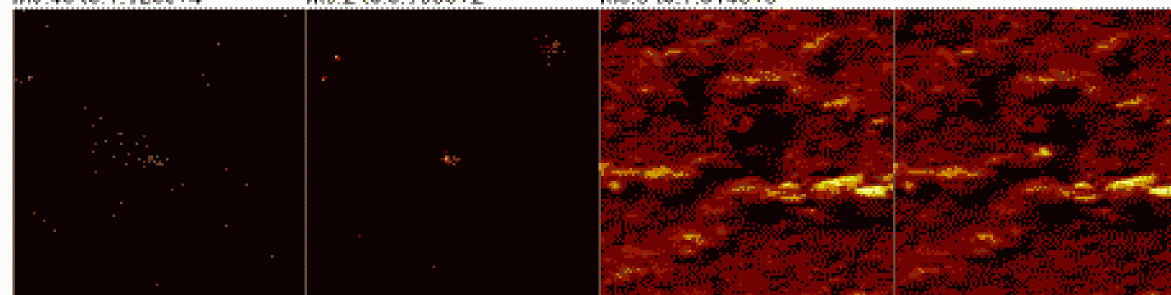


Image, Positive Secondary Ion Polarity

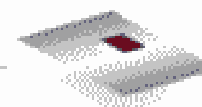
Field of view: 72.3 x 72.3 µm²; Substance: Particle 1 O2 6 min; Polarity: positive; File: R54323A.MIF







Particle 1 - after 6 min Sputtering



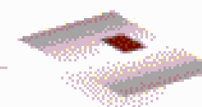
Correlation Analysis: 3 Colour Overlay

field of view: 72 x 72 µm²

Pb	
Al	
Cu	

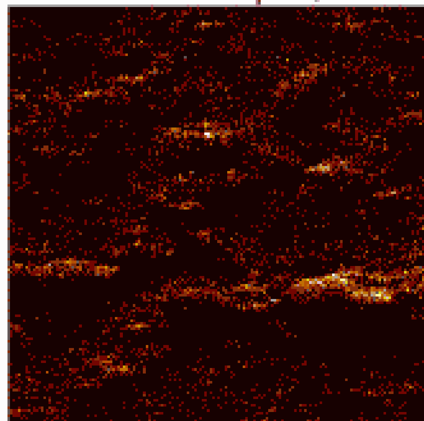
data taken from R54323a.MIF

Particle 1 - after 6 min Sputtering

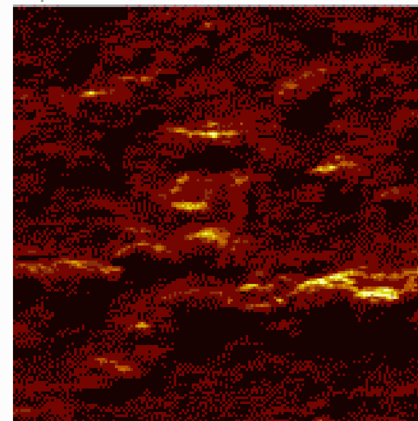


Image, Negative Secondary Ion Polarity

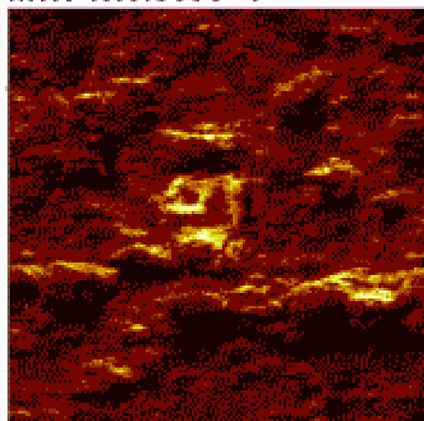
FoV: 72.3 x 72.3 µm²; Particle 1 O2 6 min; Polarity: negative; File: R54325A.MIF



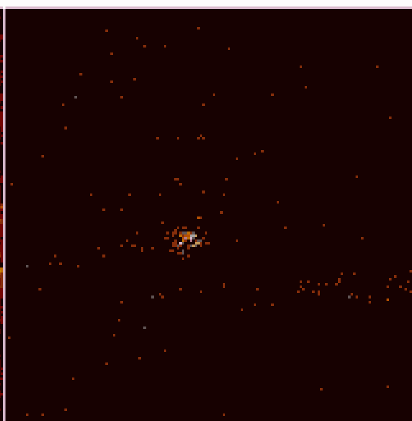
Cu
mc:9 tc:3.863e+4



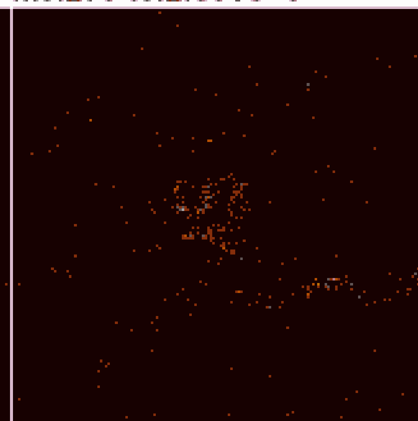
total ion
mc:279 tc:2.856e+6



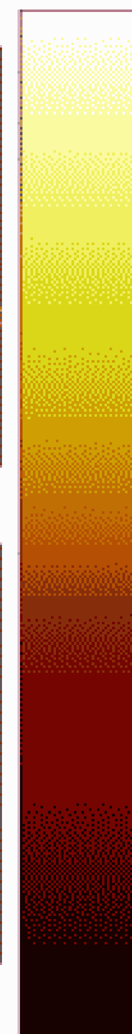
O
mc:114 tc:1.524e+6



AlO
mc:2 tc:3.800e+2



Pb
mc:2 tc:5.610e+2



tascon GmbH · Münster, Germany

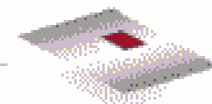
Particle 1 - after 16 min Sputtering

Measurement Conditions (Surface Imaging)

- Instrumentation:
  - IONTOF „TOF.SIMS 5“
- Analytical Conditions:
  - Pre-Sputtering: O_2^+, 2 keV; 560 nA on 300 x 300 µm²
  - Sputter Time: 10 minutes (total: 16 minutes)

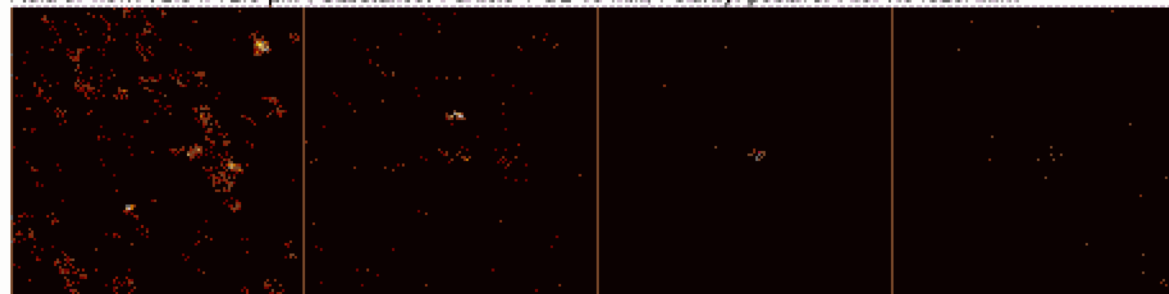
  - Ion / Energy: Bi_1^+, 25 keV
  - Mode: burst alignment mode (nominal mass resolution, focus approx. 300 nm)
  - Analysis Current: 0.2 pA
  - Area: 72 x 72 µm²

Particle 1 - after 16 min Sputtering

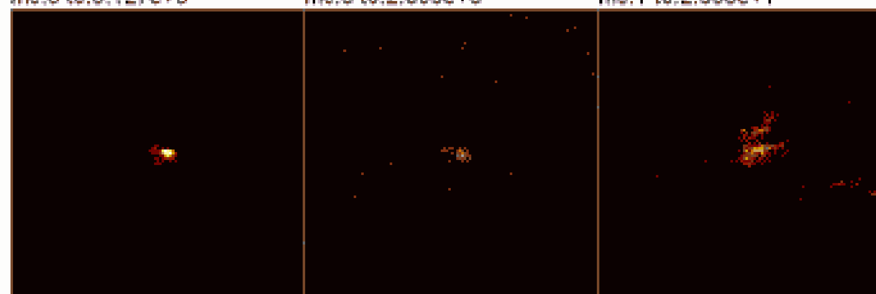


Image, Positive Secondary Ion Polarity

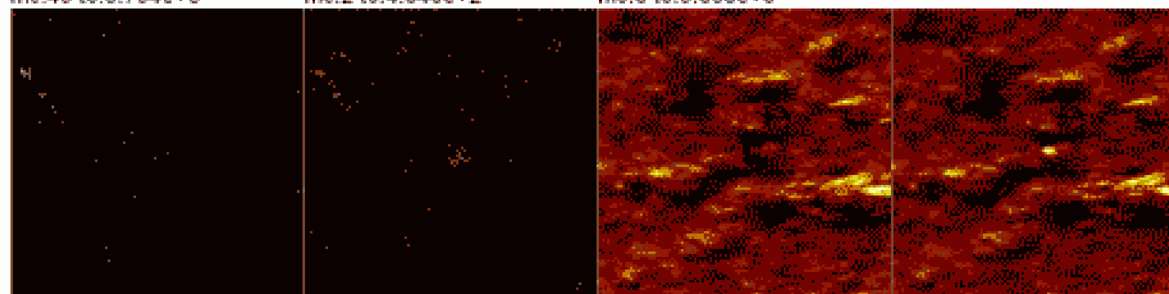
Field of view: 72.3 x 72.3 µm²; Substance: Particle 1 O2 16 min; Polarity: positive; File: R54329A.MIF



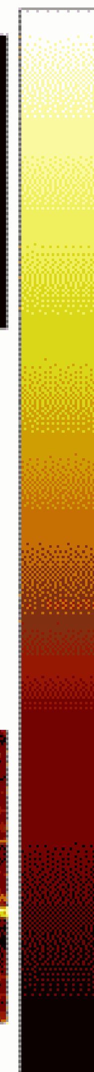
Na	Ca	B	Ti
mc:5 tc:8.127e+3	mc:3 tc:2.066e+3	mc:1 tc:2.300e+1	mc:1 tc:3.000e+1



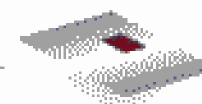
Al	Si	Pb
mc:49 tc:8.754e+3	mc:2 tc:4.940e+2	mc:8 tc:5.850e+3



Cr	Fe	Cu	total ion
mc:1 tc:7.000e+1	mc:2 tc:5.680e+2	mc:73 tc:1.123e+6	mc:86 tc:1.216e+6



Particle 1 - after16 min Sputtering



Correlation Analysis: 3 Colour Overlay

field of view: 72 x 72 µm²

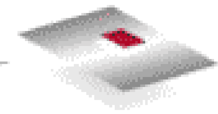
Pb

Al

Cu

data taken from R54329a.MIF

Particle 1 - after Sputtering



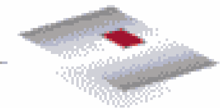
Measurement Conditions (3D Microarea Analysis)

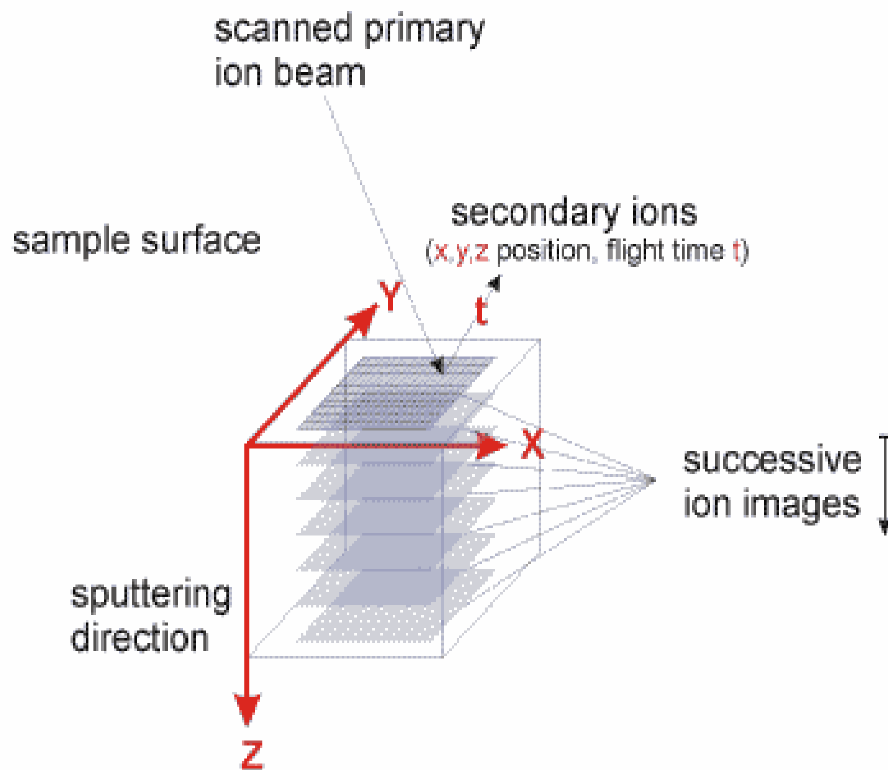
- Instrumentation:
  - IONTOF „TOF-SIMS IV“
- Sputter Erosion:
  - Ion/Energy: O_2^+, 2 keV
  - Current: 560 nA
  - Area: 300 x 300 µm²
- Analytical Conditions:
  - Ion/Energy: Bi^+, 25 keV
  - Mode: burst alignment
  - Current: 0.2 pA
  - Area: 39 x 39 µm²
- This experiment was performed on the already pre-bombarded area.

tascon GmbH · Münster, Germany

3D Analysis

Analysis Principle

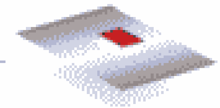




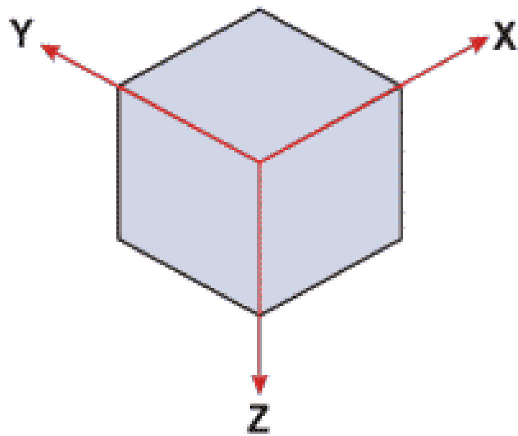
Principle of 3D Analysis

- Combination of SI Imaging and Depth Profiling
- Storage of all detected SI masses as function of cube co-ordinates x, y, z
- Possibility to reconstruct SI images in different depths (XY layers) as well as XZ- and YZ-cuts

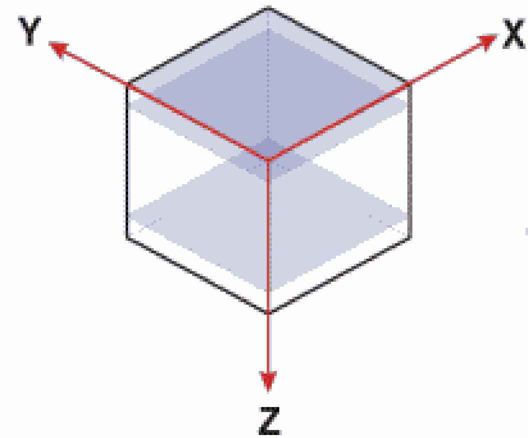
3D Analysis



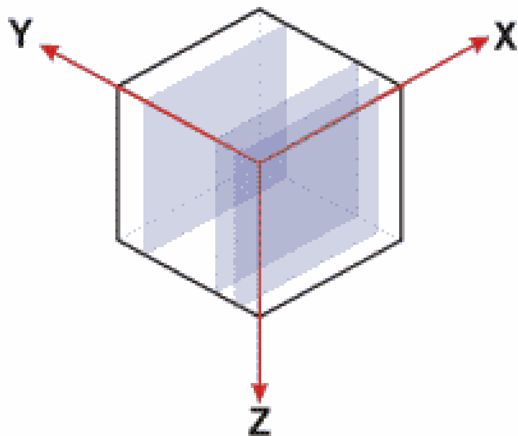
3D-Analysis: Co-Ordinates and Cross Sections



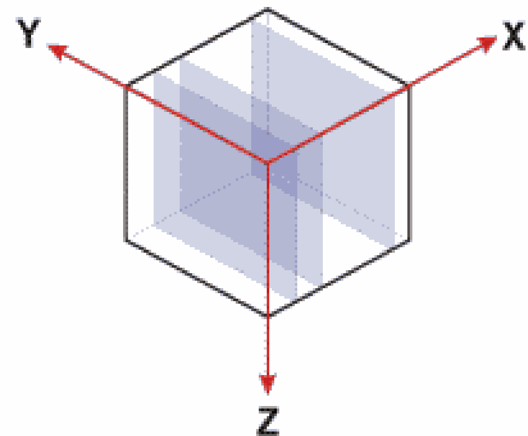
3D Cube



XY Layer
(in depth Z)

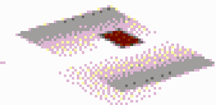


XZ Section



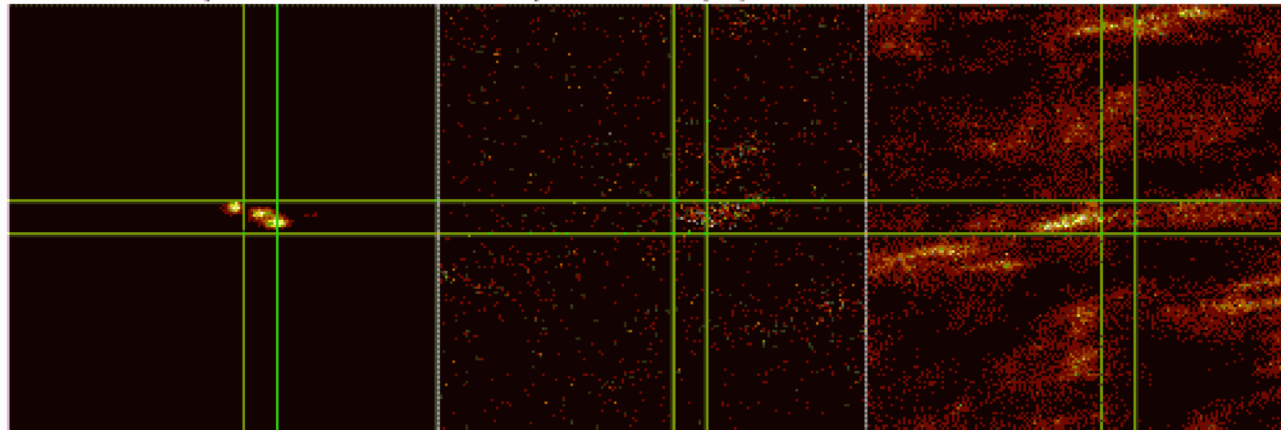
YZ Section

Particle 1 - 3D Analysis (after Sputt.)

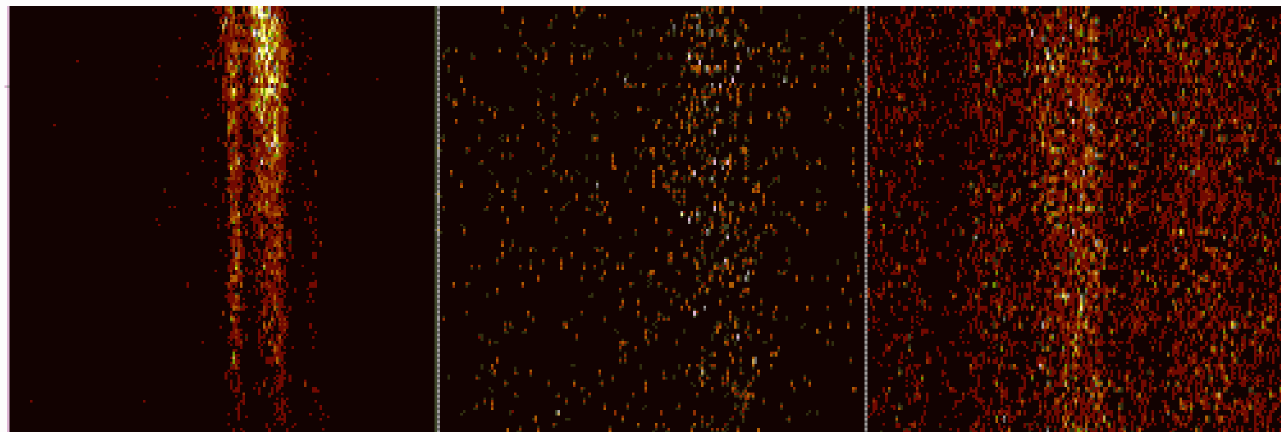


Depth Profile, Positive Secondary Ion Polarity

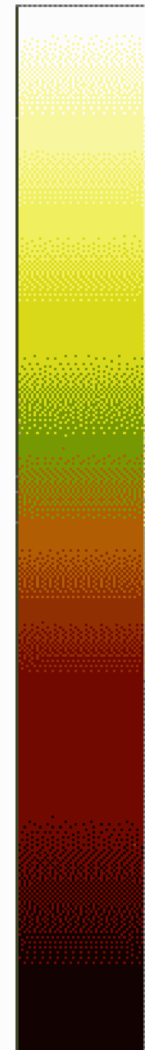
FoV: 39 x 39 µm²;Particle 1 3D analysis; Polarity: positive; File: R54333A.MIF



Al (all depths added) Pb (all depths added) Cu (all depths added)

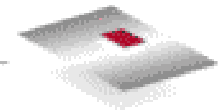


Al XZ section Pb XZ section Cu XZ section



tascon GmbH · Münster, Germany

Particle 1



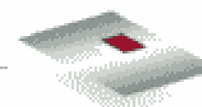
Results of Depth Profiling

- In the particle mainly Pb and Al is found. Pb is best visible at the surface whereas the Al intensity is higher in the centre of the particle.
- The Pb containing area is larger than the Al containing area.
- In the centre of the particle additionally B, Si, Ca, Ti, Cr and Fe is found.
- The Al intensity is dropping during the 3D profile (thus the particle was completely sputtered).
- Throughout the complete sputter process a low intensity of Pb prevails.

- It is possible that the pre-analyses (SEM/EDX, Auger) removed some of the particle's surface (Compare results of particle 2).

tascon GmbH · Münster, Germany

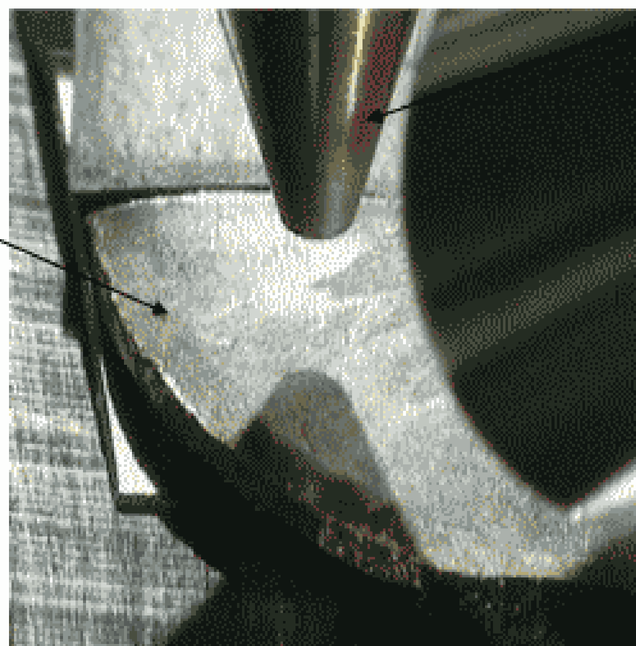
ToF-SIMS Analysis of Particles



Optical Image of Sample 232 (Overview)

sample

secondary ion extractor

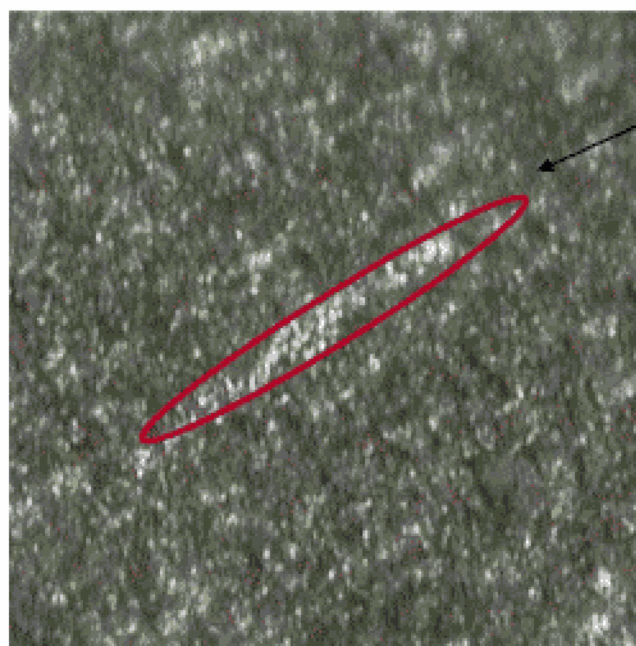


54337.bmp

ToF-SIMS Analysis of Particles

Optical Image of Sample 232 (Detail View Particle 2)

Field of View approx. 700 x 850 µm²

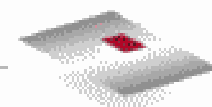


splash line

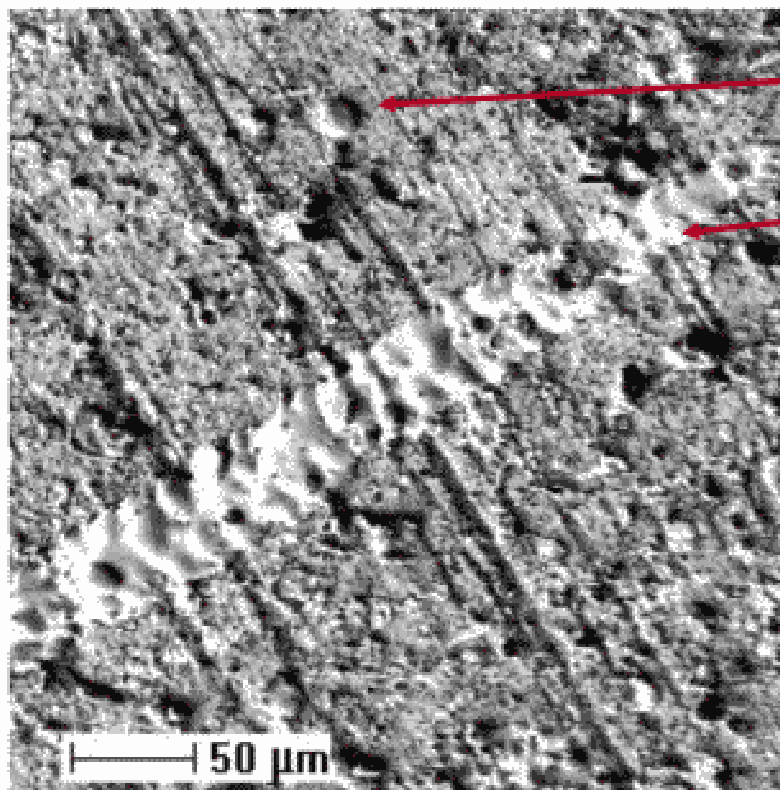
54338.bmp

tascon GmbH · Münster, Germany

Particle 2 - without Sputtering



Ion Induced SE Image



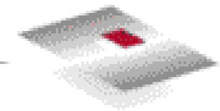
particle

splash line

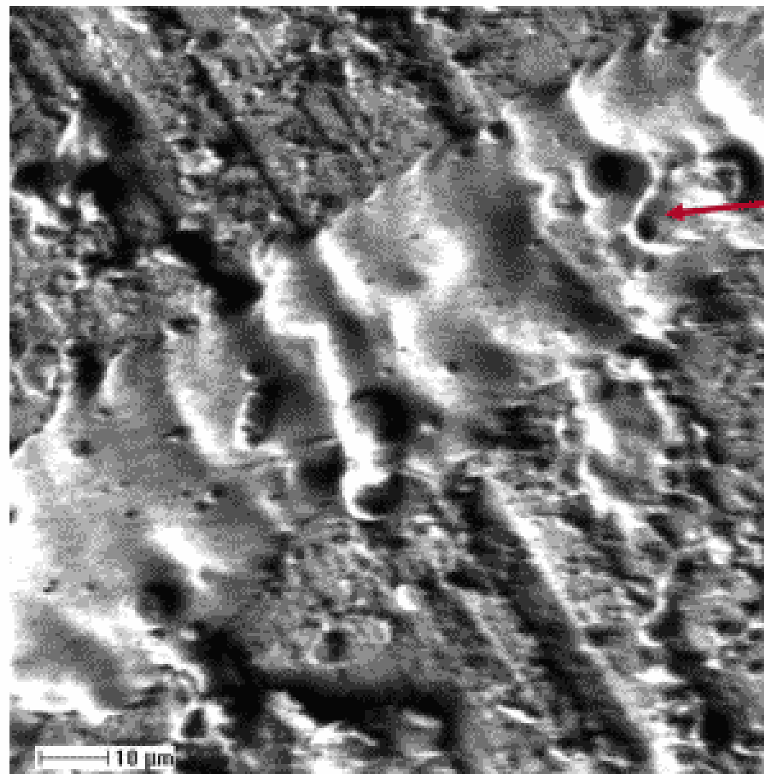
54339.bmp

tascon GmbH · Münster, Germany

Particle 2 - without Sputtering



Ion Induced SE Image



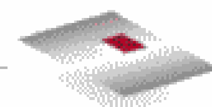
splash line

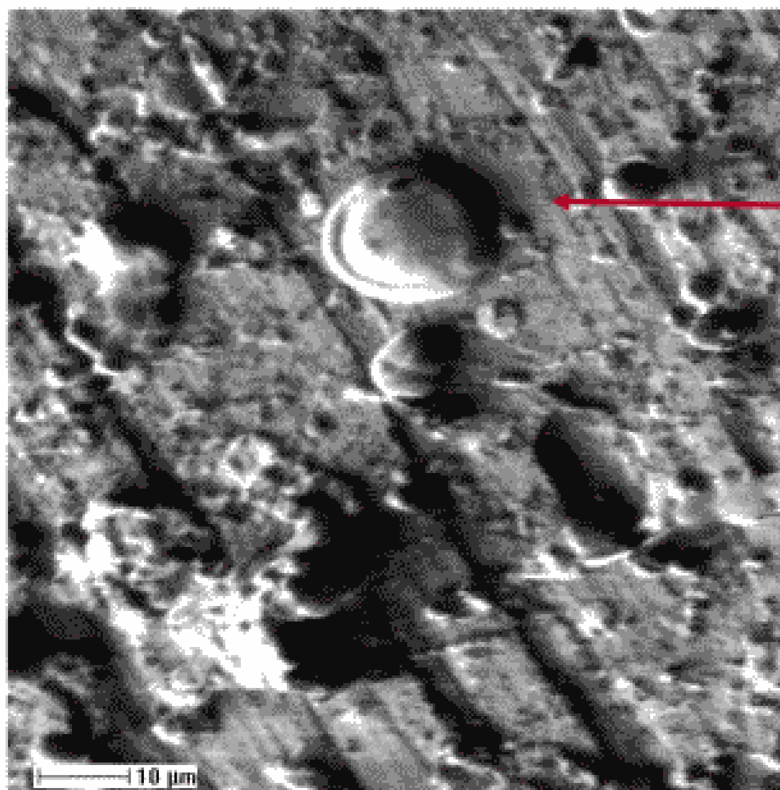
54339.bmp

tascon GmbH · Münster, Germany

Particle 2 - without Sputtering

Ion Induced SE Image

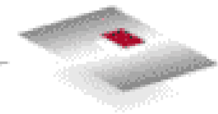




particle

54340.bmp

Particle 2 - without Sputtering



Measurement Conditions (Surface Imaging)

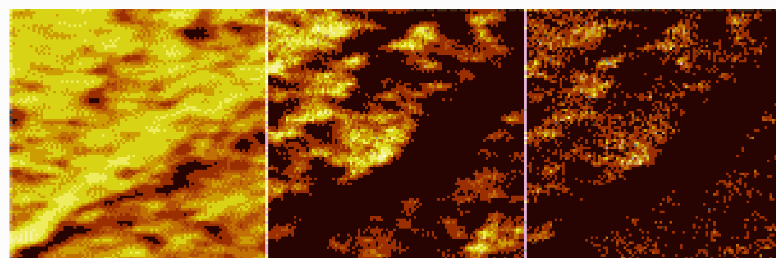
- Instrumentation:
  - IONTOF „TOF.SIMS 5“
- Analytical Conditions:

- Ion / Energy:	Bi_1^+, 25 keV
- Mode:	bunched mode (high mass resolution, focus approx. 3 µm)
- Analysis Current:	0.4 pA
- Area:	242 x 242 µm²

Particle 2 - without Sputtering

Image, Positive Secondary Ion Polarity

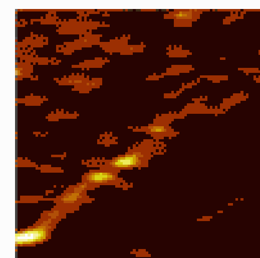
Field of view: 242.0 x 242.0 µm²; Substance: Particle 2 Surface; Polarity: positive; File: R54335A.MIF



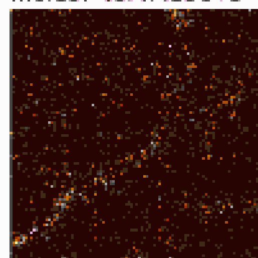
Cu
mc:397 tc:4.439e+6

Na
mc:65 tc:3.310e+5

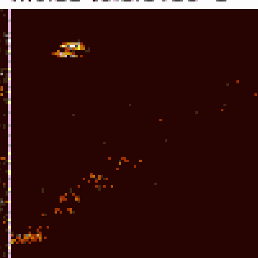
K
mc:13 tc:4.349e+4



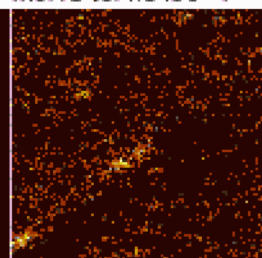
total ion
mc:2297 tc:1.019e+7



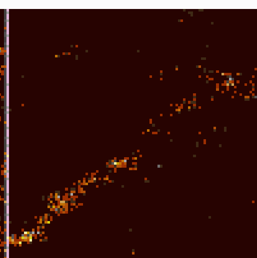
Mg
mc:2 tc:1.569e+3



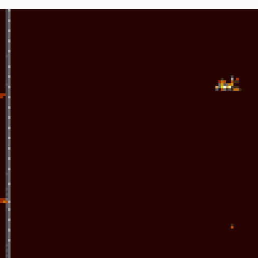
Al
mc:7 tc:4.508e+3



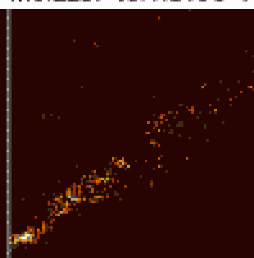
Si
mc:7 tc:1.812e+4



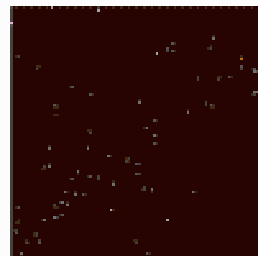
Ca
mc:5 tc:4.244e+3



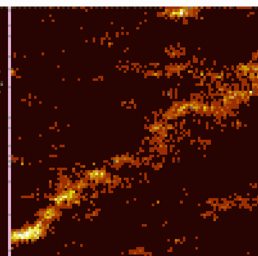
Ti
mc:3 tc:1.600e+2



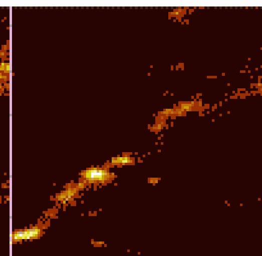
Fe
mc:4 tc:2.099e+3



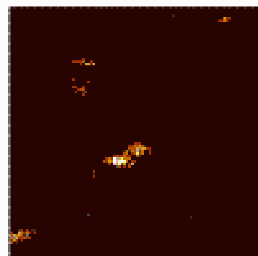
Ni
mc:1 tc:7.000e+1



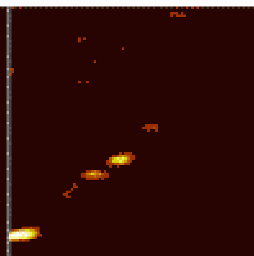
Zn
mc:19 tc:5.659e+4



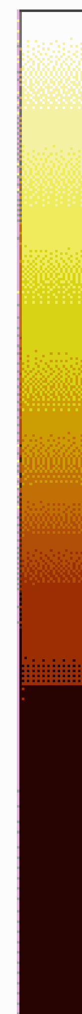
Pb
mc:43 tc:9.668e+4



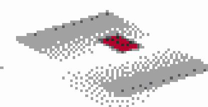
Siloxane
mc:9 tc:3.318e+3



C_xH_y
mc:204 tc:2.892e+5



Particle 2 - without Sputtering



Correlation Analysis: 3 Colour Overlay

field of view: 242 x 242 µm²

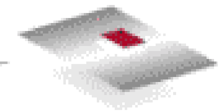
Al

Ti

Pb

data taken from R54335a.MIF

Particle 2

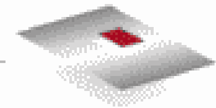


Results of Surface Analysis

- The splash line mainyl consists of Cu. Additionally, the elements Mg, Al, Si, Ca, Fe, Ni, Zn and Pb are detected.
- On parts of the splash line organic hydrocarbons and polysiloxane is found. This may be a handling contamination.
- The particle mainly consists of Al.
- Addiontally a position on the splash line is found which is rich in Ti. In the corresponding SE image no particle topography is detected at this position.

tascon GmbH · Münster, Germany

ToF-SIMS Analysis of Particles



Disclaimer

- The measurements were performed by specially trained personnel using state-of-the-art instrumentation.
- Nevertheless, we cannot give any guarantee for the correctness of the drawn conclusions.
- We would like to point out that the results refer only to the samples analysed by us.
- This report shall not be reproduced except in full without our written approval.
- In case this report was sent electronically it shall be regarded as **simplified test report** according to ISO/IEC 17025:2005. Its contents is identical with that of our archived and signed original test report. We will gladly send the original report to your attention if so asked. If you do not request the receipt of the original within 14 days you express your consent to accept the simplified test report as legally binding.

End of report A10561 (total number of pages: 44)