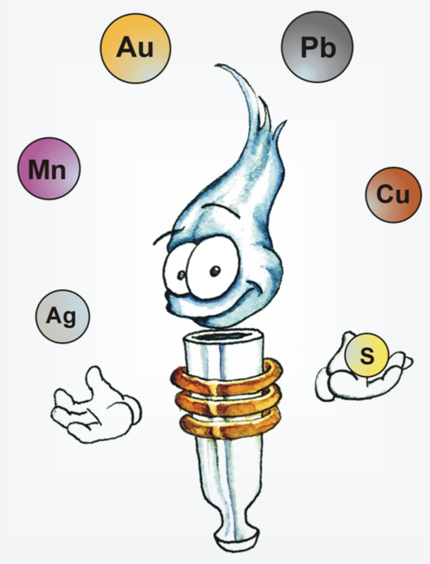
WESTFÄLISCHE WILHELMS-UNIVERSITÄT MÜNSTER

Development of Imaging Detection Techniques in Glow Discharge Optical Emission Spectroscopy



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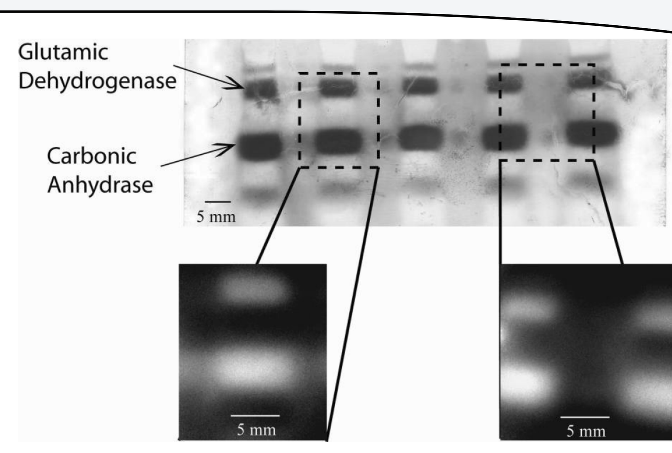
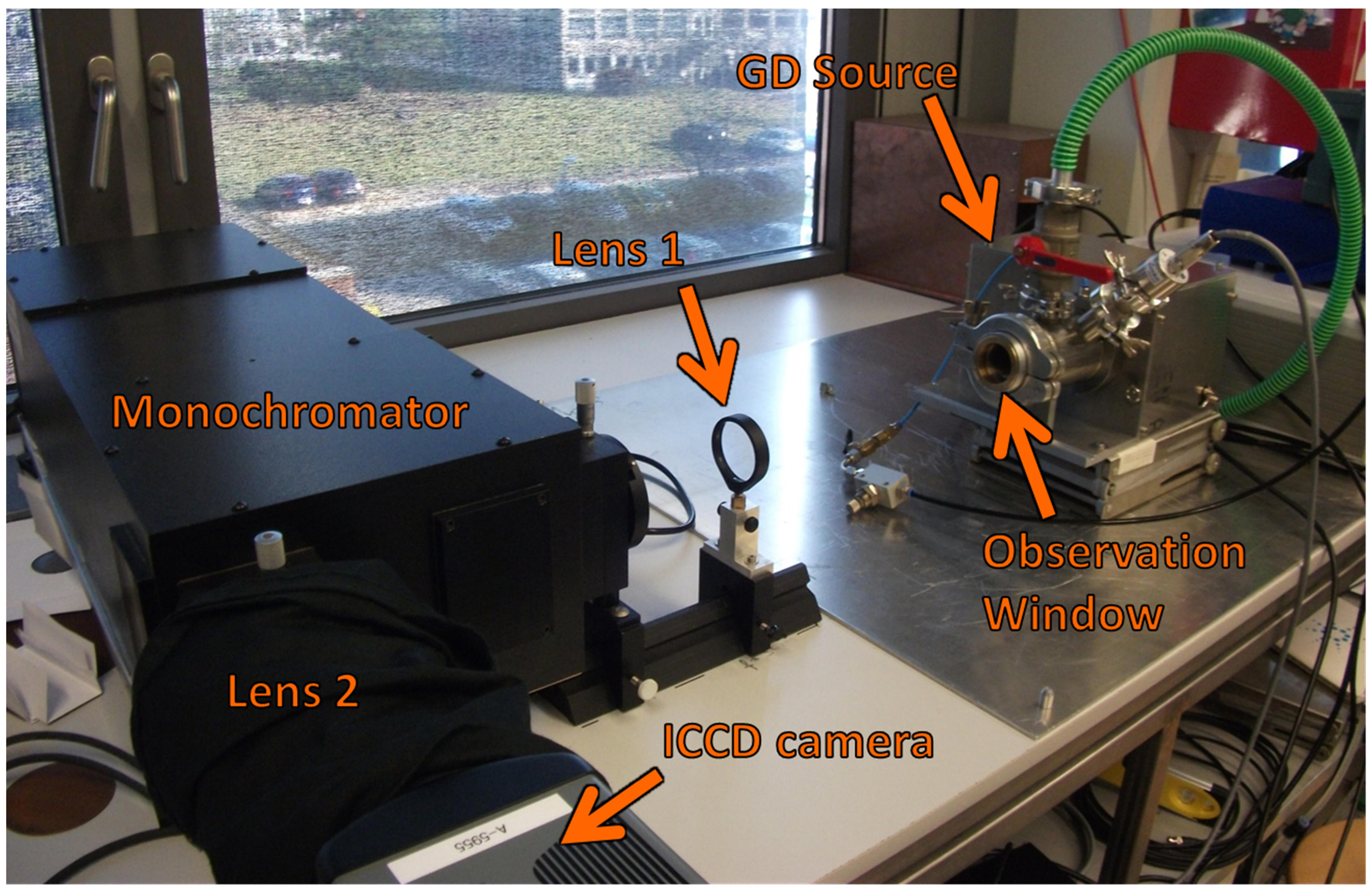
Introduction

The elemental distribution in solid samples can be very important. Especially, in material science the properties of materials can vary dramatically with the elemental composition at the surface. Furthermore, metal containing biomolecules, which can be separated with gel electrophoresis, are of increasing interest in elemental speciation analysis. A common method to analyze technical samples, dried gels or blotting membranes is LA-ICP-MS. This method is sensitive and powerful but requires point-by-point measurement and is very time consuming. An alternative method especially for surface analysis is glow discharge optical emission spectrometry (GD-OES). The central aim of this project is, therefore, the development of a large-area GD source with monochromatic-imaging detection as a new highly sensitive alternative method.



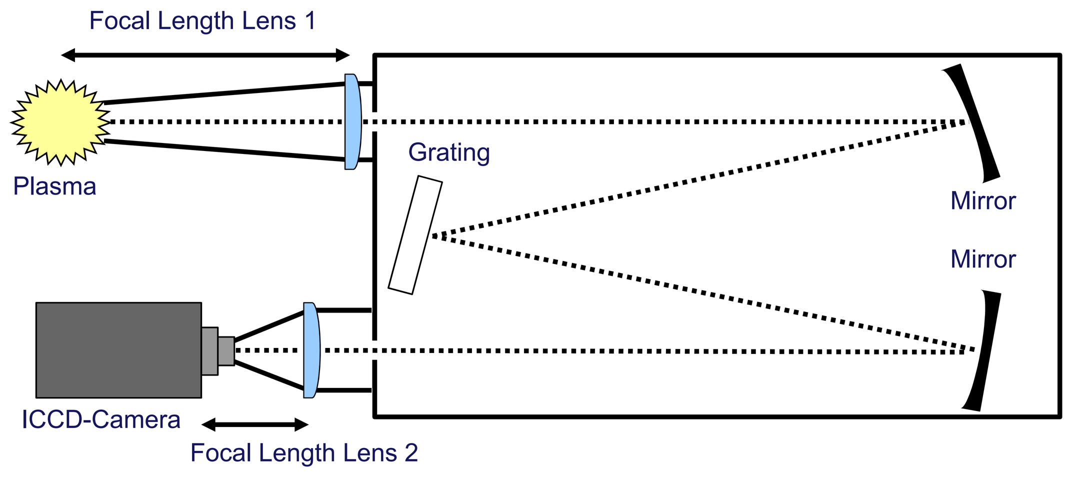
Previous work: Photograph of Western blot and Ag(I) emission by rf-GD. (Picture taken from [1]).



Instrument setup. Pulsed 4-cm GD source with monochromatic imaging spectrometer (MIS).

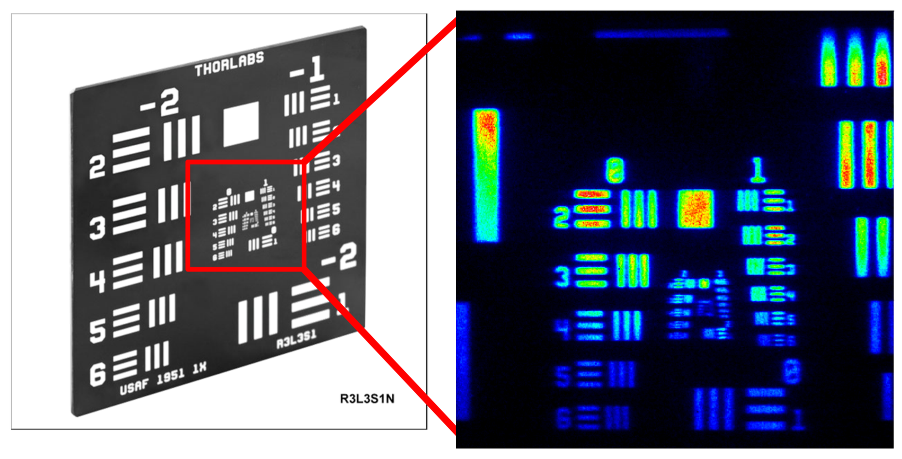
Monochromatic Imaging Spectrometer: How it works

Wavelength separation via monochromator. Lowest detectable wavelength is 200 nm. Good wavelength resolution (down to 0.04 nm; line scanning).

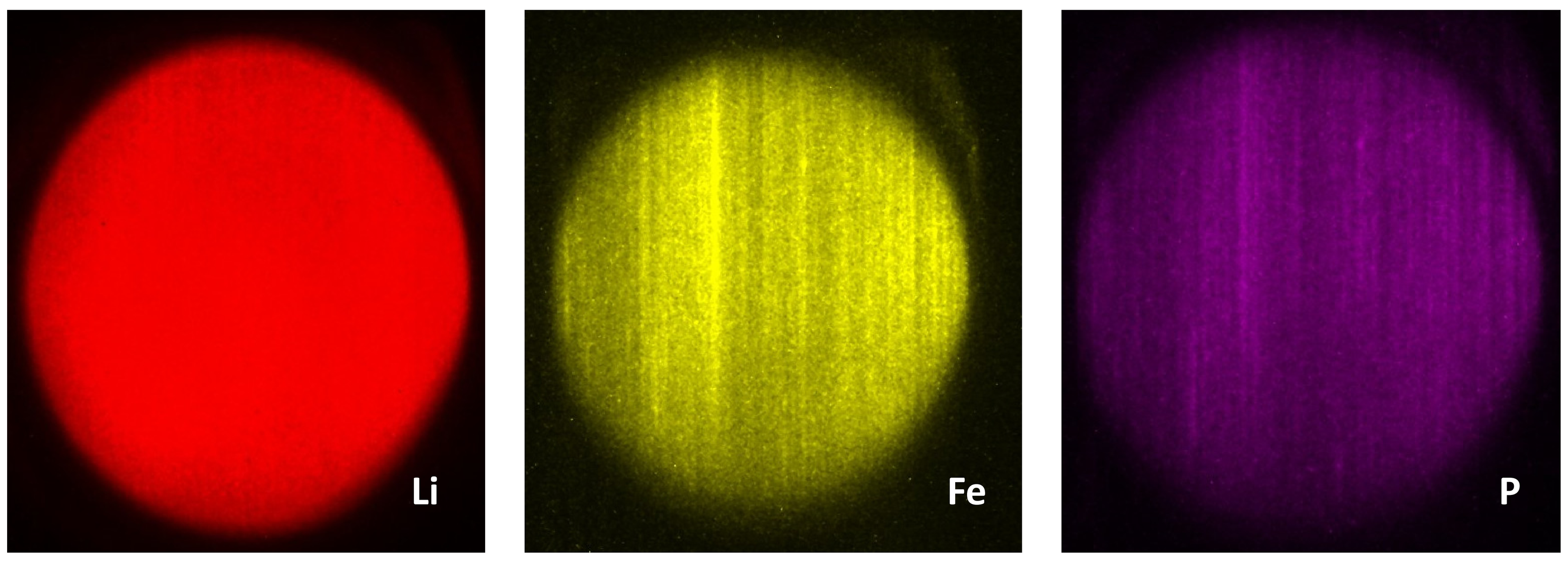


Spatial resolution: USAF 1951 backside illuminated (W-lamp):

- horizontal: 450 µm
- vertical: 250 µm

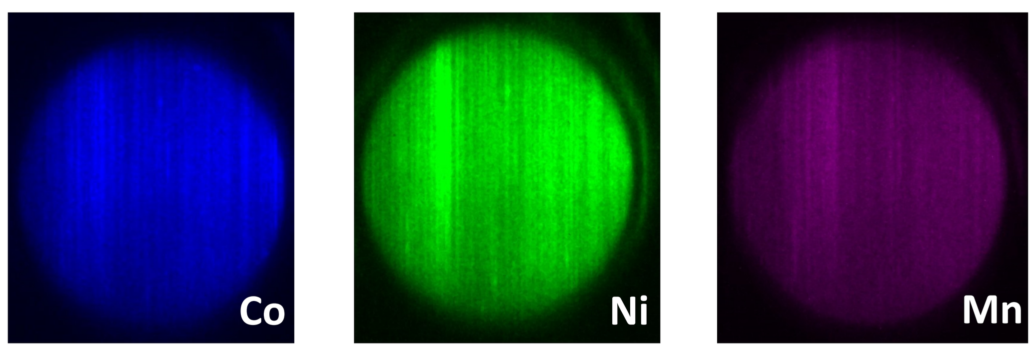


Results



First measurements of large cathode areas.
Successful analysis of various elements (Li, P, Fe, Ni, Co, Mn) in lithium ion battery cathodes.
Homogeneous distribution of all elements at the surface.

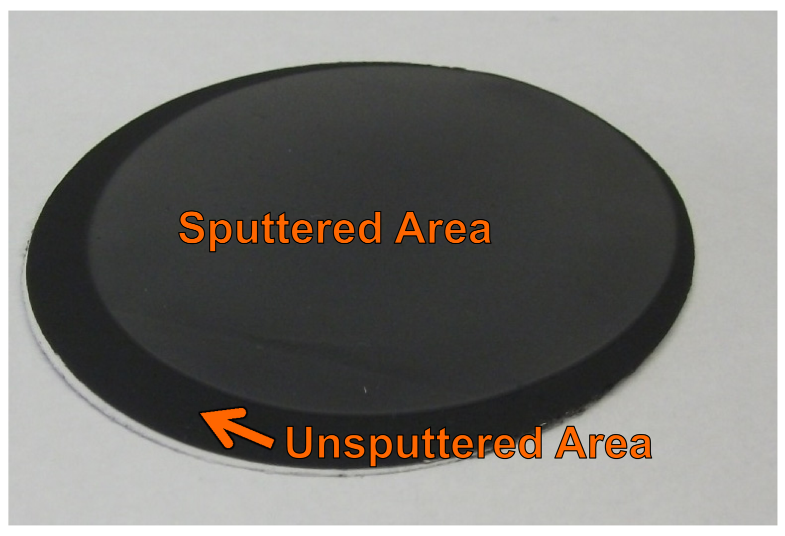
top: Spatial distribution of Li, Fe, P in LFP.
right: Monochromatic images of Co, Ni, Mn in NCM.



Application: Lithium Ion Battery Samples

The cathode materials LFP ($LiFePO_4$) and NCM ($LiNi_{0.33}Co_{0.33}Mn_{0.33}O_2$) were analyzed with pDCGD-OES. Beside the different cathode materials the samples contained elemental carbon as conductor and PVDF as binding component. The battery materials were mixed and then deposited in thin films on Al foil.

LFP: 90% $LiFePO_4$, 5% C, 5% PVDF (Binder)
NCM: 90% $LiNi_{0.33}Co_{0.33}Mn_{0.33}O_2$, 5% C, 5% PVDF (Binder)



Technical Setup

Function Generators: Peaktech 4055 (Ahrensburg, Germany), pulse frequency 1 kHz (rectangular function), 0.2% duty cycle, pulse length 20 µs
DC-Generator: IRCO Model M3kS-20N, 3kV negative Pulse generator (Instrument Research Company, Columbia, MD, USA)
Glow Discharge Source: in-house built plasma source, cathode diameter 40 mm, water cooled Cu-cathode
Monochromator: Acton SP2750 (Princeton Instruments, Trenton, NJ, USA), 75 cm
ICCD-Camera: PI-MAX 3 1024i (Princeton Instruments, Trenton, NJ, USA)
Observed wavelengths: λ(P) = 215.41 nm; λ(Ni) = 221.65 nm; λ(Co) = 238.89 nm; λ(Mn) = 257.61 nm; λ(Fe) = 374.48 nm; λ(Li) = 460.24 nm

Literature

[1] G. Gamez, S. J. Ray, F. J. Andrade, M. R. Webb and G. M. Hieftje, *Anal. Chem.*, **2007**, *79*, 1317-1326.

Future Perspectives

Further improvement of spatial resolution in the plasma.
Development of quantification strategies in various matrices.
Investigation of matrix effects on spatial resolution (e.g. oxygen).
Determination of LOD and LOQ for different elements.

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We thank the machine workshop and Michael Kießhauer from the Institute of Inorganic and Analytical Chemistry at the University of Münster The travel grant from the German Chemical Society (GDCh) is gratefully acknowledged.
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