C K-edge XANES-measurements using an off-axis reflection zone plate with a Laser-Produced Plasma Source

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Motivation

The electronic structure of molecules and biological samples can be used as an indicator for their functionality and is one of the key issues of current research, e.g. in life science. X-ray absorption spectroscopy (XAS) of the K-edges of C, N and O radiation is a well-established and often used research method for such kind of samples at synchrotron sources.

A newly developed laser-produced plasma (LPP) source [1] offers the possibility to realize XAS in the soft X-ray region in the laboratory. We present the feasibility of X-ray absorption near-edge structure (XANES)-measurements with the LPP source at the C K-edge. Two wavelength dispersive schemes were tested for their applicability. Investigations of different polymer thin foils are realized and structural characteristics due to the chemical composition can be made visible.

Instrumentation

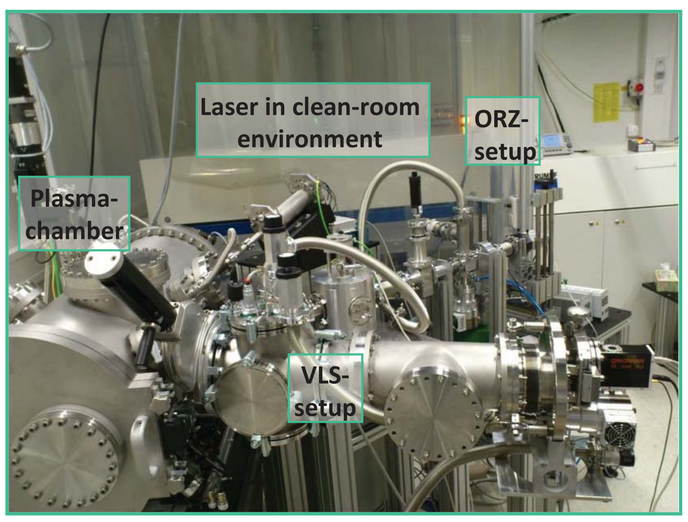
Laser System & Plasma Chamber

TRUMPF

Laser parameter:

Pulse energy	220 mJ
Pulse duration	240 ps, 1.2 ns, 12 ns
Repetition rate	100 -200 Hz
M^2	< 1.2
Laser focus size	10x10 µm²

Top: View inside the plasma chamber. Laser beam propagation, position of plasma generation and X-ray emission are indicated.



Left: Present configuration of the LPP source. Two beamlines for simultaneous measurements are available. Here, two setups for XAS-measurements are realized. Back: setup with an **off-axis reflective zone plate (ORZ)** as a dispersive element. Front: **varied-line-space-grating-spectrometer (VLS)** setup.

Setups for XAS-measurements

ORZ setup:

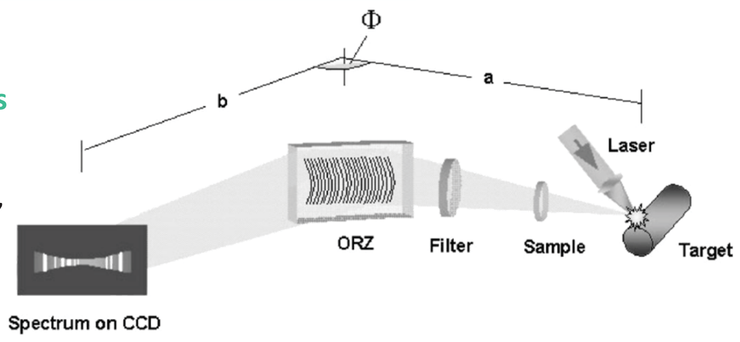
• x-ray optical element: **Off-axis reflective zone plate**

• designed for 4.03 nm (308 eV, C He-α emission line)

→ $\lambda / \Delta\lambda = 1000$

• achieved resolution for the C K-edge (284.2 eV) [2]:
$E/\Delta E = 560$

• 200 nm Al-filter (*Lebow*)



VLS-spectrometer setup:

• Calibrated **Varied-line-space grating** (*Hitachi)*: 2400 lines / mm

• CCD camera (*Greateyes*):
13 x 13 µm², 2048 x 512 pixel

• 200 nm Al-filter (*Lebow*)

• electrical shutter (*Melles Griot*)

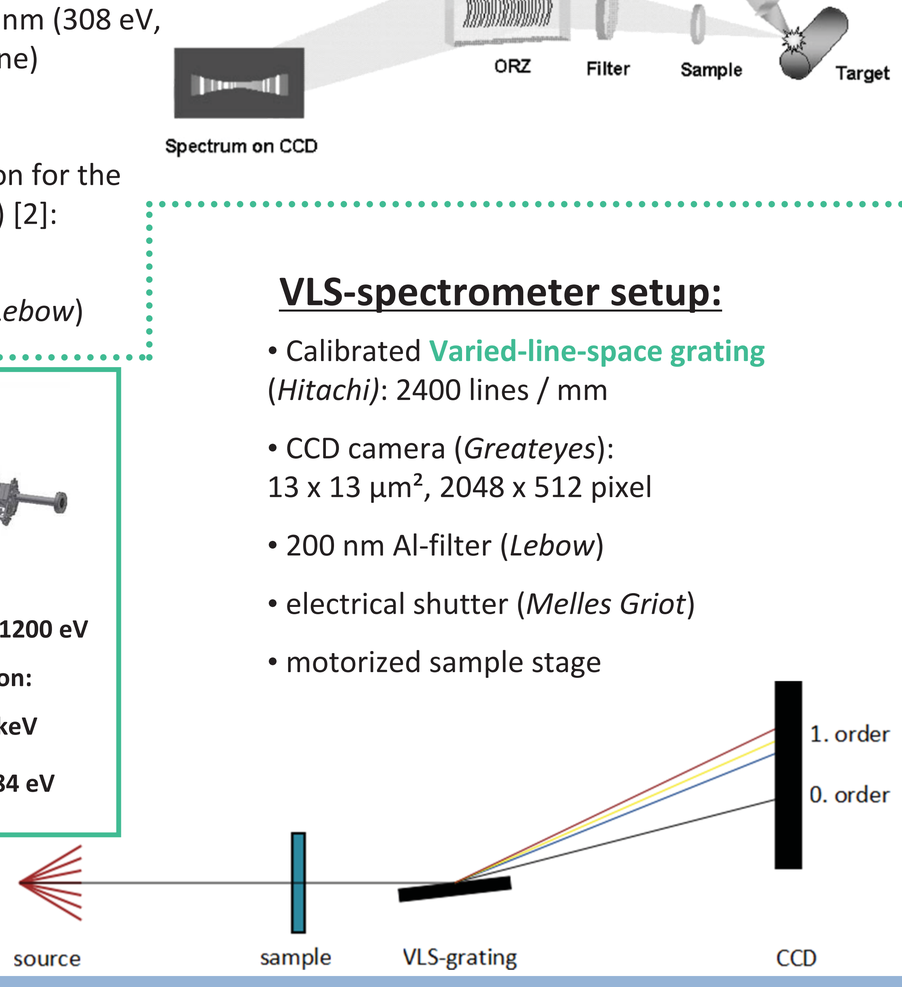
• motorized sample stage

Energy range: 200 – 1200 eV

Energy resolution:

ΔE = 5 eV @ 1 keV

ΔE = 0.5 eV @ 284 eV (C K-edge)



Results

Simultaneous measurements of K-absorption edges with the VLS-setup

• Sample: 112 nm polyimide foil
(LUXEL Corporation)

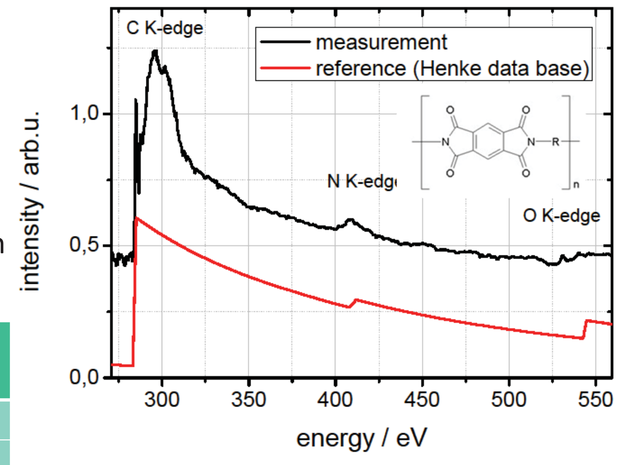
• Absorption edges in detected energy region: C, N, O

• Accumulation time: 20 s

• Energy values of K-edges lie in between Ref. 1 and 2

	Energy eV	Reference 1 eV	Reference 2 eV
C K-edge	283.3	284.2	284
N K-edge	408.3	409.9	402
O K-edge	539.2	543.1	537

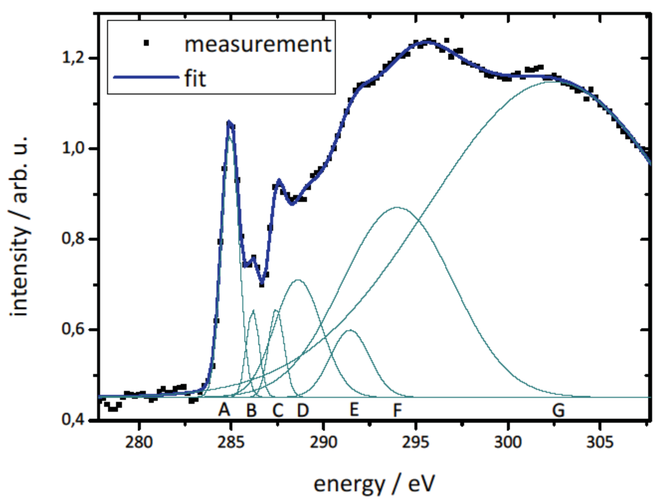
[Ref. 1: Henke data base, Ref. 2: McMaster data base]



• Detailed XANES-spectrum of the C K-edge

• Fitted with seven Gaussian curves (solid line)

• $E / \Delta E \approx 450$ @ C K-edge



	Energy eV	Assignment	Reference data* eV
A	284.9	1s → π *	285.2
B	286.2	1s → π *	286.6
C	287.4	1s → π *	287.4
D	288.6	1s → π *	289.2
E	291.4	1s → σ *	291.9
F	294.0	1s → σ *	295.4
G	302.5	1s → σ *	303.1

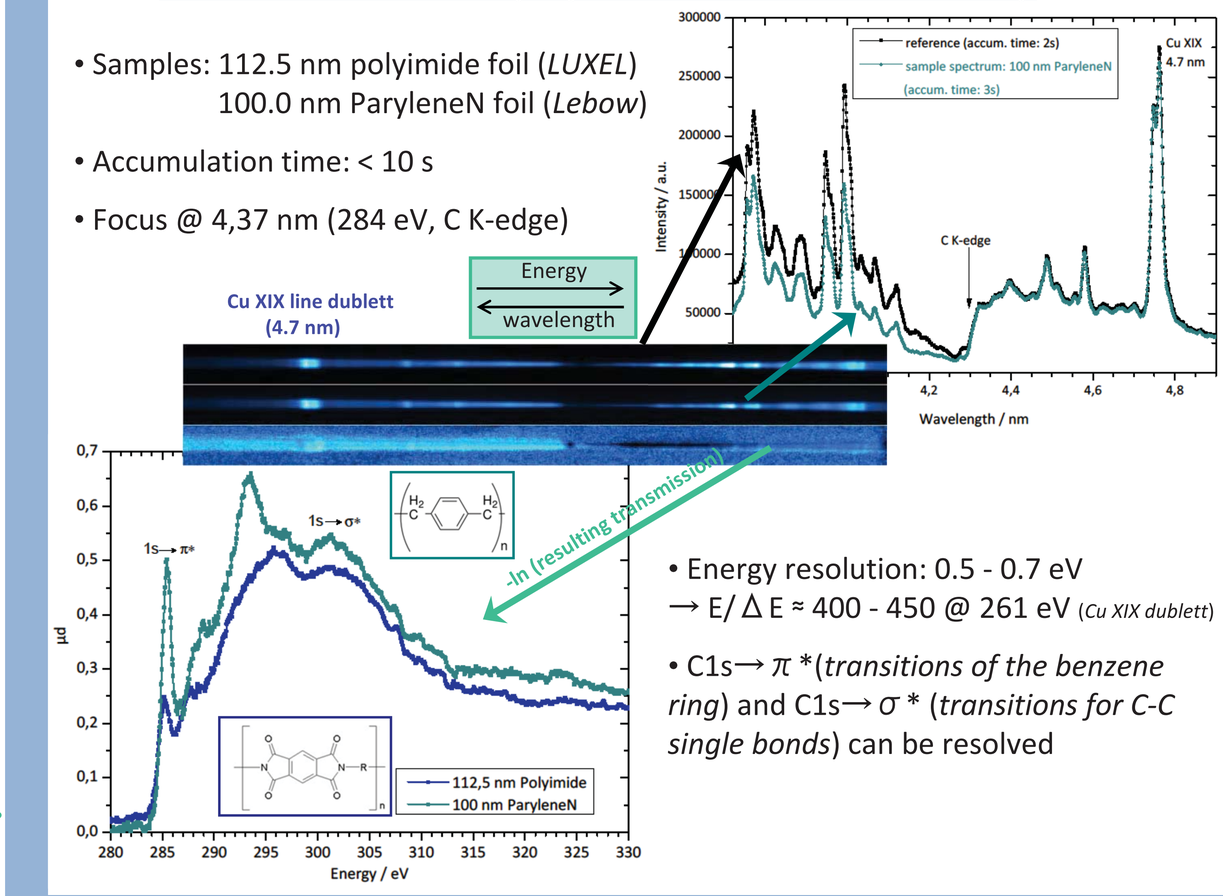
*** J. L. Jordan-Sweet, Chem. Phys. 89, 2482 (1988)**

Differentiation of thin polymer-foils with the ORZ-setup

• Samples: 112.5 nm polyimide foil (*LUXEL*)
100.0 nm ParyleneN foil (*Lebow*)

• Accumulation time: < 10 s

• Focus @ 4,37 nm (284 eV, C K-edge)



• Energy resolution: 0.5 - 0.7 eV
→ $E/\Delta E \approx 400$ - 450 @ 261 eV (*Cu XIX dublett*)

• C1s→ π *(*transitions of the benzene ring*) and C1s→ σ * (*transitions for C-C single bonds*) can be resolved

Conclusion & Outlook

	VLS-Spectrometer	ORZ-Setup
Energy range	200 – 1200 eV	250 – 350 eV
Accumulation time	< 1 min	< 10 s
$E / \Delta E$ total range	250 – 450	350 – 450
$E / \Delta E$ @ C K-edge	450	400

VLS: higher resolution, compact system, broad energy range ⟷ **ORZ:** short measuring time, can be optimized for a specific absorption edge

• with both wavelength dispersive schemes different polymers could be distinguished

• for fast measurements at specific absorption edges calculations for optimized ORZ are in progress

• measurements on biological samples in native environment will be carried out in the near future

[1] I. Mantouvalou, R. Jung, T. Tuemmler, H. Legall, T. Bidu, H. Stiel, W. Malzer, B. Kanngießer, and W. Sandner, Rev. Sci. Instrum. 82, 066103 (2011)

[2] U. Vogt, T. Wilhein, H. Stiel, and H. Legall, Rev. Sci. Instrum. 75, No. 11, (Nov. 2004)

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