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LumStimAge project: Application of TL/OSL imaging in dating and characterisation of heritage objects.

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HAL Authorization



The C2RMF has acquired a imaging system of thermally stimulated luminescence (TL) and optically stimulated luminescence (OSL) for heritage materials. The use of TL/OSL imaging is challenging due to the low light intensity and the fast kinetics of the observed phenomena.

Hardware

TL/OSL reader

Lexsyg Research (*Freiberg Instruments GmbH*)
A modular reader with separated chamber for samples storage and measurements

Automatic detector changer:

- PMT UV-Vis Hamamatsu H7360-02
- EMCCD

B-source (RFQ2) Sr-90 1.85 GBq

T° up to 710 °C @ 0.1 - 20.0 °C/s

Blue LED 458 ± 5 nm (max. 100 mW/cm²)

IR LED 850 ± 20 nm (max. 300 mW/cm²)

2 filterwheels

Hardware

Camera device

Camera Andor TM iXon Ultra
Model DU-897U-CS0-#EX

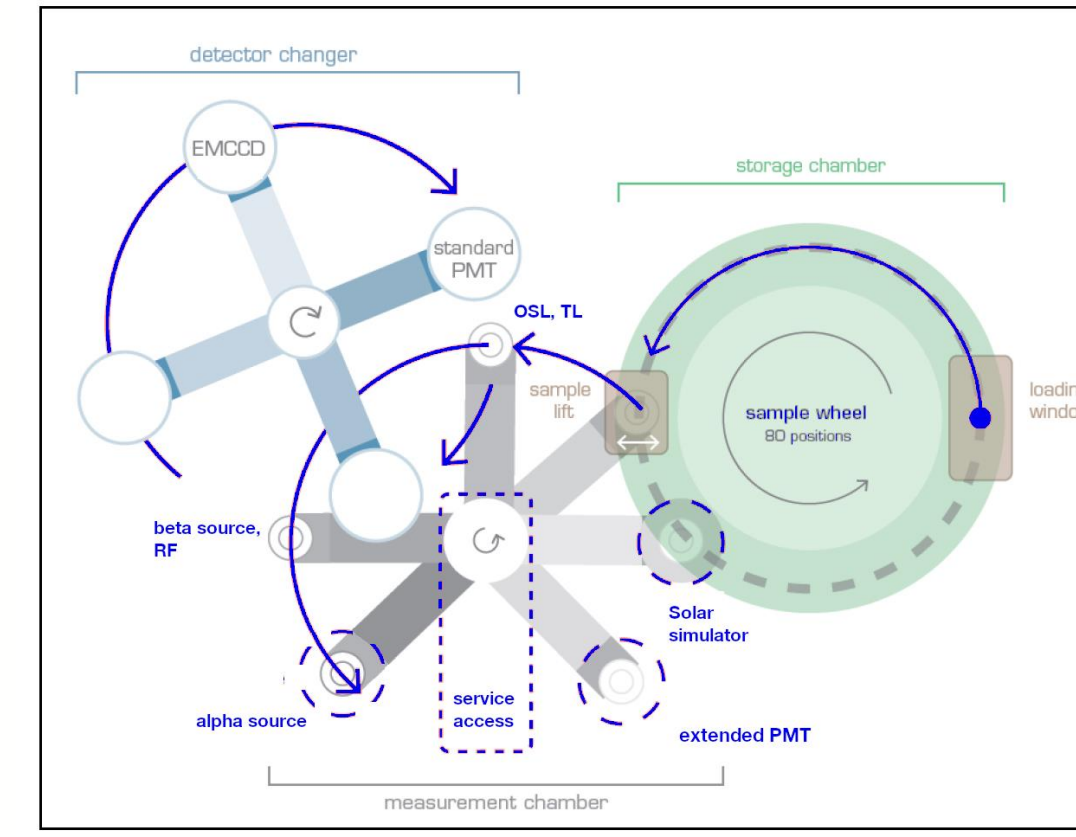
Sensor E2V/CCD97-00-1-175

512 × 512 (16 μm × 16 μm)

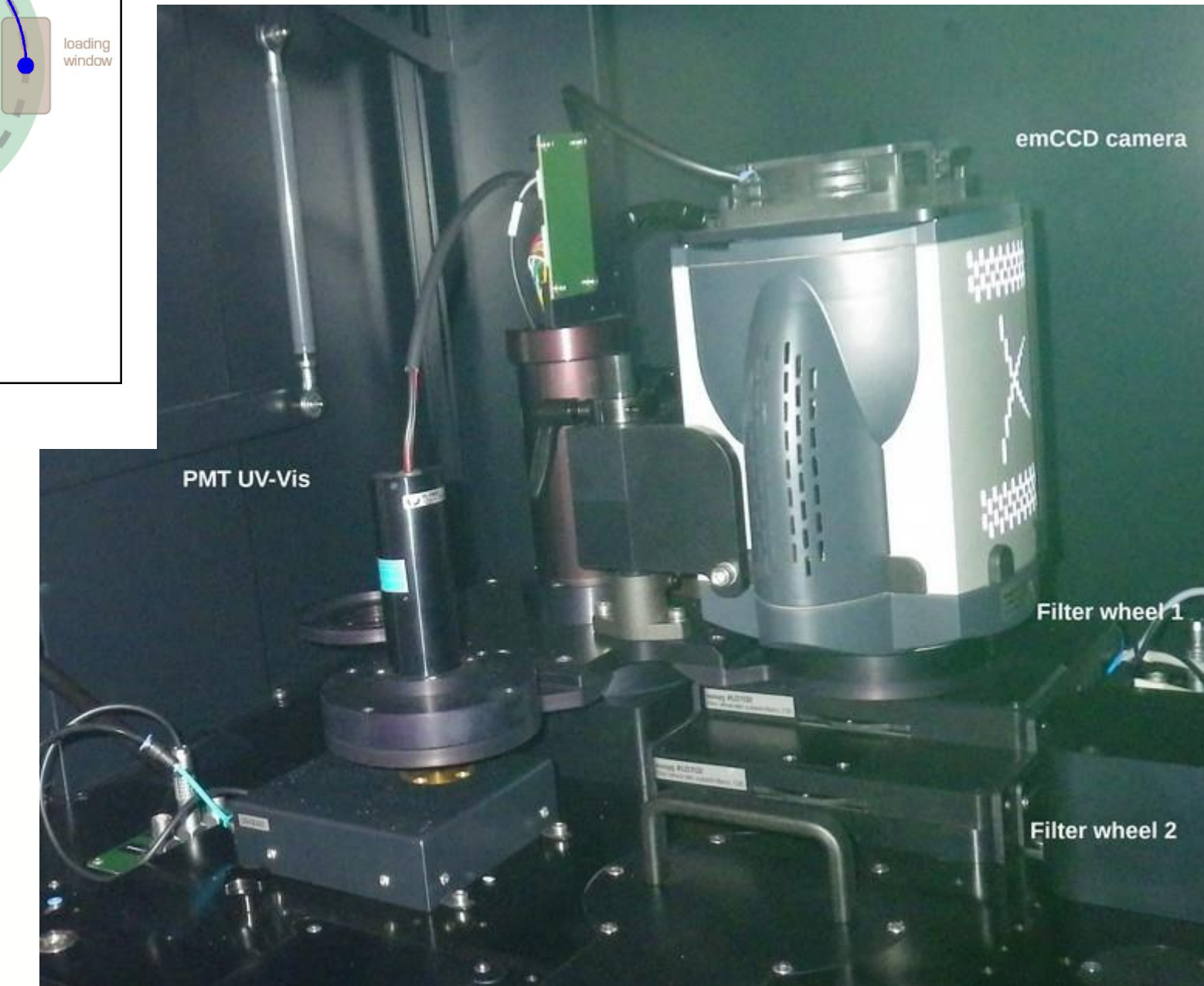
Back-illuminated, Dual AR coated

broadband VUV-NIR wedged

Air cooling down to 80 °C



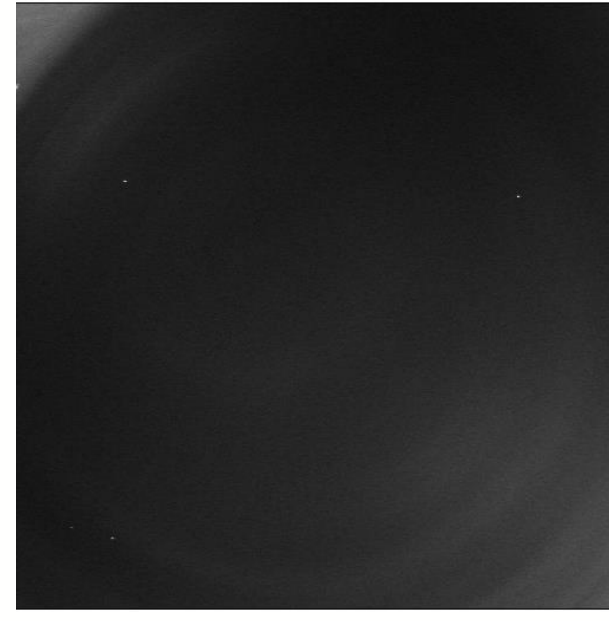
Sample handling modified from instruction manual v.1.53 figure 5 © FreibergInstrument GmbH



Filter and fluorescence

A signal in the form of concentric bands masking the luminescence of the grains was observed under infrared stimulation. After testing different filter sets, it was found that this signal was linked to the HC414/46 filter and was attributable to spurious fluorescence.

Filter set name	IRSL/TL-340nm	IRSL/TL-410nm	IRSL/TL-565nm	IRSL/TL-wideband blue
Filterwheel 1	BG39	BG39	BG39	BG25 x BG39
Filterwheel 2	HC340/26	HC414/46	HC575/25	KG3
Spurious emission	low	high level	very low	n



Spurious fluorescence from a HC414/46 3.5nm filter observed on a non-irradiated disc © Zink

Discs and cups

Various types of container can be used for our samples. The nature of the sample and the way it is fixed must be taken into account, as well as the type of disc coating.

- Discs and cups are made of stainless steel for working temperatures of up to 700°C;
- fine grains 4-10μm (large aliquot): a monolayer of grains is deposited in acetone on disk, which they cover completely. The appearance of the disk is irrelevant;
- small aliquot (dia. 2-4mm): the fine grains cover only part of the disc. The disc should be polished to avoid reflection from stimulation towards the camera;
- dosimetric pellets (dia. 5mm): these pellets are placed in cups and fixed with silicone spray to prevent them from moving. Their luminescence is enough intense for the appearance of the cup to be irrelevant;
- coarse grains 100-300μm: they are mounted on polished disc using silicone spray.

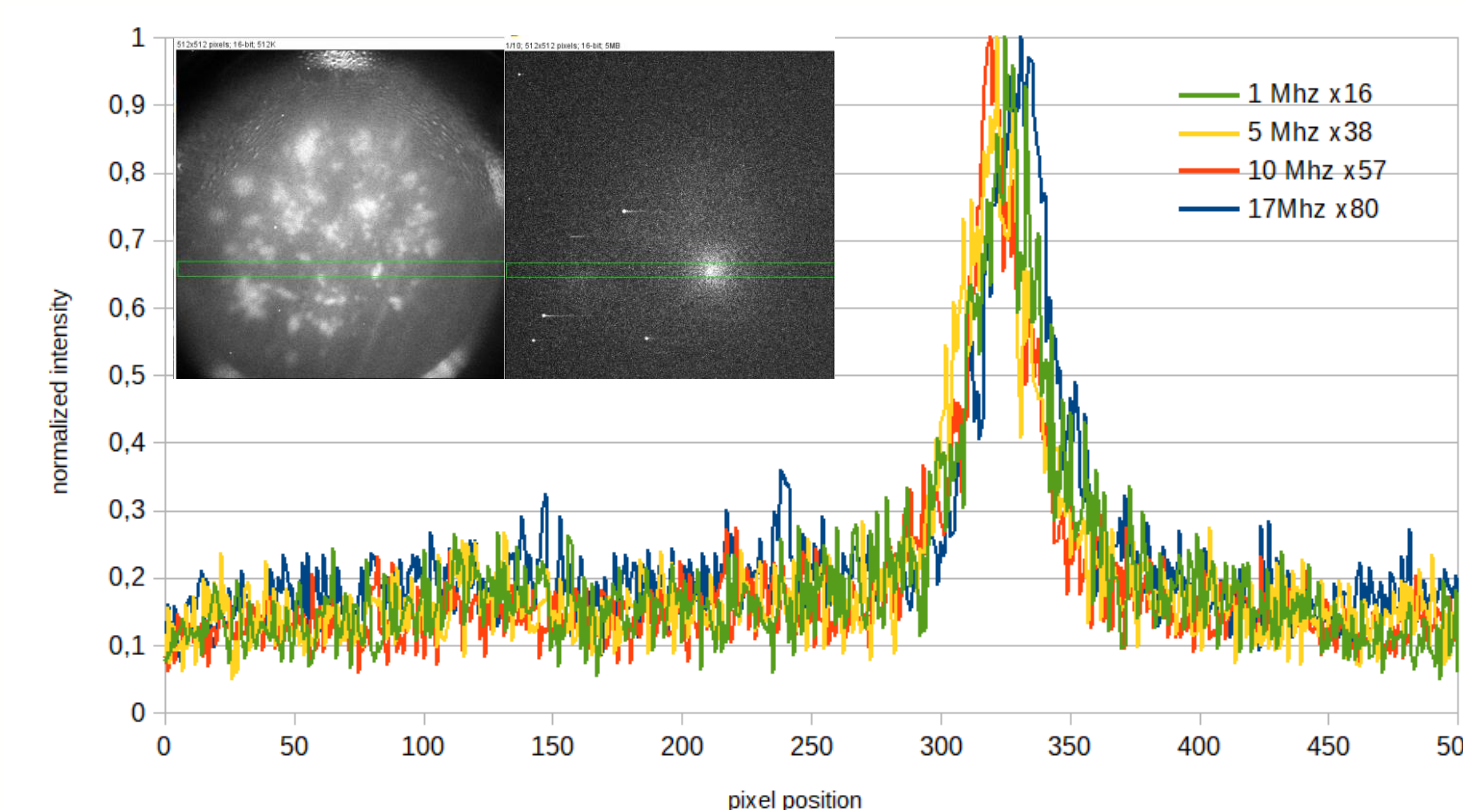
Optimizing luminescence

The first task was to determine the appropriate parameters to optimize the signal.

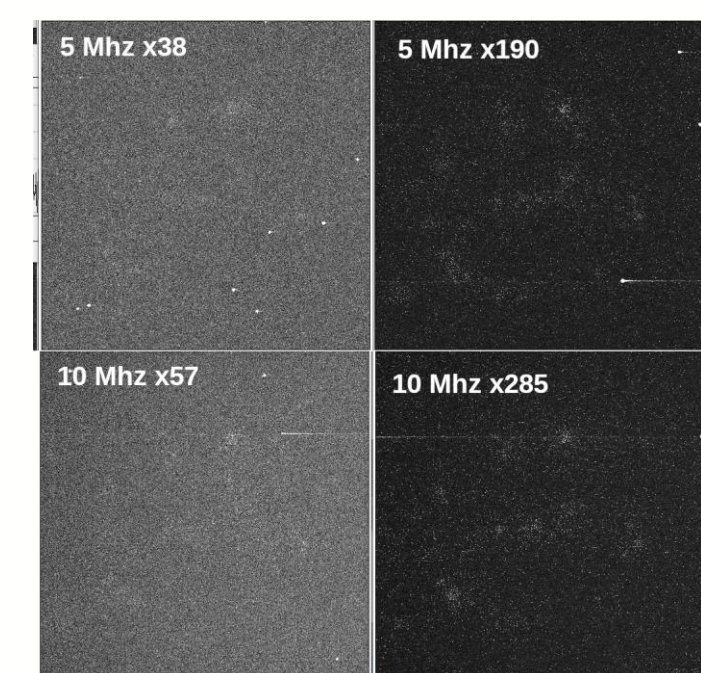
- Shift speed: 1.7μs (**recommended**)
- Vertical clock voltage Amp: Normal (**recommended**)
- PreAmplifier setting: 3 (ca. 4-5 e-/count)

EM gain and readout
Test on fluvial sediment from Somme river (France) irradiated with 21 Gy and 210 Gy

Readout rate	Read noise (calibration sheet)	EM gain (RN x1e- rms)	EM gain (RN x5e- rms)
1 MHz	15.5	X 16	-
5 Mhz	37.9	X 38	X 190
10 Mhz	57.4	X 57	X 285
17 MHz	79.8	X 80	-



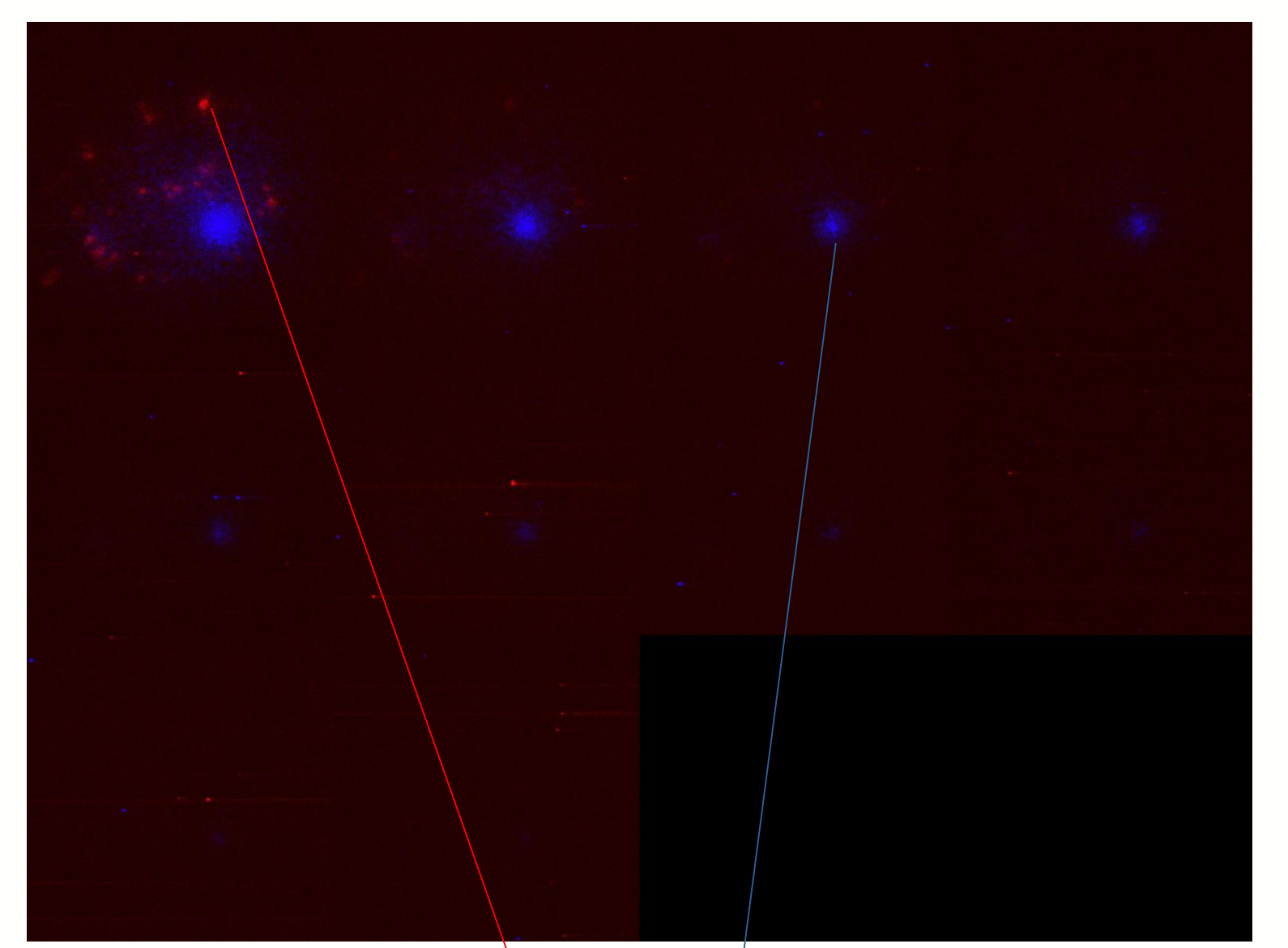
Normalized plot profiles (insert photo and OSL 1-10s, irr. 210 Gy, 10 Mhz x57 with selected profile) © Zink



IR-OSL 1-10s, irr. 21 Gy © Zink

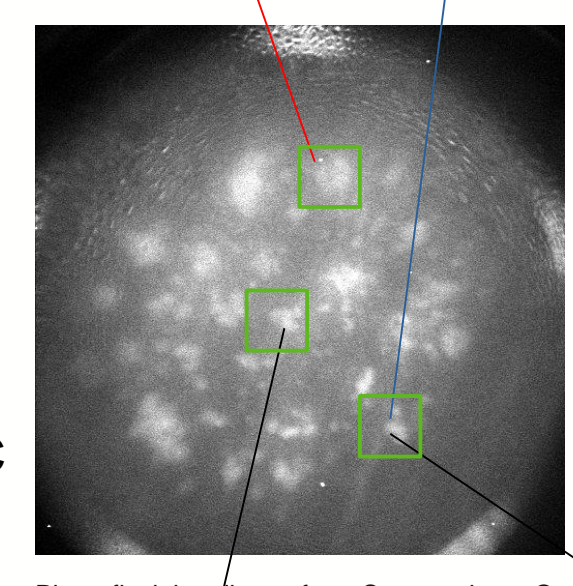
Selected values
Readout rate 5 Mhz
EM gain x190

Application OSL / TL from sediment



Composite IR-OSL (red) and BL-OSL (blue) images 0-100s, 1 image/10s, irr. 210 Gy, 5 Mhz x190 © Zink

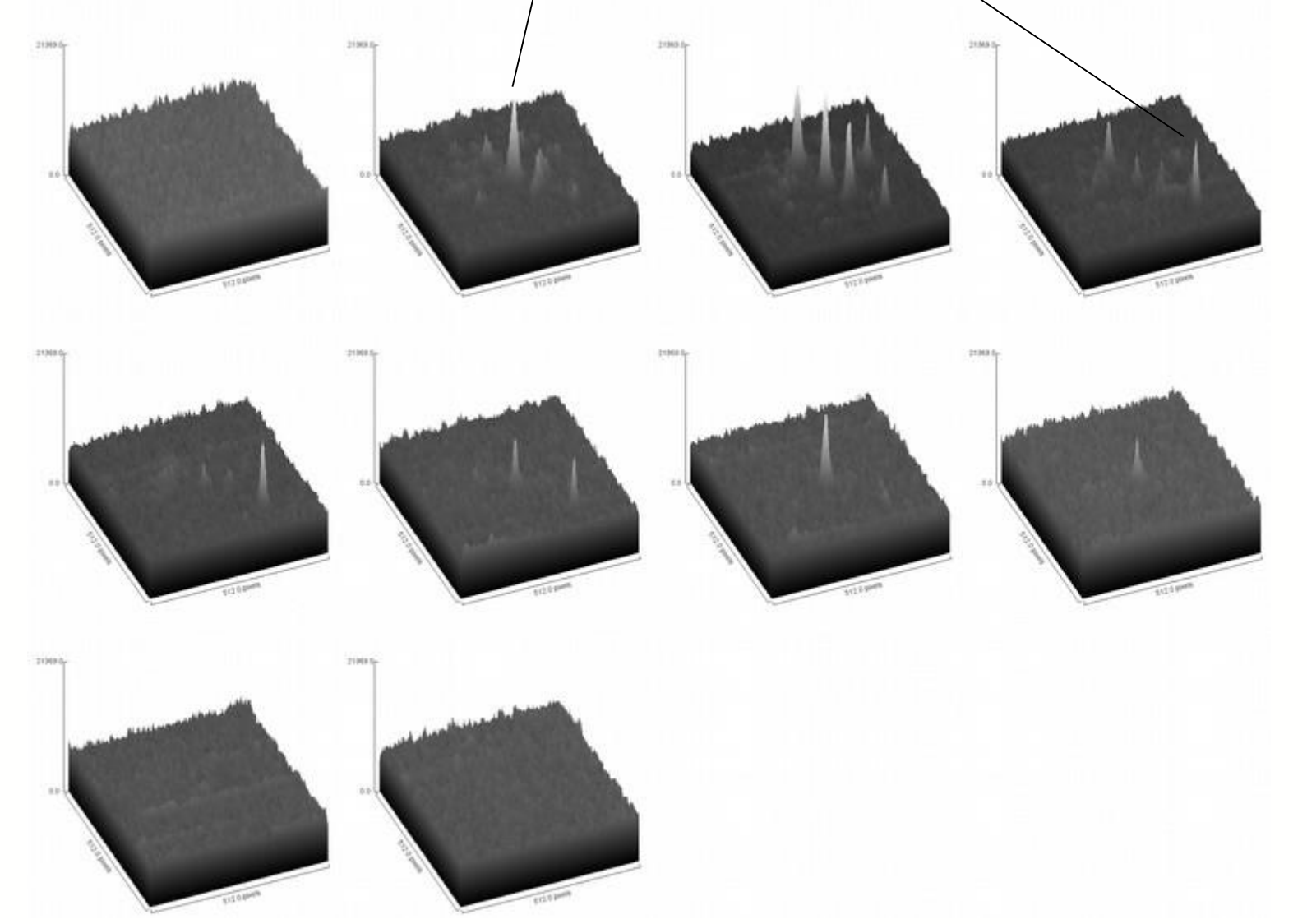
this grain responds under IR-OSL



this grain presents a TL with two peaks at 100°C and 400°C

this grain has a high BL-OSL and a TL with a peak at 250°C

Photo fluvial sediment from Somme river © Zink



Thermoluminescence 25 - 500°C 5%/s 1image/50°C irr. 115 Gy, 5 Mhz x190 uncorrected from background emission © Zink

Conclusion

The main hardware parameters have been optimized. Binning, time exposure, temperature cooling being sample dependent. The next step is to improve image processing for low-intensity measurements (sample irradiated around 1 Gy). The system can already be used for qualitative studies of polymineral samples in various forms