Message ID: **182** Entry time: **Mon Jul 10 14:15:13 2023**

Experiment Date:	2023 07 10
Duration (Days):	5
User:	Stjepko Fazinic, Iva Bozicevic Mihalic, Anja Miokovic
Accelerator:	Tandetron
Beam Line:	Old uProbe
Project:	Hi-REXS (HRZZ projekt)
Experiment Title:	HR PIXE Na compounds + Zn, Cr in paint layer cross-section (Mudronja's sample 27608)
Beam:	2MeV H, 3 MeV He
Method:	HR PIXE, PIXE

Beam deflection was connected to the horizontal deflector with -700 V.

GreatControl: X Binning = OFF, Y Binning = OFF, Readout Speed = 500 kHz, Gain = Max Sensitivity, Correct Bias = UNCHECKED, T_{ccd}= -70 ºC, T_{back}= 23 ºC, Chiller at 18 ºC

Chamber positioned according to the black marks on the floor that were drawn in February.

Surface barrier detector is added to the chamber for RBS.

10.7.2023.

Beam: 2 MeV H^+ TDT

Focus: Me 17.3 A, Ox 34.7 A

Diffraction crystal: Beryl(1010) at 10.5 cm, peeking out of holder 3 mm, with blue dot facing the main door

Samples: NaBr+Zn (position 1), Na_2SO_4+Zn (position 2), Na+Zn (position 3), NaF+Zn (position 4), NaBr (position 6)
All samples except Na+Zn are in the form of pellets. Zn powder was put on top of Na compound powder before pressing it into a pellet, so Zn is present only on one side of each pellet.
Na+Zn is prepared the same way as in March (inside of the Ar balloon) and once suitable piece of Na is cut Zn powder is pressed on top of it.
Sample is taken to the chamber inside of the bag filled with Ar. Before that chamber is vacuumed roughly and then filled with N_2. Sample is taken out of the bag as close as possible to the chamber door and immediately put inside.

We are using SDD spectra to help us decide in which position we want to measure each sample.

WARNING! NaF = "Toxic if swallowed. Wash skin thoroughly after handling."

FILE	SAMPLE	N_{frames}	t_{exp}/s	COMMENT
2307028	Na?	1	10	I~2 nA, NaKa line visible in Vista at ch~570; we inspect part of Na+Zn sample without Zn to check if we have pure Na or if it has oxidized, in SDD spectrum we see no Zn
2307029	Na?	1	7	
2307030	DARK	10	7	
2307031	Na?	200	7	I~2 nA, +SDD spectrum (same name)
2307032	Na?	50	7	I~2 nA
2307033	Na?	200	7	- || -, conclusion after Matlab analysis: some form of Na oxide, not pure Na!

Since we had Zn on top of Na we could not scrape off the surface layer of the sample after putting it inside of the chamber. We think that could be the reason for formation of oxide.
We will try to use small holder made from Zn (Andro made it) into which a piece of Na can be pressed. That way the surface layer of Na can be scraped off after the sample is put inside of the chamber.

Positions of samples is changed: Na inside Zn holder (position 1), Na_2SO_4+Zn (position 2), NaBr+Zn (position 3), NaF+Zn (position 4), NaBr (position 6)

FILE	SAMPLE	N_{frames}	t_{exp}/s	COMMENT
2307034	Na+Zn?	1	10	I~2 nA; as we were vacuuming the chamber we were seeing some reaction happening, resulting with orange color of the sample
2307035	DARK	10	10	
2307036	Na+Zn?	150	10	+ SDD spectrum; we assume that sodium zincate has been produced: 2NaOH + Zn -> $2Na_2ZnO_2$ + H_2 (at atmospheric pressure it happens on 550°, but we have vacuum, link: Zinc reacts with aqueous sodium hydroxide to form dihydrogen and _____.A. Zinc oxideB. Sodium zincateC. Zinc hydroxideD. Sodium precipitate (vedantu.com))

Conclusion: We are not able to prepare pure Na. It could be that there is too much moisture in air now so our Na sample is oxidizing faster than in March. We can try again in autumn.
Also, there is possibility that we will have to try to mix Na with other Zn compound with which it won't react.

11.7.2023.

Beam: 2 MeV H^+ TDT

Focus: Me 16.4 A, Ox 34.7 A

Diffraction crystal: Beryl(1010) at 10.5 cm, peeking out of holder 3 mm, with blue dot facing the main door

Current on metal before measurement ~6 nA (Andro's slits closed a bit to reduce the current)

Samples: Na_2SO_4+Zn (position 2), NaBr+Zn (position 3), NaF+Zn (position 4), NaBr (position 6)

We use SDD spectrum to help us determine good position for measurement.
We search for position where we see both SK and ZnK peaks in SDD spectrum.

FILE	SAMPLE	N_{frames}	t_{exp}/s	COMMENT
2307037	Na_2SO_4+Zn	1	20	I~4 nA, NaKa line visible in Vista at ch~570, we are not sure about Zn -> starting to collect and then we will know from Matlab analysis
2307038	Na_2SO_4+Zn	1	8	
2307039	DARK	10	8	
2307040	Na_2SO_4+Zn, 3 different positions!	130	8	I~4 nA; energy window used in Matlab analysis: [200,300]; 1st position (frames 0-25): beam spot is shining a lot -> only Na; 2nd position (frames 39-90): beam spot barely shining -> Zn mush higher than Na; 3rd position (frames 102-129): beam spot shining with low intensity, I(SK)/I(ZnKa)=2.5/1 in SDD spectrum, I(ZnLa)/I($NaKa_{12}$)=1.8/1 in HR spectrum -> we are satisfied with 3rd position!
2307041	DARK	10	6	
2307042	Na_2SO_4+Zn, decided position	350	6	+ SDD spectrum
2307043	- || -	450	6	+ SDD spectrum; after some time I($NaKa_{12}$)>I(ZnLa)
2307044	- || -	450	6	+ SDD spectrum, current fell and then was increased to ~6 nA
2307045	- || -	34?	6	measurement stopped because in the meantime we analysed collected frames and concluded that growing of Zn lines became to slow -> moving to new position!
2307046	Na_2SO_4+Zn, newly decided position	450	6	I~7 nA, +SDD spectrum, new position is close to the carbon tape
2307047	- || -	450	6	SDD spectrum collected in the same file as previous one(2407046.dat)
2307048	- || -	450	6	+ SDD spectrum
2307049	- || -	450	6	- || -
2307050	- || -	320	6	- || -

12.7.2023.

Beam: 2 MeV H^+ TDT

Focus: Me 16.6 A, Ox 34.8 A

Diffraction crystal: Beryl(1010) at 10.5 cm, peeking out of holder 3 mm, with blue dot facing the main door

Samples: NaBr+Zn (position 1), Na_2SO_4+Zn (position 2), Na+Zn (position 3), NaF+Zn (position 4), NaBr (position 6)

We use SDD spectrum to help us determine good position for measurement.
We search for position where we see both FK and ZnK peaks in SDD spectrum.

FILE	SAMPLE	N_{frames}	t_{exp}/s	COMMENT
2307051	NaF+Zn	1	5	I~7 nA, measuring sample on the position near the carbon tape, I(ZnKa)/I(FK)=70/1 in SDD spectrum
2307052	DARK	10	5	
2307053	NaF+Zn	1	3	exposure time is reduced because we were seeing too many events in one frame in Vista
2307054	DARK	10	3	
2307055	NaF+Zn, 2 different positions!	720	3	very unstable beam, +SDD spectrum (which is restarted after moving); 1st position (frames 0-50): same as previous one, at some point we lost Na peak in HR spectrum; 2nd position (frames 58-719): I(ZnLa)/I($NaKa_{12}$)=2.3/1 in HR spectrum-> we are staying here!
2307056	NaF+Zn, decided position	638?	3	+SDD spectrum, stopped because beam we lost the beam
2307057	- || -	720	3	I~5 nA (finally more stable), +SDD spectrum
2307058	- || -	720	3	I~5-4.5 nA, +SDD spectrum
2307059	- || -	720	3	I~4 nA, +SDD spectrum, Matlab analysis of this round shows change in peaks intensities: I($NaKa_{12}$)/I(ZnLa)=2/1,
2307060	- || -	720	3	I~4 nA, +SDD spectrum

13.7.2023.

Beam: 2 MeV H^+ TDT

Focus: Me 16.6 A, Ox 34.6 A

Diffraction crystal: Beryl(1010) at 10.5 cm, peeking out of holder 3 mm, with blue dot facing the main door

Current on metal before measurement ~5 nA

Samples: NaBr+Zn (position 1), Na_2SO_4+Zn (position 2), Na+Zn (position 3), NaF+Zn (position 4), NaBr (position 6)

We use SDD spectrum to help us determine good position for measurement.
We search for position where we see both BrL and ZnK peaks in SDD spectrum.

FILE	SAMPLE	N_{frames}	t_{exp}/s	COMMENT
2307061	NaBr+Zn, 1st position	30	5	measuring sample on the position near the carbon tape; I(ZnKa)/I(BrLa)=19/1 in SDD spectrum, I(ZnLa)/I($NaKa_{12}$)=1/1 in HR spectrum
2307062	NaBr+Zn, 2nd position	30	3	I(ZnKa)/I(BrLa)=30/1 in SDD spectrum, I(ZnLa)/I($NaKa_{12}$)=3/1 in HR spectrum
2307063	DARK	10	3	
2307064	NaBr+Zn, 2nd position	180?	3	I~3.6 nA but falling (again unstable), +SDD spectrum, stopped because current was falling way too fast
2307065	DARK	10	1	
2307066	NaBr+Zn, 2nd position	200	1	I~7 nA > falling slower now, it is constantly needed to change values of switching magnets
2307067	- || -	900	1	
2307068	- || -	900	1	I~7 nA, +SDD spectrum (everything up to frame 69 of this round was collected in one file - 2307064.dat)
2307069	- || -	900	1	I~7 nA, +SDD spectrum
2307070	- || -	900	1	- || -; there is a change in peaks intensities in SDD spectrum: I(ZnKa)/I(BrLa)=4.6/1, but in HR spectrum Zn peak is still higher than Na: I(ZnLa)/I($NaKa_{12}$)=1.6/1

2307071	- || -	900	1	I~7 nA, +SDD spectrum; I(ZnKa)/I(BrLa)=10/1 in SDD spectrum (it seems that ration changes over time but it does not seem to be effecting HR spectrum resolution)
2307072	- || -	900	1	

14.7.2023.

Beam: 3 MeV $^4He^{2+}$ TDT

Focus: Me 20.8 A, Ox 42.7 A

Diffraction crystal: Beryl(1010) at 10.5 cm, peeking out of holder 3 mm, with blue dot facing the main door

Current on metal before measurement ~3 nA

Samples: NaBr+Zn (position 1), Na_2SO_4+Zn (position 2), Na+Zn (position 3), NaF+Zn (position 4), NaBr (position 6)

We use SDD spectrum to help us determine good position for measurement.
We search for position where we see both FK and ZnK peaks in SDD spectrum.

FILE	SAMPLE	N_{frames}	t_{exp}/s	COMMENT
2307073	NaBr	1	20	I~2 nA, NaKa line visible in Vista at ch~470
2307074	NaBr	1	10	I~3 nA (but it falls rapidly), Andro's slits were opened a bit
2307075	DARK	10	10	
2307076	NaBr	150	10	I~3-4.5 nA, it is constantly needed to change values of switching magnets
2307077	NaBr	300	10	- || -
2307078	NaBr	300	10	- || -
2307079	NaBr	150	10	- || -

We have some extra time to check Cr in Mudronja's sample 27608.

Beam: 2 MeV H^+ TDT

Focus: Me 16.2 A, Ox 34.5 A

Scanning: X 10.0, Y 9.55

Diffraction crystal: LiF(220) at 9.8 cm initially, peeking out of holder 3 mm

Current on metal before measurement ~1.3 nA

Different samples holder with: Sample 27608, Cu mesh 400, Cr foil, quartz, paper

FILE	DETECTOR	SAMPLE	N_{frames}	t_{exp}/s	COMMENT
2307080	SDD	Cu mesh 400	/	/	SS=5x0.1, SDD shaping time=0.5 us
2307081	SDD	Cr	/	/	- || -
In February we had some artefact in SDD spectrum on 4 keV. We want to check what is with that now. -> For shaping times under 8 us we do not see it, only then it appears. We also wanted to test installed RBS detector. -> We had not luck with it. It does not seem to respond to a change in bias voltage.					
2307082	CCD	Cr	1	30	I~1 nA, CrKa line visible in Vista at ch~640
2307083	CCD	Cr	1	10	LiF at 10.1 cm -> CrKa line at ch~350
2307084	SDD	Sample 27608, 1st map	/	/	SS=1x1, mapping edge of the sample with the thin orange line (**maps in Spector are flipped both horizontally and vertically**!)
2307085	SDD	Sample 27608, 2nd map	/	/	SS=0.5x1, same area but zoomed in
2307086	CCD+SDD	Sample 27608, ROI in 2nd map	150	10	orange line at the edge of the white part of the sample; I~0.7 nA -> HR spectrum is collecting very slow so Andro's slits were opened a bit -> I~3 nA
2307087	CCD+SDD	- || -	50	10	I~2.8 nA
2307088	CCD+SDD	- || -	100	10	- || -
2307089	SDD	Sample 27608,	/	/	SS=0.5x1, mapping part of the sample around the green line

		3rd map			
2307090	CCD+SDD	Sample 27608, ROI in 3rd map	100	10	green line in the central part of the sample; I~2.6 nA and slowly decreasing
2307091	CCD+SDD	- || -	300	10	I~2.4 nA