Message ID: **199**    Entry time: **Mon Feb 19 15:57:36 2024**

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|---|---|
| Experiment Date: | 2024 02 19 |
| Duration (Days): | 5 |
| User: | Anja Miokovic, Stjepko Fazinic, Iva Bozicevic Mihalic, Marija Tkalcevic |
| Accelerator: | Tandetron |
| Beam Line: | Old uProbe |
| Project: | Hi-REXS (HRZZ projekt) |
| Experiment Title: | HR PIXE |
| Beam: | 2MeV H |
| Method: | HR PIXE, PIXE, RBS |

Chamber positioned according to the black marks on the floor.

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

PIXE: Coarse gain=1 k; Fine gain=3; Shaping time=2 us; covered with Al (1 mm thick) mask with hole of 2r=1.7 mm

# 19.2.2024.

**Beam: 2 MeV H$^+$ TDT**

Focus: Me 17.1 A, Ox 35.2 A (collimator slits open, Ando's slits closed for best focus)

Scanning: x=10.0, y=9.55

## 1) Testing new crystal: TIAP (001) -> energy: 0.5-0.9 keV

Diffraction crystal: TIAP(001) on 11.1 cm, peeking out of holder for 3 mm

RBS: Bias=+50 V; Coarse gain=200; Fine gain=4.85; Shaping time=1 us; covered with teflon mask (3 mm thick) with hole of 2r=3 mm

Samples: Mn (1), $MnO_2$ (2), Cr (3), Fe (4), quartz (5), Cu mesh 400 (6)

I~2 nA on the metal before the measurement

| FILE | SAMPLE | DETECTOR | $t_{exp}$/s | $N_{frames}$ | COMMENT |
|---|---|---|---|---|---|
| 2402090 | Cu mesh 400 | SDD | / | / | SS=5x0.1 |
| 2402091 | DARK | CCD | 10 | 10 | |
| 2402092 | Fe | CCD, SDD, SBD | 10 | 50 | FeLa not visible in Vista, but visible in Matlab analysis after ~30 collected frames on ch~450 |
| 2402093 | Fe | CCD, SDD, SBD | 10 | 300 | I~1.5 nA |

Conclusion:
-> Low-energy X-rays are being absorbed within foil that we have over CCD chip.
-> Measurements of wanted elements and compounds would be too slow with this setup, we will not do it now.

## 2) Inspecting Mg and Ge purity

RBS (fine gain changed): Bias=+50 V; Coarse gain=200; Fine gain=7.08; Shaping time=1 us; covered with teflon mask (3 mm thick) with hole of 2r=3 mm

Samples: Mg chunk - half sanded, half not (1), $MnO_2$ (2), Ge? - pellet from powder (3), Fe (4), quartz (5), Cu mesh 400 (6)

Collecting only PIXE+RBS spectra. One point analysis except for file 2402097.

Andro's slits opened to increase the rate. -> I~10 nA

| FILE | SAMPLE | COMMENT |
|---|---|---|
| | | |

| | | | | | |
|---|---|---|---|---|---|
| 2402094 | Mg - sanded part | | | | wrong position, camera moved so we were not on the position we thought we were |
| 2402095 | **Mg - sanded part** | | | | I~7 nA; RBS rate~300 Hz; **RBS spectrum corresponds to pure Mg!** |
| 2402096 | **Ge? - pellet from powder** | | | | I~5,5 nA; **RBS spectrum corresponds to GeO$_2$!** |
| 2402097 | Ge? - pellet from powder | | | | SS=5x0.1, scanning to check homogenity of the sample |

# 20.2.2024.

**Beam: 2 MeV H$^+$ TDT**

Focus: Me 16.5 A, Ox 34.7 A (collimator slits open, Ando's slits closed for best focus)

Scanning: x=10.0, y=9.55

Diffraction crystal: Beryl(1010) on 11.3 cm, peeking out of holder for 3 mm

RBS: Bias=+50 V; Coarse gain=200; Fine gain=7.08; Shaping time=1 us; covered with teflon mask (3 mm thick) with hole of 2r=3 mm

Samples: Ge - sanded from Ge detector (1), GeO$_2$ - pellet from powder (2), Mg+Ge (3), MgO+Ge (4), quartz (5), Cu mesh 400 (6)

Ge in mixtures is from Ge detector. Mg is sanded from Mg chunk.

I~1.5 nA on the metal before the measurement

| FILE | SAMPLE | DETECTOR | $t_{exp}$/s | $N_{frames}$ | COMMENT |
|---|---|---|---|---|---|
| 2402098 | Cu mesh 400 | SDD | / | / | SS=5x0.1 |
| 2402099 | Mg+Ge | CCD | 20 | 1 | MgKa$_{1,2}$ line visible in Vista on ch~680 |
| When crystal is moved forward MgKa$_{1,2}$ line disappears. -> Left half of the frame is unavaiable. Chamber is moved a bit so that beam spot is closer to the crystal. | | | | | |
| 2402100 | MgO+Ge | CCD | 20 | 1 | With Beryl on 11.6 cm it is possible to see MgKa$_{1,2}$ line on ch~310, but whole |
| 2402101 | MgO+Ge | CCD | 20 | 1 | frame is still not avaiable. |

In November of 2022, when we measured Mg+Ge, Beryl was peeking out of holder for 4 mm. We decided to pull it out as far today.

Diffraction crystal: Beryl(1010) on 11.6 cm, peeking out of holder for 4 mm

I~2.4 nA on the metal

| FILE | SAMPLE | DETECTOR | $t_{exp}$/s | $N_{frames}$ | COMMENT |
|---|---|---|---|---|---|
| 2402102 | MgO+Ge | CCD | 20 | 1 | MgKa$_{1,2}$ line visible in Vista on ch~170 |
| First ~150 channels are not avaiable. That is good enough. It will be possible to see both Ge and Mg lines in the same frame. Beryl on 11.45 cm. | | | | | |
| 2402103 | MgO+Ge | CCD | 10 | 1 | MgKa$_{1,2}$ line visible in Vista on ch~730 where we want it |
| 2402104 | Ge | CCD | 10 | 1 | GeLa line visible in Vista on ch~260 |
| 2402105 | DARK | CCD | 3 | 10 | |
| 2402106 | Ge | CCD, SDD, SBD | 3 | 124 | I~3 nA, after Matlab analysis: GeLa on ch~180; stopped because students came to visit |
| Beryl moved a little bit backwards. Željko increased current so we reduced $t_{exp}$. | | | | | |
| 2402107 | DARK | CCD | 2 | 10 | |
| 2402108 | Ge | CCD, SDD, SBD | 2 | 300 | I~4 nA |
| 2402109 | Ge | CCD, SDD, SBD | 2 | 600 | - || - |
| 2402110 | GeO$_2$ | CCD, SDD, SBD | 3 | 720 | I~1.8 nA |
| 2402111 | Mg+Ge | CCD, SDD | 3 | 30 | looking for the best position on the sample; 1. position -> PIXE: I(GeKa)/I(GeLa+MgKa)~0.17, HR PIXE: I(GeLa)/I(MgKa)~1.4 -> we want a little bit more Mg events |
| 2402112 | Mg+Ge | CCD, SDD | 3 | 30 | 2. position -> PIXE: I(GeKa)/I(GeLa+MgKa)~0.24, HR PIXE: no Mg peak |
| 2402113 | Mg+Ge | CCD, SDD | 3 | 30 | 3. position -> PIXE: I(GeKa)/I(GeLa+MgKa)~0.09, HR PIXE: no Ge peak |
| 2402114 | Mg+Ge | CCD, SDD, SBD | 3 | 1080 | 4. position -> PIXE: I(GeKa)/I(GeLa+MgKa)~0.18, HR PIXE after curvature correction: I(GeLa)/I(MgKa)~0.61 -> good position; I~3.5 nA |
| 2402115 | MgO+Ge | CCD | 3 | 30 | PIXE: I(GeKa)/I(GeLa+MgKa)~0.2, GeLa on ch~390 -> moving Beryl a bit forward |
| 2402116 | MgO+Ge | CCD | 3 | 10 | GeLa on ch~330, we are satisfied |
| 2402117 | DARK | CCD | 5 | 10 | |
| 2402118 | MgO+Ge | CCD, SDD, SBD | 5 | 1000 | I~1.7 nA |

| 2402119 | MgO+Ge | CCD, SDD, SBD | 5 | 250 | - \|\| - |
|---|---|---|---|---|---|

Energy window used in Matlab analysis: [240, 360]

# 21.2.2024.

**Beam: 2 MeV H$^+$ TDT**

Focus: Me 16.5 A, Ox 34.7 A (collimator slits open, Ando's slits closed for best focus)

Scanning: x=10.0, y=9.55

Diffraction crystal: Beryl(1010) on 11.4 cm, peeking out of holder for 4 mm

RBS: Bias=+50 V; Coarse gain=200; Fine gain=7.08; Shaping time=1 us; covered with teflon mask (3 mm thick) with hole of 2r=3 mm

Samples: Mg$_2$Si+Ge (1), MgB$_2$+Ge (2), MgSO$_4$+Ge (3), MgBr$_2$+Ge (4), quartz (5), Cu mesh 400 (6)

Ge in mixtures is from Ge detector.

| FILE | SAMPLE | DETECTOR | t$_{exp}$/s | N$_{frames}$ | COMMENT |
|---|---|---|---|---|---|
| 2402120 | Cu mesh 400 | SDD | / | / | SS=5x0.1 |
| Samples were changed after focusing the beam. Beam parameters also needed to be changed. Focus was then checked again on Cu mesh and it was not good anymore. Beam refocused (Me 16,6 A, Ox 34.8 A). | | | | | |
| 2402121 | Cu mesh 400 | SDD | / | / | SS=5x0.1 |
| 2402122 | Mg$_2$Si+Ge | CCD, SDD, SBD | 3 | 720 | I~5 nA, but very unstable; PIXE: I(GeKa)/I(GeLa+MgKa)~0.16; **Caution**: Spector was not stopped on time. It was running after this set was done and while the beam was being optimised. |
| 2402123 | Mg$_2$Si+Ge | CCD, SDD, SBD | 3 | 400 | I~4.1 nA and much more stable then before |
| 2402124 | MgSO$_4$+Ge | CCD, SDD, SBD | 3 | 720 | I~3.3 nA; PIXE: I(GeKa)/I(SKa)~0.09 |
| 2402125 | MgSO$_4$+Ge | CCD, SDD, SBD | 3 | 720 | I~3.2-3.0 nA |
| 2402126 | MgSO$_4$+Ge | CCD, SDD, SBD | 3 | 360 | I~3 nA |
| 2402127 | MgB$_2$+Ge | CCD, SDD, SBD | 3 | 1000 | I~4.3 nA; PIXE: I(GeKa)/I(GeLa+MgKa)~0.12 |
| 2402128 | MgB$_2$+Ge | CCD, SDD, SBD | 3 | 500 | - \|\| - |

Energy window used in Matlab analysis: [240, 360]

# 22.2.2024.

**Beam: 2 MeV H$^+$ TDT**

Focus: Me 16.6 A, Ox 34.7 A (collimator slits open, Ando's slits closed for best focus)

Scanning: x=10.0, y=9.55

Diffraction crystal: Beryl(1010) on 11.4 cm, peeking out of holder for 4 mm

RBS: Bias=+50 V; Coarse gain=200; Fine gain=7.08; Shaping time=1 us; covered with teflon mask (3 mm thick) with hole of 2r=3 mm

Samples: Mg$_2$Si+Ge (1), MgB$_2$+Ge (2), MgSO$_4$+Ge (3), MgBr$_2$+Ge (4), quartz (5), Cu mesh 400 (6)

Ge in mixtures is from Ge detector.

I~2.5 nA on metal before measurement

| FILE | SAMPLE | DETECTOR | t$_{exp}$/s | N$_{frames}$ | COMMENT |
|---|---|---|---|---|---|
| 2402129 | Cu mesh 400 | SDD | / | / | SS=5x0.1 |
| Current drastically fell. After Željko optimised the beam again focus was not good anymore. Beam refocused. | | | | | |
| 2402130 | Cu mesh 400 | SDD | / | / | SS=5x0.1 |
| 2402131 | DARK | CCD | 8 | 10 | |
| 2402132 | MgBr$_2$+Ge | CCD, SDD, SBD | 8 | 360 | I~2.4 nA; PIXE: I(GeKa)/I(BrKa)~1.17; beam was lost at frame~250?, but after few minutes it was back |

| 2402133 | MgBr$_2$+Ge | CCD, SDD, SBD | 8 | 313 | I~2-2.4 nA; pause for visit of students |
|---|---|---|---|---|---|
| 2402134 | MgBr$_2$+Ge | CCD, SDD, SBD | 8 | 360 | I~2.4-3 nA |

New samples: Mg$_3$N$_2$+Ge (1), MgWO$_4$+Ge (2), quartz (5), Cu mesh 400 (6)

| FILE | SAMPLE | DETECTOR | $t_{exp}$/s | $N_{frames}$ | COMMENT |
|---|---|---|---|---|---|
| 2402135 | Cu mesh 400 | SDD | / | / | SS=5x0.1 |
| 2402136 | Mg$_3$N$_2$+Ge | SDD, SBD | / | / | SS=4x1 |
| 2402137 | Mg$_3$N$_2$+Ge - ROI | SDD, SBD | / | / | position where there is no Ge, checking if the powder is still only Mg$_3$N$_2$ or if it has reacted with something -> **RBS spectrum looks like pure Mg$_3$N$_2$** |
| 2402138 | Mg$_3$N$_2$+Ge | CCD, SDD, SBD | 5 | 98 | I~3.8 nA; PIXE: I(GeKa)/I(GeLa+MgKa)~0.16; **Problem**: there are no events in the upper part of the MgKa$_{1,2}$ line. **Caution**: Spector was not stopped on time, it was running when bias was removed from SBD. |

We decided to heat up CCD to check if something got stuck on it. It was heated to 16°C and then cooled again to working temperature (-70°C).
We also checked vacuum when letting the beam on the sample. -> It is NOT getting worse. Nothing should be evaporating.
Note: We see backgorund events in the same part of the frame. That means that there should not be any problem with the CCD.
Crystal was moved a bit to the front and still no events in the upper part of the MgKa$_{1,2}$ line. -> Maybe there is some problem with the sample.

| 2402139 | Mg$_3$N$_2$+Ge | CCD, SDD, SBD | 5 | 770 | I~3.2 nA; still no events in the upper part of the MgKa$_{1,2}$ line, but we decided to continue with the measurement |
|---|---|---|---|---|---|
| 2402140 | Mg$_3$N$_2$+Ge | CCD, SDD, SBD | 5 | 385 | |
| 2402141 | Mg$_3$N$_2$+Ge | CCD, SDD, SBD | 5 | 350 | |

Energy window used in Matlab analysis: [240, 360]


# 23.2.2024.

**Beam: 2 MeV H$^+$ TDT**

Scanning: x=10.0, y=9.55

Diffraction crystal: Beryl(1010) on 11.4 cm, peeking out of holder for 4 mm

RBS: Bias=+50 V; Coarse gain=200; Fine gain=7.08; Shaping time=1 us; covered with teflon mask (3 mm thick) with hole of 2r=3 mm

Samples: Mg$_3$N$_2$+Ge (1), MgWO$_4$+Ge (2), quartz (5), Cu mesh 400 (6)

Ge in mixtures is from Ge detector.

I~2 nA on metal before measurement

| FILE | SAMPLE | DETECTOR | $t_{exp}$/s | $N_{frames}$ | COMMENT |
|---|---|---|---|---|---|
| 2402142 | Cu mesh 400 | SDD | / | / | SS=5x0.1; focus is not satisfactory, we will try get better focus |
| 2402143 | Cu mesh 400 | SDD | / | / | SS=5x0.1; we are satisfied |

We have a lot of problems with the beam. Not enough current on LE side of the accelerator.
Yesterday cathode was changed but maybe it was not inserted properly. Željko was thinking about changing it again but decided no to.
He optimized the beam again, changing the parameters also on LE side. We focused again. (Me 16.6 A, Ox 34.8 A)

| 2402144 | Cu mesh 400 | SDD | / | / | SS=5x0.1 |
|---|---|---|---|---|---|
| 2402145 | MgWO$_4$+Ge | CCD, SDD, SBD | 5 | 500 | I~3.3 nA; PIXE: I(GeKa)/I(WLa)~1.9, but it seems that this ratio is not constant during measurement (maybe the beam is moving slightly?); **Caution**: Spector was not stopped on time. It was running after this set was done and while the beam was being optimised. |
| 2402146 | MgWO$_4$+Ge | CCD, SDD, SBD | 5 | 500 | |
| 2402147 | MgWO$_4$+Ge | CCD, SDD, SBD | 5 | 500 | I~2.6 nA |

Current fell a lot, we could not improve it. Decided to stop TDT and finish for the day.

Energy window used in Matlab analysis: [240, 360]