WP3-B2 Activity

CRYSTAL GROWTH

Test of TeO_2 crystal samples completely processed at production factory

Capelli Silvia

TEST AIM

Test radiopurity and bolometric **performances** of crystal samples, **completely processed by the producer**, following an **agreed protocol**.

The protocol is intended to:

- further improve crystals bulk purity (^{232}Th, ^{238}U, ^{190}Pt...)
- implement at producer the crystal surface treatment
- have a clear codification and check of all production phases:

1. TeO_2 powder production
2. Crystal growth
3. Crystal surface treatment
4. Crystal storage and shipment

1.+2. TeO_2 powder production and crystal growth

- radio-purity requirements to be fulfilled (sampling check) for:
  1. raw materials (Te metal),
  2. reactants (acids, water, other liquids),
  3. intermediary products (TeO_2 powder)
  4. final product (TeO_2 crystals)
- Test of three different dry-up and calcination processes (2 crystals/type):
  - I. standard one with 80°C dry-up + 690°C calcination in Pt crucible
  - II. 600°C in ceramic without dry-up
  - III. 240°C dry-up in teflon + 690°C calcination in Pt crucible

- After 1st growth (standard Bridgman in Pt crucible): ~1/3 ingot selected taking a part more central than usual (to reduce impurity content)
- After grinding, washing,drying and calcination the powder showed a reduced Pt content
- Finally a 2nd growth was performed

3. Crystal surface treatment

Procedure similar to one already tested in LNGS for a previous R&D measurement (RAdioativityDetector):

1. washing with water in ultrasonic bath
2. surface lapping with two Al_2O_3 powders:

   big granularity (1 mm lap off) + small granularity (0.1 mm lap off)

   => the small granularity powder didn't work !!!

   The used powders are new.

   They have been selected for radiopurity reasons, but didn't work properly
3. chemical etching with very clean acids (HNO_3 4 mol) in order to remove the dirty part of the surface

4. polishing with a very clean SiO_2 powder, already used for RAD, and a new lapping pad (10-20 μm lap off)

4. Crystal storage and shipment

- At the end of the polishing step, the crystals were stored immediately in sealed, clean, polyethylene bags with 80mb vacuum. All the crystals were then put in a polycarbonate box under vacuum.

- Produced crystals must be stored underground before shipping.

- During shipping, any contact between crystals and air must be avoided

- Any excessive exposure to cosmic rays must be avoided (total exposure has to be no longer than 4 months)

Test Set-up

- CUORE-like mounting system
- Copper frames covered with polyethylene film
- 3 planes 4-crystals each:

  - 6 crystals grown and mechanically treated at producer following the new protocol

  - 6 old style crystals (Cuoricino like growth and surface treatment performed at LNGS with the RAD procedure)

- The 6 new style crystals were provided with 2 sensors each
- The 6 old style crystals were provided with 1 sensor each

Detector performances

- All the new style detectors were working
- Three old-style detectors didn't work, and one lost the stabilization heater connection

The new style crystals appear to have quite lower pulse amplitudes (despite the quite low working temperature) and poorer energy resolutions

Detector	Pulse Height μV/MeV	FWHM [Low E] KeV	FWHM [2615keV] KeV	FWHM [5.4 MeV] KeV	
BC1-T1	13	8	14	36	
BC1-T2	93	5	17	37	
BB2-T2	10	11	18	61	
BB2-T1	25	9	40	74	
BA1-T2	35	11	23	39	
BA1-T1	46	5	21	44	
BA2-T1	99	16	26	98	
BA2-T2	30		49	110	
BB1-T2	42	11	39	79	
BB1-T1	49	8	35	75	
BC2-T1	31	17	32	56	
BC2-T2	19		58	100	
B64	204	5	8		Old-style
B48	564	3	7		Old-style

Background

- Excess of counts on the right side of the 2615 keV peak for both new and old-style crystals

  => at present no explanation...it could be due to a variation of the calibration in time

- No evident improvement with respect to ^{190}Pt internal contamination

  (poor statistics doesn't allow to compare the different detector rates)

- Higher rate in 4-5 MeV region with respect to RAD.

  Similar rate with respect to Cuoricino.

- 4-5 MeV structures could be ascribed to ^{238}U contamination both in crystals' bulk and surface.

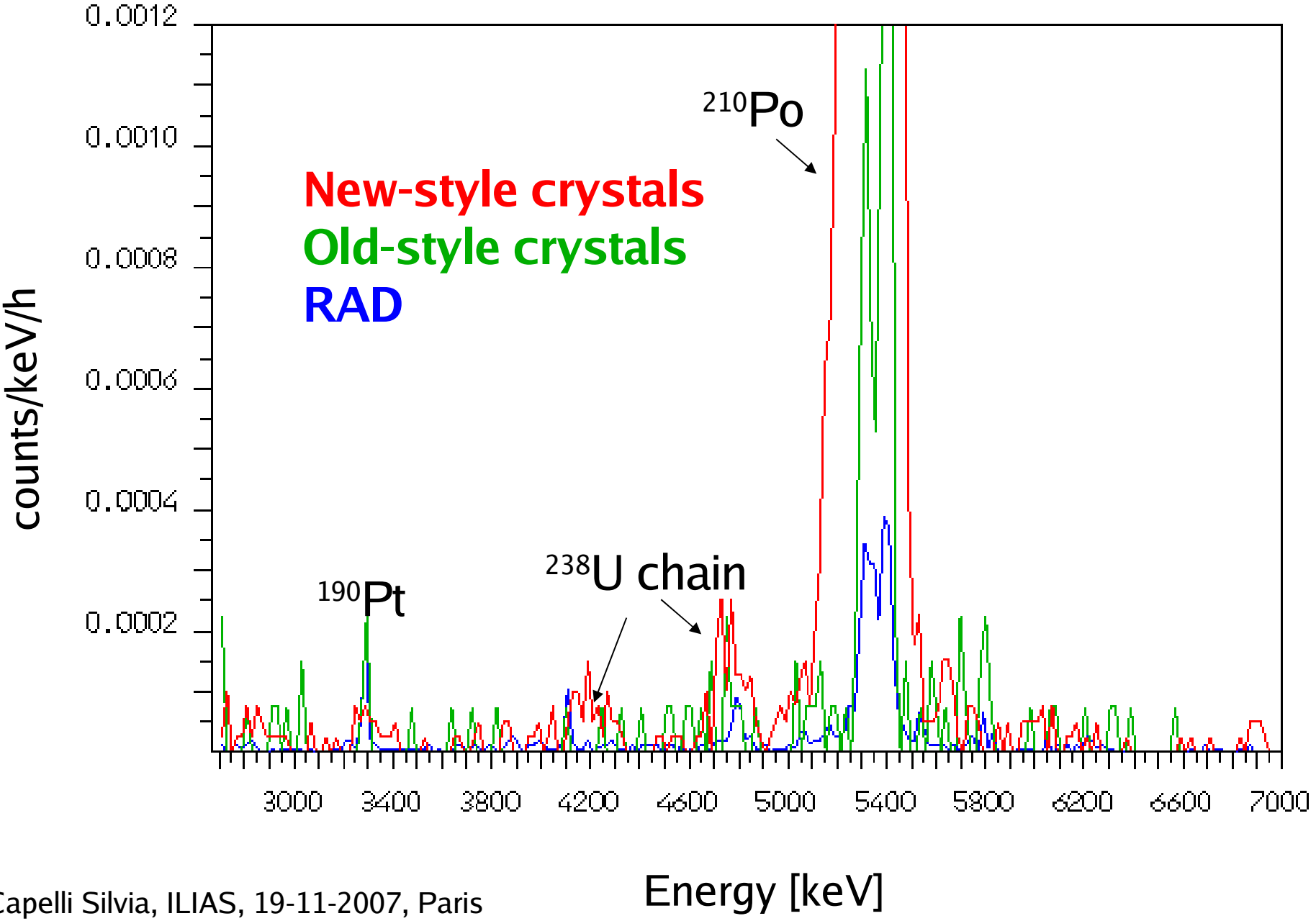
- Huge contamination in ^{210}Po (it's usually observed for new crystals)

α region integrals

Energy range	KeV	Continuum 2700-3200	err.	^{190}Pt 3200-3400	err.	Continuum 3400-3900	err.
Anticoinc.	Cuoricino	0.117	0.003	0.515	0.009	0.118	0.003
Coincidence	Cuoricino	0.012	0.001	0.017	0.002	0.009	0.001
Anticoinc.	RAD	0.064	0.014	0.373	0.052	0.082	0.016
Coincidence	RAD	0.023	0.008	0.037	0.016	0.009	0.005
Anticoinc.	OldStyle	0.269	0.095	0.336	0.168	0.101	0.058
Coincidence	OldStyle	0.000	0.000	0.000	0.000	0.000	0.000
Anticoinc.	NewStyle	0.297	0.082	0.393	0.149	0.157	0.059
Coincidence	NewStyle	0.050	0.034	0.000	0.000	0.006	0.011

Energy range	KeV	U and Th peaks 4000-5000	err.	^{210}Po peak 5000-6000	err.	U and Th peaks 6000-8000	err.
Anticoinc.	Cuoricino	0.517	0.004	0.964	0.005	0.198	0.002
Coincidence	Cuoricino	0.101	0.002	0.182	0.002	0.073	0.001
Anticoinc.	RAD	0.168	0.016	0.725	0.033	0.027	0.004
Coincidence	RAD	0.009	0.004	0.149	0.015	0.006	0.002
Anticoinc.	OldStyle	0.303	0.071	2.350	0.199	0.084	0.027
Coincidence	OldStyle	0.017	0.017	0.034	0.024	0.008	0.008
Anticoinc.	NewStyle	0.625	0.084	142.000	1.261	0.118	0.026
Coincidence	NewStyle	0.028	0.018	1.170	0.115	0.017	0.010

α region comparison plot



Why this bad result...

The crystals showed a strange "yellow" surface, probably attributable to the cleaning procedure:

1. the Al_2O_3 powders have been selected for radiopurity reasons but, even if chosen with the same granularity of the chinese ones, didn't work properly:
   - large granularity powder is rather good though gives scratches
   - small granularity powder gives too many scratches and could not be used

2. the selected SiO_2 powder works extremely slow:
   - too small granularity
   - too soft
   - bad coupling with selected polishing pads (soft)

=> the bad surface could be the reason for the obtained bad performances and the high alpha background

Future plans

A **new run** with 12 crystals is going to start within the **end of this month**:

- 4 new crystals, with Cuoricino-like growth and RAD-like surface treatment, completely done at producer

(lapping with old Chinese powders + polishing with SiO_2 and old lapping pad)

=> to verify that the chinese factory can guarantee a good and reproducible standard crystal production

- 6 of the presently tested New-style crystals but with RAD-like surface treatment, performed at LNGS

=> to check if the bad performances are due to a surface problem

- 2 Old-style crystals (the two already used in this run)

=> for comparison