#### Report

## about research activity of D.V. Poda within P2IO post-doctoral fellowship at CSNSM (Orsay) during 05.11.2013 – 05.09.2014

## 1. Project goals

A post-doctoral position is in the framework of a LUMINEU project. The main purpose of the LUMINEU (Luminescent Underground Molybdenum Investigation for NEUtrino mass and nature) is to set the bases for the realization of a next-generation neutrinoless double-beta decay experiment with unprecedented sensitivity. To reach this goal, the LUMINEU is going to develop an advanced particle detector with high detection efficiency, high energy resolution and high capability to discriminate an alpha-induced background. A scintillating bolometer — detector with a heat-light double read-out — is considered as a device which perfectly satisfies all the requirements needed for realization an experiment with "zero" background at the range of interest. To succeed, there are several important sub-goals to be completed:

- a) Development of large volume radiopure zinc molybdate (ZnMoO<sub>4</sub>) crystal scintillators containing the favorable double beta active nuclide <sup>100</sup>Mo;
- b) Fabrication and optimization of the scintillation light detectors in terms of energy threshold, size, reproducibility and time response;
- c) Development of phonon sensors based on several technologies: NTD (Neutron Transmutation Doped) germanium thermistor, TES (transition edge sensor) based on superconducting NbSi thin films, and MMC (metallic magnetic calorimeter);
- d) Construction, low temperature test, and optimization of single module with the aim to achieve the highest bolometric performances;
- e) Realization of a pilot experiment with ZnMoO<sub>4</sub> scintillating bolometers containing ~1 kg active mass of <sup>100</sup>Mo.

The research within the fellowship is mainly focused on the optimization of very low-temperature light-detector technologies, aiming at improving the sensitivity and the reproducibility with respect to the state-of-the-art and, obviously, it involves activity over b)–d) sub-goals listed above.

## 2. Description of work achieved

The fellow mainly took part in construction of detectors, their assembling into cryostats, low temperature tests, data analysis, and preparation of drafts of the papers. All achievements obtained with the fellow's participation and related to the LUMINEU are described below.

### 2.1. Upgrade of wiring in a cryostat dedicated to the LUMINEU

- New wiring for the detector lines between junction points "300K" "4K" "<sup>4</sup>He/<sup>3</sup>He mixing chamber" in the cryostat were performed by using microcables with twisted pairs and shield has been done;
- New contacts on the detector "anti-vibrating plate" of the cryostat have been installed;
- New measurement with scintillating bolometer based on 24 g ZnMoO<sub>4</sub> crystal (used as reference sample) demonstrates good operability of the updated system.

#### 2.2. Low-temperature test of light detectors based on ultra-pure Ge wafers at CSNSM

• Two new light detectors with  $\emptyset$  44 mm, covered by a thin SiO layer, have been tested at a base temperature of 16 mK. Middling performances of one light detector (signal amplitude was 1.36  $\mu$ V/keV, but baseline resolution was FWHM = 3.1 keV) were caused by significant microphonic noise due to not enough hard tying of the wafer in a copper holder. While, another light detector demonstrates acceptable performances: signal – 2.48  $\mu$ V/keV,

the energy resolution of 5.9 keV peak of  ${}^{55}$ Fe – FWHM = 0.75(8) keV, the energy resolution of baseline – FWHM = 0.65(1) keV;

- A light detector (Ø 44 mm) with new string-based suspension of the Ge wafer has been successfully tested;
- A light detector with deposited circular electrical contacts has been tested with the aim to study the efficiency of the Neganov-Luke effect (amplification of light signal in order to get higher signal-to-noise ratio, lower threshold, and higher discrimination capability). The detailed data analysis is foreseen.

#### 2.3. Low temperature studies with scintillating bolometer based on LUMINEU precursor

- Scintillating bolometer was constructed from 313 g ZnMoO₄ crystal (precursor of the LUMINEU programme) grown by the low-temperature gradient Czochralski technique at the Nikolaev Institute for Inorganic Chemistry (NIIC, Novosibirsk, Russia). Two germanium photodetectors Ø 50 × 0.25 mm were fixed at the opposite side of the crystal for registration of emitted scintillation light. The crystal and the light detectors were equipped with NTD temperature sensors;
- The aboveground low temperature test at CSNSM demonstrates good operability of the scintillating bolometer cooled down to 17 mK. The device was calibrated by gammas from natural radioactivity (mainly <sup>226</sup>Ra and its daughters) and <sup>232</sup>Th source with low activity. Perfect performances were achieved with one light detector: signal 1.01  $\mu$ V/keV, the energy resolution of 5.9 keV peak of <sup>55</sup>Fe FWHM = 0.29(2) keV. The ZnMoO<sub>4</sub> bolometer is characterized by reasonable performances, f.e. the signals were registered with amplitude 25  $\mu$ V/MeV; the energy resolution (FWHM) of 2615 keV peak of <sup>208</sup>Tl was 26(3) keV. The measured light yields relative to gamma(beta) and alpha particles are 0.77(11) keV/MeV and 0.118(2) keV/MeV, respectively. Such behavior is caused by not so good quality of the crystal and dominated pile-up effect during the aboveground measurements (due to large volume and high density of the crystal);
- The device was installed into the EDELWEISS set-up (dedicated to dark matter search) located at the Modane underground laboratory (LSM, France). The calibration (<sup>133</sup>Ba source) and background data collected from October 2013 to February 2014 (elapsed time  $\approx$  851 hours), the base temperature was regulated at  $\approx$  19 mK. The ZnMoO<sub>4</sub> detector shows significantly better performances at very low background rate caused underground conditions: the amplitude of signals was 106  $\mu$ V/MeV, baseline FWHM = 3.3(1) keV;
- First calibration measurements with <sup>232</sup>Th gamma-source (nominal activity  $\approx 0.6$  kBq) made from tungsten thoriated wires was performed in January 2014 (the EDELWEISS dark matter experiment uses only <sup>133</sup>Ba source which emits gammas with energy below 0.4 MeV). These measurements demonstrate high spectrometric performances of the bolometer at the energy region close to the expected signal of neutrinoless double beta decay of <sup>100</sup>Mo (at  $\approx 3$  MeV). In particular, the energy resolution for 2.6 MeV gamma-peak was FWHM = 9(2) keV. In addition, a full separation of gamma(beta) events from registered alpha-particles was achieved. This feature is very important for neutrinoless double-beta decay searches because it allows to decrease significantly the background rate at the range of interest by rejecting of alpha-events caused by radionuclides from internal/surface contamination of crystal and surface pollution of constructing elements of bolometer;
- The low background measurements were used to estimate a radioactive contamination of the tested ZnMoO<sub>4</sub> crystal. The radiopurity of even precursor already satisfies the LUMINEU requirements, in particular only trace contamination of <sup>210</sup>Po (activity of 0.62(3) mBq/kg), <sup>226</sup>Ra (0.026(5) mBq/kg), and <sup>228</sup>Th (0.010(3) mBq/kg) was registered, while only upper limits in the range of 0.003–0.014 mBq/kg were set on activity of other alpha-radionuclides from U/Th-chains, <sup>147</sup>Sm, and <sup>190</sup>Pt.

# **2.4.** Development of scintillating bolometers based on the new LUMINEU crystals, including ones grown from isotopically enriched compound

- Advanced middle volume ZnMoO<sub>4</sub> crystal boules were grown in the NIIC (Novosibirsk, Russia) from deeply purified molybdenum and two cylindrical samples ( $\emptyset$ 50×40 mm, with mass of 334 and 336 g) were produced for new bolometric tests. Moreover, first crystal boule (length 95 mm, mass 171 g) was developed in the NIIC from deeply purified molybdenum enriched in <sup>100</sup>Mo to 99.5% (Zn<sup>100</sup>MoO<sub>4</sub>) and two samples (D24×30 mm, mass of 59 and 63 g) were cut from the boule;
- Two scintillating bolometers based on the advanced ZnMoO<sub>4</sub> crystals and one bolometers array with two Zn<sup>100</sup>MoO<sub>4</sub> samples were constructed at CSNSM. Each device contains one photodetector ( $\emptyset$ 50×0.25 mm). The NTD-based temperature sensors were used for registration of phonon signals. Two NTD thermistors were glued for each ZnMoO<sub>4</sub> crystal, while other detectors were assembled by one NTD. In addition, the bolometers were equipped also by one heating device for monitoring of thermal response;
- First low temperature test of  $Zn^{100}MoO_4$  scintillating bolometers array was performed at cryogenic laboratory of CSNSM. The array was tested at three base temperatures: 13.7 mK (over 18.3 h), 15 mK (4.8 h), and 19 mK (24.2 h). The detector was calibrated by gammas from natural radioactivity (mainly <sup>226</sup>Ra and its daughters) and weak <sup>232</sup>Th source. The best bolometric performances of the array were achieved at 13.7 mK. The performances of light detector were following: signal  $-0.41 \mu V/keV$ , the energy resolution of 5.9 keV peak of  ${}^{55}$ Fe – FWHM = 0.42(2) keV, the energy resolution of baseline – FWHM = 0.28(1) keV. Two Zn<sup>100</sup>MoO<sub>4</sub> detectors demonstrate similar performances: the amplitude of signals is 86 and 96 µV/MeV; the energy resolution (FWHM) of 609 keV peak of <sup>214</sup>Bi is 5.0(5) and 10(1) keV; the energy resolution of baseline (FWHM) is 1.4(1) and 1.8(1) keV. The similar values for the light yield relative to gamma(beta) particles were measured at all working temperatures:  $\approx 1.0(1)$  keV/MeV for both crystals. The analysis of selected alpha events indicates encouraging radiopurity of the tested crystals; only <sup>210</sup>Po with activity 1.1(3) mBq/kg is observed. Summarizing, these results demonstrate the good reproducibility of the behaviour of the two detectors and the absents of deterioration of their performances due to the enrichment (as it was observed for TeO<sub>2</sub> bolometer);
- All three scintillating bolometers were installed into the EDELWEISS set-up at the Modane underground laboratory (in particular, the fellow was involved in dismounting of the set-up, installation of additional polyethylene shield, cabling in the cryostat and providing of their thermalization, test of cables, mounting of bolometers);
- Calibration measurements by using <sup>133</sup>Ba source and different settings (pulse profile, sampling rate, polarization of NTD sensors) were carried out in July in order to check operability of the new LUMINEU detectors and to find optimal working parameters. Preliminary data analysis demonstrates normal operability of bolometers based on non-enriched ZnMoO<sub>4</sub> crystals, while the Zn<sup>100</sup>MoO<sub>4</sub> array has very huge microphonic noise which deteriorates its performances (probably, it is related with non-standard copper holder);
- Background measurements were realized in August, in particular the fellow was involved in 10 days shift (tuning pre-settings before each start of the run and control of data taking);
- Preliminary analysis demonstrates good bolometric performances of the non-enriched ZnMoO<sub>4</sub> crystals and their encouraging radiopurity. For example, the energy resolution of 356 keV peak of <sup>133</sup>Ba is FWHM  $\approx$  5 keV for both samples, only <sup>210</sup>Po with activities 0.6(5) and 1.1(3) mBq/kg is clear visible in alpha spectra of the detectors. Detailed analysis of all accumulated data is foreseen in September.

### 2.5. Investigation of W-doping effect on bolometric properties of zinc molybdate crystals

- Low temperature test of scintillating bolometer array constructed from two ZnMoO<sub>4</sub> crystals (Ø20×40 mm, one of them was doped by W) and one Ge-based light detector was performed at CSNSM. Both samples were grown in the NIIC (Novosibirsk, Russia);
- The measurements show that W doping does not deteriorate the bolometric performances of ZnMoO<sub>4</sub> crystal, and therefore can be used to improve significantly the crystal growth process.

# **2.6.** Study of lithium molybdate based scintillating bolometer as a perspective detector for rare event search

- Deeply purified molybdenum oxide and commercial high purity grade lithium carbonate were used to produce initial Li<sub>2</sub>MoO<sub>4</sub> compound. The crystal was grown by low-temperature gradient Czochralski technique at the NIIC (Novosibirsk, Russia). A cylindrical scintillating element (∅40×40 mm) was produced from the Li<sub>2</sub>MoO<sub>4</sub> crystal boule;
- First low temperature tests of scintillating bolometer based on middle volume Li<sub>2</sub>MoO<sub>4</sub> crystal and extra thin Ge light detector (thickness is 25 μm) was performed at CSNSM. The used light detector was produced in the Institut d'Astrophysique Spatiale (Orsay, France). Both the Li<sub>2</sub>MoO<sub>4</sub> crystal and the light detector were equipped with NTD temperature sensors. The device was tested over 126 h in several runs with base temperatures 16.5–15.2 mK. The detector was calibrated by gammas from natural radioactivity (mainly <sup>226</sup>Ra and its daughters) and low intensive <sup>232</sup>Th source (used in one run);
- The light detector demonstrates excellent performances: signal  $(2.56-6.60) \mu V/keV$ , the energy resolution of 5.9 keV peak of <sup>55</sup>Fe FWHM = (0.30-0.37) keV, the energy resolution of baseline FWHM = (0.07-0.08) keV (depending on the run);
- The crystal demonstrates good bolometric performances and the best scintillation at very low temperature ever got with such type of material. In particular, the measured amplitude of signals is (40–62)  $\mu$ V/MeV; the energy resolution of 609 keV peak of <sup>214</sup>Bi is FWHM = (5–6) keV; the energy resolution of baseline is FWHM = (2–3) keV (depending on the run). The light yields relative to gamma(beta) particles and alphas+tritons (result of <sup>6</sup>Li(n, $\alpha$ )<sup>3</sup>H reaction) were estimated to be 0.616(1) keV/MeV and 0.1755(3) keV/MeV, respectively. The measured values of light yield are in ≈1.5 times higher than the best published ones. The measurements demonstrate that Li<sub>2</sub>MoO<sub>4</sub>-based scintillating bolometer is extremely promising detector for rare event search (neutrinoless double beta decay of <sup>100</sup>Mo, solar hadronic <sup>7</sup>Li axions), as well as, for weak neutron flux monitoring in cryogenic underground experiments.

## **3.** Publications

- A.S. Barabash et al., Enriched Zn<sup>100</sup>MoO<sub>4</sub> scintillating bolometers to search for 0v2β decay of <sup>100</sup>Mo with the LUMINEU experiment, Accepted to Eur. Phys. J. C (2014); arXiv:1405.6937.
- LUMINEU and EDELWEISS collaborations, *The LUMINEU double-beta-decay experiment in the EDELWEISS setup: first tests and prospects*, To be submitted to Astropart. Phys.
- N. Coron et al., *Performance of advanced Li*<sub>2</sub>*MoO*<sub>4</sub> *crystal scintillator for*<sup>100</sup>*Mo neutrinoless double beta decay search*, To be submitted to Astropart. Phys.

## 4. Relevance of the project within P2IO

The main objective of the LUMINEU is related to investigation of neutrinoless double beta decay of <sup>100</sup>Mo with unprecedented sensitivity. The searches for this rarest hypothetical process

are included to scientific thematic "Symmetries in the subatomic world" within the mission "EXPLORE" of P2IO. The project is devoted to developing of the advanced particle detector namely, scintillating bolometer — and therefore includes implementation of three main development lines: 1) sensitive bolometric material based on high radiopure zinc molybdate crystal scintillator enriched in isotope of <sup>100</sup>Mo; 2) ultra-pure germanium-based light detector with high performances; 3) innovative temperature sensors (NTD, TES, MMC) for these detectors. Certainly, such activity involves construction of prototypes, investigation of their performances at very low temperatures, optimization, fabrication of final detectors, and performing of a pilot low background experiment with the aim to search for neutrinoless double beta decay of <sup>100</sup>Mo. Obviously, all these tasks are fully correspond to the P2IO technological thematic "New generation sensors and their results" within the mentioned mission. The realization of such ambitious research requires a strong cooperation of top-class laboratories / institutions and this is again the case of the LUMINEU, which involves partnership between following French institutions: CSNSM, CEA, IAS, and ICMCB. Three of them are incorporated to P2IO network, and therefore the LUMINEU is also relevant to the P2IO mission "TRANSFORM". The LUMINEU is an interdisciplinary project which cuts the boundaries between different fields of physics and has a valuable impact on strengthen of all partners involved in its realization, thereby it fits with the mission "STRUCTURE". According to all presented above, one can conclude that the LUMINEU, as well as the research project within the fellowship, are totally relevant to the P2IO.