Improvement of radiopurity level of enriched $^{116}$CdWO$_4$ and ZnWO$_4$ crystal scintillators by recrystallization

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Abstract

As low as possible radioactive contamination of a detector plays a crucial role to improve sensitivity of a double beta decay experiment. The radioactive contamination of a sample of $^{116}$CdWO$_4$ crystal scintillator by thorium was reduced by a factor $\approx 10$, down to the level 0.01 mBq/kg ($^{228}$Th), by exploiting the recrystallization procedure. The total alpha activity of uranium and thorium daughters was reduced by a factor $\approx 3$, down to 1.6 mBq/kg. No change in the specific activity (the total $\alpha$ activity and $^{228}$Th) was observed in a sample of ZnWO$_4$ crystal produced by recrystallization after removing $\approx 0.4$ mm surface layer of the crystal.

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1. Introduction

High quality large volume cadmium tungstate crystal scintillators were developed from cadmium enriched in $^{116}$Cd to 82% ($^{116}$CdWO$_4$) for the Aurora experiment to investigate the double beta ($2\beta$) decay of $^{116}$Cd [1]. Three crystal scintillators (586 g, No. 1; 589 g, No. 2; 326 g, No. 3) have been cut from the crystal boule (see Fig. 1). The experiment is in progress with two scintillators (No. 1 and No. 2) installed in the DAMA/R&D low background set-up at the Gran Sasso underground laboratory (LNGS) of the INFN (Italy) (average depth $\approx$ 3650 m w.e. [2]). Preliminary results of the experiment were reported in conference proceedings [3, 4, 5].

The radiopurity level of the crystal scintillators plays a crucial role in double beta decay experiments. The radioactive contamination of the crystal scintillators No. 1 and No. 2 was estimated from the experimental data of the Aurora experiment [1, 6, 7], while the radioactive contamination of the crystal sample No. 3 was measured by HPGe detector [1] and also in the present work by scintillation counting method (see Sections 3 and 4). The contamination of the rest of the melt remaining in the platinum crucible after the crystal growth was measured with the help of the GePaolo ultra-low background HPGe $\gamma$ spectrometer of the STELLA (SubTErranean Low Level Assay) facility at LNGS [8]. The radioactive contamination of the crystal samples and of the rest after the crystal growth is presented in Table 1. The contamination of the rest material by potassium, radium and thorium is substantially higher than the contamination of the crystals. An increase of radioactive contamination by thorium along the crystal boule from the growth cone to the bottom of the boule was observed too. The observed variation of the thorium specific activity, and a much higher contamination of the rest after the crystal growing process, can be explained by segregation of the radioactive elements in the CdWO$_4$ crystal growth process, which provides a possibility to improve the radiopurity level of the $^{116}$CdWO$_4$ crystal scintillators by re-crystallization. It should be stressed that multiple crystallization is well-known approach to improve optical quality (see, e.g., [9]) and radiopurity level [10, 11] of crystal scintillators. Thus, the $^{116}$CdWO$_4$ crystal sample No. 3 was recrystallized to study the impact of the second crystallization on the radioactive contamination of cadmium tungstate crystals.
Figure 1: (Color online) Boule of enriched $^{116}$CdWO$_4$ crystal and the crystal samples produced for the double beta decay experiment.

Table 1: Specific activities of radioactive contaminants in the different parts of the $^{116}$CdWO$_4$ crystals produced from the boule, and in the rest of the melt after the crystal growth. The limit on activity of $^{228}$Ra in the sample No. 3 was estimated by analysis of $^{228}$Ac $\gamma$ quanta in the data accumulated by the HPGe detector [1]. Reference date is May 2014.

| $^{116}$CdWO$_4$ sample, mass (see also Fig. 1) | Radioactive contamination (mBq/kg) | Method                  |
|----------------------------------------------|-----------------------------------|-------------------------|
| No. 1, 586 g                                | $^{40}$K ≤ 0.9, $^{226}$Ra ≤ 0.005 | Scintillation counting  |
| No. 2, 589 g                                | $^{40}$K ≤ 0.9, $^{226}$Ra ≤ 0.005 | Scintillation counting  |
| No. 3, 326 g                                | $^{40}$K ≤ 1.2, $^{228}$Ra ≤ 0.1, $^{228}$Th ≤ 1.3 | Scintillation counting |
| Rest of the melt after the crystal growth, 264 g | $^{40}$K 27(11), $^{226}$Ra 64(4), $^{228}$Th 9(2) | HPGe $\gamma$ spectrometry |

Reference:  
[1]
As regards the zinc tungstate (ZnWO$_4$) crystal scintillators, they are promising detectors to search for double beta decay [12, 13], dark matter [14, 15, 16], eka-elements [17], and investigation of rare alpha decays [18]. The detector has a very low level of radioactive contamination [19]. The effect of the recrystallization procedure on the radioactive contamination of the ZnWO$_4$ crystal was also investigated in Ref. [19]. Although one could expect an improvement of the material purity level after a second crystallization, the radioactive contamination of the sample obtained by recrystallization was even slightly higher, on the contrary of the case of CdWO$_4$. This could be explained by a concentration of radioactive impurities in a thin surface layer of the ZnWO$_4$ crystal. In Ref. [20] a substantial reduction of cadmium tungstate crystal scintillators radioactive contamination was achieved by removing a thin (0.8−1.5 mm) surface layer of the crystals. In the present work a possible concentration of radioactive impurities in a surface layer of the ZnWO$_4$ crystal obtained by recrystallization was also checked.

2. Samples of $^{116}$CdWO$_4$ and ZnWO$_4$ crystal scintillators

The sample No. 3 of the $^{116}$CdWO$_4$ crystal was recrystallized by the low-thermal-gradient Czochralski technique [21, 22, 23] in a platinum crucible 50 mm in diameter with 99.93% Pt purity level. The crystallization rate was 1.5 mm/h. A crystal boule with mass 286 g (see Fig. 2, left) was grown, which is 88% of the sample before recrystallization. The boule was cut into three parts as shown in Fig. 2, right. The central part with mass 195 g (60% of the sample before recrystallization) was used to test the radioactive contamination. The side surface of the sample was made opaque by grinding paper to improve the scintillation light collection.

On the other hand, a ZnWO$_4$ crystal sample with mass 141 g was obtained by recrystallization – with ordinary Czochralski method – of a 699 g ZnWO$_4$ crystal. Both crystals were used in the experiments to search for double beta decay.

1 Indeed, the ratio surface/volume is higher in the smaller crystal sample, which could explain the higher value of the specific total α activity in the ZnWO$_4$ crystal after recrystallization.

2 The low yield of ZnWO$_4$ crystal (20%) is typical for ordinary Czochralski method, in contrast to the low-thermal-gradient Czochralski technique (used for the $^{116}$CdWO$_4$ re-cristallization) which allows to obtain crystal boules with the mass of up to ~90% of the initial charge [24].
Figure 2: (Color online) Left: Boule of enriched $^{116}$CdWO$_4$ crystal obtained by recrystallization. The conic part of the boule is the beginning of the crystal growth. Right: The boule cut into three parts. The side surface of the central part (used in the present study) is diffused to improve the light collection.

The radioactive contamination of the samples is summarized in Ref. [19]. The total alpha activity in the recrystallized sample, 0.47(7) mBq/kg, is even higher than that of the initial crystal, 0.18(3) mBq/kg. Such a difference in the specific activity could be explained by concentration of uranium, thorium and their daughters in the surface layer of the crystals. The side surface layer $\approx 0.4$ mm of the recrystallized sample was removed by a diamond-needle file to test the assumption that the impurities were concentrated in a thin surface layer. Then the side surface of the crystal was made opaque by grinding paper. The mass of the crystal after the treatment was 133 g.

All the crystal samples were carefully cleaned using special low radioactive detergents, ultra-pure nitric acid and water before the low background measurements described in the next section.

3. Low background measurements

The radioactive contamination of the samples was measured in the DAMA/CRYS set-up at the Gran Sasso underground laboratory. This is a low background facility realized for low counting experiments using scintillation detectors. The passive shield of the set-up is made of high purity copper (11 cm), lead (10 cm), cadmium (2 mm), and polyethylene (10 cm). The set-up is sealed and continuously flushed by high purity nitrogen gas to prevent
the detector to be in contact with the environmental air. The inner volume available for scintillation detector(s) installation is $(82 \times 16 \times 14) \text{ cm}^3$.

The $^{116}$CdWO$_4$ crystal scintillator before the recrystallization and the ZnWO$_4$ crystal were viewed by a low-radioactive 3 inch green enhanced photomultiplier (PMT, Hamamatsu R6233MOD) through a high purity quartz light guide $\varnothing 7.6 \times 10 \text{ cm}$.

The $^{116}$CdWO$_4$ crystal scintillator after the recrystallization was installed inside a cavity ($\varnothing 47 \times 59 \text{ mm}$) in the central part of a polystyrene light guide $\varnothing 66 \times 312 \text{ mm}$. The cavity was filled with high-purity silicon oil. Two high-purity quartz light guides $\varnothing 66 \times 100 \text{ mm}$ were optically connected to the opposite sides of the polystyrene light guide. The scintillation light was collected by two low-radioactive 3 inch PMTs (EMI9265B53/FL).

An event-by-event data acquisition system based on a four channels 14 bit transient digitizer (CAEN DT5724D) records the time of each event and the pulse profile with a sampling rate 100 MS/s.

The energy scale and energy resolution of the $^{116}$CdWO$_4$ and ZnWO$_4$ detectors were measured with $^{22}$Na, $^{60}$Co, $^{137}$Cs, $^{133}$Ba, and $^{228}$Th $\gamma$ sources.

4. Data analysis, results and discussion

4.1. Pulse-shape discrimination

The $\alpha$ events in the CdWO$_4$ and ZnWO$_4$ detectors were selected with the help of the pulse-shape discrimination procedure based on the optimal filter method proposed by E. Gatti and F. De Martini [25]. For each signal $f(t)$, the numerical characteristic of its shape (shape indicator, $SI$) was defined as: $SI = \sum f(t_k) \times P(t_k) / \sum f(t_k)$. The sum is over the time channels $k$, starting from the origin of signal and up to 30 $\mu$s, and $f(t_k)$ is the digitized amplitude (at the time $t_k$) of a given signal. The weight function $P(t)$ was defined as: $P(t) = [f_\alpha(t) - f_\gamma(t)]/[f_\alpha(t) + f_\gamma(t)]$, where $f_\alpha(t)$ and $f_\gamma(t)$ are the reference pulse shapes for $\alpha$ particles and $\gamma$ quanta. By using this approach, $\alpha$ events were clearly separated from $\gamma$ ($\beta$) events as shown in Fig. 3 where the scatter plot of the shape indicator versus energy is depicted for the data of the low background measurements over 2394 h with the $^{116}$CdWO$_4$ detector before the recrystallization. The events with energy 2–4 MeV outside the $\gamma$ ($\beta$) and $\alpha$ belts are caused by the fast decay chains (Bi-Po events): $^{212}$Bi–$^{212}$Po (daughters of $^{232}$Th) and $^{214}$Bi–$^{214}$Po ($^{238}$U).

The energy spectra of $\alpha$ particles selected by the optimal filter method from the data accumulated with the $^{116}$CdWO$_4$ crystal scintillators before
and after the recrystallization are presented in Fig. [4]. The spectra were fitted by the model, which includes α peaks of 232Th, 238U and their daughters. Equilibrium in the 232Th and 238U chains was assumed to be broken. The results of the fits are presented in Fig. [4] and in Table [2]. One can see a clear decrease of the total alpha activity in the 116CdWO4 crystal scintillator after the recrystallization by a factor ≈ 3. Segregation of uranium and lead in the 116CdWO4 crystal growth process was also observed in work [26], where a sample produced from the growth cone was tested as low temperature scintillating bolometer.

4.2. Time-amplitude analysis to estimate 228Th activity

The activity of 228Th in the crystals was estimated with the help of the time-amplitude analysis (see e.g. Ref. [27]). The arrival time, the energy

Figure 3: (Color online) Shape indicator (see text) versus the energy accumulated over 2394 h in the low-background set-up with the 116CdWO4 crystal scintillator before the recrystallization. The two sigma intervals for the shape indicator values corresponding to γ quanta (β particles) and α particles are shown. The α events are clearly separated from γ and β events. The counts distributed in the energy region 2 – 4 MeV outside of the α and γ(β) belts are events of Bi–Po decays.
Figure 4: (Color online) The energy spectrum of α events selected by the pulse-shape discrimination from the data accumulated in the low-background set-up with the $^{116}$CdWO$_4$ crystal scintillator before (upper figure, time of measurement 2394 h) and after the recrystallization (lower figure, 1623 h).

and the pulse shape of each event were used to select the fast decay chain in the $^{228}$Th sub-chain of the $^{232}$Th family: $^{224}$Ra ($Q_\alpha = 5.789$ MeV, $T_{1/2} = 3.66$ d) $\rightarrow$ $^{220}$Rn ($Q_\alpha = 6.405$ MeV, $T_{1/2} = 55.6$ s) $\rightarrow$ $^{216}$Po ($Q_\alpha = 6.906$ MeV, $T_{1/2} = 0.145$ s) $\rightarrow$ $^{212}$Pb. As a first step, the α events within an energy interval 0.8 − 1.85 MeV were used as triggers, while for the second events a time interval 0.001 − 1.0 s and the energy interval 0.8 − 1.95 MeV were required. The α events were selected in the $\pm 2.327\sigma$ ”alpha” region. The energy spectra of $^{220}$Rn and $^{216}$Po α particles, and the distribution of the time intervals between the events were selected at this step.

As a next step, all the selected pairs ($^{220}$Rn − $^{216}$Po) were used as triggers in order to find the events of the decay of the parent α active $^{224}$Ra. A 1−112 s time interval (74.01% of $^{220}$Rn decays) was chosen to select α events in the energy interval 0.6 − 1.6 MeV. Here we took into account the quenching in the CdWO$_4$ and ZnWO$_4$ scintillators for α particles [28] to choose the
energy intervals where the $\alpha$ events of $^{224}$Ra, $^{220}$Rn, and $^{216}$Po are expected. The energy spectrum of $^{224}$Ra $\alpha$ particles and the distribution of the time intervals between $^{224}$Ra and $^{220}$Rn decays were obtained at the second step of the time-amplitude analysis.

The $\alpha$ peaks and the distributions of the time intervals between events obtained by the time-amplitude analysis of the data measured with the $^{116}$CdWO$_4$ crystal scintillator before the recrystallization (see left column in Fig. 5) are in agreement with those expected for the chain. The activity of $^{228}$Th in the crystal before the recrystallization was calculated as 0.10(1) mBq/kg. The results of the time-amplitude analysis of the data accumulated with the $^{116}$CdWO$_4$ detector after the recrystallization are presented in Fig. 5 (right). The specific activity of $^{228}$Th after recrystallization decreased by $\approx 10$ times to the level of 0.010(3) mBq/kg, which is the result of the strong segregation of thorium in the CdWO$_4$ crystal growth process. It should be stressed that thorium is the most harmful radioactive contamination for double beta decay experiments due to a large energy release ($Q_\beta = 4999$ keV) in the beta decay of $^{208}$Tl (daughter of $^{228}$Th from the $^{232}$Th family).

The summary of radioactive contamination of the $^{116}$CdWO$_4$ crystals before and after the recrystallization process (or limits on their activities) is given in Table 2.
4.3. Radioactive contamination of ZnWO₄ crystal scintillator with removed surface layer

The total α activity in the ZnWO₄ crystal after the surface layer treatment was estimated with the help of the pulse-shape discrimination of the data accumulated over 1445 hours to be 0.45(3) mBq/kg, i.e. the same as before the surface treatment: 0.47(7) mBq/kg. The activity of ²²⁸Th was estimated by using the time-amplitude selection of the events as ≤ 0.003 mBq/kg (only one ²²⁴Ra → ²²⁰Rn → ²¹⁶Po event was selected), while a specific activity 0.002(1) mBq/kg was measured in the ZnWO₄ crystal before the surface layer treatment.
Therefore, no reduction of the specific activity of $^{228}$Th and of the total $\alpha$ activity of $^{232}$Th and $^{238}$U and their daughters was observed after removing the 0.4 mm surface layer. Taking into account the rather similar chemical and physical properties of ZnWO$_4$ and CdWO$_4$ crystals, one can assume that the concentration of uranium and thorium in a thin (a few tenths of a millimeter) surface layer of the enriched $^{116}$CdWO$_4$ crystal scintillators reported in Ref. [20] can be explained by a pollution of the $^{116}$CdWO$_4$ crystal due to diffusion of radioactive elements in the crystal during the annealing process. In fact, in such an annealing process the $^{116}$CdWO$_4$ crystal boule was in direct contact with the ceramic oven at high temperature during approximately one day. An R&D to test this assumption is in progress aiming the development of highly radiopure cadmium and zinc tungstate crystal scintillators for low counting experiments.

5. Conclusions

The recrystallization of a sample of cadmium tungstate crystal enriched in $^{116}$Cd reduced the thorium contamination of the sample by one order of magnitude to the level of 0.01 mBq/kg ($^{228}$Th). The total alpha activity due to uranium, thorium, and their daughters decreased by a factor $\approx 3$ to the level of 1.6 mBq/kg. This finding, as well as the much higher contamination of the rest of the melt remaining in the platinum crucible after the crystal growing process by potassium, radium and thorium, indicates strong segregation of the radioactive elements in the CdWO$_4$ crystals growing process. This feature can be used in order to produce highly radiopure CdWO$_4$ crystal scintillators for high sensitivity double beta decay experiments.

Moreover, possible concentration of radioactive elements (the activity of $^{228}$Th and the total $\alpha$ activity of uranium, thorium and their daughters) in a thin, $\approx 0.4$ mm, surface layer was not observed in the ZnWO$_4$ crystal. Therefore the concentration of $\alpha$ active uranium and thorium and their daughters in a thin surface layer of the enriched $^{116}$CdWO$_4$ crystal scintillators reported in Ref. [20] was not due to the growing process but most likely due to the radioactive elements diffusion into the $^{116}$CdWO$_4$ crystal during the annealing process in a ceramic oven.

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