Growth of long undoped and Ce-doped LuAG single crystal fibers for dual readout calorimetry

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Abstract

Undoped and Ce3+-doped Lu3Al5O12 (LuAG) fibers were grown to evaluate their potential use in new particle physics experiments, such as dual-readout calorimeters. The choice of grown crystals was made to detect scintillation (doped LuAG) and Cherenkov radiation (undoped LuAG). Growth conditions for obtaining fibers with improved quality were found based on measurements of attenuation length of the fibers and cathodoluminescence measurements. The effect of annealing on attenuation length for LuAG and LuAG:Ce was also studied. In addition, we also evaluated a possibility to substitute LuAG by the cheaper mixed (Lu,Y)3Al5O12 (LuYAG:Ce) fibers.

Keywords: A1 Doping, A2 Growth from melt, B1 Oxides, B3 Scintillators

1. Introduction

Current and future calorimeters need novel crystalline materials and concepts for ionizing radiation detection. The excellent performance of the CMS electromagnetic calorimeter based on PbWO4 (PWO) contributed to the discovery of the Higgs boson [1]. New concepts for next generation experiments include combined electromagnetic (EM) and hadron calorimeter based on dual readout by detecting the Cherenkov and scintillation light [2]. One of the approaches is to use crystal fibers instead of bulk crystals [3, 4]. The construction at FAIR, the announcement of the LHC upgrade (HL-LHC) and intensive developments of the ILC Program [5] will require cheap and radiation hard material, capable for mass production.

The production of scintillator materials shaped as fibers has a long history and has collected an extensive list of applications in case of plastic/organic scintillators. However, the low effective Z of the
atomic components limits the sensitivity to electromagnetic probes. Since several years, enabled by the micro pulling down technique, fibers based on inorganic crystalline scintillator can be grown with continuously improving quality. The main challenge is the optimization of the fiber growth technology to reduce crystal imperfection and achieve homogeneous distribution of Ce$^{3+}$ activation centers \[6\]. In particular, the manufacturing of LuAG:Ce and LYSO:Ce, which are bright scintillators with high radiation hardness and compactness due to a short radiation length, imposes additional complications due to their high melting point and the large difference in geometrical dimensions of the host and Ce$^{3+}$ ions. For both materials, which are considered for a new generation of sampling calorimeters with dual read-out for hadron detection or pure electromagnetic calorimetry and additional applicability in PET-tomography, significant progress has been achieved [7-9]. Previously grown LuAG:Ce fibers had shown good results on the attenuation length measurements [9].

The present work is focused on growing and testing the reproducibility capabilities of a series of long (≥22 cm) and homogeneous undoped and Ce-doped, scintillating fibers with improved attenuation length. The requirement of the 22 cm length was elaborated basing on the length of PbWO$_4$ crystals currently installed in the CMS experiment at CERN. The choice of 2 mm diameter fibers was a compromise between the granularity (smaller diameter means better spatial resolution) and a number of fibers in the calorimeter (smaller fiber diameter requires more fibers to fill the given volume and also the higher number of photodetector channels).

2. Experimental

2.1. Fiber growth by the micro-pulling down technique

The LuAG fibers were grown from the melt by the micro-pulling down technique. All the experiments were done in inert Ar atmosphere using Ir crucible and afterheater. The details of the growth technique were described in [9]. Crushed parts of undoped, cerium-doped LuAG crystals previously grown by the Czochralski and micro-PD methods were used as raw material. The concentrations of Ce in melt did not exceed 0.1 at.%. The ~2.2 mm dia. fibers were grown, similar to the diameter of crucible capillary die. The melt meniscus thickness under the capillary did not exceed 0.05 mm. The fibers were grown with the rates 0.3-0.5mm/min to find the optimum balance between pulling rate and crystal quality. In order to estimate the seed orientation effect the fibers were obtained with the seed orientations [111] and [100]. In the case of Ce-doped fibers, LuAG:Ce seeds were used. The length of grown fibers reached 25 cm while 100% of the melt in the crucible was crystallized. The temperature gradient was measured using W/W-Re thermocouple.
2.2. Attenuation length measurements

Attenuation length was taken as the indicator of fiber optical quality. This parameter was measured by the two procedures described below.

2.2.1. LED bench

The undoped and Ce-doped LuAG fibers attenuation length measurements were carried out using a custom made setup under excitation with blue light (475 nm). LED driver (SP5601 from CAEN) was used; the light was transported with a clear fiber to the sample. Both extremities of the LuAG-based fibers were coupled to MPPCs, also called SiPMs, (Model S10931-050P from Hamamatsu). The output of these MPPCs were amplified with a dedicated instrumental amplifier (gain of 180) operated in a differential mode. Signals (of both left and right photodetectors) were then acquired with a digitizer (Model DT5720 from CAEN). The fibers were moved with a translating stage (Model M-413.32S from PI).

2.2.2. Xe lamp bench

Preliminary results on attenuation length of some LuAG:Ce fibers were obtained using the custom made setup with Xe lamp excitation at 350 nm. The beam from the source was focused on the side of the fiber. The scintillation light propagated through the fiber was registered on its end face. The signal was transferred by the optical fiber to the monochromator (Triax series 320) and a CCD camera.

2.2.3. Cathodoluminescence

The Ce radial distribution in Ce-doped fibers was evaluated by the distribution of the cathodoluminescence (CL) intensity across the fibers. It was measured with an AVT Pike F-100B CCD camera, with a magnification 3.95X. LuAG:Ce samples were cut perpendicularly to the growth axis. Before the installation on the holder the side not exposed to the electron beam was covered with black paint in order to avoid presence of reflected light. The beam from the electron gun EMG-4212 Kimball Physics was left unfocused in order to have a uniform electron flux on the entire sample surface.

3. Results and discussion

3.1. Undoped LuAG fibers growth

Figure 1 shows the undoped LuAG fibers grown at different pulling rate in the range 0.3-0.5mm/min along [100] and [111] direction. To combine a homogeneous diameter and stable regime, the power
applied to the RF heater should be increased during the growth process. Otherwise the fiber diameter becomes unstable. Such effect is caused by the increase of heat sink from the meniscus with the elongation of the fiber. Empirically we found that, in the case of LuAG fiber, to compensate the heat sink increase, the power should be gradually increased by ~0.85 % from the beginning (connection step) till the end of the growth of 22-25 cm long fiber. Macroscopically, the grown undoped LuAG fibers don’t present any visible defects such cracks and bubbles. The undoped fibers were selected for Cherenkov light readout, the convolution has to be compared with the emission spectra of Cherenkov light (~1/λ²). Figure 2 shows the attenuation curves as a function of the pulling rate at 475 nm. The best results are obtained at low pulling rate (0.3mm/min). The average results give 8.7 cm attenuation length for fibers grown at 0.5mm/min, as opposed to 27.6 cm when grown at 0.3mm/min. The largest attenuation length of 38 cm was obtained with fiber v31 pulled at 0.3mm/min along [111] direction and more generally the best performing fibers were obtained with the fibers grown at this low pulling rate. This pulling rate is very likely to be the critical value to get fibers without defects and a minimum level of stress, resulting in good light propagation along the fibers. The attenuation length of the fiber grown along [100] and [111] are similar and we did not observed seed effect orientation on the attenuation.

2. Ce-doped LuAG fibers growth

Ce-doped LuAG fibers of length higher than 22 cm were grown with pulling rate 0.3 mm/min along [111] and [100] directions (Figure 3). With well optimized growth parameters [9], green transparent fibers, with homogeneous diameter (variation less than 1%) and free of macroscopic defects were grown. The growth conditions for Ce-doped LuAG fibers has to be adjusted as compared to the undoped LuAG fibers. During the growth of Ce-doped LuAG fibers the power increase was two times larger than in the case of undoped fibers growth (~1.9 % of power increase relatively to the seeding stage) showing a difference melt behavior between the two compositions. Obviously, this significant difference was explaining the larger attenuation length observed on Ce-doped fibers due to different radiative heat sink through the fibers. The best results achieved with 0.1 at.% Ce concentration and pulling rate of 0.3 mm/min. At higher Ce concentration, stronger dopant segregation affect the light transmission along the fibers and (LuCe)AlO₃ perovskite inclusions with CeO₃ free particles were observed which strongly increase the light attenuation through the fiber. We have noted that the perovskite phase could be nucleated only if the starting charge contains a high cerium concentration (>1.5at%).

The regions with these perovskite inclusions can be observed on the attenuation plots. Comparing the fiber vc02 with no visible inclusions (figure 4) to the fiber vc10 containing inclusions (figure 5), the smooth light absorption absorbed for vc02 is very different to the attenuation plot of vc10 which
contains bumps where the inclusions are located. The Ce doping concentration in the LuAG host matrix is therefore a key parameter for predicting attenuation characteristics. The comparison of figures 4 and 5 bring us to conclude that a good fibers quality without inclusions presence and segregation effect is crucial to obtain reasonable light attenuation length. In addition such defects are localized in the surface area and inside the fibers, in this case surface diffusion is otherwise favoring the light escape through the lateral faces. Also we note that the problem of inclusion appearance is inherent to the doped fibers, while no such inclusions are observed in the undoped fibers with no intentional admixtures. For a low pulling rate \((v \leq 0.3 \text{ mm/min})\) the crystallization interface is plane. This condition ensure a good surface regularity and a low level of defects. At speed higher than the critical maximum growth rate, constitutional undercooling generates defects such thermal stress. The fibers grown with a low pulling rate have a constant diameter and a smooth surface. This is mainly related to the stable molten zone and the interface. So, it is very important to optimize the optimum regime in which fibers can be grown without defects.

There is a tendency to improve the attenuation length through controlling the thermal gradient. The normal growth rate of the fiber-melt interface is essentially controlled by the rate at which the pull fiber is raised. To reach the condition of steady growth with constant diameter requires that the net heat flow through the interface exactly balances the rate of evolution of latent heat due to the crystallization process [10]. For flat interfaces (which is the case here), this condition can be described by the following equation:

\[
\rho_s \Delta H_m v + K_l G_l = K_s G_s
\]

Where \(\rho_s\) is the density of the solid, \(\Delta H_m\) the latent heat of fusion, \(v\) the pulling rate, \(K_{s,l}\) are the thermal conductivities of liquid and solid phases and \(G_{s,l}\) the axial temperature gradients in the solid and liquid phases at the crystallization interface (meniscus height). High axial temperature gradient \((G_s)\) which increases with decreasing fiber diameter are connected to high pulling rate.

**Figure 6** represents the attenuation results for 4 fibers grown under different axial thermal gradient corresponding to high thermal gradient at about 60°C/mm (vc1, vc2) and lower thermal gradient about 20°C/mm (vc16, vc21). The fibers are sequentially numbered starting from the beginning of the experiments. The fibers vc16 and vc21 grown in optimized thermal conditions and low thermal gradient (0.3mm/min) have smoother attenuation (38 cm) curve at 350 nm excitation higher than the fiber grown at high thermal gradient (17 cm). So it is possible to improve the light propagation inside the fiber by controlling the axial thermal gradient through controlling pulling rate.
3.3. Effect of annealing

The Lu substitution by Ce into the crystal structure of LuAG introduces distortion due to the different radii of dopant and host ions. Whatever the shape of the crystals, such distortions in Ce-doped LuAG crystals will create some stress during the cooling of the crystal, it is the major source of dislocation in the as grown fibers. These microscopic defects can be one reason explaining observed lower light propagation in some of the grown fibers. In addition, high thermal gradient related to pulling rate can also induce stress in the crystal allowing to transmission and light propagation decreasing of the pulled fibers. It is known that annealing crystals after growth process could be a good way to eliminate such defects, redistribute dopant and to improve crystals optical performance. To investigate the effect of thermal annealing on the attenuation length, 8 fibers (4 undoped LuAG and 4 LuAG:Ce) grown with different pulling rates and crystallographic orientations were annealed during 24 hours in air atmosphere at 1200°C. Table 1 summarize the results. The undoped LuAG fibers seem to be not affected by the considered annealing treatment. Regardless the growth rate and the grown direction, we did not observe any improvement in the light propagation through undoped LuAG fibers. On the another hand, the attenuation length of Ce-doped LuAG can significantly be improved, especially for fibers grown with the 0.3 mm/min rate. The largest attenuation length of more than 1 meter is achieved in LuAG:Ce fibers grown along [100] direction (Figure. 7). This value is a factor of 2 larger than the fiber grown along [111]. This is a strong indication that annealing the grown fiber significantly improves the attenuation length of Ce-doped LuAG fibers grown at low pulling rate. The attenuation improvement can be connected to better redistribution of Ce dopant in the annealed fibers. In addition, this results show that annealing does not strongly improve the attenuation of the fibers grown at 0.5mm/min and consequently the most important factor remains the pulling rate. Thus the high pulling rate may introduce a drastic change of grown- in defects. In addition new defects may be created which need high temperature and long time to be eliminated.

However, no structural change through the annealing could be detected by the powder x-ray diffraction. In addition, no coloration change was observed.

3.4. Cerium (Ce) dopant distribution in the fibers.

During Ce-doped LuAG fibers pulling from the melt, the cerium concentration in the liquid changes and this leads to a continuous variation of cerium dopant in the fiber. In the case of Ce-doped LuAG melt, convectional mixing or diffusion are present in the liquid. The concentration of Ce in the melt is C₀ and immediate diffusion /mixing of the liquid is considered. In this case, the crystallized fraction appears with a dopant concentration kₘC₀ less than the concentration C₀ in the melt (ks is segregation
coefficient). The exceeding dopants stay in the melt and, through instantaneous diffusion, consequently increase the concentration in the melt. In such case, two possibilities can be considered: either the convective movements in the meniscus are negligible, then the transport is diffusive, either they are preponderant; the liquid is then assumed homogeneous [11]. When diffusion occurs, conservation of the dopant mass in each of the phases can be written in the frame of the solid–liquid interface:

$$\frac{\partial C_l}{\partial t} = D \left( \frac{\partial^2 C_l}{\partial Z_l^2} \right) + U_{\text{liquid}} \left( \frac{\partial C_l}{\partial Z_l} \right)$$

where $D$ is the solute diffusion coefficient in the liquid and $U$ the fluid velocity. This gives the concentration profile in the liquid in the meniscus or in the capillary, with a boundary layer of the form [12].

$$C_l = C_0 + (C^*-C_0) e^{-\frac{U_{\text{liquid}} Z_l}{D}}$$

with $C^*$ the concentration at the interface or at the capillary die.

In the case of Ce-doped LuAG fibers grown by the micro-PD method, a substantial gradient of the Ce concentration might appear in the radial direction [13] and have a negative impact on the fiber attenuation length. Distribution coefficient of activator can be improved by decreasing the ionic radii difference between activator and host ions. Regarding complex oxides it has been illustrated, for example, in [14, 15]. In this connection the transfer to mixed $\text{Lu}_3-x\text{Y}_x\text{Al}_5\text{O}_{12}:\text{Ce}$ (LuYAG:Ce) crystal with larger averaged radius of host cations ($r(\text{Lu}^{3+})=0.977, r(\text{Y}^{3+})=1.019, r(\text{Ce}^{3+})=1.143$ for eightfold coordination [16]) is a way to improve homogeneity of $\text{Ce}^{3+}$ distribution. In addition, it is noteworthy that lutetium substitution with yttrium reduces fiber cost, since the yttrium oxide is much cheaper. Cathodoluminescence microscopic imaging of the fiber cross section is an easy method to evaluate the activator distribution in the fibers (assuming the proportionality between the activator concentration and the emission intensity). For the cathode luminescence experiments we used samples fabricated from LuAG:Ce fibers grown at the different rates. For reference we used $\text{Lu}_{3-x}\text{Y}_x\text{Al}_5\text{O}_{12}:\text{Ce}$ (LuYAG:Ce) sample with the Y content 85 at. % and Ce concentration 0.15 at.% grown under the same conditions. All the regions, from which the samples were cut, are ~1 cm distant from the beginning of the fibers.

Regarding the brightness distribution at the shots the most of activator is concentrated at the fiber periphery (Table 2). Meanwhile, the periphery-to-center ratio of cathodoluminescence intensity is ~2 times for 350 µm/min growth rate and ~4-5 times for 0.5 mm/min growth rate. The similar intensity variation by 4-5 times is observed with LuYAG:Ce grown with 0.35 mm/min growth rate. Therefore, by ~1.5 times lower growth rate is required in LuYAG:Ce to sustain the $\text{Ce}^{3+}$ radial distribution like in LuAG:Ce. This means that, a part of profit in cost of raw material, mixed crystal have to be produced more slowly. Impact of these two factors must be accounted for development of mass production.
technology. The radial Ce concentration showed a concave curvature for Ce-doped LuAG grown at 0.35mm/min and nearly flat profile for Ce-doped LuYAG grown at 0.35mm/in and Ce-doped LuAG grown at 0.5mm/min. But slightly higher concentration in the periphery of the fiber. The melt comes down a very long narrow and long capillary die from the main melt container. The narrow capillary die is the way path from the melt main reservoir to the grown fiber [17]. The remaining melt above the pulled fiber cannot ascend through the capillary die into the main body of the crucible. Any additional cerium dopant concentration is swept to the peripheral area are forced into the growing fiber causing transversal segregation cerium behavior. Most of the values reported for Ce segregation in LuAG are different than unity. This fact greatly complicates the growth of homogeneous Ce-doped LuAG crystals.

**Conclusions**
The possibility capabilities of mass production of LuAG and LuAG:Ce fibers with the lengths over 22 cm was demonstrated for utilization in dual-readout detectors in high energy physics experiments. Optical attenuation of the scintillation light within the fiber is a critical parameter, since the scintillating signal has to propagate along entire fiber. The growth process was optimized to obtain the LuAG and LuAG:Ce fibers without cracks and visible inclusions and with the enhanced attenuation lengths over 22 cm. The best results were demonstrated with fibers grown at the 300 µm/min rate along the [100] direction. The attenuation length over 1 m at X-ray excitation can be achieved with LuAG:Ce after thermal annealing at 1200 °C in air. Meanwhile, such annealing is revealed inappropriate for undoped LuAG fibers. Following the cathodoluminescence data achieved in (Lu\(_{1-x}\)Y\(_x\))Al\(_5\)O\(_{12}\) fibers samples this compound should be grown by ~1.5 times lower pulling rate compared to LuAG:Ce to obtain an appropriate Ce radial distribution. The further work will be devoted to determination of radiation hardness of the fibers in harsh radiation environments.

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**Figures (Captions)**

Figure 1. Undoped LuAG fibers grown by µ-PD technique.

Figure 2. Luminescence signal intensity vs. the distance from excited area to the photodetector in the undoped LuAG fibers at 475 nm excitation. The attenuation lengths are quoted in the legend. Fibers grown with the following pulling rates are denoted as: 0.35 mm/min – “square”, 0.3 µm/min – “stars”, 0.5 mm/min – “triangles”, 0.5 µm/min grown with [100] seed – “rhombuses”.

Figure 3. Ce-doped LuAG fibers under UV lamp illumination.

Figure 4. Luminescence signal intensity vs. the distance from the excited area to the photodetector in the LuAG:Ce vc02, fiber at 350 nm excitation. The intensity slope corresponds to the visible inclusion on the photo.

Figure 5. Luminescence signal intensity vs. the distance from the excited area to the photodetector in the LuAG:Ce vc10 fiber at 350 nm excitation.

Figure 6. Luminescence signal intensity vs. the distance from the excited area to the photodetector at 475 nm LED excitation for LuAG:Ce fibers.

Figure 7. Effect of annealing on attenuation of the vc20 fiber. The attenuation lengths are indicated.
Table 1. Fibers annealing effect on the attenuation length

| Crystal          | LuAG | LuAG:Ce |
|------------------|------|---------|
| Growth rate, mm/min | 0.3  | 0.5     | 0.3   | 0.5   |
| Growth orientation |      | [100]  | [111] | [100] | [111] | [100] | [111] |
| Attenuation length, cm |      | Before annealing | After annealing |
|                   | 15 | 21 | 9,5 | 29 | 13 | 17 | 13 | 18 |
|                   | 14 | 11 | 7  | 24 | 104 | 50 | 14 | 23 |

Table 2. Photos and radial distributions of cathodoluminescence intensity in some fibers.

| Crystal          | Growth rate, mm/min | Diameter, cm | Normalized intensity |
|------------------|---------------------|--------------|----------------------|
| LuAG:Ce C35 0.35 mm/min |                     |              |                      |
| LuAG:Ce C19 0.5 mm/min |                     |              |                      |
| LuYAG:Ce C6 0.35 mm/min |                     |              |                      |
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