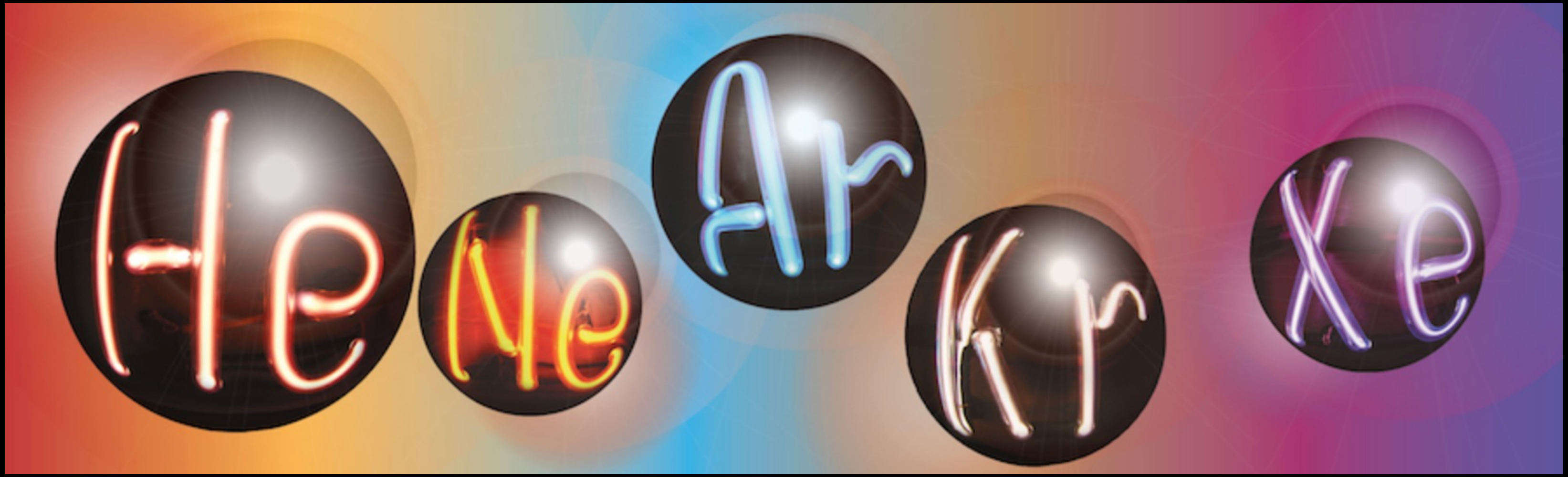
INSTRUMENTS AND METHODS FOR COATING OF WAVELENGTH-SHIFTING MATERIALS USED FOR NOBLE LIQUID TECHNOLOGY



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INFN-Naples

LIDINE 2023: LIght Detection In Noble Elements - Conference

Sep 20 – 22, 2023 --- Madrid - Spain

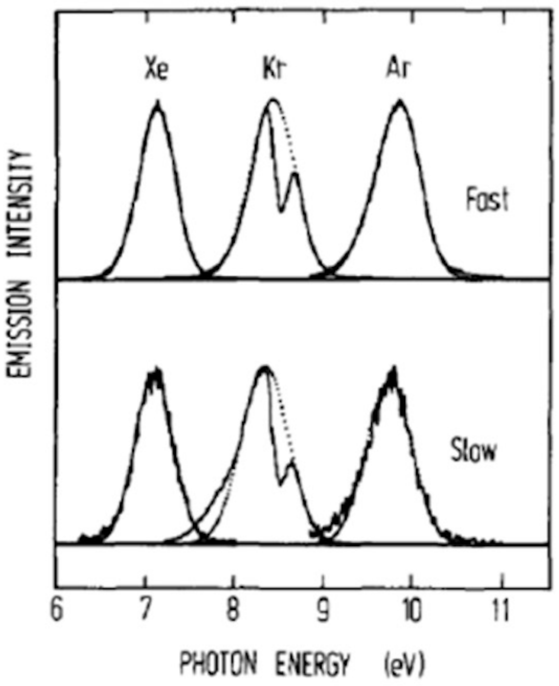
Outlines

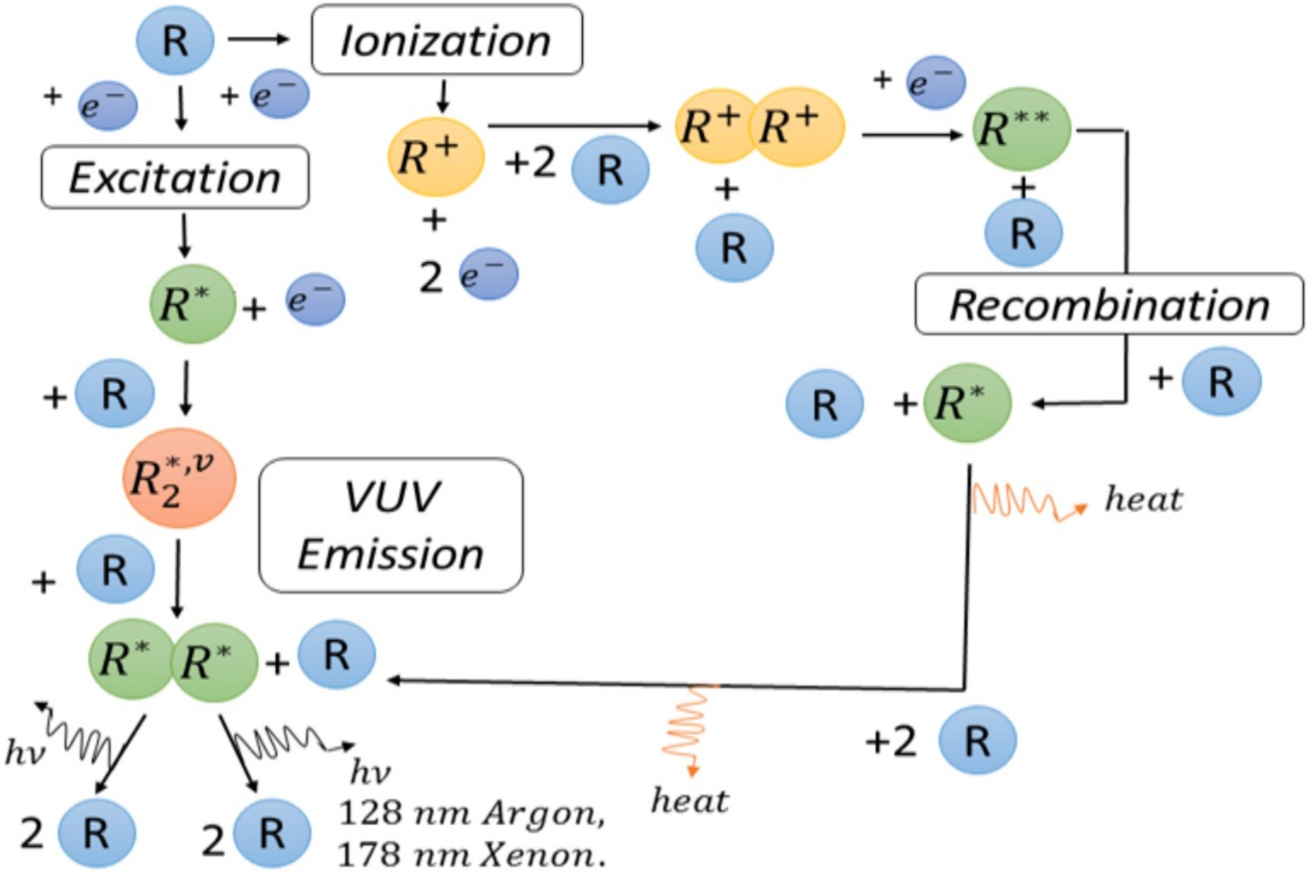
- Scintillation Light in Noble Liquids
- Wavelength-shifting Materials
- Most common WLS Deposition Techniques
- WLS Vacuum Deposition Design
- WLS Coating Procedures
- Evaluation of the WLS Process
- Factor Affecting WLS Deposition
- Main Noble Liquid Experiments using WLS Deposition
- References
- Conclusions

Scintillation Light in Noble Liquids

- Charged particles crossing the noble liquid volume produce excitation and ionization followed by recombination and both the processes lead to the emission of VUV light
- Noble liquid scintillation light emitted in the VUV range needs to be converted to be detected by «conventional» photosensors

- Argon: λ=128 nm, τ_S=6 -10 ns, τ_T=1500 ns
- Xenon: λ=178 nm, τ_S=3 ns, τ_T=27 ns

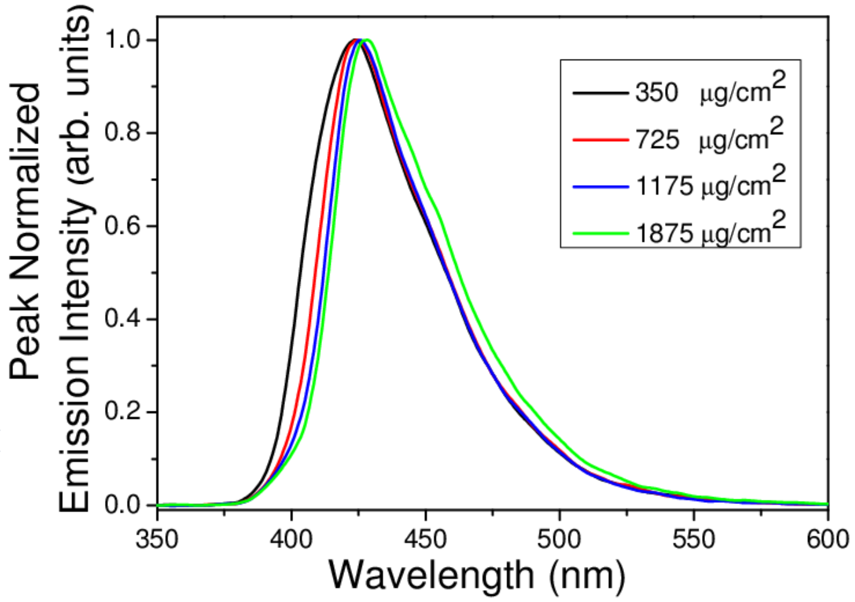


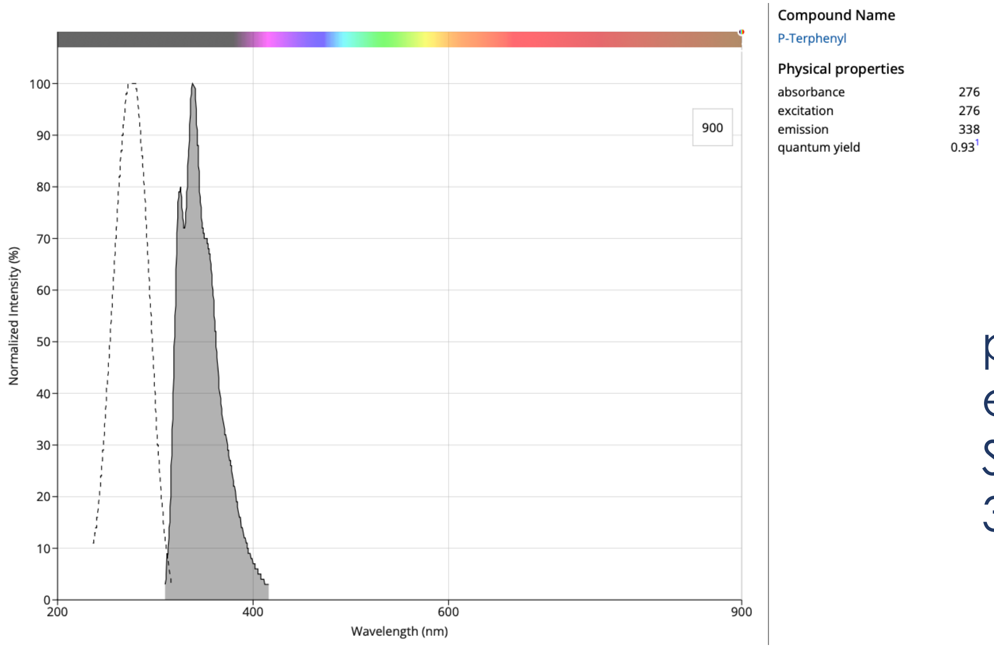


Wavelength-shifting Materials

- The use of wavelength-shifting coatings are of primary interest in the area of liquified noble gases detectors (Ar and Xe) used as active medium in the neutrino physics and dark matter experiments
- WLS materials, such as TetraPhenyl-Butadiene and p-Terphenyl, are especially used for conversion in the visible range
- WLS are very important in the case of large area detectors since only a fraction of the surface can be instrumented

TPB emission Spectrum: 420 nm





p-TP emission Spectrum: 350 nm

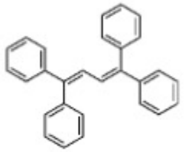
Wavelength-shifting Materials

- WLS materials, such as TPB and p-TP, are in form of powder
- TPB and p-TP are quite expensive in small amount
- Different techniques have been studied and developed to coat substrates with these materials

Product Specification

Product Name:
1,1,4,4-Tetraphenyl-1,3-butadiene suitable for scintillation, ≥99%

Product Number:	**185213**
CAS Number:	1450-63-1
MDL:	MFCD00004766
Formula:	C28H22
Formula Weight:	358.47 g/mol



TEST	Specification
Appearance (Color)	White to Off White
Appearance (Form)	Powder or Solid
Infrared spectrum	Conforms to Structure
Purity (HPLC)	≥99.0 %

Product Specification

Product Name:
p-Terphenyl – ≥99.5% (HPLC)

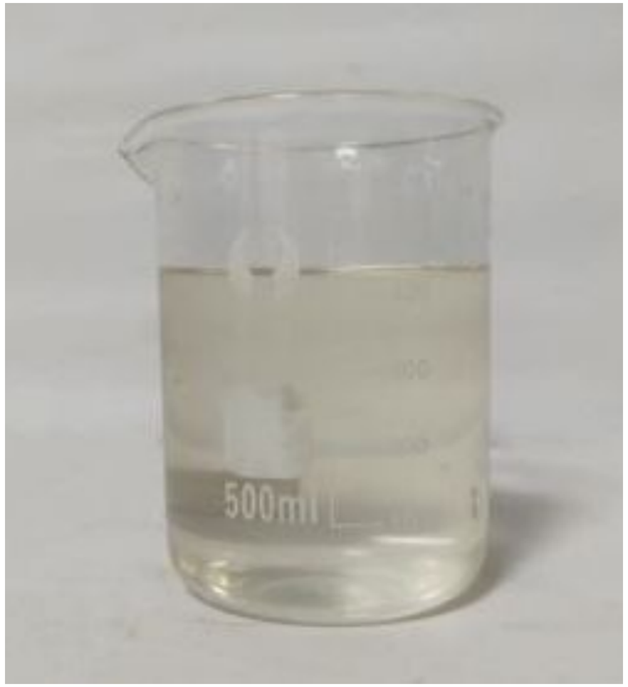
Product Number:	**T3203**
CAS Number:	92-94-4
MDL:	MFCD00003061
Formula:	C18H14
Formula Weight:	230.3 g/mol

TEST	Specification
Appearance (Color)	White
Appearance (Form) Crystalline Powder or Crystalline Granules	Conforms to Requirements
Infrared spectrum	Conforms to Structure
Purity (HPLC)	≥ 99.5 %

WLS Deposition Techniques

Different techniques have been studied a developed to coat substrates with WLS materials

- Vacuum evaporation
- Dipping in liquid mixtures
- Spraying diluted WLS
- Direct evaporation and dilution into the noble liquid (under study)



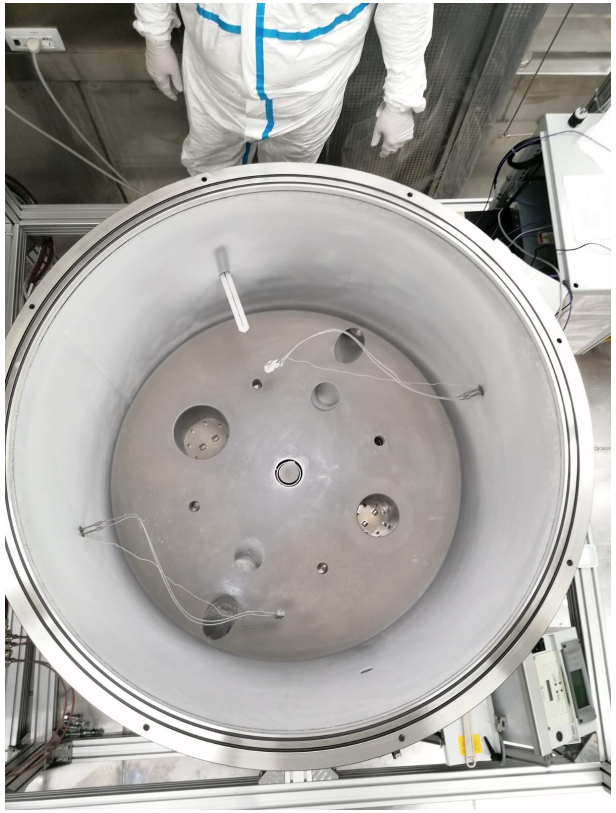
WLS Deposition Techniques

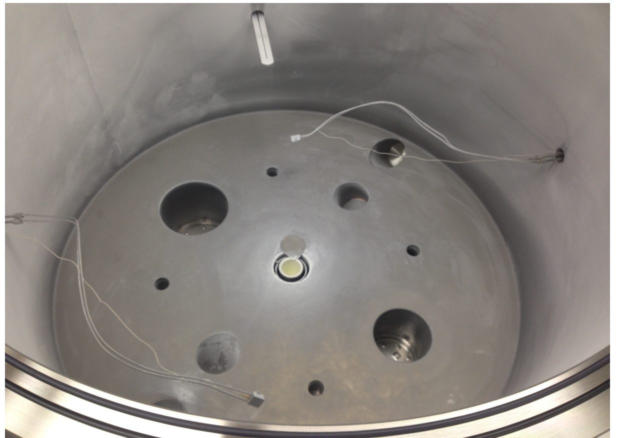
Technique	Spraying diluted WLS	Dipping in liquid mixtures	Vacuum evaporation
Reactants	WLS+Toluene	WLS+Toluene+polystyrene	WLS
Recipe	5 g of WLS per 1 l of toluene Warm the solution for ~1 h 0,5 cm^3 of solution sprayed on 1 cm^2 substrate	Mixtures with 100 cm3 of toluene+2 gr of polystyrene+400mg of WLS	Vacuum in the chamber Warming-up of the crucible Monitoring of the process
Materials	Fudes silica, glass, metals No plastic	Fudes silica, glass, metals No plastic	Any solid substrate
Set-up	Spraying system No vacuum	Dipping and rotating system for drying the mixture Procedure repeated 2 times	Evaporation system
Estimated relative efficiency (wrt evaporation)	~30% of the evaporation coating	~30% of the evaporation coating	To be evaluated
Coating uniformity/Features	Fast process	Robust matrix on the surface	Depending on the distance from the crucible, etc.

WLS Deposition Techniques

Vacuum deposition through evaporation system

- Stainless steel vacuum chamber
- Vacuum pumps (rough+turbopump(+cryopump))
- Crucible(s)+heating control unit(s)
- Quartz monitoring system
- Rotating motor
- Structure holding the chamber
- Crane for lifting the flange
- VUV lamp for a quick qualitative response
- Ancillaries





WLS Vacuum Deposition Design

Evaporation chamber can be designed considering the following cross-correlated parameters:

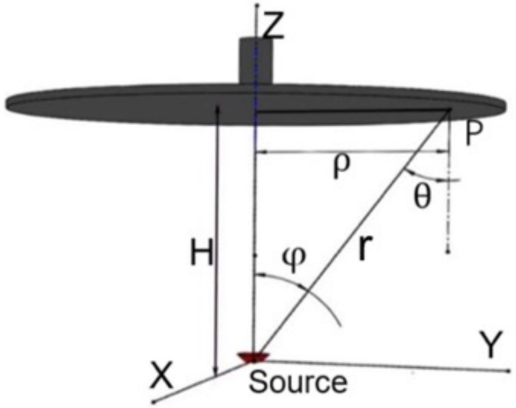
- Uniformity of the coating
- Size/dimensions and shape of the chamber
- Vacuum level
- Number of samples and/or materials deposited per cycle

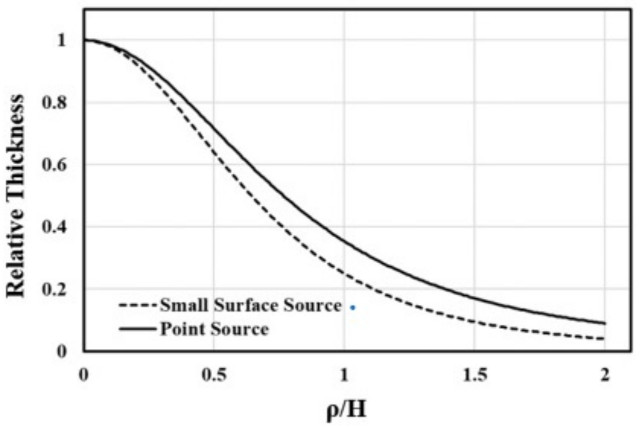
- Cylindrical symmetry chambers are the widest used and can ensure higher uniformity
- The height of the chamber impacts on the uniformity and on the number of evaporation sources
- The transverse dimensions are an important requirement for the number and the sizes of pieces to be coated
- Large evaporators need several hours to reach the vacuum level matching the requested mean free path for the evaporated molecules to coat the substrate

WLS Vacuum Deposition Design

Example of cylindrical symmetry chamber

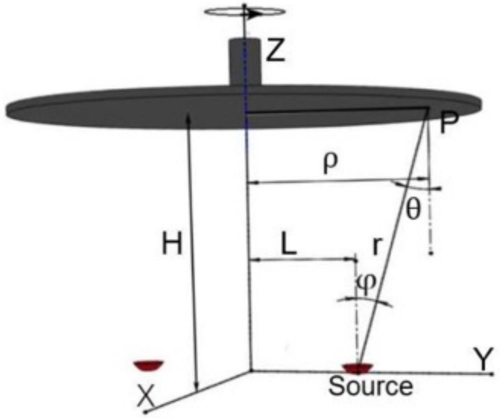
- Samples to be evaporated are placed on a disk holder positioned in the top circle of the cylinder
- Crucible(s) positioned on the opposite bottom circle of the cylinder

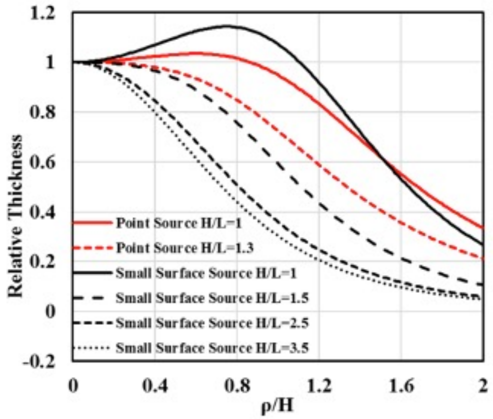


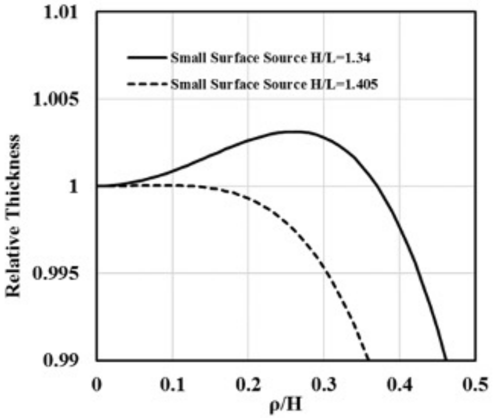


Coating uniformity as function of the chamber dimensions

Centered crucible at the bottom flange





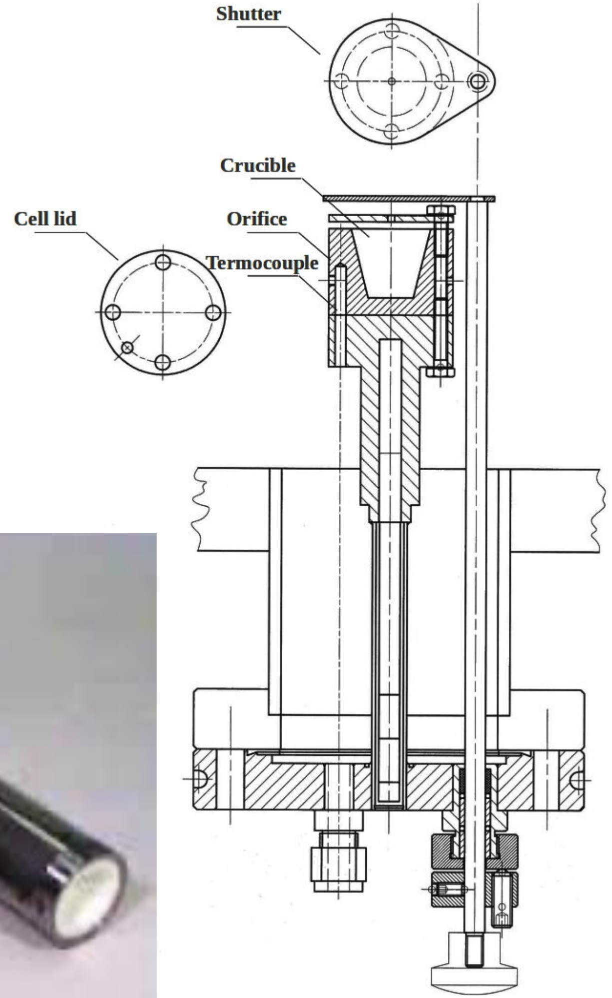


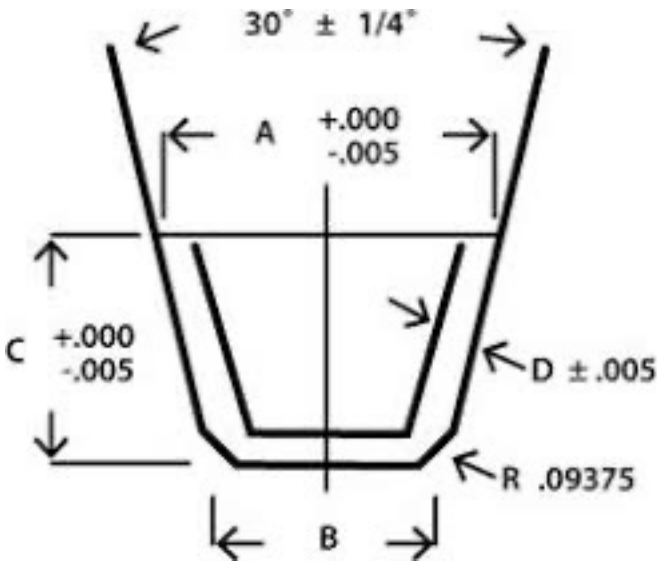
Off-axis crucible at the bottom flange

WLS Vacuum Deposition Design

Effussion cell

- Most common are Knudsen effusion cells
- Heating controllers and temperature monitors
- Different types of crucibles:
  - Allumina, boron nitride , carbon, ceramic, OHFCopper, fused silica, intermetallic, molybdenum, tantalum
  - Geometry and thickness depending on the material

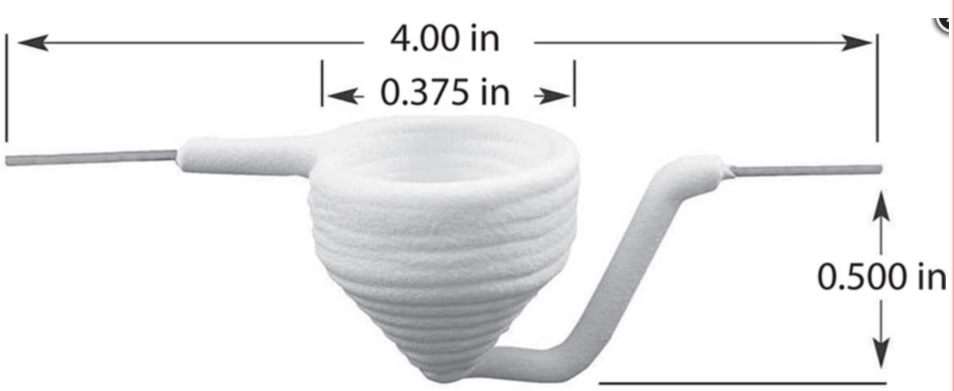


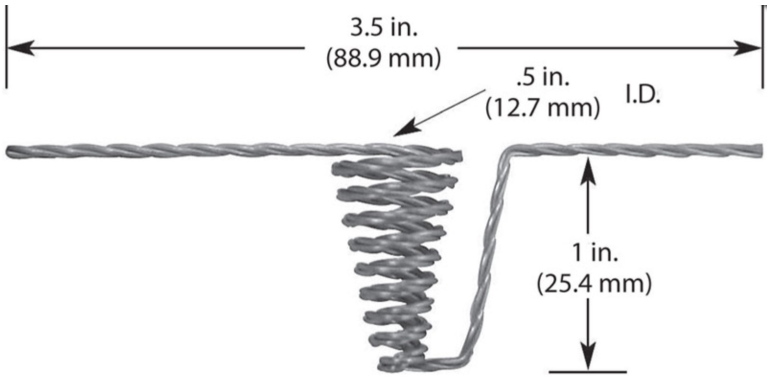


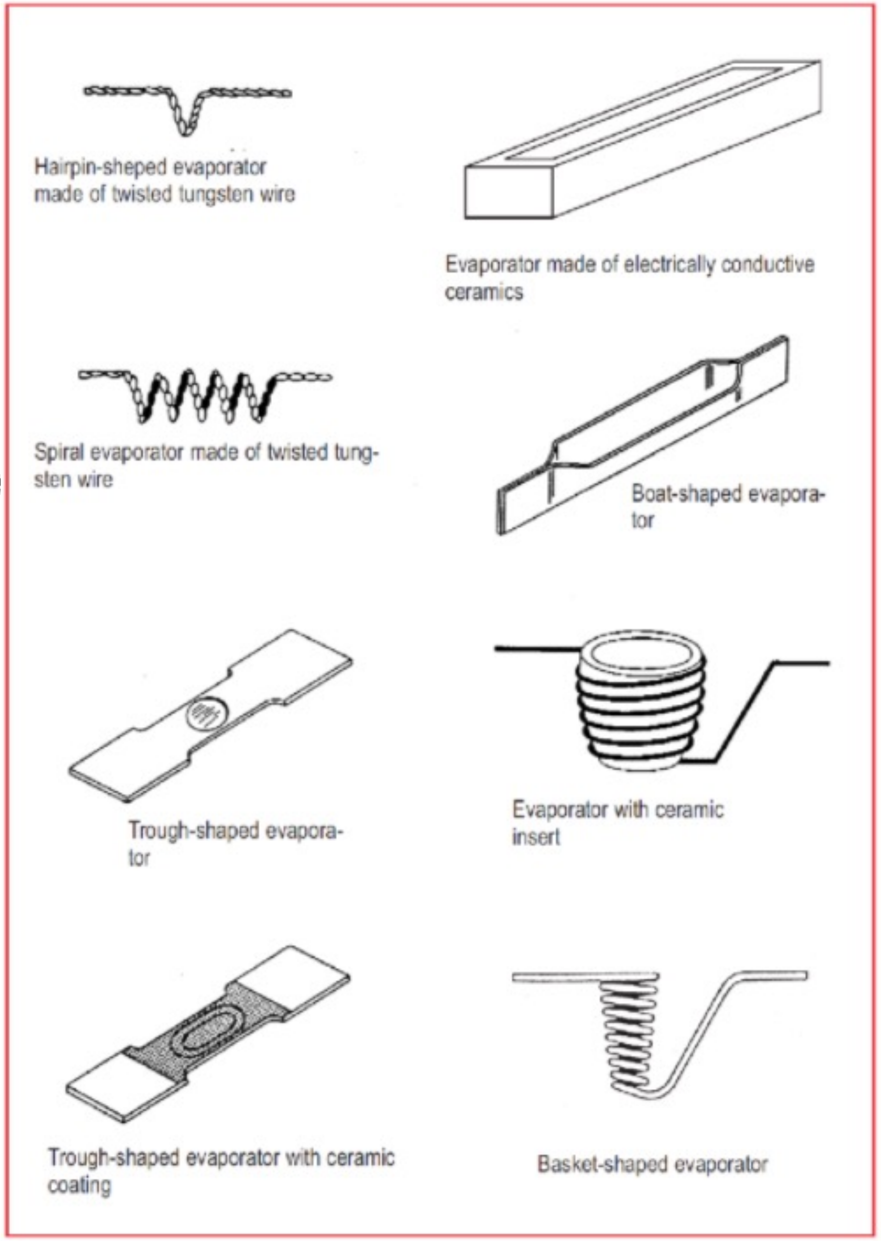
WLS Vacuum Deposition Design

Effussion cell

- Different types of heater:
  - wires
  - boats
- Different types of heating materials:
  - Alumina coated
  - Tungsten



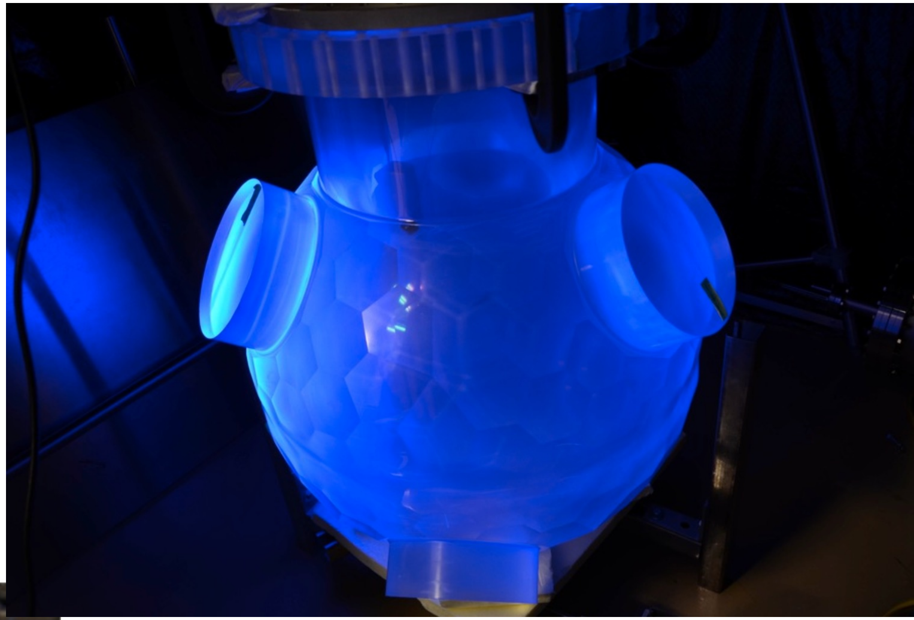


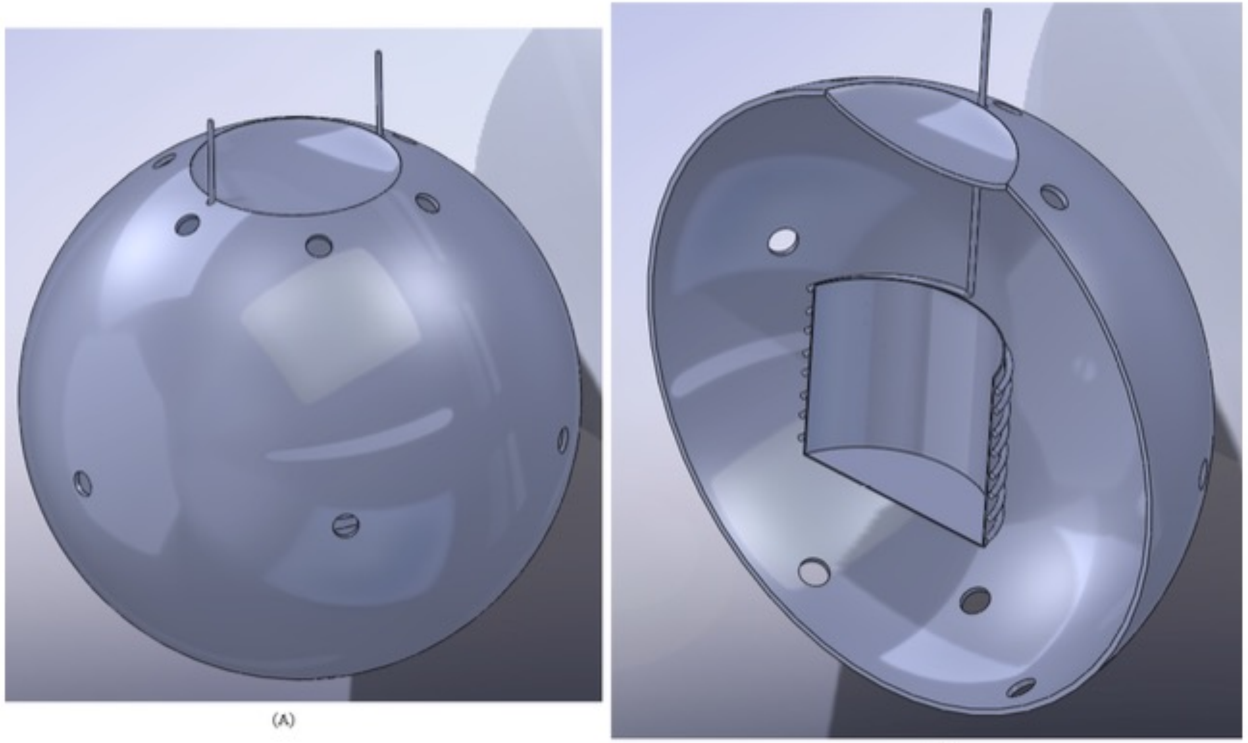


WLS Vacuum Deposition Design

Effusion cell

- Custom source for direct evaporation:
  - Selection of proper materials
  - Choice of geometry





(A)

(B)

Figure 12: (A) Isometric View of Final Assembly (B) Cross Section View of Final Assembly

Figure 3. Final stainless steel TPB deposition source. The coil heater is held in thermal contact with the sphere with compressed tabs.

Figure 4. The inner copper crucible to hold the TPB powder is attached to a stainless steel top hat and lowered into the spherical source.

WLS Vacuum Deposition Procedure

Vacuum deposition through evaporation system: scheme of operations

- Preparation of the substrates to be coated (cleaning, handling, etc.)
- Perparation of the reference samples
- Handling of the substrates and samples
- Insertion in the evaporation chamber of the parts
- Start the process (vacuum, evaporation, ventilation)
- Check the samples (weigh, visual inspection)
- Control the quality of the coating (intrumentation for quantitative measurements)
- Ensure safety during the operations

Istituto Nazionale di Fisica Nucleare

Laboratori Nazionali del Gran Sasso

DarkSide Operational Group Process Procedure

Wavelength shifter evaporation

Last Revision Date: June 2015

Procedure Author(s):

Marco Carlini

Reviewed by:

Nicola Canci (Evaporator Manager)

Andrea Ianni (Site Manager)

Federico Gabriele (GLIMOS/RAE)

Augusto Goretti (Operation Manager)

Last Revised and Approved by:

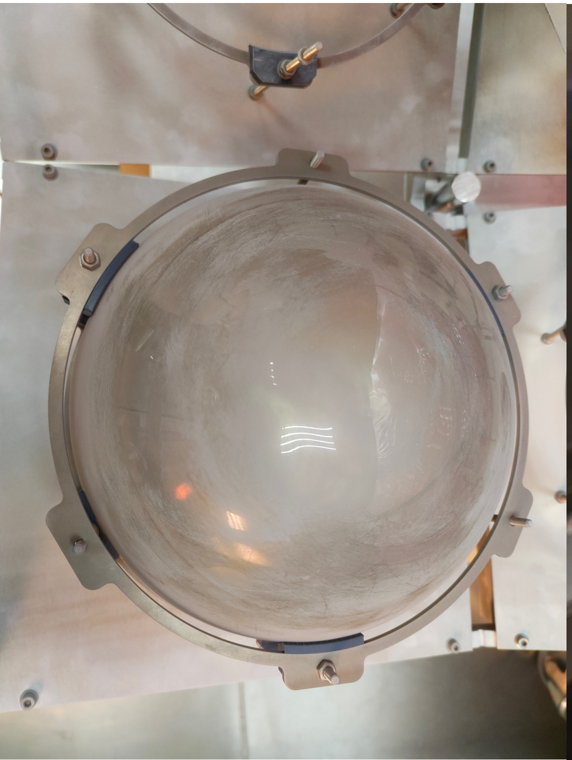
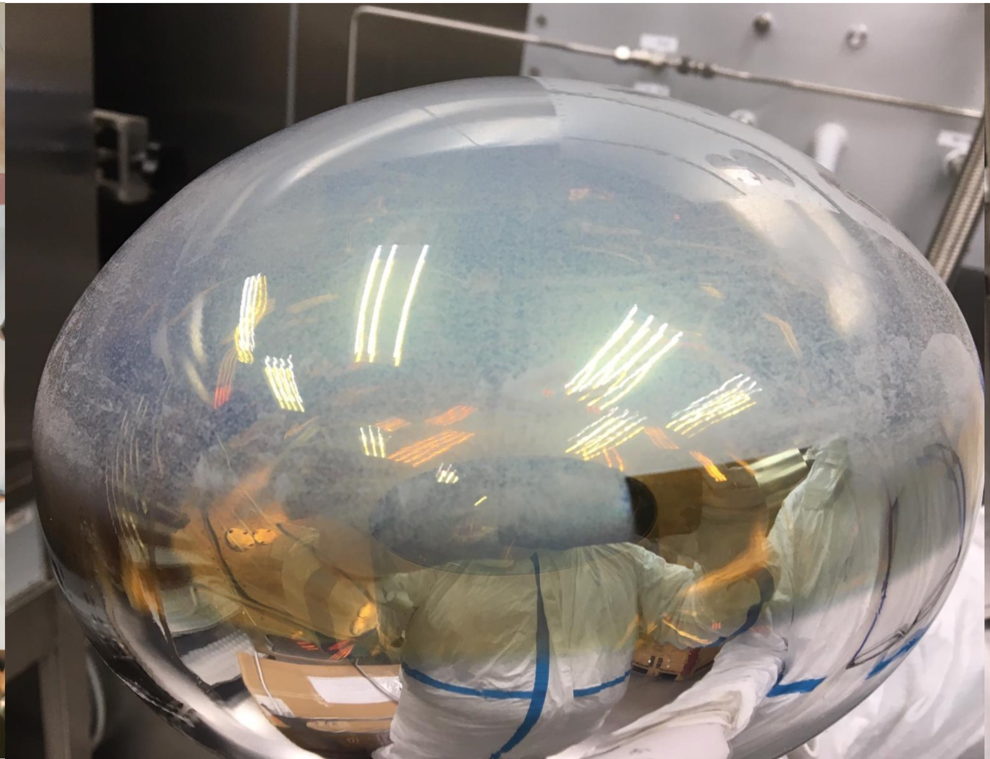
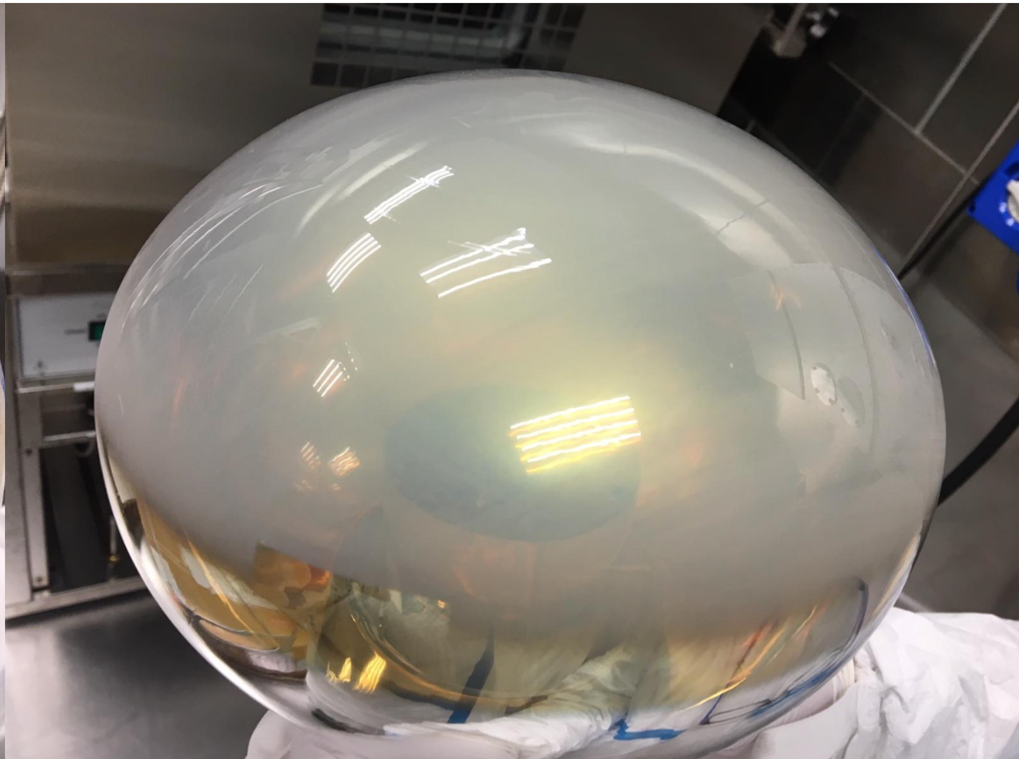
Andrea Ianni

Procedure validity: from Revision Date to End of Project

WLS Vacuum Deposition Procedure

Cleaning of the materials

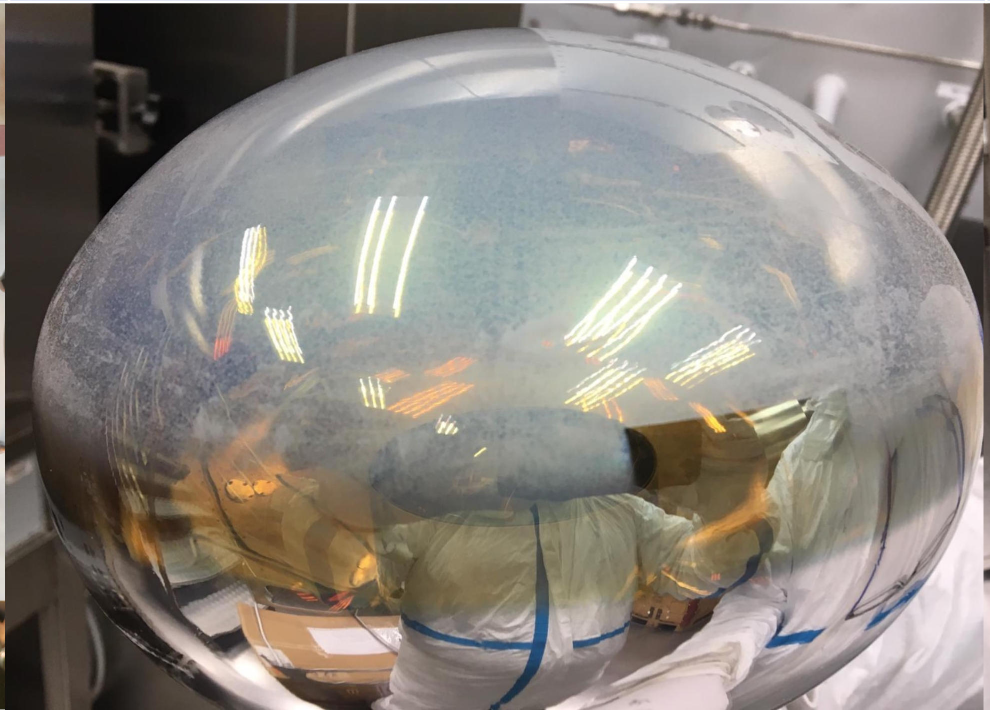
- Cleaning is one of major issue prior the coating
- Cleaning procedures and processes depend on the materials to be coated (plastic, glass, quartz, metal), ad hoc cleaning processes are often used
- Backing and vacuum can be worth and/or necessary before and/or after the coating

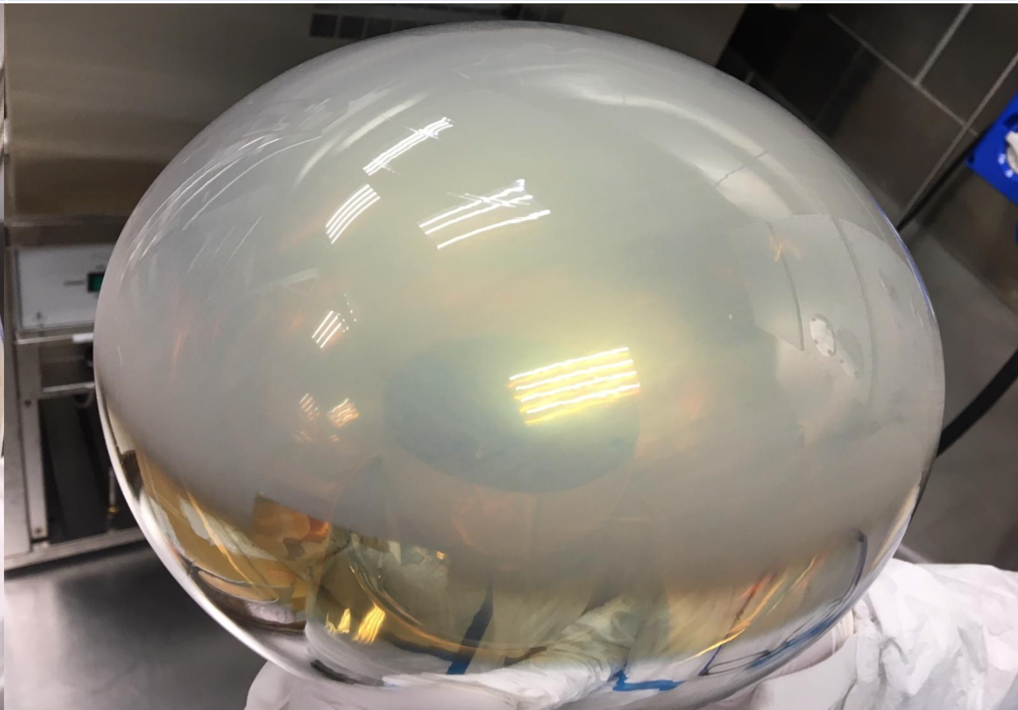




WLS Vacuum Deposition Procedure

Cleaning of the materials: example of PMTs

Cleaning Method		
Ultraclean wipe with Ethanol	Demi Water and Soap (Elma Clean 65) at 1% - Rinsing with demiwater - Towel changed and again new Demi Water and Soap (Elma Clean 65) at 1% - Rinsing with demiwater FC0011: directly in the vacuum oven at 40 C for about 18 hours	Demi Water and Soap (Elma Clean 65) at 1% - Rinsing with demiwater - Towel changed and again new Demi Water and Soap (Elma Clean 65) at 1% - Rinsing with demiwater FC0007: rough drying with a CR towel on the photocathode surface in some well defined directions and in the vacuum oven at 40 C for ~18 hrs

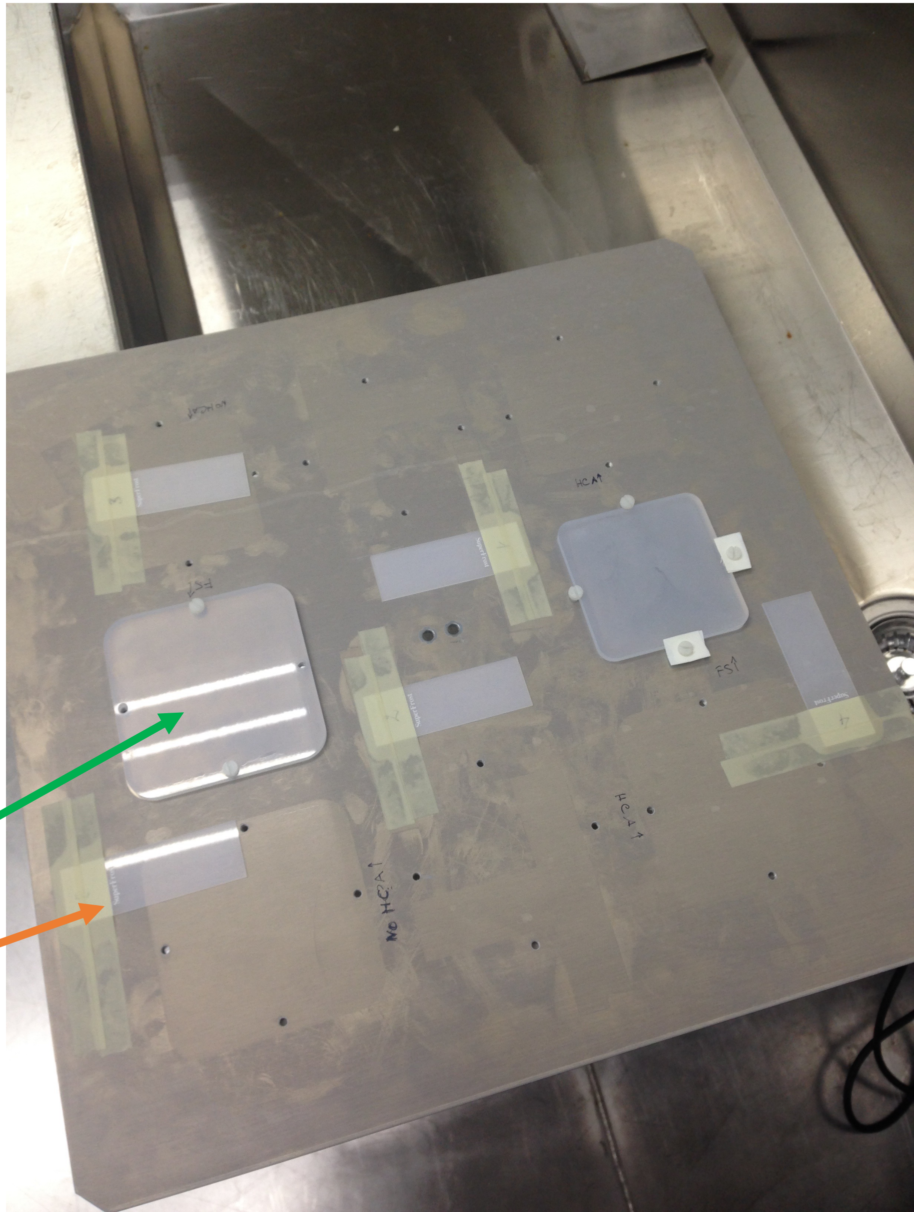




WLS Vacuum Deposition Results

Evaluation of the WLS coatings in terms of

- Visual inspection
- Weight/mass density
- Coating uniformity
- Characterization of optical properties (at room and low temperatures)
    - Reflectance
    - Absorbance
    - Transmittance
- Characterization of electrical properties (WLS are dielectric insulating materials)
- Characterization of mechanical properties (adherence)



Fused Silica Windows

Glass Samples

WLS Vacuum Deposition Results

Amount of the WLS mass density

- Coatings used in low temperarure environment
- WLS and substrate respond in different way to the shrinkage
- Define a mass density preserving:
- Temperature resistance and robustness
- Conversion properties and reflectivity/transparency
- Uniformity
- Mass density of 100 ÷ 1000 μg/cm^2 offers quite the same performance
  - Density$<$100 μg/cm^2 →less conversion
  - Density$>$1000 μg/cm^2 →less transparency
- Mass density of 200 ÷ 500 μg/cm^2 offers best performance
  - Density preserve the conversion and transparency
  - WLS diluted in the bulk of the noble liquid does not impact on the optical features

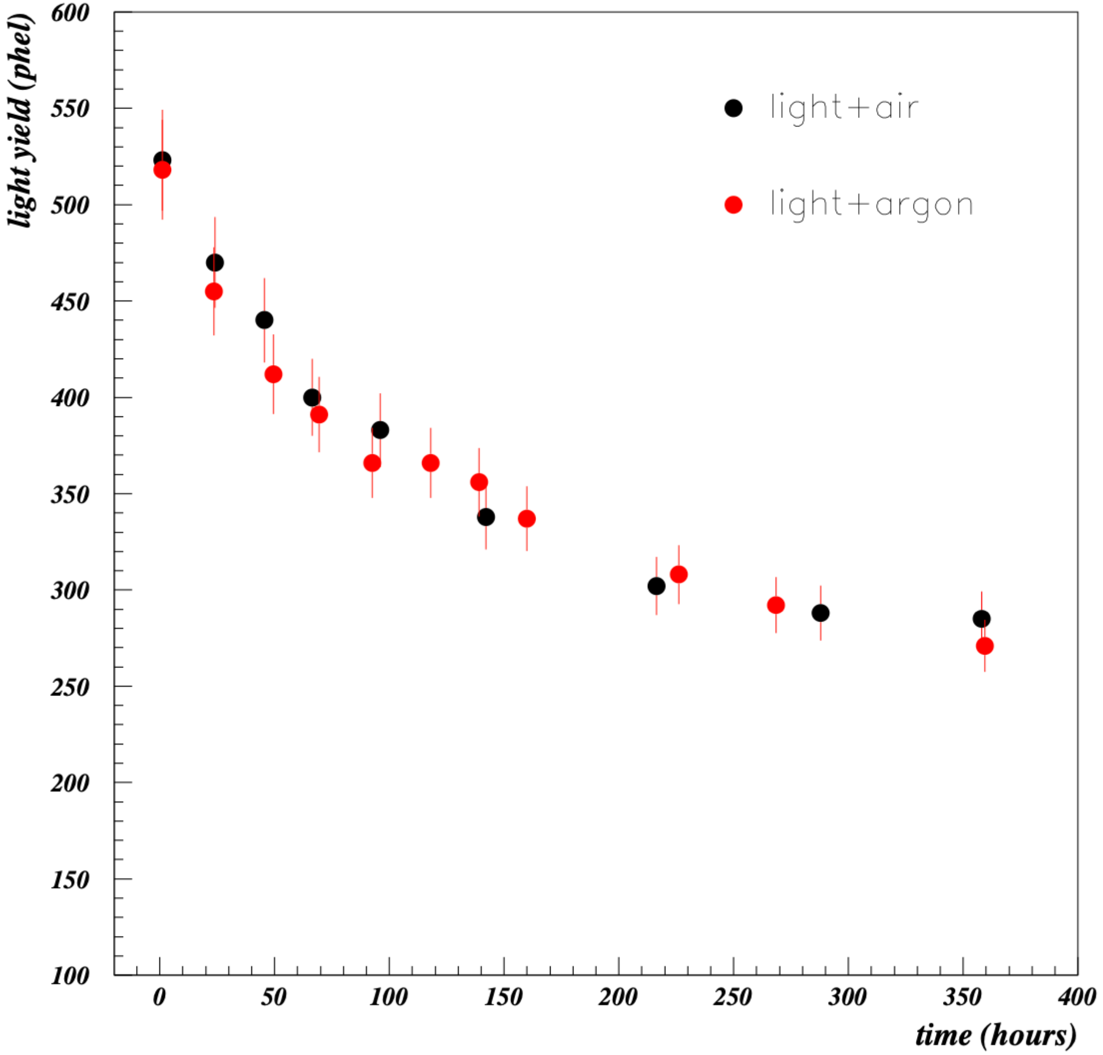
Factor Affecting WLS Deposition

Moisture and light have impact on the coated substrates

- TPB and p-TP degradate if exposed to environment with high humidity percentage
- High intensity UV light leads to a reduction of the yield (till 40%)
- Impurities can drive the degradation process
- Use of dry storage and/or inert atmosphere (mainly gaseosus nitrogen) mitigate the dicrease
- UV-blocking shields prevent the WLS degradation
- Pure materials/best scintillation grade WLS limit the performances loss

- Avoid to use different WLS in the same coating system without a deep proper cleaning

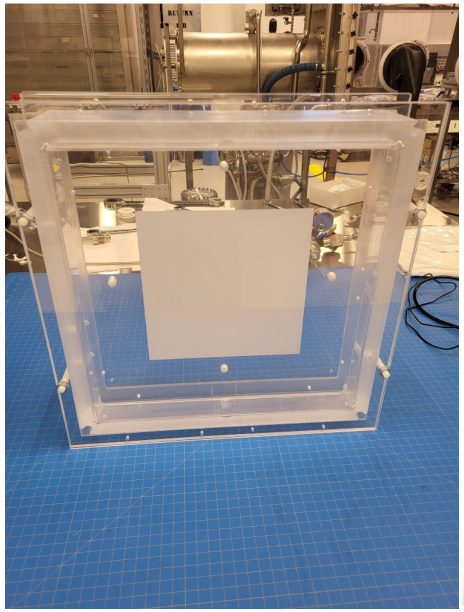
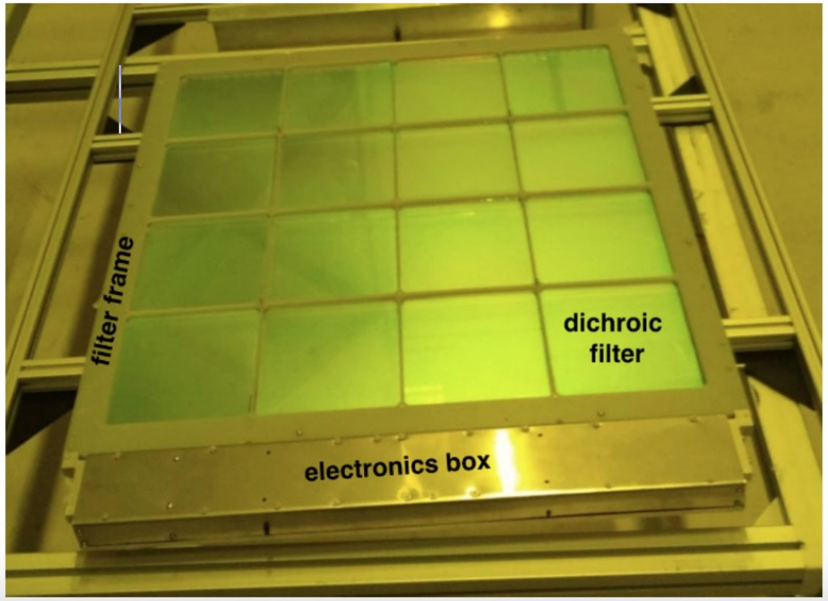
Case of TPB coated samples



Experiments using WLS Deposition

Noble liquids experiments using WLS coatings

- WLS coatings are mainly used by liquid argon experiment (VUV scintillation light at 128 nm)
  DarkSide-20k, DArT/ArDM, DEAP3600, DUNE, ICARUS, LEGEND, MicroBoone, SBND
- Xenon experiments also use WLS to improve the light efficiency collection
  NEXT
- Several R&D noble gases prototypes make use of WLS deposition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List on main references:

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Conclusions

- Large volume noble gases detectors are mainly focused on the dark matter search and neutrino physics
- Deep UV scintillation light from noble gases needs to be shifted to be detected by photosensors, namely photomultiplier tubes and silicon devices
- TetraPhenyl-Butadiene and p-TerPhenyl are the most common wavelength shifting materials, able to convert the scintillation light respectively to 420 and 350 nm
- These organic compounds can be deposited on both, the photosensors and the walls/parts of the detector containing the liquified noble gases
- Different techniques can be used, the most common is performed through vacuum deposition by using evaporation chamber equipped with crucibles
- Dedicated set-ups have been built and specific techniques have been adopted to produce wavelength-shifting coatings on highly reflecting material substrates and/or optical filters/windows
- Ad-hoc procedures have been developed for the production, handling and storage
- Proper characterization studies of the coatings in terms of uniformity, stability and optical properties have been explored
- Improvements on the systems and techniques are under study to further enhance the features of the deposition process and the performances of the coatings

Conclusions

- Hope to see you to the next LIDINE Conference for a possible futher discussion on this subject