Results of irradiation, Epoxy characterisation and some information

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CMS week March 15-19 1999

Results of gamma, neutron and proton irradiation (10 years of CMS running)

Glue	Averaged absorption length (cm)			
	before irrad.	**gamma** 4000 Gy	**neutron** 2 10^{13} n/cm^2	**proton** 2 10^{13} p/cm^2
Histo-mount	13 ± 4	9 ± 2	13 ± 4	**1.48±0.06**
NOA61	0.40±0.01	0.40±0.01	0.40±0.01	0.40±0.01
TSE3250	150±130	**43 ± 23**	110±80	110±80
x-38-406	120±100	**21 ± 8**	60±23	110±80

Notes:

1) The specimens consist of 1mm thick glue layer on 3mm thick fused silica substrate
2) The absorption length is calculated from the transmittance spectrum once the refractive indices of glue and substrate are known
3) The table reports the absorption length averaged on the PWO scintillation spectrum

For CMS a glue can be considered **“Radiation Hard”** if it does not cause the degradation of the light collection along 10 years of running.

The radiation hardness can be evaluated by means of the ratio "detected to emitted photons" before and after the irradiation in the simplified system:

PWO//glue//Si_3N_4(65nm)//Si

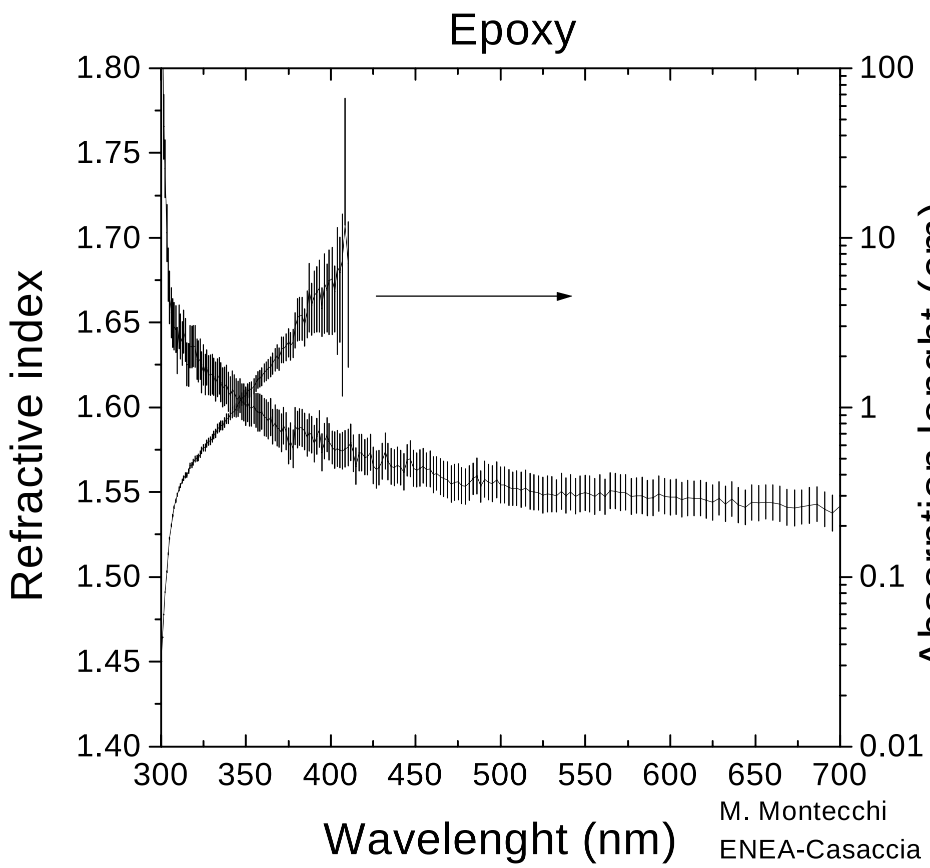
Before irradiation

Glue	Detected/Emitted (%)	
	0.3 mm	**1.0 mm**
Histomount	11.9	11.9
NOA61	11.2	10.9
TSE3250	9.4	9.4
x-38-406	10.2	10.2

After the irradiation these values are confirmed

⇒ all these glues can be considered "radiation hard" for CMS!

Complex refractive index and absorption length of EPOXY



n (430 nm) = 1.57 ± 0.01

<Λ> = 11 ± 6 cm

***Epoxy* has a refractive index greater than *Silicon Resin* (≈1.47 @430 nm) and is harder**

Determination of the glue complex refractive index from spectrophometric measurements

1) spectral transmittance (*T*) and reflectance (*R*) at normal incidence of both **bare substrate** and **glue(1mm)//SiO_2(3mm)**
   (P.E. λ9 equipped with a 15cm integrating sphere)

2) n_{sub} and k_{sub} are the solutions of

$$\begin{cases} T(n_{sub}, k_{sub}, d_{sub}, \lambda) = T_{\exp}(\lambda) \\ R(n_{sub}, k_{sub}, d_{sub}, \lambda) = R_{\exp}(\lambda) \end{cases}$$

   d_{sub} is measured with a caliper

3) n_{glue} and k_{glue} are the solutions of

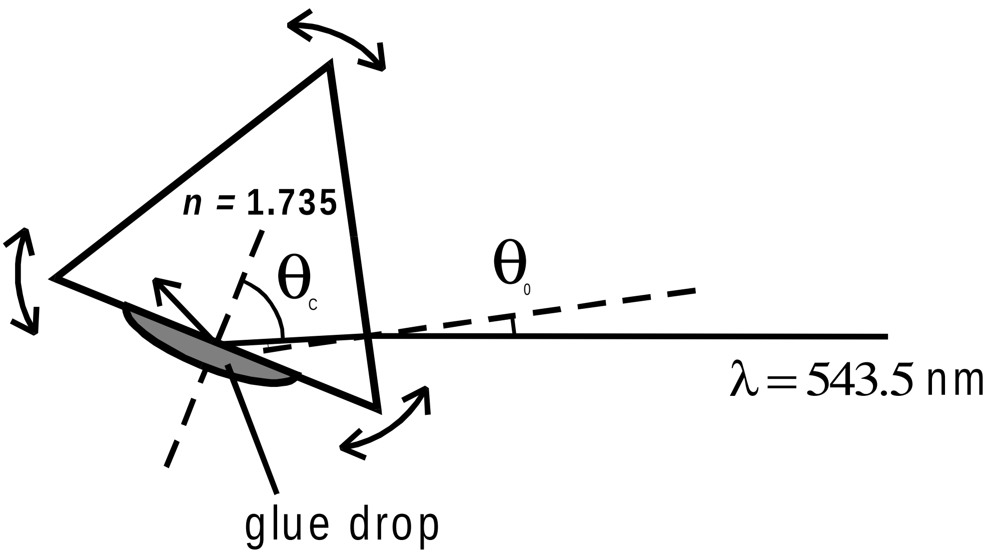
$$\begin{cases} T(n_{glue}, k_{glue}, d_{glue}, n_{sub}, k_{sub}, d_{sub}, \lambda) = T_{\exp}(\lambda) \\ R(n_{glue}, k_{glue}, d_{glue}, n_{sub}, k_{sub}, d_{sub}, \lambda) = R_{\exp}(\lambda) \end{cases}$$

   d_{glue} is measured with an optical microscope

4) the equation systems are solved with a very reliable program developed in ENEA-Casaccia

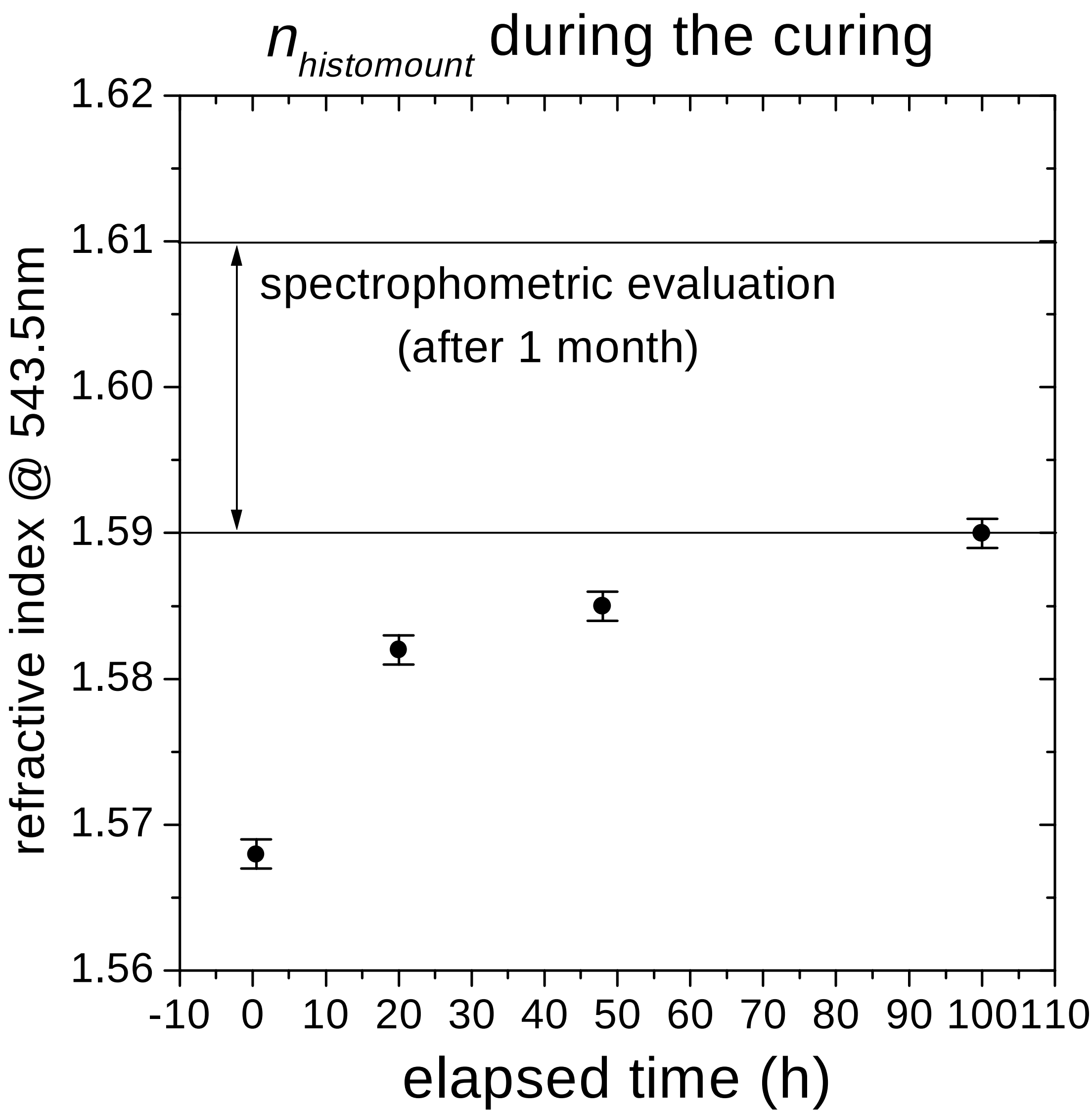
N.B.: as general rule: ***n↔R*** ***k↔T***

Glue refractive index from the critical angle



$$n_{glue} = n\, sin(60° + asin(sin\theta_0 / n))$$

- The light source is a green HeNe laser (λ=543.5nm).
- Equilateral prism of high refractive index glass; the prism refractive index (*n*) was determined from the angle of minimum deviation (before gluing).
- The prism is mounted on a goniometer-rotor.
- θ_0 is increased up to the extinction of the transmission in the glue.



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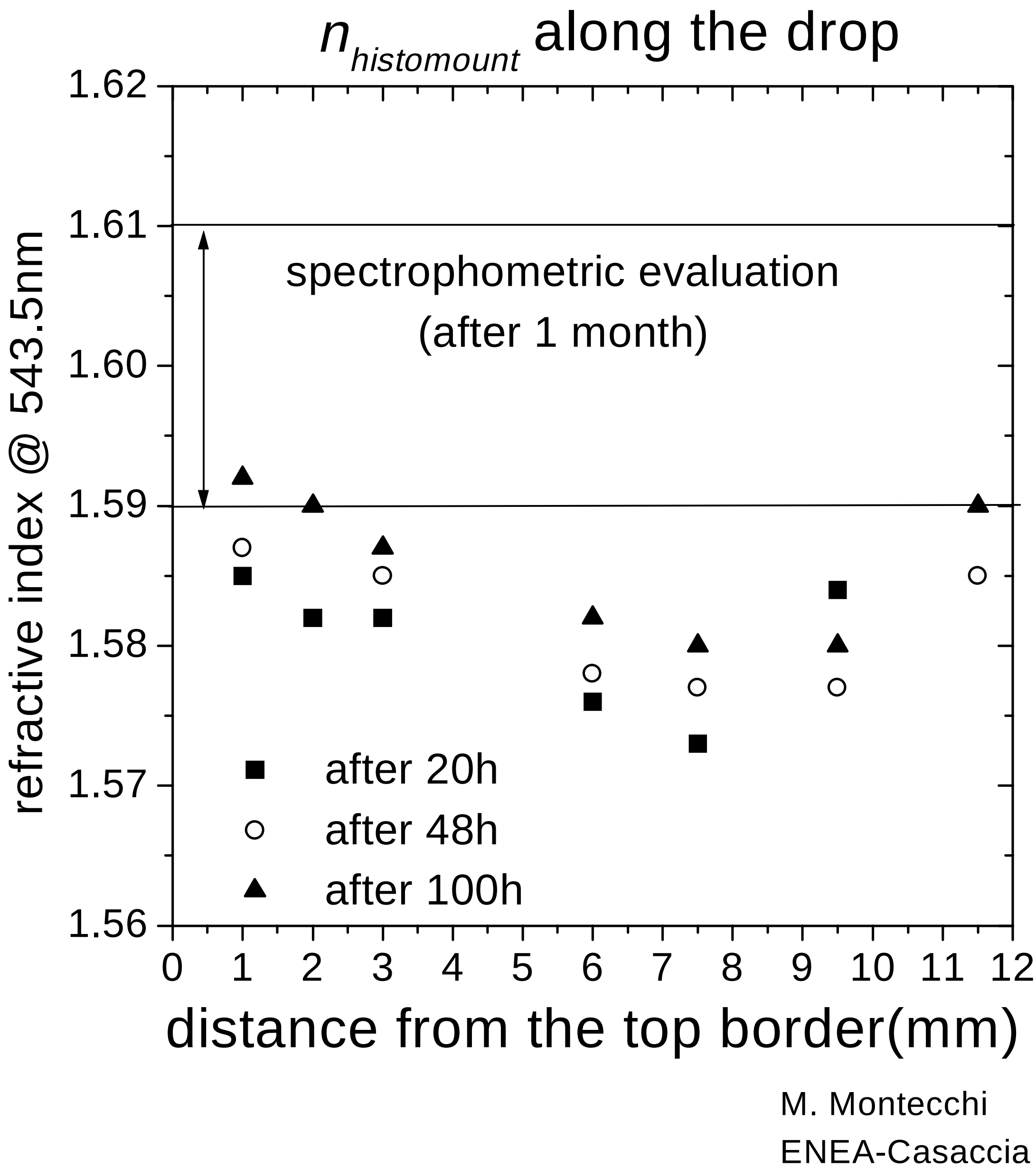
- $n$histomount increases during the curing
- good agreement between spectrophotometric and prism methods



- the curing speed depends on the glue thickness
- the curing can be monitored with the critical angle method also in the realistic situation

prism(PWO?)//glue//APD

Some news

1) National Diagnostic seems to be not interested to give us technical support.

2) *Histomount* shows a good mechanical adhesion on PBT (capsule).

3) *Histomount*, once well cured, is **very rigid** but also **very fragile!**

4) Thermal ageing is going on 2 specimen sets (80 and 110 °C), but there are serious problems with *Histomount*: **air bubbles** **cracks** and **disjunction** at the interface glue//substrate appear:

- **air bubbles**: probably *Histomount* is not well cured in the deep region, even 3 months later the layering, because of the "bottle-effect"
- **cracks**: shrinking?
- **disjunction**: probably are due to the different thermal dilatation between glass and *Histomount*

Open questions

1) **How takes the *Histomount* curing in the system PWO//glue//APD?**

Monitoring of the curing with the prism in the real condition (prism//glue//APD)

+ critical angle related to the APD readout

2) **If it takes a long period, how much change the APD readout?**

Simulation based on the monitored n_{glue}

3) **Does the *Histomount* solvent etch the APD window?**

Dip of APD window specimens in solvent
(The chemical analysis of *Histo.* is going on)

4) **Are air bubbles, cracks and disjunction (observed in thermal ageing test) representative of the ageing at RT?**

I will test **glass//glue(0.3mm)//glass** at temperature less than the solvent boiling point.

Gluing choice and next steps

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Considered glues for CMS (March ’99)

glue	manu-facturer	curing	*n* 430 nm ± 0.01	<Λ> (1) (cm)	D/E (%)	aging	on bare APD	used in
Histomount **(one part)**	National Diagnostic	12 h @ RT solvent	**1.63**	13±4	11.9	γ OK n OK p OK		**Monit. ’99** (2)
NOA 61 (one part)	Norland Optical Adhesive	UV curing 5mW/cm^2 (350-380nm) 10 min	**1.59**	0.40 ±0.01	11.2	γ OK n OK p OK		
Melmount 1.6 (one part)	Cargille	thermo-plastic liquid @ 70°C	**1.59**	9 ± 3	11.4	γ OK n OK p OK T~OK	OK	(2)
TSE 3250 (one part)	GE Bayer Silicones	4 h @ 100°C	1.47	150 ± 130	9.4	γ OK n OK p OK		
rtv 615 (two part)	GE Bayer Silicones	(24 h) 6-7d @ RT 1h@100°C	~ 1.4					Monit. ‘98 (3)
X-38-406 (two part)	Shin Etsu	4 h @ 100°C	1.53	120 ± 100	10.2	γ OK n OK p OK		
Epoxy (two part)	Shin Etsu	? @ 150°C **nitrogen**	**1.57**	11± 6			OK	Hama. PIN& APD (4)
Silicon resin	Shin Etsu		~1.47				OK	Hama. APD (5)

Notes:

1) <Λ> is the glue absorption length averaged on the PWO scintillation spectrum.
2) Melmount and Histomount were sent to Hamamatsu to be used as material for the protective window.
3) This glue was used in monitor98 as optical coupling medium; another glue (silicone) ensured the mechanical sealing. The signal observed with the electron beam was systematically decreasing, but at the present the cause is not definitively ascertained.
4) Used in new APDs and in some calibrated Hamamatsu PIN. Along several months Yuri Musienko observed the **degradation** of the quantum efficiency in the 350-500nm range of a new PIN protected with 0.2 mm epoxy layer. Is epoxy not able to prevent the Si degradation?

5) Used in old Hamamatsu APD.

Requirements for the use in CMS

1) $n_{\text{glue}} \geq n_{\text{APD\_window}}$

2) **Low absorption in PWO scintillation wavelength range**

3) **Good mechanical adhesion with PWO, APD and capsule.**

4) **Chemical compatibility with APD**

5) **No degradation of light collection and mechanical adhesion along 10 years of CMS running:**
   - radiation hard
   - negligible aging
   - no disjunction or air bubbles formation

Next steps

1) **Monitoring of the *Histomount* curing in a more realistic situation** (prism//glue//APD)

2) **Dip of *Epoxy* in *Histomount* solvent**

3) **Epoxy irradiation and results** (April?)

4) **Results of thermal aging** (April?)

5) **Cumulative accelerated aging (irradiation + thermal) of**
   - **PWO(slide)//glue//APD (with capsule)**
   - **Prism(PWO?)//glue//APD (with capsule)**

   (summer 99?)

6) **Monitoring ’99** (old APDs and capsules)

7) **Module #0** (new APDs and capsules): realistic test for gluing recipe, tools and *Histomount*

8) **LY monitoring along several months for PWO(full size)//glue//APD(with capsule)**