Outcome of glue tests

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Gluing task force

ECAL week May 3-7, 1999

Considered glues for CMS (May ’99)

glue	manu-facturer	Curing	*n* 430 nm ± 0.01	<Λ> (cm) (1)	D/E (%) (2)	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	OK? (7)	**Monit. ’99** (3)
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 T OK		
Melmount 1.6 **(one part)**	Cargille	Thermoplastic Liquid @ 70°C	**1.59**	9 ± 3	11.4	γ OK n OK p OK T~OK	OK	(3)
Epoxy **(two part)**	Shin Etsu	? @ 150°C **nitrogen**	**1.57**	11± 6	11.0	γ OK n OK p OK T OK	OK	Hama. PIN& APD (4)
Silicon resin	Shin Etsu		~1.47 (6) 1.420 @ 543.5nm				OK	Hama. APD (5)

Notes:

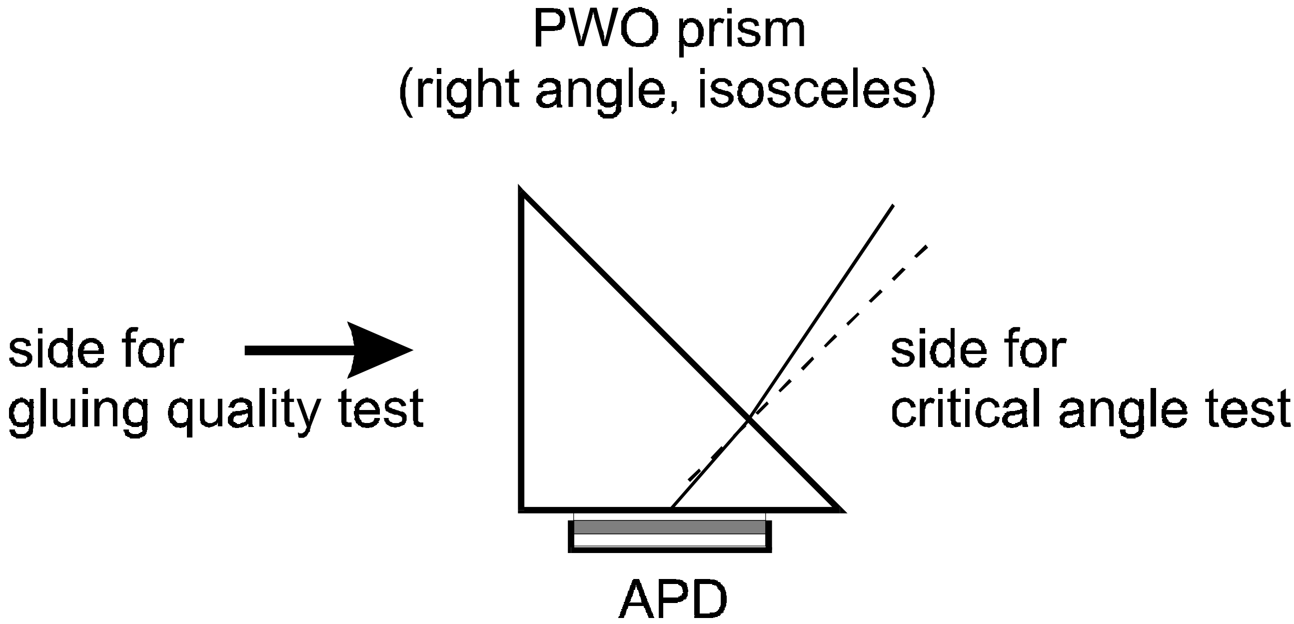
1) <Λ> is the glue absorption length averaged on the PWO scintillation spectrum.
2) Calculated for the simplified system PWO//glue(0.3mm)//Si_3N_4(65nm)//Si where PWO and Si are semi infinite media and the interposed materials infinitely extended; the reported value is the average on the PWO scintillation spectrum, the incidence angle and the polarisation.
3) Melmount and Histomount were sent to Hamamatsu to be used as material for the protective window in Sept. ‘98.
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 APDs.
6) *n*~1.47 was determined by the best fit of the APD spectral reflectance; in order to fit the APD reflectance by using the recent experimental value @ 543.5nm, the refractive index of the Si_3N_4 film should be slight lower (-0.05) than the one reported in literature.
7) Since April 13 1999, a bare APD glued with *Histomount* to a glass plate is working and its properties are unchanged.

News on glues at May 99

- *Histomount* directly purchased from Zymed shows lower *n* (1.50 @ 543.5 nm) than the one distributed from National Diagnostic (*n*~1.60).
- *Histomount* consists in vinil toluene and xylenes (solvent).
- Xylenes:
  a) etches the *Silicon Resin* window of the APD (after few days);
  b) temporary kills the APD (~1 day);
  c) softens *Epoxy* specimen.
- Since April 13 ‘99, a bare APD glued to a glass plate with *Histomount* is working and its properties are unchanged.
- In PWO//APD (no capsule) after 1 day the most of Xylenes is evaporated (monitoring with PWO prism).

- *Epoxy* is rad. hard for γ, neutrons and protons.
- *Epoxy* and *NOA61* are low degraded after 15 years @ RT.
- Some glues have been recently considered.

Tests with PWO prismatic specimens



a) Gluing quality tests: *bubble-viewer* and *bubble-meter*.

b) Measurements of the critical angle (total reflection) and the effective cut off angle (APD readout).

NB: n_{ord} @ 543.5 nm =

2.282 ± 0.003 by minimum deviation angle

2.26 ± 0.01 by spectrophotometry

PWO(prism)//*Histomount*//APD#237

a) Gluing quality tests:

• ***Bubble-viewer*:**

one small air bubble ∅<1mm

NB: air bubbles are between PWO and glue

• ***Bubble-meter*:**

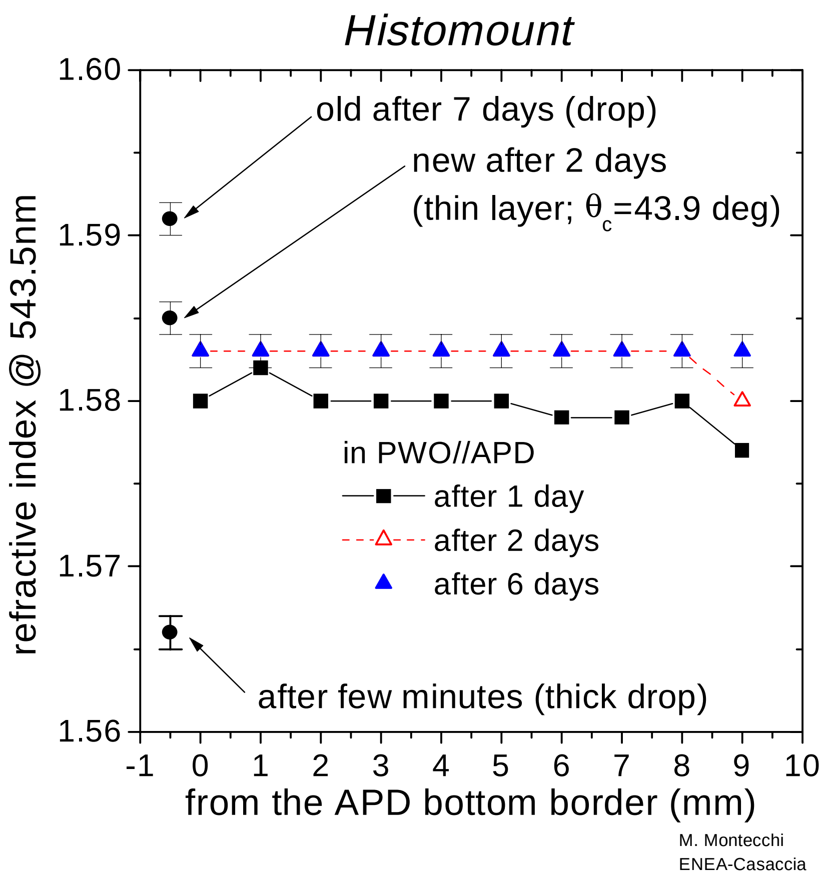
$$\eta_{EXP} = \mathbf{1.156 \pm 0.002}$$

from simulation:
η=1.1617 perfect
η=0.9799 air layer between PWO and glue

$$\Rightarrow \varnothing \sim 0.5 \text{ mm}$$

PWO(prism)//*Histomount*//APD#237

a)



⇒ the most of Xylenes is evaporated after 1 day!

b) Critical angle:

Due to the *silicon resin* window, the cut-off angle of the APD readout occurs at

$$\theta_C = 38.5 \text{ deg}$$

$$\Rightarrow n_{\text{Silicon Resin}} = 1.420 \text{ @ } 543.5 \text{ nm}$$

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

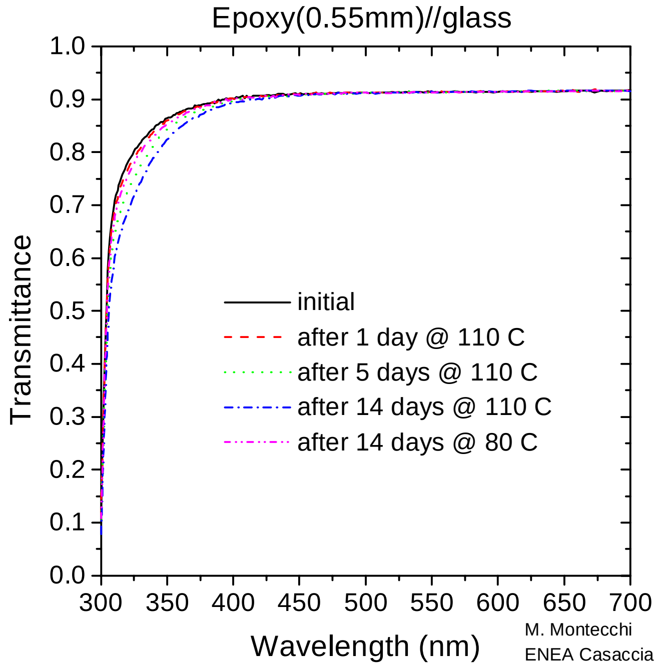
Glue	Averaged absorption length (cm)			
	before irrad.	**gamma** 4000 Gy	**neutron** $2\ 10^{13}$ n/cm^2	**proton** $2\ 10^{13}$ p/cm^2
Epoxy	11 ± 6	6 ± 2	11 ± 6	**1.68±0.11**
Histo-mount	13 ± 4	9 ± 2	13 ± 4	**1.48±0.06**
NOA 61	0.40±0.01	0.40±0.01	0.40±0.01	0.40±0.01

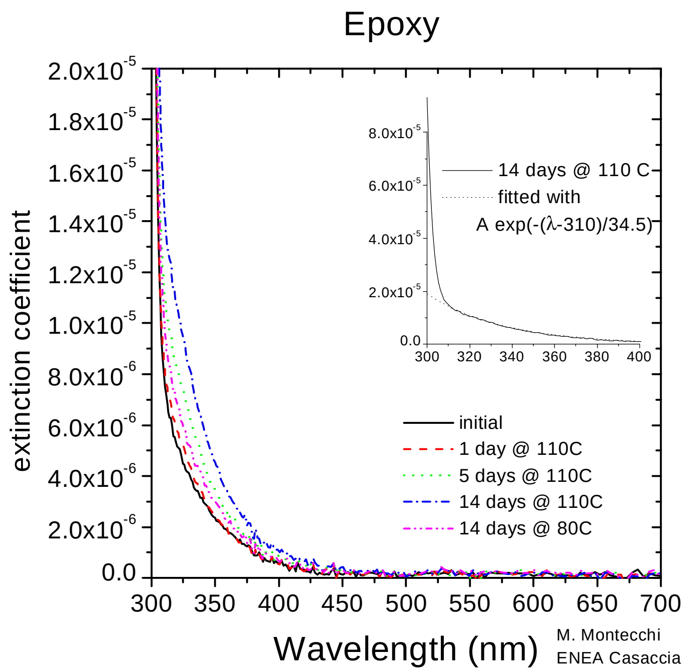
Notes:

1) The absorption length is calculated from the transmittance spectrum once the refractive indices of glue and substrate are known
2) 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, that is if the absorption length is much greater than the glue thickness: **$\Lambda >> 0.03$ cm**

Thermal ageing of *Epoxy*



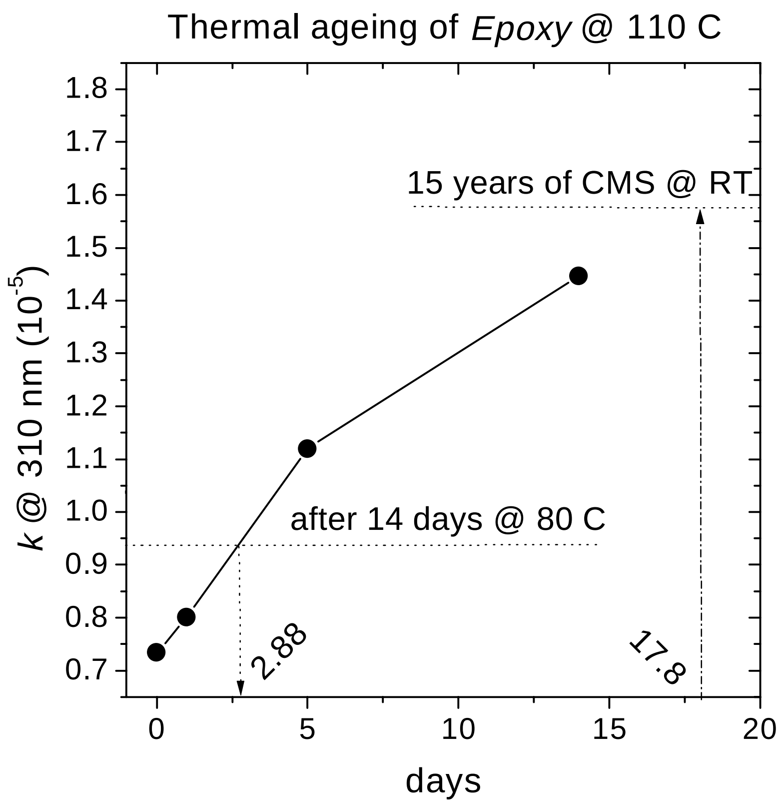


above 310 nm $k(\lambda)$ is well fitted with

$$k(\lambda) = k(310) \exp\{ -(\lambda-310)/34.5 \}$$

Condition	k(310nm)
initial	0.734 ± 0.003
14 days @ 80 C	0.948 ± 0.004
1 day @ 110 C	0.801 ± 0.003
5 days @ 110 C	1.120 ± 0.004
14 days @ 110 C	1.447 ± 0.004

... thermal ageing of *Epoxy*



assuming the Arrhenius low $\Delta t = C\exp(Ea/kT)$:

- **Ea/k = 7126 °K is obtained from**

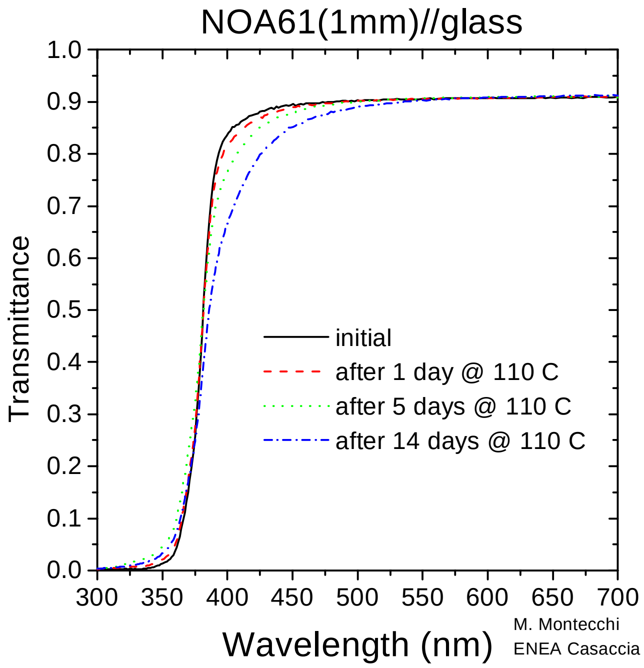
  14days @ 80 C ≡ 2.88days @ 110 C

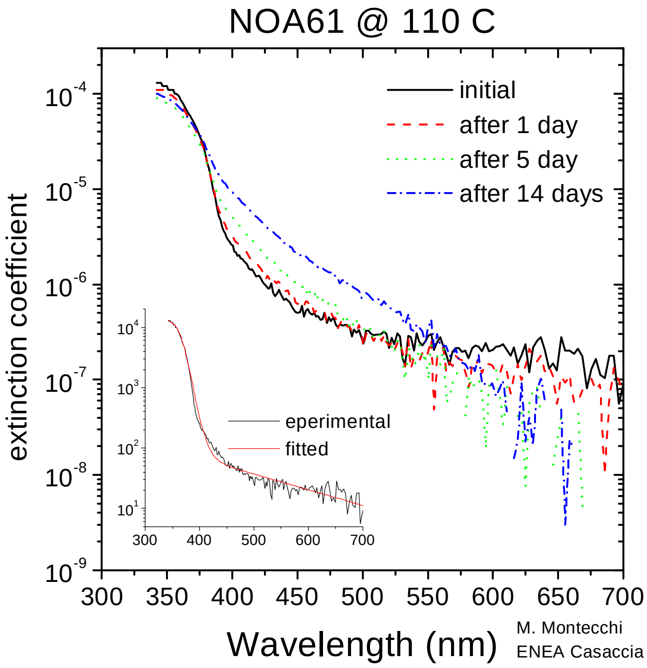
- **(ageing @ 110 C)/(ageing @ RT) = 303**
- **15 years @ RT ≡ 17.8d @ 110 C**
- **after 15 years @ RT**

  ***k*(310nm) ~ 1.6 10^{-5}**

  <Λ> : 11 ± 6 → 4.0 ± 0.7 cm

Thermal ageing of *NOA61*

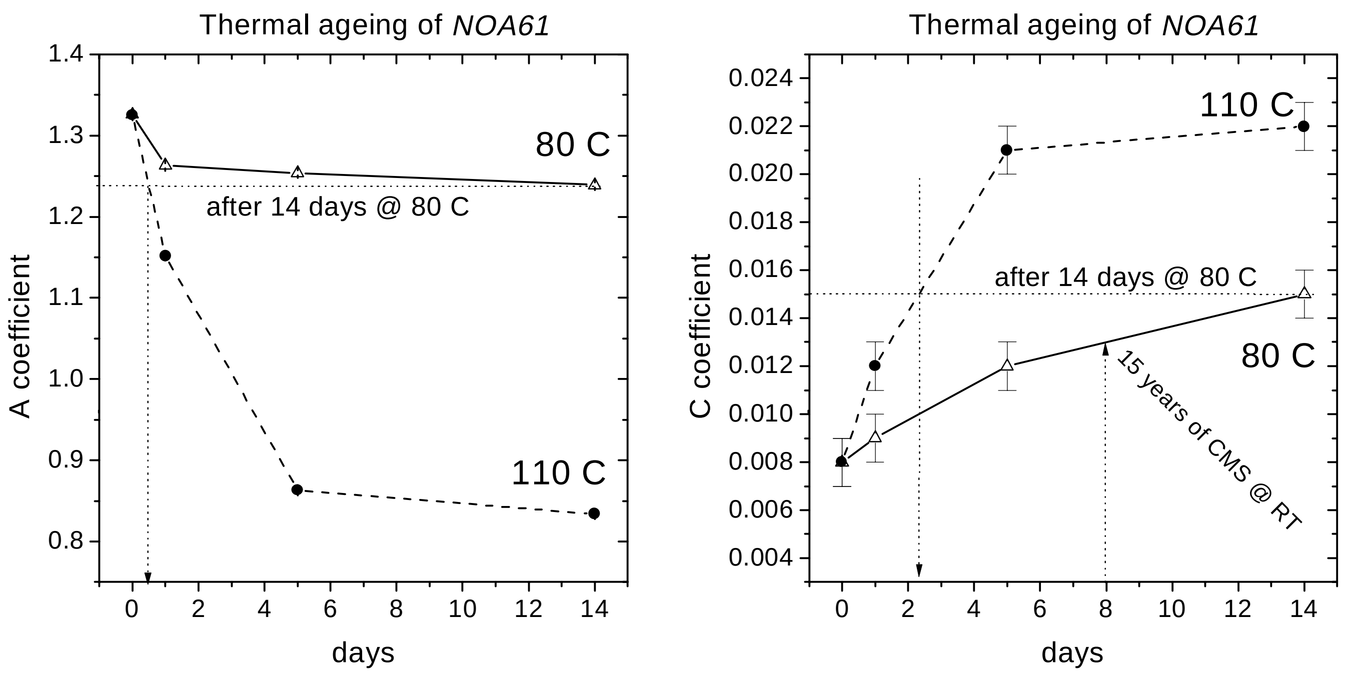




Two different ageing processes modify the transmittance spectrum: the first acts on the slope (+), the second on the knee (-); they are well described, respectively, by *A* and *C* coefficients of

$$k(\lambda) = \frac{A}{1+\left(\lambda/366\right)^{44}} + 0.00488\exp\{C(454-\lambda)\}$$

..... thermal ageing of *NOA61*



Assuming the Arrhenius low, from

A: 14days @ 80C ≡ 0.5day @ 110 C

C: 14days @ 80C ≡ 2.3days @ 110 C

⇒ $E_a(A)/k$ = 15000 K $E_a(C)/k$ = 8140 K

A: 15 years @ RT = 0.9 day @ 80 C

⇒ A = 1.269

C: 15 @ years @ RT = 8 days @ 80 C

⇒ C = 0.013

After 15 years @ RT:

- **<Λ> : 0.398 ± 0.7 → 0.407 ± 0.7 cm**
- **D/E = 11.3% for 0.03 cm of glue and does not change after proton irradiation**

Some glues recently examined

- **Shin Etsu:** KE103, 2-part, RT curing

  low refractive index (1.40@543.5nm)

- **Summer Optics:**

1) Lens Bond M62, 2-part, RT curing:

   looks brown in the bottle

2) Lens Bond UV 69, 1-part, UV curing:

   brown in the bottle + too high UV power

- **Epotek:**

1) 301: looks clear also after 2h@80C, λ_{CUTOFF} ~ 350nm
2) 301-2: brown after 1day@80C
3) 302: green, λ_{CUTOFF} ~ 420nm

N.B.: 2-part glues have to be mixed and show the same problems of *Histomount* (solvent, bubbles, long curing time for thick layer)

Next steps

- Continuation of the investigation on the *Histomount* purchased from National Diagnostic (monitoring of the curing in the real condition, compatibility with bare APD, thermal ageing, optimisation of the gluing recipe....).

- Add higher statistics testing (ageing, ageing + irradiation, survival of APD ageing,…)

- Deeper investigation of *Epotek* 301?