

Test for the Measurement of Diffusion Coefficient of Water in Kapton Foils for the Gem Detector of the Upgraded High-Pseudorapidity Muon Detection in CMS

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Abstract

GEM detectors are the subject of careful studies and discussions within the CERN scientific community, with a view to their possible installation on several experiments of LHC, including CMS. These chambers consist of three layers microholes of Kapton (50 μm) sheets, Copper-cladded on both surfaces (5 μm per coat), spaced a few millimeters between them.

A systematic study of the materials making up the detector is in progress in order to monitor the possible change of properties and behaviors of the chambers, as a result of interaction with process fluids and of radiations. This study is focused on the contact with ambient air and moisture, therefore we wanted to determine the value of the diffusion coefficient of water in the detector polyimide. The presence of this compound inside the detector sheets can determine a change of mechanical and electrical properties.

The determination of the diffusion coefficient will allow then to describe, later, a model of adsorption and diffusion within the entire sheet geometry GEM, gaining concentration profiles and the time required to saturate the system.

1 - Experimental Setup

Two specimens of kapton Apical were cut with size (15.3 x 10) cm² (Sample I) and (15.2 x 10) cm² (Sample II), both with a thickness of 50 μm. We used a model XP204 Analytical Balance, with a maximum capacity of 220 g, a minimum weight of 8 mg and a tolerance of 0.1 mg. A metal basket is located inside the scale, supplied with the instrument, which helps in the measures of weight.

Precision of the weight measurement was determined by performing a large number of readings in controlled environmental conditions, and found $\sigma = 0.2$ mg. After preparation in the oven for about 36 h at 110° C, measures were carried out, to get the measurement of the sample time zero: dry weights were (1087.3±0.1) mg (Sample I) and (1079.1±0.1) mg (Sample II).

The system to control the humidity (described in details in the Appendix) consists of an air cylinder connected, a bubbler (for having wet air) and two small vessels containing Silica Gel (for dry air); it is possible to regulate separately, in a manual way, both the bubbler and the vessels for the drying the air, so a desired value of RH is allowed. Inside the container a hygrometer with thermometer, for control of the conditions inside the system, and two small metal clamps were located; the clamps allowed to hold the samples in order to expose both sides of the larger gas flow controlled humidity. For each weight measurement, the sample was removed from its holder and placed inside the microgram balance without contact with nor base in order not to bias the measurement.

The times at which the measurements were made were as diluted, both to avoid contaminating samples, both to reduce the experimental errors, because over time the kinetics of diffusion suffers a sharp slowdown. Note also that, in order to compare data that were obtained, specially chosen to carry out the measurements at different time intervals for the two samples (e.g. the Sample I was initially monitored every 10 minutes, while the Sample II every 20 minutes).

Measurement were taken at 45-50% RH and (20±2)°C temperature, to reduce typical operational environment conditions. Humidity was kept constant in container during the time of measurement.

The experiment lasted a total of 10 hours: after this time it was possible to say with considerable confidence that now they had reached equilibrium conditions, no significant weight changes, noting the samples from at least a few hours.

2 - Results and Discussion

Measurement values for the two samples are listed in the following tables (Table 1 and 2); all graphs of correlations, fitting parameters and results for each sample are present (Figure 1-6) The correlation used to this geometry, taken from literature studies of Crank [1] (the thickness of the polymer film is $L = 2l$)

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2} \exp \left[\frac{-D\pi^2 t}{4l^2} \right] \quad (1)$$

where it is possible to obtain the diffusion coefficient by the slope of the regression line, having previously linearized in the form

$$\ln\left(1 - \frac{M_t}{M_\infty}\right) = \ln\left(\frac{8}{\pi^2}\right) - \frac{D\pi^2}{4l^2} t \quad (2)$$

The procedure shows an error of less than 0.1% and therefore negligible.

Conditioning: 36h at 110°C, RH=45-50% Sample I: 10X15.3X50E-4 cm								
W_polymer [mg]	ε	Time [s]	M(t) [mg]	M(t)/M(inf)	ε	ln(1-M(t)/M(inf))	ε	Y _{sper} -Y _{calc}
1087.3	1.88E-01	0	0	0.00	0.00	0.00	0.00	0.82
1091.7	1.89E-01	600	4.4	0.44	7.69E-05	-0.59	-1.02E-04	0.31
1093.2	1.89E-01	1200	5.9	0.60	1.03E-04	-0.91	-1.57E-04	0.06
1095.3	1.89E-01	1803	8	0.81	1.40E-04	-1.65	-2.86E-04	-0.61
1094.9	1.89E-01	2400	7.6	0.77	1.33E-04	-1.46	-2.53E-04	-0.35
1095.4	1.90E-01	3000	8.1	0.82	1.42E-04	-1.70	-2.95E-04	-0.52
1094.8	1.89E-01	3600	7.5	0.76	1.31E-04	-1.42	-2.45E-04	-0.16
1094.9	1.89E-01	4200	7.6	0.77	1.33E-04	-1.46	-2.53E-04	-0.13
1095.1	1.89E-01	4200	7.8	0.79	1.36E-04	-1.55	-2.68E-04	-0.22
1095.3	1.89E-01	4800	8	0.81	1.40E-04	-1.65	-2.86E-04	-0.25
1094.6	1.89E-01	5400	7.3	0.74	1.28E-04	-1.34	-2.31E-04	0.14
1095.4	1.90E-01	6600	8.1	0.82	1.42E-04	-1.70	-2.95E-04	-0.08
1095.4	1.90E-01	8105	8.1	0.82	1.42E-04	-1.70	-2.95E-04	0.10
1095	1.89E-01	9660	7.7	0.78	1.35E-04	-1.50	-2.60E-04	0.49
1096	1.90E-01	10800	8.7	0.88	1.52E-04	-2.11	-3.65E-04	0.02
1096.1	1.90E-01	13200	8.8	0.89	1.54E-04	-2.20	-3.80E-04	0.22
1095.9	1.90E-01	14400	8.6	0.87	1.50E-04	-2.03	-3.51E-04	0.53
1096.6	1.90E-01	18300	9.3	0.94	1.63E-04	-2.80	-4.85E-04	0.23
1097	1.90E-01	22140	9.7	0.98	1.70E-04	-3.90	-6.75E-04	-0.41
1097	1.90E-01	25200	9.7	0.98	1.70E-04	-3.90	-6.75E-04	-0.04
1097.1	1.90E-01	28800	9.8	0.99	1.71E-04	-4.60	-7.95E-04	-0.30
1097.1	1.90E-01	32400	9.8	0.99	1.71E-04	-4.60	-7.95E-04	0.14
1097.2	1.90E-01	36000	9.9	1.00	1.73E-04			

Table 1: Results of each measurement of weight for Sample I and mathematic manipulations for the following fitting. M(t) is calculated as difference between W_{polymer}(t) – W_{polymer}(t=0)

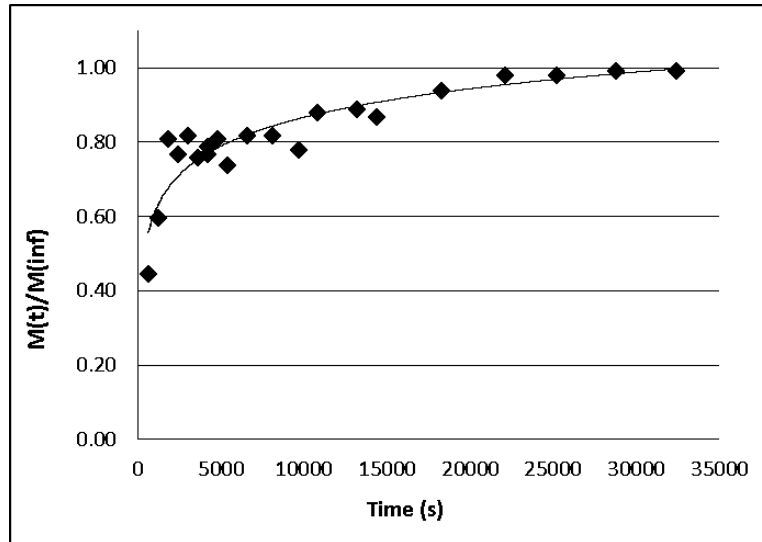


Figure 1: Trend for saturation of Sample I

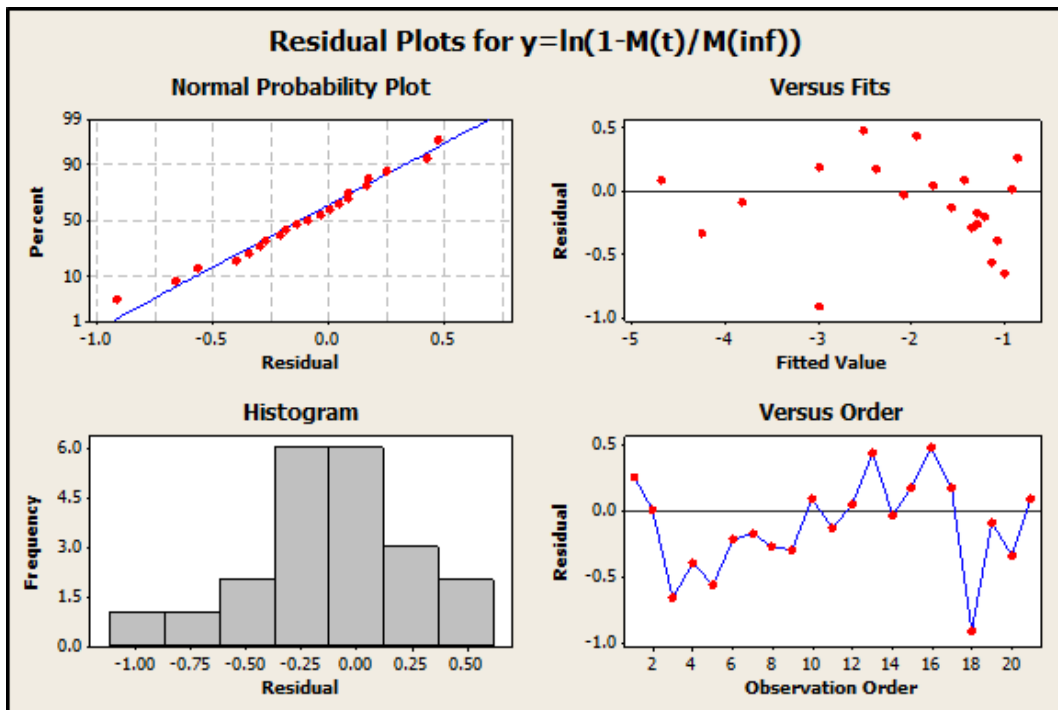


Figure 2: Fitting data for Sample I using (2)

	(3.06±0.34)E-10 cm²/s		Diffusion Coefficient		
	$y = a \cdot x + b$				
a	-1.21E-04		-7.70E-01		b
σ_a	1.32E-05		0.09		σ_b
R ²	0.81		1230.71		σ

Analysis of Variance						
Source	DF	SS	MS	F	P	
Regression	1	1.26E+08	1.26E+08	83.38	0	
Residual Error	19	2.88E+07	1.51E+06			
Total	20	1.55E+08				

Figure 3: Fitting parameters and Diffusion Coefficient for Sample I; DF is the degrees of freedom, SS the sums of squares, MS the mean squares, $F = (MS \text{ regression}) / (MS \text{ residual error})$ and P a parameter that must be smaller than 0.05

The parameters *a* and *b* are shown in (2)

Conditioning: 36h at 110°C, RH=45-50% Sample II: 10X15.2X50E-4 cm								
W_polymer [mg]	ϵ	Time [s]	M(t) [mg]	M(t)/M(inf)	ϵ	$\ln(1-M(t)/M(\text{inf}))$	ϵ	$Y_{\text{sper}} - Y_{\text{calc}}$
1079.1	1.87E-01	0	0	0.00	0.00	0.00	0.00	0.56
1085.4	1.88E-01	1220	6.3	0.40	6.94E-05	-0.51	-8.87E-05	0.13
1087.3	1.88E-01	2400	8.2	0.52	9.04E-05	-0.74	-1.28E-04	-0.01
1088.3	1.88E-01	3560	9.2	0.59	1.01E-04	-0.88	-1.53E-04	-0.08
1088.9	1.88E-01	4800	9.8	0.62	1.08E-04	-0.98	-1.69E-04	-0.09
1089.1	1.88E-01	6000	10	0.64	1.10E-04	-1.01	-1.75E-04	-0.04
1091	1.89E-01	6900	11.9	0.76	1.31E-04	-1.42	-2.45E-04	-0.39
1091.1	1.89E-01	9645	12	0.76	1.32E-04	-1.45	-2.50E-04	-0.22
1090.7	1.89E-01	12000	11.6	0.74	1.28E-04	-1.34	-2.32E-04	0.04
1091.6	1.89E-01	14400	12.5	0.80	1.38E-04	-1.59	-2.75E-04	-0.04
1093.1	1.89E-01	18360	14	0.89	1.54E-04	-2.22	-3.85E-04	-0.41
1093.5	1.89E-01	22260	14.4	0.92	1.59E-04	-2.49	-4.31E-04	-0.41
1093	1.89E-01	25210	13.9	0.89	1.53E-04	-2.17	-3.75E-04	0.12
1092.5	1.89E-01	29400	13.4	0.85	1.48E-04	-1.92	-3.32E-04	0.65
1094.8	1.89E-01	32400	15.7	1.00	1.73E-04			
1094.8	1.89E-01	36000	15.7	1.00	1.73E-04			

Table 2: Results of each measurement of weight for Sample II and mathematic manipulations for the following fitting. M(t) is calculated as difference between $W_{\text{polymer}}(t) - W_{\text{polymer}}(t=0)$

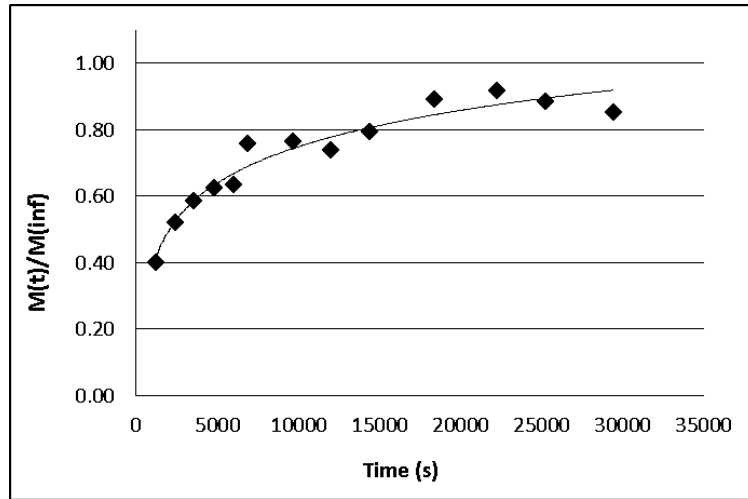


Figure 4: Trend for saturation of Sample II

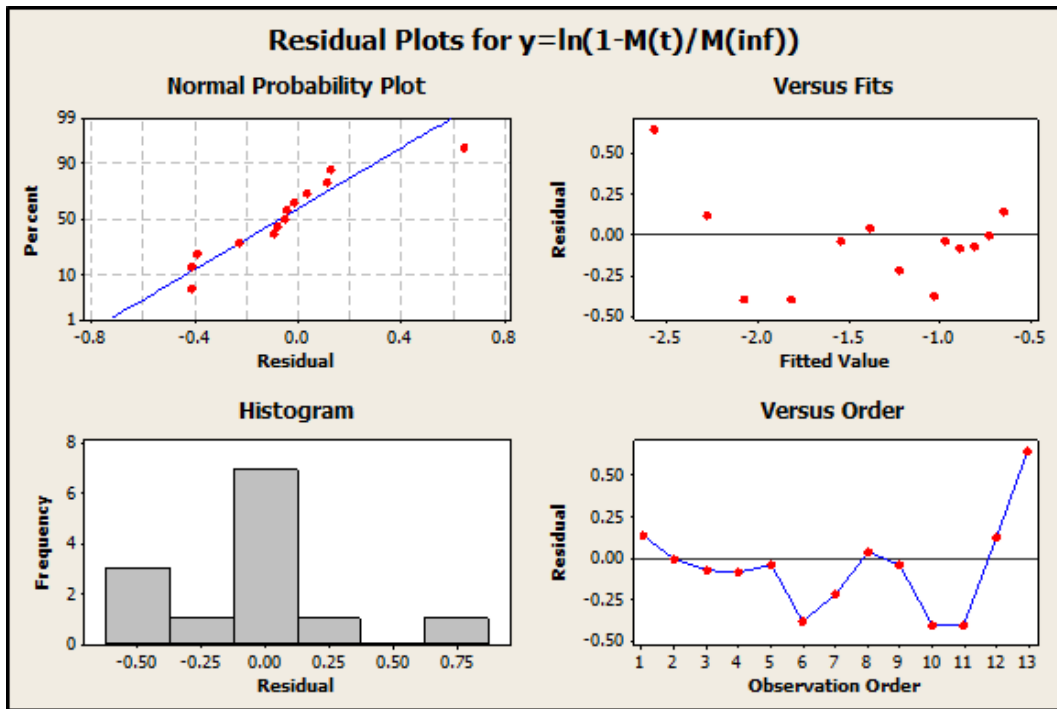


Figure 5: Fitting data for Sample II using (2)

	(1.73±0.21)E-10 cm²/s		Diffusion Coefficient		
	$y = a \cdot x + b$				
a	-6.84E-05		-5.61E-01		b
σ_a	8.34E-06		0.56		σ_b
R ²	85.90		1048.11		σ

Analysis of Variance						
Source	DF	SS	MS	F	P	
Regression	1	7.39E+07	7.39E+07	67.27	0	
Residual Error	11	1.21E+07	1.10E+06			
Total	12	8.60E+07				

Figure 6: Fitting parameters and Diffusion Coefficient for Sample II; DF is the degrees of freedom, SS the sums of squares, MS the mean squares, F= (MS regression) / (MS residual error) and P a parameter that must be smaller than 0.05

The parameters *a* and *b* are shown in (2)

We found, for sample I and sample II respectively

$$D_I = (3.06 \pm 0.34) 10^{-10} \text{ cm}^2/\text{s} \quad (3)$$

$$D_{II} = (1.73 \pm 0.21) 10^{-10} \text{ cm}^2/\text{s} \quad (4)$$

and average

$$D = (2.11 \pm 0.18) 10^{-10} \text{ cm}^2/\text{s} \quad (5)$$

It is reported that, during the test, there have been problems with the Sample II that, during the measurement n. 7, at the time of their removal from the metal clamp, was ripped up from Center along the larger side: despite the attention times after stopping the champion, to avoid touching this piece the walls of the container or to fold onto itself, the measurements were suffering from certain error, as shown by the regression statistics of the points later.

Despite this, the data obtained and the value of the diffusion coefficient are as expected from tests with the same purpose but conducted under different conditions or with different modes (usually by indirect parameters, such as the electrical properties), found in literature.

3- Conclusion

This article has presented a preliminary study on diffusion phenomena within the GEM [2]. In particular, it was determined the diffusion coefficient of water inside the polyimide.

Contrary to what was previously done by other authors [3-5], has tried to proceed with measures of weight, in accordance with ASTM standard directives [6] on the water adsorption polymeric materials. The results are $D_I = (3.06 \pm 0.34) 10^{-10} \text{ cm}^2/\text{s}$ for the Sample I and $D_{II} = (1.73 \pm 0.21) 10^{-10} \text{ cm}^2/\text{s}$ for Sample II, yielding to average $D = (2.11 \pm 0.18) 10^{-10} \text{ cm}^2/\text{s}$. The magnitude of both values is consistent with other evidence (indirect) reported in the literature [3-5].

Errors that were introduced in the measures arise from frequent disruption of the system as a result of weight measures which were to be conducted. In fact, every time that happened, the sample had to be removed from container to controlled humidity, not always negligible effects on weight measurement that you obtained. In an effort to reduce the errors, the samples were all weighed once. Analysis conducted earlier on the same samples showed a standard deviation of 0.19 mg for each measurement. It must however kept in mind that the systematic error that is committed on the measurement is certainly greater than the statistical error.

We will continue with the study and experimentation, in order to obtain results that are more precise and less affected by errors, especially systematic, recreating the same experimental setup with continuous weighing mode, so you do not have to move the sample from its environment.

4- Reference

- [1] J. Crank, *The Mathematics of Diffusion*, Second Edition, Clarendon Press, England (1975)
- [2] R. Bouclier, M. Capeans, W. Dominik, M. Hoch, J-C. Labbé, G. Million, L. Ropelewski, F. Sauli and A. Sharma, *The Gas Electron Multiplier (GEM)*, IEEE Trans. Nucl. Sci., Vol. 44, n. 3, pp.646 -650 (1997)
- [3] G. Rabilloud, *High-performance Polymers: Chemistry and Applications, Volume 3*, Editions Technip (2000)
- [4] Jongchul Seo, Haksoo Han, *Water sorption behaviour of polyimide thin films with various internal linkages in the dianhydride component*, in *Polymer Degradation and Stability* 77, pp. 477-482 (2002)
- [5] H. Han, H. Chung, C.C. Gryte, T.J. Shin, M. Ree, *Effects of Precursor Origins on Water Sorption Behaviors of Various Aromatic Polyimides in Thin Films*, *Polymer* 40, pp. 2681-2685 (1999)
- [6] ASTM International, D 570-98, Standar Test Method for Water Absorption of Plastics