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Ageing tests and recovery procedures of silica aerogel

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ABSTRACT

Silica aerogel has been extensively used in RICH detectors for the identification of charged particles over the momentum range between 1 and 10 GeV/c. Tiles of hygroscopic aerogel with large transverse dimensions ($20 \times 20\,cm^2$) and refractive index $n = 1.03$ have recently been produced for use in the LHCb experiment, allowing pion–kaon identification up to 10 GeV/c. The tiles have excellent optical properties (clarity factor better than $0.006\,\mu m^4/cm$ and homogeneity $\sigma(n-1)/(n-1) \sim 1\%$ within the tile). Extensive R&D tests on aerogel samples have been performed. Samples have been exposed to intense irradiation (proton, neutron and gamma), to humid air, to standard black varnish (used to paint the inner surface of RICH detectors), and to C_4F_{10} and CO_2 gases. The optical properties of the aerogel have been monitored during these tests and, when required, recovery procedures have been investigated and applied. In particular, regeneration of the tiles has been realized through exposure to dry atmosphere (gaseous N_2) or through baking for several hours at 500 °C. The measurements demonstrate that the optical properties have been successfully restored to their values at the production stage, and in no case permanent degradation has been observed.

1. Introduction

Particle identification over the momentum range between 1 and 60 GeV/c will be performed in the LHCb experiment [1] using the RICH 1 detector [2,3]. RICH 1 is equipped with two Cherenkov radiators, solid silica aerogel and gaseous C_4F_{10}. The aerogel samples have been produced by the Boreskov Institute of Catalysis (Novosibirsk).

Silica aerogel is a solid material made of SiO_2 with a very low density. Due to its transparency and its refractive index which is tunable within a wide range to match the physical requirements, it is an appealing material for RICH detectors. The refractive index is tuned during production by calibrating the density.

Photon scattering within the aerogel block limits the performance of this material as a Cherenkov radiator. The dominant contribution comes from the Rayleigh scattering mechanism with a cross-section proportional to λ^{-4}, where λ is the photon wavelength. The transmittance $T(\lambda)$ is parameterized by

$$T(\lambda) = Ae^{-C \cdot t/\lambda^4} \tag{1}$$

where t is the thickness of the block and A is a constant for the material, which is wavelength-independent. The clarity factor C is used to specify the optical quality of the sample.

The RICH 1 solid Cherenkov radiator consists of a wall of 50 mm thick hygroscopic aerogel tiles. The aerogel refractive index n is 1.030, with exceptionally good clarity, typically $C = 0.0050\,\mu m^4/cm$ or better. The LHCb tiles have the largest transverse size ever produced, up to $200 \times 200\,mm^2$.

2. Ageing effects and long-term stability

Due to its position inside the detector, the aerogel radiator will be exposed to a significant particle flux, up to 3.5×10^{12} particles/cm^2/year. Possible ageing of aerogel due to intense irradiation has been investigated [4]. Aerogel tiles have been exposed to very intense γ radiation from a ^{60}Co source (dose up to ~230 kGy corresponding to about 30 years of data taking) and to proton and neutron high intensity beams (fluence up to 5.5×10^{13} particles/cm^2). The transmittance has been monitored, studying the clarity factor as a function of the increasing dose of irradiation. No detectable degradation of the optical parameters was observed for γ and proton irradiation. However, a small worsening of the clarity due to neutron irradiation has been observed. For a fluence corresponding to the LHCb lifetime, the clarity factor in this case increases by about 5%. However, this is not a concern for the particle identification performance.

The behaviour of hygroscopic aerogel when exposed to humid air has also been explored [4]. Here a modification of the aerogel optical properties is expected. The tests revealed that a prolonged exposure to humid air changes the optical properties of the

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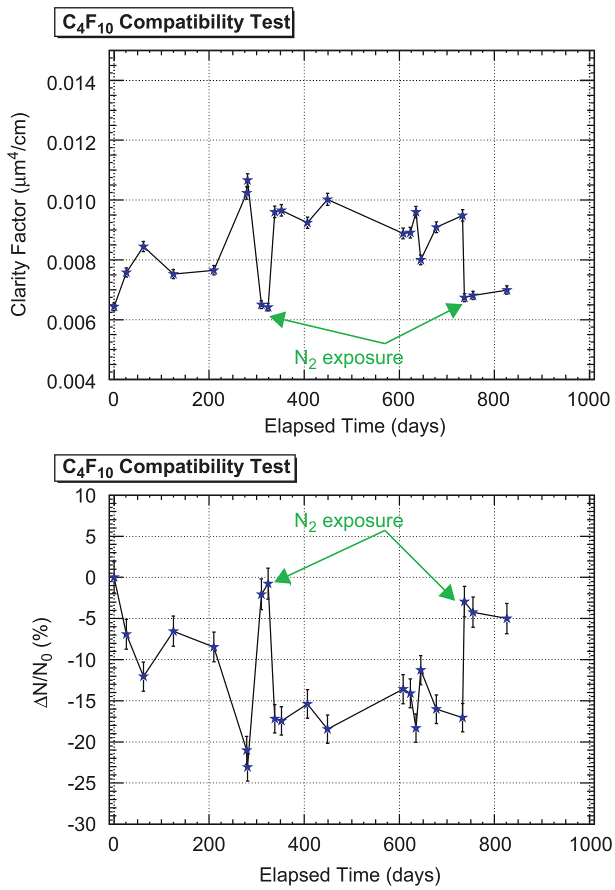

Fig. 1. Variations of the clarity factor (top) and photoelectron yield (bottom) as a function of time during the C_4F_{10} gas exposure test.

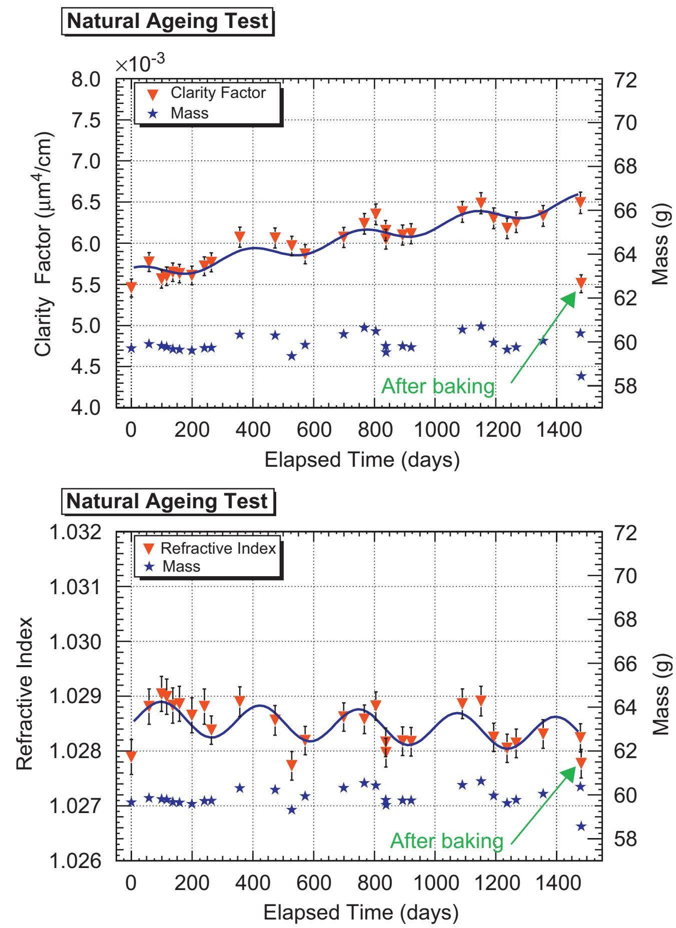

Fig. 2. Variations of the clarity factor (top) and refractive index (bottom) as a function of time in the natural ageing test. The refractive index is measured at $\lambda = 543.50$ nm. The results of the fit to Eq. (2) are superimposed.

aerogel, but the clarity can be completely restored by baking the exposed sample at high temperature. The integration of the aerogel into the RICH 1 detector will therefore be done with special care to prevent humidity absorption.

Inside the RICH 1 detector volume, the aerogel radiator will be in contact with the gaseous C_4F_{10} radiator. Generally, air fills the porous structure of the aerogel solid, and a replacement of air with C_4F_{10} is expected. Possible effects have been investigated. Several tiles have been stored in a C_4F_{10}-filled box with the clarity factor being periodically monitored to evaluate possible effects due to the gas. The behaviour of C and the relative variation of the number of photoelectrons $\Delta\mathcal{N}/\mathcal{N}_0$ expected in LHCb as a function of elapsed time to C_4F_{10} exposure are shown in Fig. 1. The number of photoelectrons $\mathcal{N}$ is analytically evaluated by integrating the Cherenkov spectrum convoluted with the transmittance of the aerogel which is clarity-dependent and considering all the others factors such as quartz window transparency, mirror reflectivities, photon detector (HPD) quantum efficiency and geometrical active area coefficient.

As shown in Fig. 1, the clarity coefficient rises with C_4F_{10} exposure time, with a corresponding decrease in the photoelectron yield. Following this rise, between day 281 and 310, the tile was kept in a dry N_2 atmosphere to check the possibility of restoring the aerogel to its initial optical conditions. The N_2 regeneration demonstrates that there is no permanent degradation of the clarity. On resumption of the C_4F_{10} exposure, it can be seen that the clarity again degrades, before reaching stability. The N_2 regeneration procedure is then repeated successfully at the end of the test.

Natural ageing variations have been studied by monitoring a tile kept in a humidity-controlled laboratory. The typical relative humidity of the air within the lab was in the range 15–35%. Results are reported in Fig. 2. During four years of monitoring, the clarity variation is ~4% per year. The fluctuations of n and C can be ascribed to uncontrolled absorption of water vapour or rejection of humidity previously absorbed, depending on the relative humidity level of the lab during measurements. The seasonal modulation is well described by the function:

$$y(t) = (p_0 t + p_1) + p_2 \cdot \sin(p_3 t + p_4) \quad (2)$$

where $y(t)$ can be either the clarity factor or the refractive index. The p_3 parameter is the frequency of the oscillatory pattern measured during the test. For the clarity factor, the period of the oscillatory pattern is $T = 2\pi/p_3 = (368 \pm 26)$ days, in good agreement with the seasonal hypothesis. The initial optical quality of this sample has been restored by baking.

3. Integration of aerogel in the detector

The RICH 1 detector is constructed around a gas- and light-tight vessel in which both the solid and the gaseous radiators are placed. Fig. 3 shows the internal part of the gas enclosure during a trial installation of the aerogel support mechanics.

Fig. 3. Trial installation of the aerogel support mechanics into the RICH 1 detector.

The inside of the enclosure is painted with black varnish[2] to avoid unwanted reflections of Cherenkov photons. The aerogel is exposed to possible residual outgassing of the varnish inside the vessel.

A compatibility test of the aerogel with varnish vapours was performed by monitoring both the refractive index and the aerogel transmittance as a function of time. An aerogel tile was kept in a box which has been painted black inside. To speed up the test, the aerogel was in contact with the painted surfaces and the box was locked with screws. This set-up corresponds to a more hostile environment than will be experienced in the final RICH 1 detector.

Fig. 4 shows the behaviour of the clarity factor and the refractive index as a function of exposure time to the black varnish. There is a variation of the clarity factor of the order of 70% over 425 days. The refractive index experiences a 0.1% variation, which is negligible.

At the end of the test, the tile was baked for several hours at 500 °C, and the clarity factor and the refractive index were re-measured. A typical temperature cycle used in the recovery process is shown in Fig. 5. The values of C and n are compatible with the ones measured at the beginning of the test, hence no permanent degradation has been detected.

Since the aerogel will at no time be in contact with the varnish inside RICH 1, and also there will be a break of over 12 months before the aerogel is installed into the gas enclosure which will allow time for the varnish to outgas, a more realistic compatibility test was performed. In this test the aerogel block was not in contact with the varnish, and a realistic period for the varnish to outgas was allowed to elapse. After ~200 days of exposure, a variation of 0.1% on n and of 20% on C has been observed; these numbers are compatible with the expected variation from natural ageing. No additional degradation due to the varnish has been detected.

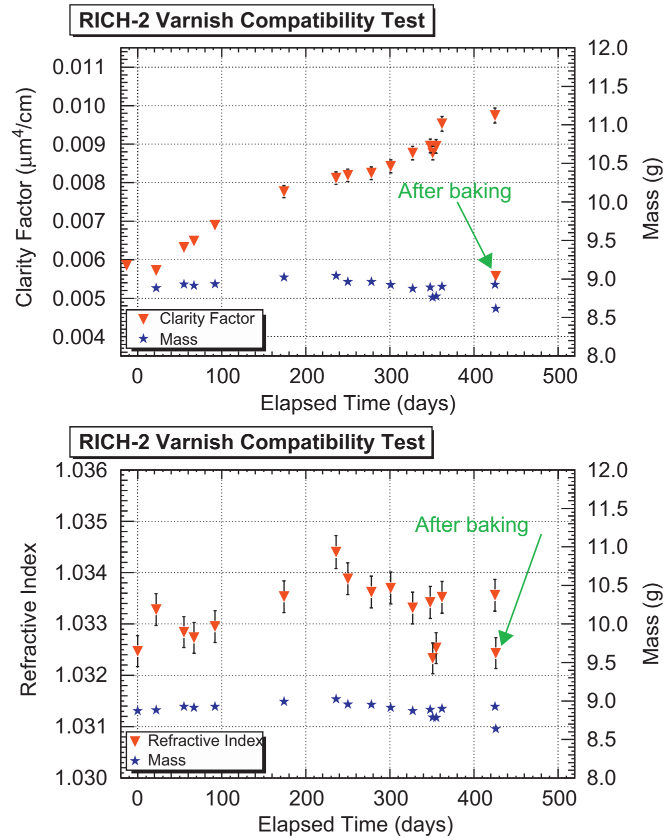

Fig. 4. The variation of clarity factor (top) and refractive index (bottom) as a function of time during the black varnish compatibility test. The refractive index has been measured at $\lambda = 543.50$ nm. During this test, the aerogel block was in contact with the black varnish.

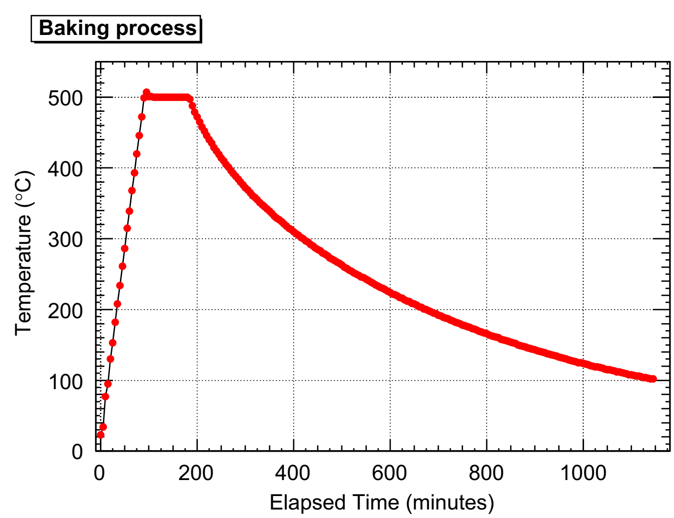

Fig. 5. Example of a temperature cycle during the recovery procedure used for one of the tested aerogel tiles.

4. Compatibility of aerogel with CO_2 gas

During any scheduled LHC shutdown, the RICH 1 gas enclosure will be filled with CO_2 instead of C_4F_{10}. This is both to conserve the fluorocarbon gas radiator during periods of inactivity and to preserve a dry atmosphere inside the gas enclosure itself. A compatibility test of silica aerogel with CO_2 has therefore been performed in which an aerogel block was kept for several weeks in a gas-tight CO_2-filled box. As expected, no visible variations were detected.

[2] The varnish is a "JalPrim Fond Adherent, Noir Mate", produced by Jallut S.A. Peintures.

5. Conclusions

Extensive ageing and robustness tests have been performed on silica aerogel to assess its compatibility with the LHCb RICH 1 environment. All relevant optical parameters have been monitored. The radiation hardness of the aerogel and its compatibility with C_4F_{10} and black varnish have been proven. No permanent degradation of the excellent optical properties has been detected in any of the performed tests. Recovery procedures such as exposure to dry atmosphere (gaseous N_2) or baking at 500 °C have shown that the original quality of the exposed samples have been restored.

The production of all the 16 tiles required for the LHCb experiment is now complete and the tiles are ready to be installed in the LHCb RICH 1 detector.

Acknowledgements

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