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AGEING OF MICROSTRIP GAS CHAMBERS:
PROBLEMS AND SOLUTIONS

R. Bouclier, M. Capeáns, C. Garabatos, G. Manzin, G. Million,
L. Ropelewski, F. Sauli, L. Shekhtman, K. Silander,
and T. Ropelewski-Temmel

ABSTRACT

The experimental set-up and the procedures used for studying the long-term behaviour of micro-strip gas chambers under sustained irradiation are described in detail. The most significant measurements on ageing obtained in a variety of conditions are reported, and a tentative interpretation of the results is presented. The relevance of these findings for the conception, construction and use of MSGCs trackers in high luminosity LHC detectors is discussed.

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1. INTRODUCTION

Most measurements with micro-strip gas chambers (MSGCs) exposed to high radiation fluxes show a gradual decrease of gain, and in many cases a fast degradation in performances, attributed to the formation in the avalanches of polymers and their deposit on the strips and the substrate [1-10], causing modifications of the electric field and affecting the operation. The diversity of results obtained by various groups, and the difficulty in consistently achieving long lifetimes is a major concern in view of the use of the devices, and the question of whether or not several thousand MSGC modules could be made to successfully withstand the harsh operating conditions of the proposed LHC experiments [11-13] has yet to be fully answered.

The doses to which detectors will be exposed in the LHC environment have been computed by Monte-Carlo simulations. Conservative estimates of the charged particle flux in the CMS tracking detector for the MSGCs closer to the beam pipe provide a value of $\sim 2 \cdot 10^4 \text{ mm}^{-2}\text{s}^{-1}$ at the full luminosity of about $10^{34} \text{ cm}^{-2}\text{s}^{-1}$. Taking into account the fraction of low momentum particles and their angular spread and mainly their curling inside the tracker due to the presence of a solenoidal magnetic field, the equivalent flux of minimum ionizing particles perpendicular to the detectors is $\sim 4 \cdot 10^4 \text{ mm}^{-2}\text{s}^{-1}$. For an avalanche size of 10^5 electrons, the corresponding current density is 0.5 nA mm^{-2} , an accumulated charge of $\sim 10 \text{ mC}$ per cm of strip for each effective year of LHC operation (10^7 s). Actual doses may be larger, due to neutron- and gamma-induced backgrounds.

Accelerated ageing tests are extensively being carried on by many groups in order to find a set of conditions that would permit MSGCs to survive ten years of continuous operation ($100\text{--}150 \text{ mC cm}^{-1}$) without significant deterioration in performances. There are nowadays enough results and sufficient knowledge to be able to ascertain, among a wide number of parameters, the main conditions for MSGCs to withstand such large doses of radiation in laboratory conditions. However, even if good results have been obtained in small and well controlled set-ups, a further step is necessary aimed at obtaining similar performances in larger and more complex installations. Presumably, this will only be possible if the fundamental processes leading to ageing, and the ways to avoid it, are better understood interpreting the results of an intensive dedicated experimental research program.

We provide in this paper a summary of the present status of our experience in this respect. Originally born under the code-name RD-10 [14, 15], the generic ageing study of gaseous detectors was merged with the more specific research on MSGCs RD-28 [16], and conducted within the Gas Detectors Development (GDD) group at CERN in collaboration with other institutes. After a detailed description of the experimental set-up and methods of measurement, we present and discuss the most significant results obtained with MSGCs operated at high fluxes. The last sections contain a brief summary of these results, and our tentative conclusions on the controversial issue of ageing.

2. GENERALITIES ON THE AGEING PROCESSES

Ageing, a permanent degradation of operating characteristics under sustained irradiation, is a well known problem met using gaseous detectors. Years of research in connection with the development and use of multi-wire proportional chambers have led to some understanding of the processes leading to ageing of gaseous detectors [17-20]. The detectors' lifetime appears to depend critically on the nature and purity of the gas mixture, on the materials used in the chamber assembly and in the gas system, on the nature of the electrodes and on the electric field strength at their surface.

Two basic mechanisms lead to wire chamber ageing:

- formation, in the avalanche plasma, of polymers issued from ions and radicals of the main gas filling and/or polluting molecules, producing deposits on the electrode surfaces. The polymerization rate can be largely enhanced by catalyzing agents, if present in the gas flow as pollutants: plasticizers used in flexible tubing are a well known example;

- direct deposition on the electrodes of heavy molecules released by materials in contact with the gas, possibly helped by the presence of strong electric fields in the detector. Residual vapour pressure in oil bubblers used in the exhaust pipes is a sample case.

Etching, a concurrent process removing surface layers, may also take place due to reactive species produced in the avalanche plasma in presence of specific molecules: carbon tetra-fluoride is particularly efficient in this respect, and has been used in wire chambers to prevent ageing processes and even to remove deposits produced by previous exposures to radiation [21, 22]. Some experience of using CF_4 mixtures in MSGCs exists [5, 7, 23-25]. Due to the etching properties of the avalanches on the electrodes, it remains to be proved however that such mixtures can be used for long-term operation in MSGCs without affecting the thin metal electrodes themselves; the observed heavy production of long-lived electro-negative molecules in CF_4 under strong irradiation [26] casts also doubts on the feasibility of large systems with moderate gas flow and serial circulation.

While the detailed mechanisms of ageing are quite complex, the effects on proportional gas detectors are straightforward: the thin layers of insulating material building up on anodes interfere with the electric field, are easily charged up under avalanche conditions and produce a rate-dependent decrease of proportional gain. Deposits on cathodes can also induce discharges by secondary electron emission (or Malter effect [27, 28]). An increasing leakage current and a decreasing detection efficiency in the irradiated areas are the clear signs of ageing; the process is usually irreversible and leads easily to fatal breakdown. All conditions being equal, ageing depends from the amount of ions produced in the avalanches and a natural scale factor for the process is the amount of accumulated charge per unit length of wire. Other factors, such as the rate of charge production and the level of gas flow have also been found to affect the

rate of ageing for a given device.

Suitably choosing the operating conditions, acceptable long-term performance in MWPCs has been achieved, up to a collected charge of several $C\ cm^{-1}$ without significant gain degradation. The ability to counter the effect of pollutants in the gas with additives such as water, alcohol, methylal and other products has also been reported, often with conflicting results (see for example Ref. [17]). Small deviations from optimal conditions (for example, pollutants released in the gas flow by sealants used in the construction of the detector or by the gas mixing and distribution system) have been found to degrade the long-term behaviour thus resulting in substantially shorter lifetimes. Micro-strip chambers appeared from the very beginning to be even more susceptible to age than their wire counterpart. One can identify some aspects specific to MSGCs that can be expected to affect their response to radiation;

- the conductor area of the strips is typically an order of magnitude smaller as compared to wires: the same amount of deposits is bound to induce a larger local modification of the electric field. Moreover, thin strips can be severely and permanently damaged by reactive species and micro-discharges induced by local charging-up processes;

- polymers or pollutants, if present in the avalanche plasma, may deposit on the insulating surface between strips and affect the electrical properties of the substrate, in a region where fields are critically high;

- the higher energy density in the avalanche plasma, a consequence of the small anode-to-cathode distance, could result in an increased efficiency of polymer production.

The first two issues may explain the shorter lifetimes observed in MSGCs as compared to MWPCs, given the rate of polymerization. Deposition of layers on the strips and on the substrate has been often observed in aged plates, and is considered responsible for the appearance of micro-discharges in the irradiated area causing irreversible damages to both anode and cathode strips (Fig. 1). The effect of the higher energy density in the avalanches is more difficult to assess. The available power in gaseous detectors is orders of magnitude higher than in standard RF cavity discharges [29], but one cannot induce from this if the polymerization rate should be larger in gaseous detectors or, on the contrary, lower due to saturation of the polymerization processes.

From the arguments presented in this general discussion one can expect ageing, if qualitatively inherent for a given set of operating conditions, to induce faster and more severe damages in micro-strip detectors than in wire chambers. Most experimental results support this statement.

It should be mentioned here also the frustrating outcome of our efforts of surface analysis by various methods of the deposits found in damaged structures; much as in the similar work done for wire chambers, the expected presence of carbon and hydrogen complexes, together with unexpected species such as silicon proved so far not very useful to help understanding the ageing mechanisms.

3. EXPERIMENTAL SET-UPS TO STUDY AGEING OF GAS DETECTORS

3.1 General layout

Three independent test stations have been built in our laboratories to implement systematic long-term irradiation studies with MSGCs. Each station includes a clean gas mixing and distribution bench, an X-ray generator, and a data acquisition and monitoring system allowing to record the detector performance as well as various physical and ambient parameters. Data from the detector under study (pulse heights, counting rate, current on electrodes) can be continuously recorded, together with those of a reference proportional counter monitor and of several ambient meters; normalization procedures, to be described later, are used to single out permanent gain variations (if any) from ambient-induced shifts. For a well behaving, non-ageing detector, one expects gain variations not exceeding a few percent over several months, and a good stability of the system is therefore mandatory. One of the set-ups, originally built for the detector research and development project RD-10 [15], includes also a sophisticated gas analysis system in order to make qualitative and quantitative gas purity measurements.

3.2 Gas mixing and distribution system

Micro-strip chambers, much as the wider family of gaseous detectors, can be operated with a wide choice of gas fillings. As suggested already by early measurements [30, 31], the purity of the gas is an essential requirement to obtain acceptable lifetimes of MSGCs under irradiation, with even more stringent requirement than for multi-wire chambers. Outgassing from materials used in the gas system appeared to substantially affect the results; standard gas mixing racks, universally used at CERN for multi-wire chambers, were found inadequate. We have developed and built several clean gas systems in order to provide suitable gas mixtures; two designs are schematically shown in Fig. 2. The most sophisticated (referred to as RD-10) includes a gas analysis line, while the simpler ones (RD-28, also used for the test beam runs) only allow controlled mixing and distribution of the gas. Entirely realized with stainless steel tubing, they were assembled avoiding as much as possible pollution sources (rubber joints, plastic tubing, etc.), and include mass flow metres and active filters to remove oxygen (and to some extent water), and micro-pore filters to stop oils and particulates. Table 1 provides a list of the major components used in the two systems.

In order to avoid pollution from residual oil vapours, bubblers have been avoided altogether and the exhaust is realized with a long line vented directly to the atmosphere. Installing a fixed, long drain pipe (24 m stainless steel, 4 mm in diameter plus 4.5 m copper, 2 mm in diameter) we have measured with the gas chromatograph (see later) the amount of air pollution due to back diffusion, as a function of gas flow (Fig. 3). For the range of flow used in our detectors (50 to 100 $\text{cm}^3 \text{ min}^{-1}$) the back diffusion is negligible, but increases substantially at lower

fluxes. In our system, the presence of the long exhaust pipe creates an over-pressure at the MSGC of about 20 Torr, a constraint that has to be taken into proper account in the design of the detectors [32].

As apparent in the table, the RD-10 system has been realized generally with higher grade elements; a large fraction (though not all) of the tubing and components could also be baked under inert gas flow with the help of electrical heating ribbons. The system also includes several additional facilities. A temperature-controlled stainless steel or glass vessel could be used to add small controlled amounts of vapours in the gas mixture to study the impact (if any) to the ageing process. A stainless steel container, the so-called outgassing box, placed upstream of the detector under test, allows to introduce in the gas flow samples of materials, candidate components for the construction of chambers, allowing systematic studies of the effects of outgassing on the detector lifetime.

In most of our tests we have used dimethyl ether (DME) as one of the components of the gas mixture. Because of the chemical activity of the vapour (a solvent), special care had to be taken in the choice of non-metallic parts in the DME line, and in particular of the sealing joints that could both swell and outgas under the action of the vapour; this subject will be discussed extensively later. A survey of the tolerance of various materials to exposure to DME was published several years ago [33].

3.3 Gas analysis

An essential component of the RD-10 set-up is a powerful analysis station that allows to sample the gas as it flows out of the test chamber, or directly from bottles through an independent line; it permits continuous monitoring of the quality of the gas mixture. The gas chromatograph (GC)¹ consists essentially of an oven containing one or more diffusion columns through which a sample of the substance to be analysed flows, together with a high purity, low mass carrier gas. A detector connected in series with the GC column is used to reveal and identify the various molecular component of the sample. In the stand-by mode, the sample gas flows through a loop in the GC that has the same impedance as the separating column. When a data acquisition cycle is started, a pneumatic valve allows the gas to flow into the column, where different species in the sample are separated by their characteristic retention times, while the oven temperature, after a given time offset, increases according to a pre-selected rate.

The chromatograph is equipped with two associated heads, a mass spectrometer detector (MSD)² and an electron capture detector (ECD)³. The system runs under control of a work station which allows the user to set up the conditions of a gas analysis, to acquire the data and to help in the analysis. The extended data base available for the MSD helps qualitative identification of the

¹ HEWLETT-PACKARD HP 5890 Series II, modified for gas analysis.

² HP 5971 Series

³ HP G1223A

species; quantitative analysis is possible if a specific calibration is performed. Both detectors can be optimized to be more sensitive to a certain range of compounds: the choice of the diffusion column (length, thickness and phase thickness), the temperature ramp and the run time selection are crucial. In its present configuration, the analysis system has certain limitations. While the presence of extra peaks in the spectra is a sign of the presence of pollutants, their absence is not sufficient evidence that the gas mixture is clean. The MSD, optimized for the detection of light hydrocarbons, is not very sensitive to heavier molecules, and the ECD is sensitive only to electronegative compounds. Complementary to the direct ageing measurement on detectors, the gas analysis is not by itself sufficient to guarantee the issue.

For the MSD the sample is carried by high purity helium. We have used two capillary columns: one optimized to identify light hydrocarbons⁴, and the other able to better separate species with higher molecular weight⁵; the choice was motivated by the fact that our ageing tests are done mostly under Ar-DME mixtures, and hydrocarbons are expected as pollutants in the DME cylinders. The analyzer, driven by the GC/MSD interface, contains under vacuum a cylindrical 70 eV electron impact ion source, ionizing and fragmenting the molecules of the sample. The interface also contains a hyperbolic quadrupole mass filter to sort the ions according to their mass-to-charge ratio (M/Z) by means of a combined DC and RF signal applied to its segments. A continuous dynode electron multiplier receives the ions that have passed through the mass filter and generates the corresponding electronic signal. Even if the GC did not separate substances such as nitrogen, water or methane, the MSD would detect their characteristic M/Z spectra, which will appear together with the spectra of other (low mass) molecules.

During the ageing tests with mixtures containing DME, some impurities can be expected to be present in the bottles; among others, small traces of freons. Their detection is difficult with the MSD, because the expected quantity is very small (<ppm); the ECD is more appropriate having higher sensitivity to electronegative substances. For quantitative analysis, a specific calibration should be made analysing known samples.

Abundant literature exists reporting possible influence of carbon halogens (freons) in the ageing process [33-36]; the high sensitivity of the ECD detector to electronegative compounds make it a good choice to spot the presence of freons as pollutants in the DME, even at a fraction of parts per billion (ppb) levels. A capillary column is used, with helium as carrier gas; a make-up gas (nitrogen with purity 99.9996%) is added to ensure a sufficient gas flow in the detector, and is bombarded in a cell by electrons emitted by a 15 mCurie ⁶³Ni source, producing a swarm of secondary electrons with thermal energies. The current induced by the free electrons is kept constant adjusting the rate of short-term voltage pulses

⁴ HEWLETT-PACKARD HP PLOT 19091P-AL5

⁵ HEWLETT-PACKARD HP PONA 19091S-001

to the cell electrodes; electronegative molecules, if present in the sample under analysis, capture some of the electrons and are revealed by an increasing frequency of the pulses. There is no information other than the presence of a signal along the retention time (abundance versus retention time); qualitative and quantitative calibrations are needed for identification. We have calibrated the detector for air, water, carbon dioxide, DME, freon 11 (F-11, CCl_3F) and freon 12 (F-12, CCl_2F_2).

Fig. 4 shows the ECD spectrum of pure argon with 10 ppm of F-11 and 10 ppm of F-12 added in a calibrated sample⁶. The first peak corresponds to F-12, and the second to F-11; identification is possible because F-11, having one more chlorine atom, is more electronegative and saturates the detector. A quantitative calibration then correlates the area of the peak with the known amount of pollutants. The spectrum provided by the MSD for the same mixture is shown in Fig. 5: above, the chromatography of the mixture and an expanded view of it, and below the mass over charge spectrum for both freons. It is worth noting the different response, in terms of amplitude, of the ECD and MSD. The column used in the detector was optimised to detect light hydrocarbons, and the MSD is not very sensitive to these compounds; this results in not well defined peaks.

Table 2 summarizes the specific conditions chosen to operate the MSD and ECD detectors during the gas analysis performed for the measurements presented in this paper.

3.4 High-rate irradiation facility

The long-term stability of MSGCs is studied by measuring the variations of gain with the chamber exposed to high radiation fluxes. While most of the radiation in LHC detectors will be composed of charged particles, laboratory tests are realized with an equivalent flux of soft X-rays, under the assumption that ageing properties are determined mostly by the amount of collected charge and not by details in the charge deposition process. The detector under test (Fig. 6) is mounted facing a collimated X-ray beam provided by the generator; the beam intensity can be adjusted by attenuation, collimation of the beam and/or variations of the current and voltage of the tube. We have used X-ray tubes with copper and iron targets⁷, having fluorescence peaks around 6 and 8 keV, respectively. Operating the tubes at a moderate high voltage (15 kV) minimizes the contribution of the bremsstrahlung continuum. Fig. 7 shows a typical pulse height spectrum recorded with a MSGC irradiated with a copper target tube; in this case, use of a thin aluminium window on the detector preserves the original energy spectrum of the tube. In some cases however, due to differential absorption in the windows, the detected X-ray energy spectrum can be distorted towards its higher energy side, a point that should be taken in proper account in the data analysis [32].

⁶ CARBAGAS

⁷ PHILIPS fine focus types PW-2217-20 and PW 2253-20.

For most measurements the MSGC was illuminated with an effective beam area between 1 and 5 mm²; detected intensities well above 10⁶ s⁻¹mm⁻² could be realized. The MSGC plates have the read-out electrodes (anode or cathode strips, depending on models) connected together in groups and grounded; one or more of the groups are equipped with pulse-height or current recording electronics. The other set of strips are connected in groups to the HV power supply through protection resistors and a filtering network to reduce noise, as shown in the figure. In order to allow high current measurements without voltage drops, the protection resistors are kept low in value (few hundred kΩ). Values of current delivered from the power supplies are also recorded; we have used for this purpose custom-built HV modules including current meters with ~0.2 nA sensitivity^{8,9}. Fluctuations in gain of the test chamber due to changes in temperature, pressure, or particle flux are corrected, as described in the next chapter, using the recorded data on a single-wire proportional monitor counter in series with the gas of the MSGC and irradiated with an attenuated fraction of the photon flux (<10 s⁻¹ per mm of wire). During ageing tests, both detectors are continuously irradiated and current, pulse height and counting rate are recorded. Together with the DC currents due to the irradiation measured on the active and on the drift electrode, low-rate pulse height distributions and leakage currents are recorded decreasing temporarily the flux on the MSGC with the help of a thin stainless steel absorber foil inserted in the X-ray beam under computer control. Pressure and temperature are also periodically registered. The graphic and analytic capabilities of the computer are used to monitor frequently the progress of the run without disturbing the data collection.

4 DATA ANALYSIS AND NORMALIZATION PROCEDURES

4.1 Generalities

The long running time, up to several months, needed for each test exposes the detector under irradiation to gain fluctuations due to changes in the ambient conditions (pressure, temperature, humidity), variations in the X-ray flux, changes in the gas composition that could mask the real effects of the ageing process or be wrongly interpreted as signs of deterioration of the counter. We have developed several methods for data reduction, aimed at removing the effects of such instabilities; each method has its own advantages and limitations. It should be emphasised that the corrections involved in the normalization procedure may exceed in value the effect of ageing itself, and therefore other, verifications of the results have been used, such as short-term gain profiles realized by mechanical scanning with the source across the irradiated region, and control measurements with detectors known to present little or no ageing.

⁸ CAEN N471 A with direct readout

⁹ BERTAN 375 P with PREMA 4000 digital multimeter

4.2 Correction based on the measurements of temperature and pressure

The gain in proportional counters, and therefore the detected current at constant rate and the pulse height distribution, depend from the gas density, function itself of the temperature T and of the pressure P . To remove ambient-induced variations, data need to be corrected for changes in temperature (mainly day–night oscillations), and pressure determined by weather patterns.

The correlation between low rate raw gain data, recorded with the monitor proportional counter, and the ratio T/P is clearly apparent from Fig. 8. In principle, one can compute the gain dependence from P and T in proportional counters using one of the expressions provided in the literature; we have reasons to believe however the functional dependence to be different in MSGCs due to the particular field structure and to changes in the substrate resistivity, and we have therefore preferred a more phenomenological approach.

The method makes use of the recorded values of temperature and pressure T_i and P_i during the run, with an ad-hoc phenomenological correlation function that we have written as follows:

$$G'_i = G_i \left[\frac{\langle T_i \rangle}{T_i} \right]^f \left[\frac{\langle P_i^{-1} \rangle}{P_i^{-1}} \right]^g$$

1)

where $\langle T_i \rangle$ and $\langle P_i^{-1} \rangle$ are the mean values of temperature and inverse pressure over the test period, and the exponents f and g are chosen experimentally in order to minimize the standard deviation of the data fitted to a smooth distribution.

For a verification, we have used the expression to correct the gain fluctuations recorded in the single wire monitor counter, not expected to experience ageing due to the low dose irradiation; Fig. 8 shows the constant gain obtained correcting the measured values according to Exp. 1 with $f=1.6$, $g=0.3$.

Fig. 9 shows the similar result obtained over the same period of time with the gain recorded in a MSGC under heavy irradiation; in this case the best correction was obtained with $f=0.2$ and $g=0.3$, and for this choice the average gain remains constant. The MSGC under test (chromium strips on S-8900 glass, operated in argon-dimethylether) was indeed not expected to suffer ageing up to very large doses. Fig. 10 shows the dependence of the corrected data distribution from the value of the parameters appearing in the expression; for the choice, we have selected the values of f and g that minimize the standard deviation of the difference between corrected data and a smoothed fit to the distribution.

The described method, despite its simplicity, suffers from the limitation of not taking into account fluctuations in the gas composition and, if applied to the measurement of current (as against pulse height), variations in the X-ray flux. Moreover, best values of the exponents in Exp. 1 vary depending on the structure of the MSGC and on the nature of the substrate .

4.3 Correction using the monitor counter

As mentioned, a single-wire proportional counter was arranged in series on the gas flow with the MSGC under test, and irradiated at a reduced rate by a second output from the generator. In principle, its gain is affected by the same sources of fluctuations as the MSGC, except those caused by the strong irradiation, and can therefore be used as term of reference for normalization. We have used the following expression to correct the data:

$$G'_i = G_i \left[\frac{\langle M_i \rangle}{M_i} \right]^h \quad 2)$$

where G_i is the measured value of gain on the MSGC, M_i and $\langle M_i \rangle$ the actual and average values of the monitor gain. The value of exponent h reflects the amount of correlation between the gain in the two counters. Fig. 11 gives an example of gain recorded on the monitor counter during a long-term irradiation period, and of the gain measured with a MSGC and corrected, using Exp. 2, with different values of the exponent. For each value of h , we have fitted a smoothed curve through the data, and computed the standard deviation of the difference to the corrected points; as shown in Fig. 12, a value $h = 0.2$ provides the best choice. A disadvantage of the method is that it does not take into account possible temperature dependence of the leakage current and of the gain on the MSGC support, a result of the corresponding changes in resistivity of the substrate; this effect appears to be more important in the case of low resistivity supports with high leakage currents. As a consequence, the value of the correlation coefficient depends on the particular structure of the MSGC.

4.4 The double beam method

This method has been developed recently to overcome some of the above mentioned limitations. Together with the main irradiated spot, a second region in the MSGC under test is simultaneously exposed to an attenuated fraction of the X-ray beam, and the pulse height distribution is recorded; assuming that ageing will not occur in the region exposed to the low dose, the ratio of pulse heights measured on the two spots provides a correction-free information on permanent changes in gain. Fig. 13 shows schematically the set-up, realized with two collimating plates, one fixed on the MSGC with an horizontal slit and a second with two holes that can be shifted in the horizontal direction. Thin stainless steel attenuation foils can be mounted on the holes to adjust the beam intensity. Fig. 14 provides an example of raw and corrected data obtained with the procedure; medium-term variations are clearly removed.

A drawback of the double beam calibration method lays in the possibility for polymers or discharges produced in the irradiated region to affect also the reference spot; runs are however usually suspended much before serious irreversible damages are detected. Complemented by the previously described calibration procedures, the double-beam method appears to provide good stability of results in long-term measurement.

4.5 Estimation of the rate of ageing

Ageing of gaseous detectors appears as a decrease of gain during the irradiation. It is common practice to express the gain variation through a quantity R [37] providing the relative gain drop normalized to the collected charge:

$$R = -\frac{1}{Q} \frac{\Delta M}{M_0} \quad 3)$$

where Q is the total charge (in C or C cm⁻¹) and $\Delta M/M_0$ the relative gain change.

The value of R appears experimentally to depend on the total collected charge in a given run, in general (but not always) decreasing with Q as shown by the example in Fig. 15; this makes it often difficult to compare results obtained in different conditions. In the present work, quoted values of R refer to asymptotic value at large collected charge; however, only the analysis of a full measurement such as the one shown in the figure can avoid drawing superficial conclusions when making comparisons of ageing rates.

5. EXPERIMENTAL RESULTS

5.1 Generalities

A vast amount of experimental data has been accumulated over the years exposing the evolving models of detectors to radiation and recording their counting characteristics. While in principle continuing care was taken to try and modify only a few, if any, parameters at each exposure, this is often not possible, for example when comparing plates made with different metals or different substrates (and usually a different manufacturing technology). Moreover, early measurements were realized in less than optimal conditions of gas purity, and only an increasing experience in the use of the analysis line could sort out unexpected sources of pollution: an example is the freon outgassing found from a Teflon seal in a supposedly all-metal valve (see later). Some key measurements were repeated to check the consistency of results, but this cannot be done systematically in view of the long time required for each point in the best conditions.

5.2 Gas filling and purity

Micro-strip chambers have been operated with a large variety of gas mixtures; to prevent fast ageing at high rates, convincing evidence suggests to avoid altogether the use of hydrocarbons as quenchers [5, 31, 39]. Dimethyl ether (DME), in mixtures with noble gases, has been found to meet both the good quenching and the ageing requirements. All measurements here described have been realized in argon-DME mixtures, mostly in a 50-50 volumetric ratio, an outcome of our previous optimization studies [40].

In view of the long duration of measurements and of the diverse installations used, it was considered essential to certify the gas purity at the

bottles' level. Argon, obtained from CERN's stockroom in research grade (A 45), has a guaranteed maximum content of O₂ lower than 5 ppm and 10 ppm of H₂O; our analysis on selected bottles confirms these values (see Fig. 16), and was only performed occasionally. We have systematically analyzed the DME bottles¹⁰ with the gas chromatograph optimized, as described in section 2.2, for the detection of light hydrocarbons and electro-negative pollutants of the freon group. Figs. 17 and 18 show an example of analysis for a bottle of average purity; the MSD detects the presence of hydrocarbons, and the ECD a clear signal for two electro-negative pollutants, identified through the mass spectrum as F-11 and F-12. The calibration described in the previous section provides then quantitative values for the halogens, while for the other pollutants only a qualitative indication has been obtained. Table 3 summarizes the results obtained for several cylinders, from different deliveries, together with the (average) certification from the producer. Pollution levels vary between bottles, particularly for air and the freons, but remain generally at a rather low level; some bottles appear exceptionally good. The difference in the abundance of hydrocarbons (such as ethene, propane, 1-propene, butane, 1-butene, 1-propene 2-methyl and 2-butene) are significant from bottle to bottle. It has also been observed that in some cases the amount of pollutants or air vary as the gas is being used, probably due to a process of selective distillation [15, 19].

On-line purification of the gas can be a solution, provided one finds appropriate filters. We have tested the effect of a freon-specific filter¹¹ on a bottle of DME containing a particularly large amount of freon 11 and 12 (cylinder # 6164, see Table 3). As revealed by the ECD spectra, see Fig. 18, F-11 is very efficiently removed, but the content of F-12 is not affected while there is an increase the abundance of water vapour. We have found however no effect of the removal on ageing. As shown in Fig. 19, a chamber made with gold strips on D-263 glass would age in a similar way before and after insertion of the filter, and if anything the result is worse for the filtered gas case. A control run made with the same bottle without filtering with our standard chromium MSGC on electron-conducting glass (Fig. 20) reveals instead no ageing at all, a behaviour that will be discussed in section 5.4. It is therefore suggested that, at least in the range of pollutants values found in the tested DME cylinders, removal of F-11 has no effect on the ageing rate, if any; Table 3 could be considered as indicative of the tolerance levels for the pollutants identified. It should be also mentioned that we have currently used DME conditioned in 45 kg bottles (22 m³ gas). In the RD-10 laboratory, with a single test station, bottles need to be replaced every 5-6 months, and therefore a typical 3-month run would make use of the same bottle. In the RD-28 laboratory instead the larger consumption required a more frequent change, about every two months; a seam in the data would reveal the moment of change but appears to have no effect on operation.

¹⁰ MESSER-GRISHEIM

¹¹ SEMI-GAS SYSTEMS

5.3 Materials outgassing

One of the difficulties in establishing firm conclusions about ageing comes from the often incompatible results obtained by different groups. One reason for this is the different degree of cleanliness of the materials used in performing the ageing tests. An example case is shown in Fig. 21 [41] where the same MSGC, exposed to radiation in the two laboratories with different degree of cleanliness, exhibited lifetimes differing by an order of magnitude. In this particular case, and in order to explain the difference, the chromatography station was temporarily moved from the "clean" RD-10 set-up to the RD-28 laboratory and helped identifying a source of pollution: the ball-bearing valves used in the gas system, was found to release traces of freon 113 (F-113, $C_2F_3Cl_3$) as it can be seen in Fig. 22 showing the correlation between gain and the peaks revealed by the chromatograph at each manoeuvring of the valve. The pollutant was clearly identified with the help of the MSD (Fig. 23); local permanent damage of the anode strips was also visible after the irradiation. Disassembly of the valves revealed the possible culprit to be a Teflon joint; we do not know if the pollutant was introduced by the manufacturing process of the Teflon itself, or was a result of the use of cleaning fluids.

Having identified the source of pollution, an attempt to clean the system was made disassembling all valves and baking them at $\sim 100^\circ\text{C}$ for a few hours. Fig. 24 shows the result of a medium-term irradiation realized in the improved RD-28 set-up with a new MSGC plate (chromium on S-8900); 30 mC cm^{-1} have been collected with no detectable gain drop.

Using the RD-10 set-up, we have subjected a wide range of materials to outgassing tests, in view of their anticipated use for the manufacturing of gaseous detectors and MSGCs in particular. Whenever possible, samples of materials are prepared as thin plates of similar surface (around 100 cm^2); epoxies are spread over inert glass supports and cured following the recommended procedure. After thorough cleaning and pumping overnight at 10^{-3} Torr to eliminate possible residuals of the cleaning fluids, samples are introduced in the outgassing box of the RD-10 set-up (see Fig. 2) and fluxed with pure argon, pure DME or with a mixture of the two. The GC is then used to analyze the out coming gas. In order to increase the sensitivity of the system, samples can be warmed up in thermal cycles; if pollution is detected, the measurement is suspended in order to avoid the risk of contamination of the system. For good materials the thermal cycles are continued until reaching 50°C ; absence of detected pollutants at this point is considered as a necessary (but not sufficient) condition for the use of the component under analysis.

In the early measurements, a single wire proportional counter was used downstream on the gas flow to check the effects of pollutants on gain, and in some cases to detect fast ageing under irradiation. Figs. 25 and 26 show example cases for two types of epoxies (from Ref. [38]). A correlation between outgassing and measured gain is clearly seen for the "bad" epoxy; in this case, the gain

remains lower after suspending the thermal cycles, a clear indication of permanent damage or ageing.

Table 4 provides a list of the materials tested so far, either for our own needs or on request from external groups. In view of the time required for an ageing measurement, we have only verified the outgassing properties for most materials, and discarded those releasing detectable pollutants. For some “good” materials, namely VECTRA and E-505, full evidence of suitability was gained manufacturing complete MSGCs and subjecting them to a long-term ageing measurements [2, 42] (see also the following sections). In interpreting the results, it should be noted that the release of pollutants at high temperature might not represent the behaviour in normal conditions, and in any case may not have adverse long-term effects; also, the limited range of sensitivity of the MSD-ECD discussed before implies that some pollutants might have gone undetected.

5.4 Dependence of ageing on the substrate material and metal of the strip

Early observations suggested that the ageing rate could be affected by the nature of the substrate and of the metal used for the strips, the combination of low resistivity supports and gold providing better results than aluminium or chromium on high resistivity substrates [1, 5, 9, 25]. This trend appeared to be particularly true when the gas purity was not optimal due to outgassing of materials. Fig. 27 [3] shows for example the comparison between ageing rates observed with plates manufactured on high resistivity boro-silicate¹² and electron-conducting glass¹³ when unsuitable frame manufacturing materials (fibreglass and epoxy) were still in use; Fig. 28 shows the better performance obtained with gold strips on low resistivity glass¹⁴, compared to chromium, again in what are believed to be similar conditions. In view however of the difficulty of comparing results accumulated over long periods of time, or obtained by other groups, we have reasons now to suspect the correctness of our interpretation of the results; subsequent work has shown indeed that the ageing rate may depend from unexpected parameters such as the current density used in the irradiation (see 5.5 and Ref. [42]) and perhaps the gas flow, not always comparable in the early measurements. We have on the other hand confirmed that, when measurement are repeated in certified clean conditions, no ageing is observed for chromium chambers manufactured on a low resistivity substrate, see Fig. 29 [42]. The measurement was realized at a rather low current density ($4.7 \text{ nA mm}^{-2}\text{s}^{-1}$ initially, continued at $9.3 \text{ nA mm}^{-2}\text{s}^{-1}$), an important factor to consider in view of the observed current dependence of ageing (see the next section). No traces of deposits or damage of the plate could be observed by optical inspection of the irradiated region with a microscope (see Fig. 30).

The dominant role of the nature (namely the resistivity) of the support

¹² DESAG D-263

¹³ SHOTT S-8900

¹⁴ SSPC NIES C1-85

has been demonstrated manufacturing plates with different metals on boro-silicate glass and exposing the detectors to high radiation fluxes in clean conditions identical to the one described above. The results obtained both with chromium and gold strips on D-263 glass are shown in Fig. 31. Clear indications of ageing appear after an accumulated charge a few tens of mC cm^{-1} ; the current density used for the measurements was 10 and 12.7 nA mm^{-2} for gold and chromium strips, respectively. The gold plate behaves slightly better, but still ages rather quickly demonstrating that the most important factor determining the ageing properties is the nature of the support and not the metal used.

The tolerance to very high exposures of MSGCs made on low resistivity substrates has been verified by many measurements; the recent development of detectors manufactured on diamond-coated glass with a surface resistivity around $10^{14} \Omega / \square$ confirms our findings [43]. Fig. 32 [44] shows the long-term behaviour of a medium size ($80 \times 80 \text{ mm}^2$ active) MSGC plate made with chromium strips on diamond-coated D-263 glass; no change of gain is detected, within the experimental errors, after a total accumulated charge close to 100 mC cm^{-1} . We do not know at the present time if the negative results obtained with boro-silicate glass are due to a modification of conductivity due to internal migration of ions (the majority charge carriers in the glass), or to an enhanced sensitivity of the gain to small modifications in the surface resistivity that could be induced by very thin deposits. It is intuitive, but remains to be demonstrated, that the polarization currents generated by a low surface resistivity can have a stabilization effect on the value of the electric field, against modifications induced by thin deposits.

Attempts to investigate the possible effects of the X-ray absorption in the substrate itself have given so far negative results; no change of surface resistivity or gain were observed on a boro-silicate MSGC irradiated in conditions of no gas gain (lower voltage or heavily quenched gas) with X-ray doses equivalent to those used for the ageing tests.

5.5 Dependence on the current density

It appears, particularly for the high resistivity supports, that a critical role in determining the ageing behaviour is played by the exposure rate, or, more precisely, the current density at which the test is performed. This is demonstrated by the set of measurements shown in Fig. 33, obtained with the same MSGC (chromium on D-263 glass) in identical conditions except for the current density used in the exposure, from 6 to 45 nA mm^{-2} . No drop in the (normalized) detected current is observed, up to large values of accumulated charge when measurements are realized at high current densities (above $\sim 30 \text{ nA mm}^{-2}$), while at lower dose rates there is considerable damage.

This effect, not observed with lower resistivity substrates, seems to be characteristic of boro-silicate glass, and can be related to the way the electric field configures itself depending of the amount of ions that have to be neutralized.

Notice also that the time scale involved in the measurements is quite different; plotting the gain as a function of time, however, does not reveal special features (Fig. 34), although one could speculate on the competing effects of various processes as ion migration in the glass, bulk damage due to radiation, accumulation of charges on thin polymer layers. The real outcome is that MSGC, manufactured on boro-silicate glass and irradiated in clean operating conditions at moderate fluxes, exhibit substantial ageing, a qualitative difference with the observations with electron conducting glass. This observation also invalidates the extrapolation to normal operating conditions (lower current densities) of measurements at very high dose rates, such as the one quoted in Ref. [25], realized at around 80 nA mm^{-2} .

The gain loss is not only a function of the current density used for the irradiation, but also of the rate at which the gain is measured. Fig. 35 shows that in the irradiated position different gain drops are measured when scans along the strips are realized using different X-ray fluxes; notice that the higher the flux used for the measurement, the lower appears to be the gain drop. This is consistent with the apparent absence of losses, when the gain is deduced from a current measurement at very high rates.

A case of near independence of ageing from the overall absorbed dose is shown in Fig. 36, where two spots irradiated at different rates (6 and 45 nA mm^{-2}) exhibit similar gain drops when measured with a low-rate scan, although the heavier irradiated one had absorbed twice the charge of the other. This has been found to be a consequence of the different time lapse between irradiation and measurement in the two cases (few minutes for the lower current run, several weeks for the higher current). The time dependence of the gain decrease in the irradiated region is shown in Fig. 37: the original gain partly recovers, a possible manifestation of local surface charging up, or of effects due to long term ions migration. Once again, none of these effects are observed when using low-resistivity supports, as discussed in the previous section.

In order to check if any detail in the operating conditions (drift field, gas composition, frame materials) was responsible of the results obtained with the boro-silicate glass, a number of ageing tests were repeated changing parameters one at the time. All measurements were realized at a gain around 10^3 . The results are summarized in Table 5, and show that none of the operating conditions, including the presence of freons in DME, sensibly affect the ageing of plates, except for the current density. With reference to the standard operating condition (first row) we have underlined the parameter modified in the various runs and the corresponding ageing rate R . It can be noticed that the ageing rate is detectable only for tests made at a current density lower than 14 nA mm^{-2} (a value of $R = 0.001$ implies a 10% gain drop after 100 mC cm^{-1}). Independently from the operating conditions, the gas mixture and the chamber frame materials, the rate of degradation is constant for low values of the current density.

5.6 Can additives help?

Abundant bibliography and anecdotal evidence exist on the effect on multi-wire chambers lifetime of different additives like methylal, alcohol, water, oxygen, hydrogen [17, 45-48]. The general trend is that most of these substances, added to the gas mixture in small concentrations, usually (but not always) extend the lifetime of the detectors, sometimes by an appreciable factor, and in some cases even restore the original operation in aged devices. The results have been variously interpreted as due to an inhibition of the polymerization processes, to a suppression of secondary photon-mediated phenomena, to a simple restoration of conductivity on damaged electrodes due to surface vapour adsorption [17, 18, 28]. There were a priori reasons to hope that similar beneficial effects could be obtained in MSGCs, thus relaxing the requirements for the purity of the gas systems.

Several attempts were made to add vapours to the main argon-DME gas mixture. The first observation is that when trying to add quantities similar to those used in wire chambers (a few percent in volume), the MSGC would discharge at rather low voltages, probably a consequence of a modification of the substrate's dielectric rigidity due to molecular adsorption. Reducing the additive to acceptable levels (well below 1% for water) we have found no improvement and even some worsening of the ageing behaviour, as shown in Fig. 38 for a 0.1% addition of water. Amazingly, the gain drop is revealed by the measured value of current during the irradiation, but not by the low rate pulse height distributions. The picture in Fig. 39 shows the extended damages suffered by the anode strips in the irradiated area after the measurement .

Larger percentages of hydrogen, a recognized inhibiting agent for polymerization, could be added but again no improvement was observed, as shown in Fig. 40. In this case, the gain drop is observed for the low rate pulse height distributions but not for the current; we do not explain this behaviour. Similar results have been reported by other groups [49, 50].

The negative results could be due to the insufficient amount of additives that can be used still maintaining a stable operation of the chamber, or to the possibility that in wire chambers the dominant action of an additive is not to prevent polymerization but rather to increase the conductivity of the deposits, a property that could have adverse effects in the MSGC geometry. It is clear that this point requires further investigation, as there are many substances that can be added to the gas in various concentrations. A positive result would significantly release the tight requirements in the quality of the gas and of the materials.

6. SUMMARY OF RESULTS

Over the years, a large amount of data have been accumulated by many groups concerned by the crucial issue of long-term survival of MSGCs in a high radiation environment. The complexity of the parameters involved in

determining the ageing properties of detectors and the often conflicting results obtained in seemingly identical conditions have not allowed so far to obtain a clear understanding of the ageing processes and a general solution to the problem. In our group, we have attempted to address the question with a strategy aiming at keeping a minimum number of variables, changing only one operating parameter at a time, and continuously recording the nature and amount of pollutants in the system, which appears to be one of the dominant factors in determining the ageing properties. Although we foresee to explore a wider range of physical parameters in the future, our present experience has been limited to the use of argon-DME mixtures at relatively large flows (50 to 100 cm³ min⁻¹), X-ray generators to emulate high rates of charged particles, and MSGCs manufactured on standard boro-silicate, electron conducting glass and diamond-coated glass with chromium or gold strips.

Our major observations are summarized in what follows:

- The amount of pollutants in the gas system plays a major role in determining the ageing properties of the detectors; outgassing from materials, epoxies, joints, tubing has to be carefully controlled before assembly and kept at ppm levels or better. Use of an on-line analysis system (gas chromatograph) is strongly recommended to allow identification and removal of pollution sources and to guarantee reproducibility of the results; a correlation has been found between ageing rates and the amount of carbon halogens (electro-negative) pollutants, probably through a catalyzing effect on other organic residues.

- A provisions for outgassing by baking, prior to the start of operation, of the largest part of the gas mixing and distribution system seems mandatory to prevent irreversible damages in the detectors; in a small system, an overnight heating (using resistive tape) to around 150 °C of all passive tubing components in presence of a pure argon flow has proved to be necessary and sufficient, allowing even to recover from accidental serious pollution of the system.

- Standard research grade argon is consistently pure enough to allow its use in open flow, with only an active filter to remove residual oxygen and water and a micro-pore filter for particulates. The amount of pollutants in DME is instead rather variable and should be checked before using the bottles; particular attention should be given to fluorinated compounds (freons) and light hydrocarbons. Within the limits of our experience (12 standard 150 liters cylinders analyzed so far), no correlation was found however between the amount of pollutants and ageing, thus providing indicative tolerance levels for pollutants. No beneficial effect of removing traces of a notoriously suspicious pollutant (freon 11) was detected, suggesting that it should not be necessary to use expensive active filters in the DME line.

- Exposure to high-flux X-rays was used to emulate the lifetime properties of detectors with an accelerated time scale, the natural unit of measurement being the amount of charge collected per unit length of strips. We have found however that the ageing rate depends on the dose rate, or current density used in

the irradiation; the effect is particularly strong for detectors built on high resistivity boro-silicate glass. In this case, while in clean operating conditions the gain (deduced from a measurement of current) remains constant at high current densities (above 30 nA mm⁻²), severe gain losses are observed at lower irradiation rates. This observation raises a question on the relevance of ageing results obtained at high dose rates, extrapolated to normal conditions; it also makes doubtful some early comparisons of performances realized at very different rates. In all recent measurement we have adopted a maximum current density of 10 nA mm⁻², an acceleration factor of about 20 compared to the anticipated LHC maximum expected rates; in view of the measurement times involved (three months to reach 100 mC cm⁻¹) this choice is reasonable but arbitrary and its validity should be checked by a comparison with data obtained in more realistic conditions.

- On boro-silicate glass substrates, and in clean operating conditions, a low rate pulse height analysis in the seemingly unaffected regions exposed to high currents immediately after the exposure reveals a strong local, rate-dependent gain loss. With the detector left on voltage without irradiation, the gain tends to recover towards its original value with a time constant of several days. This behaviour is suggestive of a temporary modification of the electrical properties of the support not caused by polymerization, and introduces an unexpected time dependence of the results making comparisons delicate.

- In optimal operating conditions (argon-DME mixtures with a clean gas system, thorough choice of manufacturing materials, moderate current density for the irradiation), no ageing has been observed in repeated exposures, up to and above 100 mC cm⁻¹ of accumulated charge, for MSGCs manufactured on low resistivity supports ($\sim 10^{14}$ Ω /square), and independently from the metal used for the strips (chromium or gold). No time or rate dependent effects, such as those described above, have been detected in this case.

- We have reasons to believe that some early observations, by our group and by others, of a reduced sensitivity to pollution-induced ageing processes of strips manufactured in gold, as against chromium, could be in fact a consequence of the very different conductivity of the substrates and current densities used for the measurements, more than the result of a reduction in the polymer formation as suggested by some authors.

7. DISCUSSION OF THE RESULTS AND CONCLUSIONS

The present paper collects and summarizes the results of several years of investigation on the use of micro-strip gas chambers for tracking detectors designed for operation in the LHC environment. We have described in detail the experimental set-ups and procedures developed to emulate and study the long-term operation under high radiation flux of the detectors, and discussed the most representative results obtained. In view of the complexity and variety of the

factors affecting the lifetime of gaseous detectors, and MSGCs in particular, and despite the large number of observations, no general solution to the ageing problem can be proposed, but only a set of rules and caveats that should allow, to the best of our knowledge, to choose the proper construction technology for the components of a detectors. Two major processes leading to a degradation of performances in MSGCs, substrate charging up and surface deposition of polymers, have been clearly identified and separated from other sources of degradation (poor quality of the artwork, field emission from edges, micro-discharges etc.). We have demonstrated that in reasonably clean conditions, and with proper choice of gases and manufacturing materials, the ageing component due to polymerization can be avoided up to at least 120 mC cm^{-1} of collected charge, equivalent to ten years of operation at LHC, regardless from the metal used for the strips. On the other hand, use of high-resistivity boro-silicate glass results in rate-dependent, long term modifications of gain, probably due to local accumulation of charges on the insulating surface between strips and/or slow migration of the majority carriers (sodium ions) in the glass.

Use of bulk or surface-conditioned electron-conducting substrates with surface resistivity around $10^{14} \Omega/\text{square}$ solves all problems of short and long term instabilities observed with detectors made on insulating supports; presumably, even in presence of moderate formation of polymer deposits, the polarization currents maintained by the resistive support largely dominate the definition of the electric field strength and structure in the region of the electrodes, thus obliterating the effect of the layers.

Our interpretation of the results is supported by the measurement shown in Fig. 32: a long-term, low current density ageing run realized in what has been referred in this paper as the "standard" RD-28 laboratory, with a moderately clean gas system. The MSGC used for the measurement had a $100 \times 100 \text{ mm}^2$ active area, chromium strips on D-263 boro-silicate glass, coated with a thin ($500 \text{ \AA}$) diamond layer with surface resistivity of $\sim 10^{14} \Omega/\text{square}$. In the same set-up, an identical uncoated device was shown to age rapidly. This demonstrates unequivocally the predominance, in defining the lifetime characteristics of micro-strip chambers, of the surface conductivity of the support.

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COMPONENT	SET-UP	TYPE & MATERIAL	SUPPLIER
Gas lines	RD-10, RD-28	Stainless steel 316L 4×6 and 2×3 mm	CERN
Line connections	RD-28 RD-10	Stainless steel plugs Stainless steel plugs, VCR and UHV	CERN CERN and SAGANA
Flow meters	RD-28 RD-10	EL-Flow T 201C-Fa Viton (Ar) and Kemraz joint (DME) Kemraz joints (Ar and DME)	BRONKHORST HIGH-TECH B.V.
Oxisorb in Argon line	RD-28 RD-10	CrO ₃ , several Viton joints CrO ₃ , 1 Viton joint	MESSER GRIESHEIM
Filters	RD-28 RD-10	Stainless steel 2µm M6SFT2SS Stainless steel TF 25 MODU.C 576011	SAGANA QUALIFLOW
No return valves	RD-28 RD-10	Stainless steel check valve, PTFE joint, sealed by Torr-Seal Stainless steel, PTFE joint	SAGANA QUALIFLOW
Manual valves	RD-28 RD-10	Stainless steel ball valve, PTFE joint Stainless steel	SAGANA NUPRO
Exhaust	RD-28 RD-10	~ 20 m copper line ~ 24 m stainless steel line	CERN

Table 1: Components of the clean gas mixers in the RD-10 and RD-28 set-ups.

PARAMETER	MSD	ECD
Injection port temperature	100 °C	100 °C
Detector temperature	250 °C	250 °C
Oven temperature	70 °C / 200 °C @ 35 °C/min	40 °C / 150 °C @ 10 °C/min
Run time	40 min	50 min
Column type	PLOT fused silica (Al ₂ O ₃ /KCl)	PORA-PLOT Q fused silica
Column dimensions	50m length, 0.32 inner diameter.	25m length, 0.53 inner diam.
Phase thickness	5 µm	20 µm
Column pressure	35 kPa	35 kPa
Signal information	Area, retention time and spectrum M/Z	Area and retention time

Table 2: Mass spectrometer and electron capture detectors conditions.

DME bottle	Air (ppm)	Water H ₂ O (ppm)	Freon 12 CCl ₂ F ₂ (ppb)	Freon 11 CCl ₃ F (ppb)	Ethene C ₂ H ₄ (ppm)	1-propene C ₃ H ₆ (ppm)	1-butene C ₄ H ₈ (ppm)	Chloro-methane CCH ₃ Cl
Nominal	no	< 50	no	no	< 50	< 50	< 50	no
675	1000	85	no	no	35	3	0.3	no
2124	1800	200	no	no	5	3	0.7	no
168	5300	50	no	no	580	80	7	yes
287	400	70	no	no	155	55	7	yes
433	300	50	0.1	no	no	8	2	yes
7688	no	100	4	no	no	3	0.5	no
2721	400	40	0.001	0.3	no	15	2	yes
6164	400	25	0.1	1	no	25	6	yes
2038	5400	80	1	0.001	155	50	5	yes
7621	13100	80	2	0.1	200	75	10	yes
6091	1600	60	2	0.2	190	65	7	yes
2004	2600	130	3	0.5	14	3	0.5	no

Table 3: Summary of the gas analysis made for twelve DME cylinders from various deliveries. The first row shows the values given by the supplier.

Material	Type Curing	Use	Surface (cm ²)	Outgassing in Ar	Outgassing in DME	Outgassing in Ar-DME	Effect in SWPC	Global result
DURALCO 4525	Epoxy Room Temp.	MSGC assembly	156			Yes @ Room T	Gain loss	Bad
DURALCO 4461	Epoxy Room Temp.	MSGC assembly	156			Yes @ Room T	Gain loss	Bad
HEXCEL EPO 93L	Epoxy Room Temp.	MSGC assembly	150	No	No			OK
HEXCEL A40	Epoxy Room Temp.	MSGC assembly	150	Yes @ T > 40 °C	Yes @ T > 40 °C			Bad
AMICON	Epoxy 1.5 h @ 80 °C	MSGC assembly	150	No	No			OK
ARALDIT AW106 HV	Epoxy 2 d @ 70 °C	MSGC assembly	176			Yes @ Room T	Gain loss	Bad
EPOTECNY E 505	Epoxy 30 m @ 80 °C	MSGC assembly	131			Yes @ Room T	No effect	OK
EPOTEK 905	Epoxy Room Temp.	MSGC assembly	150	Yes @ T > 45 °C	Yes @ T > 45 °C			Bad
EPOTEK H72	Epoxy 1.5 h @ 65 °C	MSGC assembly	150	Fast outgassing	Fast outgassing			Bad
STESALIT	Fibreglass	MSGC frame	380			Yes @ T=75 °C	No effect	OK
VECTRA	Liquid crystal polymer	MSGC frame	142			Yes @ Room T	No effect	OK
RYTON	Polysulphur phenylene	MSGC Frame	336			Yes @ Room T	Gain loss	Bad
PEEK	Poly ether ether ketone	MSGC Frame	260	No	No			OK
EPDM	Copolymer ethylene-propylene	Joint	300	Yes @ Room T	Yes @ Room T			Bad
PVDF	Fluorinated polyvinylidene	Joint	500	Yes @ Room T	Yes @ Room T			Bad
VITON	Fluorinated copolymer	Joint	412	Yes @ Room T	Yes @ Room T			Bad

Table 4: Materials and epoxies tested for outgassing with the gas chromatograph.

GOAL	Charge density (nA mm ⁻²)	X Ray gen. voltage (kV)	Anode voltage (V)	Drift field (kV cm ⁻¹)	DME %	R (cm mC ⁻¹)
Reference values	12.7	15	540	6.7	50	0.0025
Charge density	<u>32</u>	20	540	6.7	50	0.0001
% of DME	14	15	405	6.7	<u>10</u>	0.015
Charge density	<u>6</u>	15	540	6.7	50	0.0027
Charge density	<u>45</u>	20	551	6.7	50	0.0004
X Ray energy	12.7	<u>20</u>	540	6.7	50	0.0035
Drift field	13.6	20	540	<u>3.4</u>	50	0.004
<u>Frame (VECTRA)</u>	12.5	20	540	6.7	50	0.036

Table 5. Summary of the ageing tests done with an MSGC made on D-263 glass with chromium strips. In each row, underlines identify the parameter changed in respect to the baseline values (first row).

APPENDIX 1:

PRODUCERS AND MATERIALS MENTIONED IN THE TEXT

BERTAN: 121 New South Road, Hicksville, NY 11801 USA.

BRONKHORST HIGH-TECH: 1a Nijverheidsstraat, NL-7621 AK Ruurlo.

CAEN: 11 Via Vetraia, Viareggio, I-55049.

CARBAGAS: 103 Rue des Jeunes, CH-1227 Carouge.

DESAG: Deutsche Spezialglas AG, Postfach 2032, D-31074 Grünenplan.

HEWLETT PACKARD AG: 7 Rue du Bois du Lan - C.P. 365, CH-1217 Meyrin.

MESSER-GRISHEIM GmbH: Postfach 10 09 62, 170 Wörthstr., D-4100 Duisburg.

NUPRO: Arbor AG, 10 Loonstr., CH-5443 Niederrohrdorf.

PHILIPS AG: Dietikon, 12 Riedstrasse, Postfach 360.

QUALIFLOW: 175 Rue de Caducée, F-34090 Montpellier.

SAGANA: 49 Ch. Vert, F-69760 Limonest, CH-8953.

SEMI-GAS SYSTEMS: Univ. of Warwick Science Park, Warwickshire UK.

SURMET Co., 33 B Street, Burlington MA 01803 USA.

SCHOTT GLASS TECHNOLOGIES, 400 York Av., PE 18642 Duryea USA.

SSPC NILES, 25 Smirnowskaya Ul., 10952 Moscow RUSSIA.

DURALCO 4525 and 4461: Cotronics Corp., 3379 Shore Parkway, N.Y. 11235 USA.

EPO-TEK: Epoxy Technology inc., 14 Fortune Drive, Billerica, MA 01821 USA.

EPOTECNY: 10 Impasse Latécoère, F-78140 Vélizy.

ARALDIT AW 106 (hardener HV 953 U): CIBA-GEIGY AG, CH-4002 Basel.

STESALIT 4411W: Stesalit AG, CH-4249 Zullwil SO.

VECTRA C130: Hoechst High Chem; Manufactured by Nief Plastic, 10 R. Jean Rostand, F-69745 Genax.

RYTON R4 (polysulfur phenylene): Produced by Phillips Petroleum Co. USA.

PEEK (poly ether ether ketone), EPDM (copolymer ethylene-propylene), PVDF (fluorinated polyvinylidene), VITON (fluorinated copolymer): supplied by Angst Pfister SA, 52-54 Route du Bois des Frères, CH-1219 Le Lignon.

FIGURE CAPTIONS

Fig. 1: Close view of the strips in a MSGC damaged by long-term local heavy irradiation. A pattern of changing colours over the metal strips, typical of interference, indicates the presence of a thin polymer layer; the extended damages on the edges (mostly on cathodes) are probably a consequence of micro-discharges induced by the polymer formation.

Fig. 2: Schematics of the gas mixer set-ups used for the measurements; the most sophisticated one (named RD-10) includes an analysis line and other facilities to study outgassing of materials and to add vapours.

Fig. 3: Back diffusion of air into the analysis line, detected with the gas chromatograph in the RD-10 set-up, as a function of gas flow. The mechanical parameters of the exhaust pipe are indicated in the caption.

Fig. 4: Signal detected with the electron capture detector on the gas chromatograph for a calibrated mixture containing 10 ppm each of freon 11 and freon 12 in argon.

Fig. 5: Mass spectrometer detector signals for the calibrated sample containing freons (see Fig. 4). Less sensitive than the ECD, the MSD provides nevertheless the atomic composition spectra that allow to identify the pollutants.

Fig. 6: Schematics of the experimental set-up used for long-term irradiation of detectors. A collimated X-ray beam is used to expose the chamber to high radiation fluxes; an attenuated fraction of the beam can be detected in a single wire counter for monitoring purposes. Pulse height and current can be recorded on groups of electrodes in the test chamber; under computer control, a thin absorber can be inserted in the main beam to reduce the flux.

Fig. 7: Energy resolution of a MSGC detecting the attenuated 6 keV X-ray beam used for the long term irradiation. The modest resolution is mainly due to the bremsstrahlung continuum under the main fluorescence line.

Fig. 8: Correlation between raw gain measured with the single wire proportional counter, and the ratio between temperature and pressure. The data, corrected by Exp. 1 as discussed in the text, show a constant gain over long irradiation times.

Fig. 9: Example of gain correction procedure for data measured with a MSGC under strong irradiation, realized making use of the measured temperature and pressure through Exp. 1 with $f=0.2$ and $g=0.3$.

Fig. 10: Estimation of the parameters f and g appearing in Exp. 1 taking into account gain variations due to temperature and pressure, for the previous data. Individual distributions have been shifted vertically for clarity; the fitting procedure minimizes the standard deviation of the difference between corrected points and a smoothed line through the data.

Fig. 11: Gain correction for data measured with a MSGC under strong irradiation, realized making use of the gain measured with the single wire proportional counter through Exp. 2 for several values of h .

Fig. 12: Standard deviation of the fit to a smoothed function for the data of the previous figure, as a function of the parameter h ; the value $h = 0.2$ minimizes the variance of the data.

Fig. 13: Schematics of the mechanical collimator used for the double-beam calibration procedure. The pulse height measured in the reference position on a strongly attenuated beam is used to normalize the gain variations measured on the heavily irradiated test spot.

Fig. 14: Example of gain correction realized with the double-beam method; raw data (current measured at high rate) are normalized to the low-rate pulse height measurement (reference).

Fig. 15: Relative gain recorded during an ageing test, and computed values of the parameter defined in Exp. 3. In the plot, the points marked R are computed as the difference between relative gains, while the values R_c correspond to a fitted slope to the data from the origin to the point of abscissa Q.

Fig. 16: Example of ECD response of the gas chromatograph for a bottle of argon of average purity. Quantitative values for the abundance of pollutants have been obtained from an independent measurement with calibrated samples.

Fig. 17: MSD and ECD response of the gas chromatograph for a bottle of DME of average purity showing various pollutants (see also Table 3).

Fig. 18: ECD signal spectra for a DME bottle showing the freon-11 and freon-12 peaks (top), and the efficient removal of F-11 by a freon-specific filter (bottom).

Fig. 19: Comparison between ageing measured with an MSGC with gold strips on boro-silicate glass, before and after filtering away freon 11; if anything, the ageing rate is larger for the cleaner DME.

Fig. 20: Control ageing run with an MSGC made with chromium on electron-conducting glass, making use of the same DME bottle as for the previous measurement, without filtering.

Fig. 21: Comparative ageing tests (gain as a function of accumulated charge) for the same MSGC plate (chromium on electron-conducting glass) installed in two set-ups having different degree of gas purity (the RD-10 is the set-up including the gas chromatograph).

Fig. 22: Gain as a function of accumulated charge measured with a MSGC plate mounted in a slightly polluted gas distribution system. Peaks detected by the ECD of the gas chromatograph (dashed curve) reveal the release of an electro-negative pollutant in coincidence with manipulation of a valve used in the gas distribution.

Fig. 23: Retention time distribution and mass to charge ratio detected with the GC-MSD during the measurement shown in the previous figure. The pollutant has been identified as freon 113, released at the Teflon joints in valves used in the gas system.

Fig. 24: Gain as a function of accumulated charge measured with the same MSGC plate used to obtain the data shown in Fig. 20, after outgassing of the valves by baking.

Fig. 25: Gain variation measured with a single-wire proportional counter downstream from the box containing samples of materials (Araldite epoxy). A decrease in gain is detected in coincidence with each cycle of increase in the temperature of the material, clearly due to outgassing; a permanent damage (ageing) is also observed at the end of the irradiation.

Fig. 26: Long-term ageing measurement with the single-wire counter with samples of E-505 epoxy in the outgassing box, subjected to thermal cycles. No gain variations are observed during or after the measurement.

Fig. 27: Relative gain as a function of the accumulated charge measured with two MSGCs manufactured on boro-silicate (D-263) and electron conducting (S-8900) glass, assembled with conventional fibreglass frames, rubber O-rings and epoxies.

Fig. 28: Long term gain as a function of accumulated charge measured with two MSGCs made with gold and chromium strips on semi-conducting glass (C1-85 and S-8900 respectively). Both measurements were realized in the moderately clean RD-28 set-up.

Fig. 29: Long-term irradiation in clean conditions (RD-10 laboratory) of a MSGC made with chromium strips on S-8900 glass, at moderate current densities (4.7 nA mm^{-2} initially, increased to 9.3). No gain drop is observed up to 120 mC cm^{-1} , and the energy resolution (measured at low X-ray flux) remains constant.

Fig. 30: Microscopic view of the MSGC in the area irradiated for the high flux measurement described in the previous figure. No damages, deposits or discoloration could be detected.

Fig. 31: Lifetime measured on two plates made on D-263 glass with chromium and gold strips. The tests were made at moderate current densities (around 10 nA mm^{-2}) and identical conditions; both exhibit substantial ageing.

Fig. 32: Long-term irradiation of a MSGC made with chromium strips on diamond-coated D-263 glass. Raw data have been corrected for ambient variations. The measurement was performed in the RD-28 set-up, in normal cleanness conditions.

Fig. 33: Ageing of a MSGC made on D-263 glass and exposed, in different positions, to various of X-ray beam intensities; rates are expressed as current density in the irradiated spots. All other parameters are kept identical. Measurements realized above 20 nA mm^{-2} do not show ageing.

Fig. 34: The same data of the previous figure, plotted as a function of time; no particular time dependent feature is observed. While use of higher current densities is convenient to limit exposure times, it is obviously not representative of the ageing properties at lower dose rates.

Fig. 35: Gain scans along the strips measured from pulse height at different rates after a long-term irradiation of a D-263 plate; the amount of relative gain loss depends from the X-ray flux used in the measurement.

Fig. 36: Scan along a group of strips irradiated in two locations at different dose rates (45 and 6 nA mm^{-2}). The gain drop appears similar, despite the difference in the current densities used in the exposure; the scan was realized immediately

after the lower dose rate exposure, but several weeks after the higher one.

Fig. 37: Time dependence of the measured gain drop in a heavily irradiated spot of a D-263 plate. The local damage seems to partly recover after several days from the irradiation.

Fig. 38: Effect on MSGC lifetime of the addition of water to argon-DME. After a moderate exposure, the gain deduced from the current measurement drops substantially, while the average pulse height (measured at low rate) remains constant. Chromium strips on S-8900, argon-DME-H₂O (90-10-01).

Fig. 39: Microscopic view of the irradiated area for the measurement described in the previous figure; damages and deposits are visible on the anode strips.

Fig. 40: Ageing of a MSGC (Chromium on D-263) operated in Ar-DME with the addition of 0.6% of hydrogen. In this case, the degradation is observed on the pulse height spectra measured at low rate, but not on the total current.

Fig. 41: Confirmation of the major role of the surface resistivity in determining the ageing properties. A standard MSGC (chromium on D-263 glass), overcoated with a thin resistive diamond layer does not show any ageing up to 50 mC/cm of accumulated charge when exposed in the RD-28 laboratory using a normal gas distribution system.

Fig. 1

RD10 & RD28 set-up

Fig. 2

Fig. 3

Fig. 4

F11&F12 MSD

Fig. 5

Fig. 7

Fig. 8

Fig. 9

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