ETCHING OF COPPER COATED MYLAR TUBES WITH CF_4 GAS

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ABSTRACT

Using 5 mm diameter copper coated mylar straw tubes at a potential of 2.30 KV relative to a concentric 20 μm diameter gold-plated tungsten anode, it has been observed that with very low flow rates of CF_4-based gases the conductive copper cathode material may be removed entirely from the mylar surface.

1 Introduction

Experiment E871 in the B5 line of the Alternating Gradient Synchrotron at Brookhaven National Laboratory, is designed to search for very rare K_L decays with a single event sensitivity below 10^{-12}. For this search, the 24 GeV AGS proton beam producing $\sim 2 \times 10^8$ K_L^0 per spill from a water-cooled platinum target is used. Expecting chamber rates of $\sim$ 200 MHz/m^2 for this experiment, we selected 5 mm straw tubes with CF_4 based gases as a starting point for initial prototype studies. Our interest in CF_4 based gases lies primarily in their high drift velocities [1] and in their good aging characteristics [2–4].

2 Hardware Configuration

Our small test chamber is constructed of two 60 cm long, 5.0 mm diameter tubes each with a concentric 20 μm diameter gold-plated tungsten wire stretched to a tension of $\sim$40 gm. The tubes are made of 2-ply 0.5 mil mylar, the inner layer of which has a 1000 Å layer of copper vapor-deposited onto its inside surface. Cylindrical brass sleeves provide the mechanical support for and electrical contact between the straws and the endplates. Feedthroughs passing through the endplates and into the sleeves position the wire. Details of the endplate and feedthrough assembly will be provided upon request.

3 Copper Etching

Using two thin pieces of scintillator connected to phototubes, a trigger was defined with which the operating voltage of each gas was determined using a 5 mCi ^{90}Sr source. No difference in the operating voltage was observed between the two straw tubes with gases used in this study: Ar-C_2H_6 (50:50) and CF_4-CH_4 (30:70). The operating voltages for Ar-C_2H_6 and CF_4-CH_4 are 1.65 KV and 2.30 KV, respectively. Typically, the gas flow was about one volume change per minute.

The copper etching effect was first noticed accidentally: for a period of about ten hours, the gas (CF_4-CH_4) flow was off while for one of the two straws, the HV was on. Visually, the difference between the two straws was striking: one was mostly transparent and the other appeared normal. A white powder was observed on the bottom of etched tube. There are apparently no known volatile copper compounds.

Under more controlled circumstances, the etching effect was repeated with the undamaged straw. The basic procedure was simple: flow gas through the chamber, apply high voltage and note the current flowing between anode and cathode. At this point the gas flow was turned off and subsequent current measurements were made at regular time intervals.

With CF_4-CH_4 flowing at ∼20 ml/min, no measurable current was flowing (with 10 nA sensitivity) at 2.30 KV. Approximately 90 minutes after turning the gas flow off, the chamber began drawing ∼10 nA; after 2 hours it was drawing 5μA. This was repeated many times.

When the chamber was drawing 5μA of current, a small amount of fresh gas (∼2 ml) was introduced at which point the current dropped to less than 10 nA. About twenty minutes later, it was drawing measurable current again and after ∼40 minutes, it was drawing 2 μA. This also was also very repeatable.

Changing the gas to the Ar-C_2H_6 mixture and correspondingly reducing the operating voltage to 1.65 KV, the current at normal gas flow was less than 10 nA. Stopping gas flow, no measurable current was observed for 7 hours.

At this point we changed to pure CF_4 operating at the same voltage, namely 2.30 KV. Pure CF_4 began to draw current within a few minutes of turning off the gas flow and after about ten minutes the chamber was drawing ∼60 μA; see Figure 1a. About an hour later, the current reached a maximum of ∼180 μA and after about 90 minutes, it leveled off at arount 140 μA. The straw

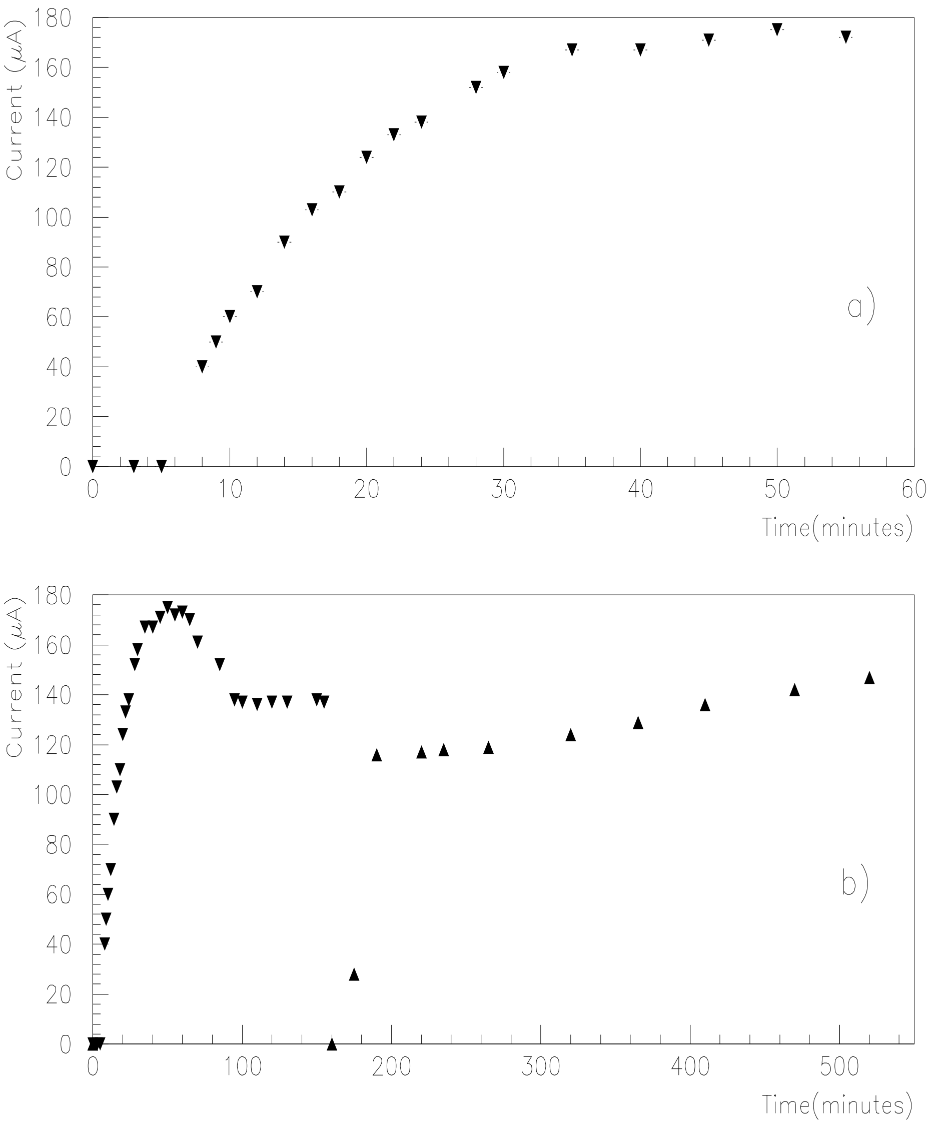

Figure 1: a) Short-term time dependence of current in straw #2 at 2.30 KV in pure CF_4 gas; b) Long-term time dependence; the copper began to thin visibly from the tube wall somewhere between 410-470 minutes on this plot. These data were taken over two days and the second set begins at t=160 minutes.

was turned off, flushed with fresh gas and the measurements started again at t=160 minutes in Figure 1b. About 310 minutes after beginning the second set of measurements, it became visibly apparent that the copper was being removed from the straw surface.

4 Suspected Mechanism

Under normal conditions CF_4 is considered a very inert gas; nonetheless, it is used[5,6] in the microelectronic fabrication industry as a component in the plasma etching of pure silicon and SiO_2. In one application of this process, CF_4 and either O_2 or H_2 are introduced into an evacuated chamber which is pumped with RF producing a plasma. In addition to ionizing the gas, the RF liberates fluorine radicals which react with the silicon to produce SiF_4. One may vary the etching rate of either the silicon or its oxide by introducing either O_2 or H_2.

Suspecting that O_2 was leaking into the chamber and reacting with the CF_4 in the high-field region near the anode wire, we built another small two-straw chamber for subsequent studies. This chamber was subjected to the same conditions as previously mentioned: pure CF_4 with HV at 2.30 KV and no gas flow. This chamber gave no indications of etching after 30 hours under these conditions. While introducing water vapor through a bubbler at room temperature did not induce etching, a pin hole in the straw tube wall did: the straw was drawing $\sim$20 μA after about 20 minutes. Repeating the exercise in a volume of N_2, the chamber drew no current. Returning the chamber again to an atmosphere of air and raising the voltage to 2.30 KV with a fresh supply of CF_4 and zero flow rate, the copper began to visibly etch after about 4 hours in the region near the hole.

It was suggested[7] that the copper was being removed through a reaction resulting in CuF_2 as a final product. A possible reaction is:

$$CF_4 + O_2 \rightarrow CO_2 + 2F_2$$

The flourine is then free to react with the copper. As mentioned, a white powder was observed in the straw after the copper was removed and CuF_2 is known to be white in its crystalline form [8].

5 Summary

In the presence of O_2 in the high-field region of a straw drift chamber, CF_4-based gases will undergo a chemical reaction with the conducting copper coating of the straws. While it is, in general, not a good idea to introduce O_2 into a drift chamber, under certain conditions complete desctruction of the chamber may result. A number of points may be made:

- CF_4 plus air in a straw chamber under high voltage removes the copper cathode material from the straw;
- ionizing radiation does not appear to be a factor;
- water vapor in the gas does not appear to be a factor;
- N_2 does not appear to be a factor.

For other reasons, the seven of eight straw chambers in experiment E871 at BNL were rebuilt for the 1996 run and to avoid problems arising from this effect two precautionary measures were taken: 1) the copper layer was increased from 1000 Å to 2000 Å; 2) all straws were individually pressure checked for leaks before installation. In addition to this, two operational changes were implemented: 1) gas flow rate was increased from 3 volume exchanges per day to 10 per day; 2) chamber voltage was ramped down between beam spills. The final chamber gas used in E871 was CF_4-C_2H_6 (50:50) with an operating voltage of 1950V (measured with an ^{55}Fe source, the gas gain is 8×10^4). Due to chamber capacitance, we were unable to ramp more than 100V down in the ~ 2 seconds available between the end of one AGS spill and the beginning of the next. As of this time, there is no indication of copper etching.

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