Aging studies for the ATLAS Transition Radiation Tracker (TRT)

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Introduction

End-cap: ~320,000 straws of about 40 cm length
Barrel: ~ 52,000 straws of 150 cm length
**Introduction: TRT operating conditions**

### ATLAS experiment at L=10^{34} cm^{-2} s^{-1}

- **Number of particles at a distance of 1 m from the interaction point:**
  - Charged: \( \sim 10^6 \text{ part/cm}^2 \text{ sec} \)
  - Photons: \( \sim 10^6 \text{ photons/cm}^2 \text{ sec} \)
  - Neutrons: \( \sim 10^6 \text{ n/cm}^2 \text{ sec} \)

- **Total dose for detector parts after 10 years of LHC operation:**
  - Neutrons: \( \sim 10^{14} \text{ n/cm}^2 \)
  - Charged particles: \( \sim 10 \text{ MRad} \)

### Some operation parameters for gas detector

- **Counting rate per wire up to:** 20 MHz
- **Ionis. current density up to:** 0.15 \( \mu \text{A/cm} \)
- **Ionis. current per wire up to:** 10 \( \mu \text{A} \)
- **Power dissipated by ionisation current per straw:** \( \sim 15 \text{ mW} \)
- **Charge collected over 10 years of LHC operation:** \( \sim 10 \text{ C/cm} \)
- **Total charge per 1 m of wire:** \( \sim 1000 \text{ C} \)
- **Total ionisation current in the detector volume:** \( \sim 3 \text{ A} \)
- **Total dissipated energy in the detector volume from ionising particles:** \( \sim 5 \text{ kW} \)

**Accessibility is very limited during ATLAS operation**

 Detector must be very robust!
Detecting elements: Straw

Reinforced straw
In order to make straw rigid four C-fibres are glued along the straw

Straw connection in the end-cap TRT
HV contacts to the straw inner surface
Detector elements: Gas mixture and straw parameters

TRT and Tracking requirements
- TR performance requires maximum content of Xe gas
- Minimum collection time requires maximum concentration of CF$_4$
- Small straw diameter: good for tracking but not optimal for TR and operation stability

Operation stability requirements
- Only combination of CF$_4$ and CO$_2$ as additives is allowed (CO$_2$ not less than 6%)
- Maximum CF$_4$+CO$_2$ concentration
- Streamer length ~ 1 mm
- Possible wire offset should not reduce straw operational stability

Straw diameter 4 mm
- Wire 30 µm
- Maximum wire offset 400 µm (goal 300 µm by construction)
- Gas mixture 70%Xe+20%CF$_4$+10%CO$_2$
Gold-damage effects

First observation in 1996 when cathode robustness to large irradiation doses was studied.

Wire - 30 μm gold plated Tungsten, current density of ~4 μA/cm, total accumulated charge of ~18 C/cm.

In some cases WO deposit was observed!

Is it a problem due only to large currents?
Gold-damage effects

Large-scale prototype:

- 61 straws of 1m length
- All straws were irradiated (60 cm of each straw) by X-ray tube
- 52 straws were in a closed loop gas system
- 9 straws were flushed
- Dose rate was 0.7 $\mu$A/cm (6 times less than maximum LHC)

After 2 C/cm

Inside irradiation zone
Gold-damage effects on wire

When does this effect can appear?
Conditions:
Mixture: 70% Xe + 27.5% CF₄ + 2.5% CO₂
Wire: Toshiba, 30 µm, ~1 µm gold
Current density: 1 µA/cm

Effective wire diameter increased by ~2 µm

Relative amplitude
Charge, C/cm
Conditions:

**Mixture:** 70% Xe + 27.5% CF₄ + 2.5% CO₂

**Wire:** OSRAM, 30 μm, ~0.5 μm gold

**Current density:** 1 μA/cm

**Total charge:** ~2 C/cm

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Conditions:

**Mixture:** 70% Xe + 6% CF₄ + 24% CO₂

**Wire:** OSRAM, 30 μm, ~0.5 μm gold

**Current density:** 1 μA/cm

**Total charge:** ~2.5 C/cm

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Gold-damage effects on wire
Gold-damage effects on wire

Edge of irradiation zone

Wire: OSRAM
Mixture: standard
Current: 5 µA/cm
Total charge: ~6 C/cm
Gold-damage effects: WO deposit

Different types of deposits

Soft deposit

Solid deposit
## Gold-damage effects: Comments

### Gold-damage effects:
- are not very regular and can be different for the same type of wire and the same test conditions.
- do not depend very much on exact gas composition (6%, 20%, 27.5% of CF$_4$)
- are not very much sensitive to gas gain or number of streamer discharges (gas gain has been changed in tests from 2 to 8$\times$10$^4$, and SLS rate changed by 2 orders of magnitude)
- are not very sensitive to dose rate (0.7 - 5 $\mu$A/cm)
- appear for very different total accumulated charges (0.5 - 6 C/cm) depending on wire and conditions
- are larger at the edges of the irradiation zone
- results in gold becoming porous after some under irradiation

### W- wire effects
- It looks like there are active chemical processes under the gold plating
- wire swelling is not seen regularly (observed just twice out of few tens of tests)
- in some tests, damage of W is observed but very often NO visible damage

### WO deposits:
- observed but not very regular
- happen very often outside irradiation zone and are larger downstream
- are sometimes very soft (disappear under EM electron beam), but sometimes solid

No F-based deposits observed at all!

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What can be the reason for the gold damage?
Gold-damage effects: Reason

What about H$_2$O and O$_2$?

1. Kapton is very transparent to H$_2$O. For 1 Volume exchange per hour concentration of H$_2$O in straw is ~1-1.5% under normal conditions
2. Air leaks into the gas volume can appear as well

In order to exclude environmental effects a special set-up with a N$_2$ envelope was used

- 70% Xe +20%CF$_4$+10%CO$_2$ mixture + additives (H$_2$O and O$_2$)
- Tests were carried out at 5 µA/cm
- Total accumulated dose was ~ 6.5 C/cm
Gold-damage effects: Reason

Toshiba W wire (1 μm Au)
Standard mixture +1.2% H₂O
6.5 C/cm

Toshiba W wire (1 μm Au)
Standard mixture +1.5% O₂ (NO H₂O)
6.5 C/cm
Gold-damage effects: Reason

What about combination of H₂O and O₂?
Tests with different types of wires: observed effects are rather varied!

OSRAM wire, 3 % Au
Standard mixture +1.2% H₂O and 1.5%O₂
0.5 C/cm
Gold-damage effects: Reason

OSRAM wire, 3 % Au
Standard mixture +1.2% H₂O and 1.5%O₂
0.5 C/cm

EDX spectrum from deposit
WO deposit!
Gold-damage effects: Reason

OSRAM wire, 3 % Au
Standard mixture +1.2% H₂O and 1.5%O₂
Total dose: 0.5 C/cm

Luma wire, 5 % Au,
Ni-substrate
Standard mixture +1.2% H₂O and 1.5%O₂
Total dose: ~3 C/cm

Presence of molecular Oxygen makes processes more complicated!
Gold-damage effects: Reason

Toshiba wire, 7% Au,
Standard mixture +~0.4% H₂O (NO O₂),
5 C/cm

After 21 C/cm

Toshiba wire, 7% Au,
Standard mixture + <0.1% H₂O (NO O₂),
20 C/cm
Gold-damage effects: Conclusions

- Main components responsible for wire damage are radicals, probably products of CF\textsubscript{4} disintegration, particularly F and H\textsubscript{2}O (or H) which may create HF acid.
- They dramatically increase rate of oxidation processes on the wire surface.
- Presence of oxygen speeds up wire degradation process (mainly due to WO deposits).
- Radicals do not affect wire without presence of H (source in our case was H\textsubscript{2}O).
- Effect very strongly depends on H\textsubscript{2}O concentration and is very much reduced for a water concentrations of about 0.4%.
- One should expect similar problems with e.g. C\textsubscript{2}H\textsubscript{2}F\textsubscript{4} gas even without H\textsubscript{2}O contamination.
- From these tests, there is no indication that F creates any solid deposits with any material, although it is known that it reacts with Au, Xe, W and O.

- All products of reactions with F are volatile (like WF\textsubscript{6}) or they react with other components creating volatile products.
- It seems HF plays the role of catalyst in the processes near the wire.
- No effects observed for water concentrations below 0.1% with various combinations of gases (Xe, CO\textsubscript{2}, CF\textsubscript{4}, O\textsubscript{2}) and wires.
- Effect appears faster for loose gold plating. For some wires one sees increase of pore size first.
- Presence of Ni increases wire degradation rate.
- Effect depends on type of wire used (producer and production technology).

Wire choice for TRT: Toshiba 30 µm W wire, 7-9% gold plating
Cathode effects

Cathode has C-loaded polyimide protection layer of ~5 µm thick with 1000 A Al under it

Straw cathode after 20 C/cm of accumulated charge

Some amount of polyimide was etched off after 20 C/cm but NO deposit on wire observed! No other cathode effects have been observed.
Detector material choices

A few steps have to be performed:

**Step 1.** Preliminary selection of the materials on the basis of their known chemical, mechanical, electrical, radiation stability and outgassing properties

**Step 2.** Selected materials are tested for outgassing before and after irradiation (up to 50 Mrad for TRT, which is a factor of 5 more than expected for 10 years at the LHC)

**Step 3.** Candidates selected after the first two steps then undergo straw ageing tests

Scope of analysis:

- Printed circuit boards
- Straws and plastic parts
- Glues
- C-fibre elements
- Other elements of the detector and gas system

**Step 1: DONE**

Complete information can be found in:
“Choice of materials for the construction of the TRT” ATL-INDET – 98-211

**Step 2: DONE**

Complete information can be found in:
“Outgassing studies of materials for TRT construction” ATL-INDET-99-011
Detector material choices

Components tested

Glues
- Araldite AW 106
- Araldite AW 134
- Araldite AW 103
- Redux 420
- Rutapox L20
- Stycast 1266
- Trabond 2115
- Traduct 2902
- Pronto

Materials
- C-fibre
- Ultem
- Polycarbonate
- Flex-rigid circuits (Kapton, FR4, solder mask)

• G10 as produced (barrel plates
• G10 machined
• Printed circuits for barrel (tension plate)
• Some others materials and components

Rejected for the reason of outgassing (Step 2) Araldite AW 106.

Next is Step 3 - Ageing studies
Consists of 2 tests for each component
- 10 mm wide beam 1 µA/cm dose rate
- 10 mm wide beam 0.1 µA/cm - the most sensitive to possible Si contamination

Final validation: all components together at 60°C in a quantity 20 times more per wire than in real detector
Detector material choices: Validation

0.1 μA/cm test

Results of one run from this test (total test duration was ~1000 hours)

Conclusions from these tests:

- No remarkable gas gain drop observed under any test conditions
- Some materials produced detected hydrocarbon outgassing but no wire deposit observed
- No Si deposit was found from G10 and FR4 materials even if they were smelling rather strongly after machining and irradiation (this outgassing is detected by GC with mass-spectrometer).
- During these tests few Si sources, which led to polymerisation effects (see slides below), were found
- It is important to note that Si deposits are never observed at 1 μA/cm dose rates because of the dominating etching process due to CF₄
Si -problem

First observation of Si - polymerisation in the straw

Run 118 - Test Peak Position vs. Integrated Charge

Comments

- Current 0.1 μA/cm
- Straw contained traces of Si-based lubricant by chance used by straw producer (occasionally)
- Amplitude drop is very fast ~10% per day!

Control manufacturing process in details!

X-RAY: 0 - 10 keV
Live: 100s Preset: 100s Remaining: 0s
Real: 144s 31% Dead

Anatoli Romaniouk,
Int. Workshop on Aging Phenomena in Gaseous Detectors, DESY, Hamburg, 2-5 Oct. 2001
Si problem: External sources of Si

1. Weak Si source:

All Si containing species are stopped at the very beginning of the straw.

- Few serially connected straws
- Gas flow from straw 5 to straw 4
- All straws are irradiated with a current density of ~50 nA/cm (~30% of max at LHC)
- At the moment corresponding to 170 h of irradiation, straw 5 was disconnected from HV

Si deposit propagation along the wire

Straw of 50 cm long connected to the gas system with weak source of Si (irradiation length of 50 cm, current density of 10 nA/cm)

- After 21h
- After 41h
- After 63h
Si problem: External sources of Si

2. Strong Si source:

If Si source is strong amplitude degradation is very rapid

- If amount of Si coming with the gas is small it might be stopped at the first few mm of the straw. But deposit then propagates along the wire affecting larger and larger areas of the straw.
- For strong Si sources, ageing is very rapid and full length of wire is affected at once.
- For strong Si sources even if current density is rather high and etching processes are dominant along the most of the straw, there appears a strong polymerisation process at the gas entry which has a tendency to propagate.

Both weak and strong Si sources are very dangerous!
Si problem: External sources of Si

Gas system components as main source of Si
(Si-based lubricant residuals)

Example: “Vogtlin” flow regulator (rotameter) specially produced by company and claimed to be free from any lubricants (particularly Si-based).

Rotameter was installed in the gas system

70 hours was enough to pollute some parts of the straw!

Graphs showing amplitude and gas gain change over time for two distances: 10 mm and 100 mm downstream from the test point.
Si problem: External sources of Si

Cleaning methods:
1. Ultrasonic bath:
   • “Vogtlin” rotameter (ageing rate of 10-15% in 100 hours). **No ageing** in 220 hours after cleaning in ultrasonic bath (see Mar’s talk).

2. “Washing” with DME
   • “Vogtlin” rotameter used in test shown in previous slide. **No ageing** during 500 hours after DME flushing through it during 1 week (few l/h). The same results for 0.8 l/h and 20 hours of flushing.
   • Small closed-loop gas-system prototype typical ageing in 200 hours ~10%. **No ageing** during 600 hours after 2 weeks operation with pure DME with 0.6 l/h gas exchange.

Can we obtain components without Si traces?

Certainly, but difficult to believe what companies claim!

There are positive examples:
   • “Scott” pressure regulator C21-08: **No ageing** in 600 h
   • “Swagelok” ball valve 316 ss-43s 6mm-SC11: **No ageing** in more than 500 h
   • “Gilmont” rotameter: **No ageing** in ~ 500 hours

Final recommendations:
   • Choose company and validate samples
   • Proper specification and control of production
   • Careful examination after delivery + sample validation in ageing tests.
   • Some parts can be ultrasonically cleaned
   • In some cases residual Si-traces can be removed of by DME after gas system is assembled (components should somehow tolerate exposure to DME)
Si problem: Si inside of the straw

What about straw produced without any lubricant?

EDX spectrum of straw cathode. Small traces of Si are observed. This Si is actually part of C-loaded polyimide layer

Under specific conditions this Si may produce some ageing effects (~5-10 % amplitude drop in 150 hours).

For instance, if straw is irradiated by a beam of 1 mm width and 5 kHz counting rate (\(^{55}\text{F-source}\)).

Dedicated tests were performed to study this phenomena

Typical set-up for Si polymerisation studies
Si problem: Si inside of the straw

For this set-up, all straws manifest ageing at different irradiation points along the straw!

In order to understand, a few additional tests were performed.

1. The same set-up was irradiated during 20 hours with a current density of $\sim 1 \mu A/cm$ (2 cm straw length) at the edge of the straw first downstream, then upstream.

Results:

1. **Downstream:** No indication of any ageing effects during at least 400 hours at 3 cm upstream of irradiation point. But some ageing effect was found at 10 cm upstream of irradiation point.

2. **Upstream:** No ageing effects observed in any straw region at least during 600 hours of irradiation!
Si problem: Si inside of the straw

Conclusions from these tests

- All straws (even with Cu-cathode) contain traces of Si, which may produce ageing effects under certain conditions.
- It may well happen that in case of a narrow beam (for instance, 1 mm) active agents released in the avalanche diffuse outside the irradiation area and react with Si producing volatile species, which transport it to the irradiation area increasing the ageing effects.
- Effect of straw cleaning indicates that cleaning agent (presumably F) survives for a rather long time and travelling with the active gas, removes Si from the cathode surface.
- Diffusion of radical exists but it is not large enough to clean the whole upstream surface of the straw.
- Si sources in the straw are rather weak and are removed after an integral charge of less than $3 \times 10^{-3}$ C (less than 1 day of LHC operation).
- There is some indication that after cleaning, a weak source of Si remains in the straw, but is not intensive enough to induce polymerisation.
Si problem: Si inside of the straw

There are always two competitive processes near the wire:

- **Si-deposition** which depends on Si-containing molecule density and ionisation current density. Si-source almost always produces a limited amount of molecules prone to polymerisation and, finally, the polymerisation rate is limited by the Si-source.

- **Si-etching** which depends only on ionisation current density and, perhaps also, on density of active species accumulated in the ionisation region.

It was found that polymerisation never happens inside the irradiation area when the active radical concentration is relatively large and etching processes are dominant. For instance, it was never observed for current density of 1 µA/cm (factor of 7 more than at LHC).

For large current densities when Si concentration is large, polymerisation is observed at the edges of the irradiated area and even far from them but not inside.

Under certain conditions (current density is ~1 µA/cm or more) etching processes dominate and existing deposits are etched away from the wires.

Polymerisation happens when concentration of the active radicals near the wire is rather small and etching effects are weak (low current densities).
**Si problem: Si inside of the straw**

**Steady state solution**

\[
\frac{dn}{dt} = D \frac{d^2n}{dx^2} - V \frac{dn}{dx} + P \delta(x)
\]

**Downstream**

\[n = \frac{P}{V} \frac{V}{eD} (x-x_0)\]

**Upstream**

\[n = \frac{P}{V} \frac{V}{eD} \]

**Diffusion coefficient** (gas in gas) is 0.1-1 cm\(^2\)/min. For minimum \(D\) and the gas flow in straw of 0.1 cm\(^3\)/min:

\(L \sim 5\) cm

**Distribution of Si sources for point like ionisation**

- Radical diffusion
- Si containing molecule diffusion

**Gas**

Straw surface seen as a source of Si impurities participating in polymerisation

**Distribution of Si sources for extended ionization**

Effective straw surface participating in polymerisation process near the wire in assumption that current stops Si-penetration form neighbour parts of the straw

**Effective straw surface participating in polymerisation process near the wire in assumption that current stops Si-penetration form neighbour parts of the straw**

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Active radical problem

3 A ionisation current in TRT at full LHC luminosity -> Plasma reactor!
Materials may be attacked by active radicals

- Conditions much worse than at LHC
- Materials may be attacked by active radicals
- Etching of coverlay on PCB
- Si deposit on gold-plated surfaces!
Active radical problem

Si crystals gold-plated metallic surface

EDX spectrum of the deposit
Conclusions

1. Wire gold-damage phenomena have quite complicated signatures, but do not appear if there is no water (nor H). Probably, H reacts with F, producing a very aggressive acid, which destroys wire gold-plating (maybe playing the role of catalyst for processes which happen at the Au-W border).

2. Cathode material is affected by active radicals in the irradiation area (not outside), but no deposit on wire and no change of straw operation properties were found.

3. Materials chosen for TRT construction if they are not in touch with radicals do not produce contamination which leads to any polymerisation in avalanche near the wire.

4. There are two competing chemical processes in action near the wire - polymerisation and etching. The resulting balance is very sensitive to the Si source intensity and the ionisation current.

5. There is some amount of Si inside the straws (few atomic mono-layers) but under the real LHC conditions, this Si should not produce and deposits on the wires.

6. External Si sources are very dangerous. Even weak sources of Si produce deposit at the gas entry, which then have a tendency to propagate along the wire.

7. One of the major sources of external Si are lubricant residuals in the gas-system components.

8. These residuals can be removed by ultrasonic cleaning procedure and sometimes by flushing with DME gas through the system or parts of it.

9. Company statements that their products do not contain traces of lubricant are sometimes untrue and validation/cleaning procedures must be foreseen.

10. Active radicals created in the avalanche live for a rather long time and attack the detector and gas-system materials.

11. Special purification elements for the removal of active radicals must be foreseen.