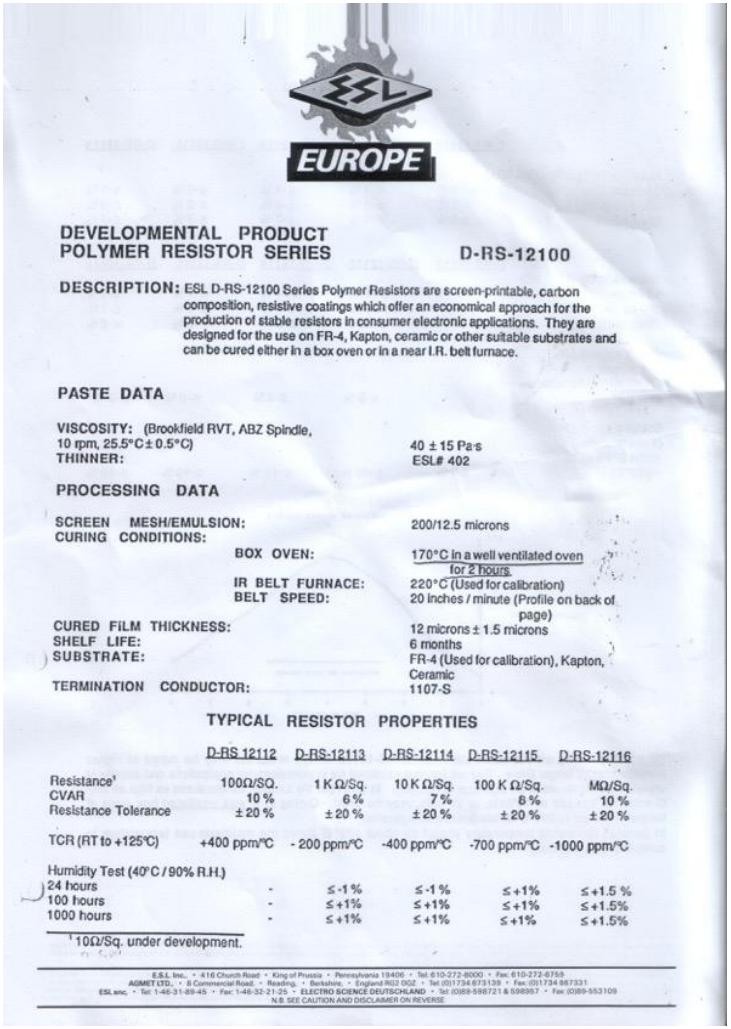
Preliminary tests of resistive materials for ProtoDUNE CPAs.

Introduction

- Tests of different materials candidate to work as cathode for ProtoDune
- Dedicated set-up to induce sparks and evaluate resistivity, at room temperature and in LAr.
- Materials under test: Micarta; Ink printed vetronite and resistive Kapton (suggested by Rui de Oliveira, CERN)

Rui De Oliveira gave us the data sheet of the resistive ink that his group is using to produce resistive surfaces of specific average resistivity with the screen print technique. A silver paste for soldering adapted to this ink is also available from the same company.



EUROPE

DEVELOPMENTAL PRODUCT
POLYMER RESISTOR SERIES **D-RS-12100**

DESCRIPTION: ESL D-RS-12100 Series Polymer Resistors are screen-printable, carbon composition, resistive coatings which offer an economical approach for the production of stable resistors in consumer electronic applications. They are designed for the use on FR-4, Kapton, ceramic or other suitable substrates and can be cured either in a box oven or in a near I.R. belt furnace.

PASTE DATA

VISCOSITY: (Brookfield RVT, ABZ Spindle, 10 rpm, 25.5°C ± 0.5°C)	40 ± 15 Pa·s
THINNER:	ESL# 402

PROCESSING DATA

SCREEN MESH/EMULSION:	200/12.5 microns
CURING CONDITIONS:	
BOX OVEN:	170°C in a well ventilated oven for 2 hours
IR BELT FURNACE:	220°C (Used for calibration)
BELT SPEED:	20 inches / minute (Profile on back of page)
CURED FILM THICKNESS:	12 microns ± 1.5 microns
SHELF LIFE:	6 months
SUBSTRATE:	FR-4 (Used for calibration), Kapton, Ceramic
TERMINATION CONDUCTOR:	1107-S

TYPICAL RESISTOR PROPERTIES

	D-RS 12112	D-RS-12113	D-RS-12114	D-RS-12115	D-RS-12116
Resistance[1]	100Ω/SQ.	1 KΩ/Sq.	10 KΩ/Sq.	100 KΩ/Sq.	MΩ/Sq.
CVAR	10 %	6 %	7 %	8 %	10 %
Resistance Tolerance	± 20 %	± 20 %	± 20 %	± 20 %	± 20 %
TCR (RT to +125°C)	+400 ppm/°C	- 200 ppm/°C	-400 ppm/°C	-700 ppm/°C	-1000 ppm/°C
Humidity Test (40°C / 90% R.H.)					
24 hours	-	≤-1 %	≤-1 %	≤+1%	≤+1.5 %
100 hours	-	≤+1%	≤+1%	≤+1%	≤+1.5%
1000 hours	-	≤+1%	≤+1%	≤+1%	≤+1.5%

[1] 10Ω/Sq. under development.

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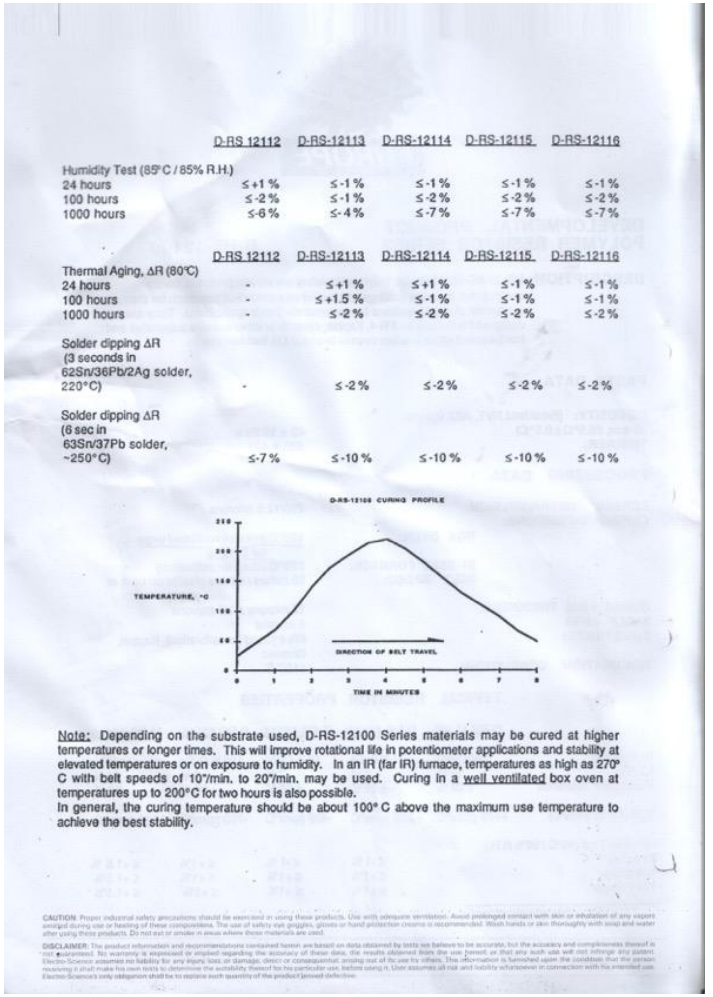


	D-RS 12112	D-RS-12113	D-RS-12114	D-RS-12115	D-RS-12116
Humidity Test (85°C / 85% R.H.)					
24 hours	≤+1 %	≤-1 %	≤-1 %	≤-1 %	≤-1 %
100 hours	≤-2 %	≤-1 %	≤-2 %	≤-2 %	≤-2 %
1000 hours	≤-6 %	≤-4 %	≤-7 %	≤-7 %	≤-7 %

	D-RS 12112	D-RS-12113	D-RS-12114	D-RS-12115	D-RS-12116
Thermal Aging, ΔR (80°C)					
24 hours	-	≤+1 %	≤+1 %	≤-1 %	≤-1 %
100 hours	-	≤+1.5 %	≤-1 %	≤-1 %	≤-1 %
1000 hours	-	≤-2 %	≤-2 %	≤-2 %	≤-2 %
Solder dipping ΔR (3 seconds in 62Sn/36Pb/2Ag solder, 220°C)	-	≤-2 %	≤-2 %	≤-2 %	≤-2 %
Solder dipping ΔR (6 sec in 63Sn/37Pb solder, ~250°C)	≤-7 %	≤-10 %	≤-10 %	≤-10 %	≤-10 %

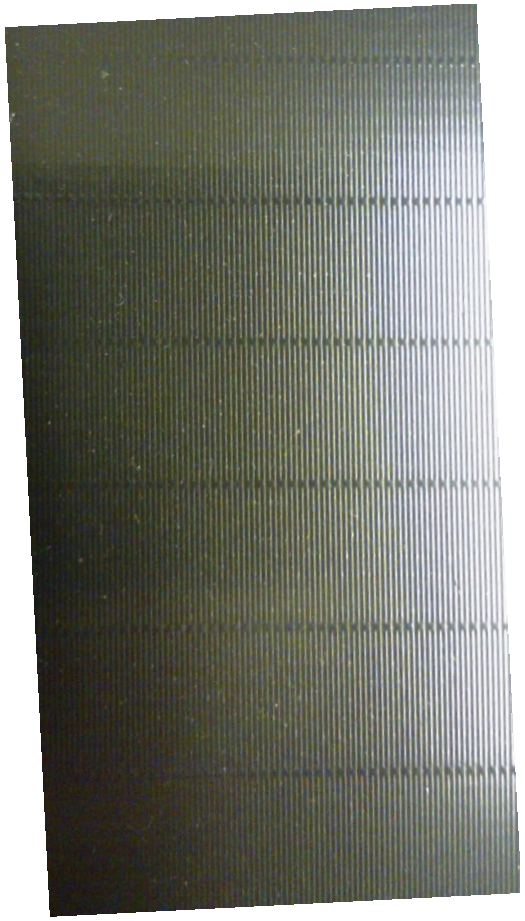
D-RS-12100 CURING PROFILE

Note: Depending on the substrate used, D-RS-12100 Series materials may be cured at higher temperatures or longer times. This will improve rotational life in potentiometer applications and stability at elevated temperatures or on exposure to humidity. In an IR (far IR) furnace, temperatures as high as 270° C with belt speeds of 10"/min. to 20"/min. may be used. Curing in a well ventilated box oven at temperatures up to 200°C for two hours is also possible.
In general, the curing temperature should be about 100° C above the maximum use temperature to achieve the best stability.

CAUTION: Proper industrial safety precautions should be exercised in using these products. Use with adequate ventilation. Avoid prolonged contact with skin or inhalation of any vapors emitted during use or heating of these compositions. The use of safety eye goggles, gloves or hand protection creams is recommended. Wash hands or skin thoroughly with soap and water after using these products. Do not eat or smoke in areas where these materials are used.

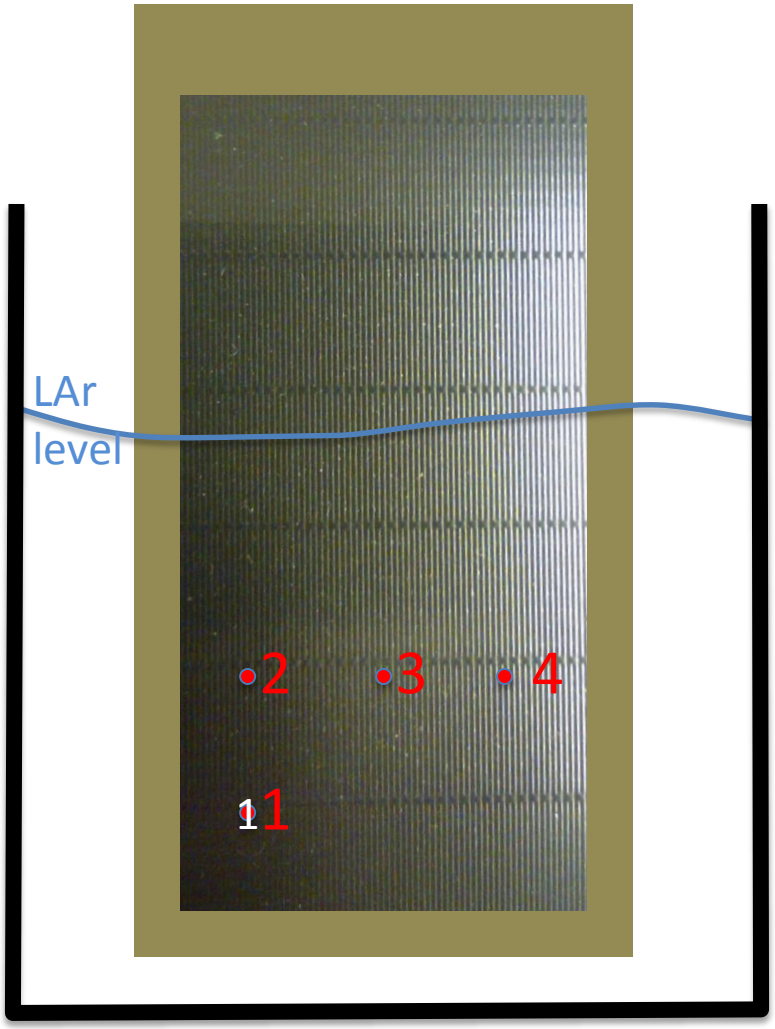
DISCLAIMER: The product information and recommendations contained herein are based on data obtained by tests we believe to be accurate, but the accuracy and completeness thereof is not guaranteed. No warranty is expressed or implied regarding the accuracy of these data, the results obtained from the use hereof, or that any such use will not infringe any patent. Electro-Science assumes no liability for any injury, loss, or damage, direct or consequential, arising out of its use by others. This information is furnished upon the condition that the person receiving it shall make his own tests to determine the suitability thereof for his particular use. Before using it, User assumes all risk and liability whatsoever in connection with his intended use. Electro-Science's only obligation shall be to replace such quantity of the product proved defective.

A sample of FR4 sheet (125x55 cm^2 area, 0.5 mm thick) was deposited with resistive ink (of few MΩ/square) in a parallel strips pattern (~250µm thick ~ 250µm spacing) to increase the average resistivity of the surface. Strips are linked together every ~cm. Given the specific layout of the printing pattern, the resistivity is expected to be different in the direction along the strips and that parallel to them.



Rui also gave us a 15x25 cm^2 sample of the Dupont resistive Kapton (3-4 MΩ*cm bulk resistivity). Thickness is 25 µm, it need to be mounted on support for tests.

Resistivity measurements at room and LAr temperatures



Measurements taken with HP-4329A High Resistance Meter			
V=100V (cross-checks at 50 V and 250 V)			
	Room Temp (25°C)	Cold (Air-GAr, -150°C)	Cold (-175°C)*
1-2	1,5	2,6	3
1-3	3	5	5,4
1-4	6	8	9,5
2-4	6	8	9,5
	Cold (-180°C, Lar waves)	Cold (-180°C, LAr prompt)	Cold (-180°C, LAr quiet)
1-2			
1-3	3,2	4	3,7
1-4	6	\	6
2-4	10	\	9.9
	11	\	10.5
All values are expressed in $10^7\ \Omega$			
*= Point (1) in LAr, point (2), (3), (4) in GAr			

Same resistance values measured at LAr and room temperatures after one night immersion in LAr. No visible damage to the screen-print pattern.

Polymer Resistive foils

DUPONT
The miracles of science™

Constructions

100XC10E7 is our standard offering for anti-static applications. It is a one mil film with a nominal surface resistivity of 5 mega ohm/sq. Two grades are available as described in **Table 2**. Custom constructions are also available, and can be produced in thickness from 1 to 5 mil, and with surface resistances from 90 to 10^{9} ohms/sq.

Table 2
Electrical Properties of Kapton® 100XC10E7 and 100XC10E5 Polyimide Film

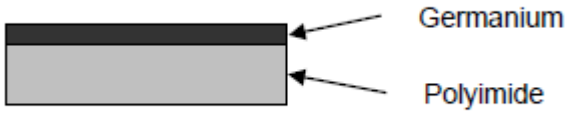
Property	Typical Value	Test Method
Film Type 100XC10E7		
Surface Resistivity Aim, mega ohm/sq.	5	ETS 870 electrometer at 100V
Resistivity Range, avg, mega ohm/sq.	.5-50	
Film Type 100XC10E5		
Surface Resistivity Aim, mega ohm/sq.	5	ETS 870 electrometer at 100V
Resistivity Range, mega ohm/sq.	0.1-1000	

Resistivity measurements on a 25~15 cm2 kapton foil provided by Rui.
Room temperature: 6 MOhm/square
Immersed in LAr: 9 MOhm/square (no change after several days immersion)

Measurements not changing after repeated immersions
Measurements taken with HP-4329A High Resistance Meter
V=100V (cross-checks at 50 V and 250 V)

Thin film

Parameter (independent of film)	Specified Value
Germanium surface resistivity	$\leq 10^9$ Ω/square (typical 10^7 Ω/square)
Transmittance (Kapton HN only)	≤ 0.20
Solar Absorptance (α) Black Kapton Side	0.93 typical
Normal Emittance (ε_N) Black Kapton Side	0.84 typical
Intermittent temperature range	-250° C to 400° C (-420° F to 750° F)
Continuous temperature range	-250° C to 290° C (-420° F to 550° F)
Outgassing: (ASTM – E595)	TML - WVR ≤ 1.0 %; CVCM ≤0.1%



Resistivity measurements on a 40x25 cm2 foil provided by Rui.
Room temperature: 4.5 MOhm/square
Immersed in LAr: 70 MOhm/square (no change after several days immersion)

Measurements not changing after repeated immersions
Measurements taken with HP-4329A High Resistance Meter
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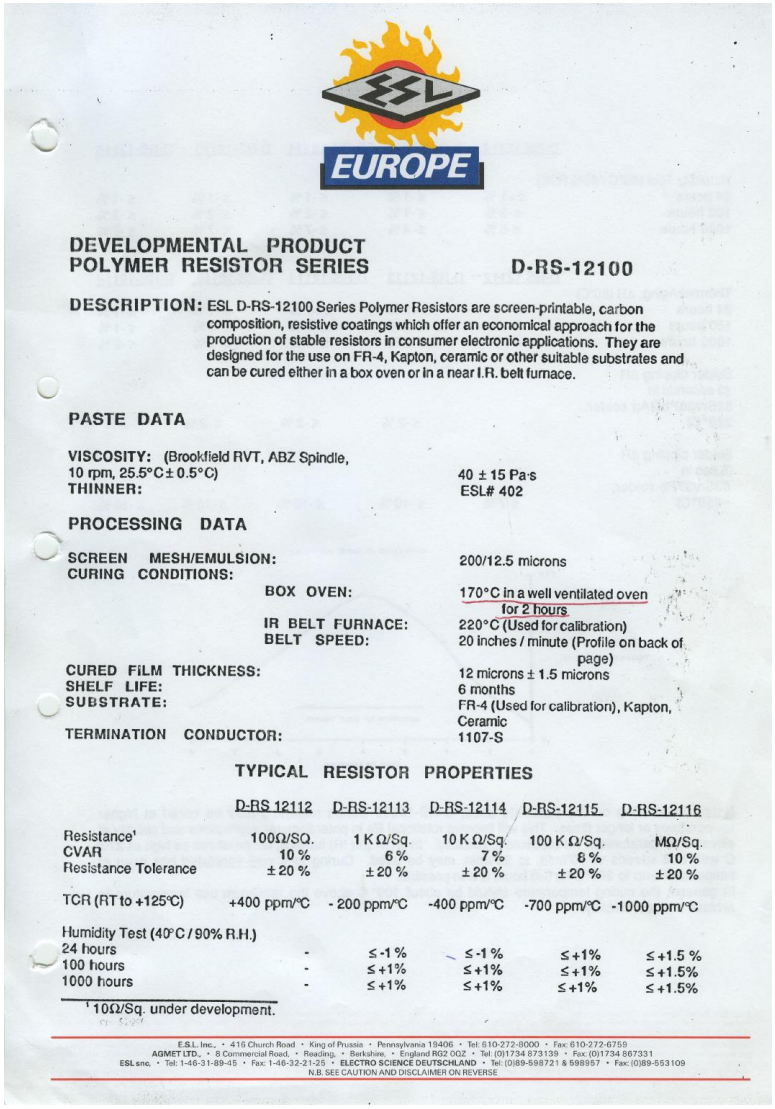
Reminder: resistive ink RS12115 on FR4



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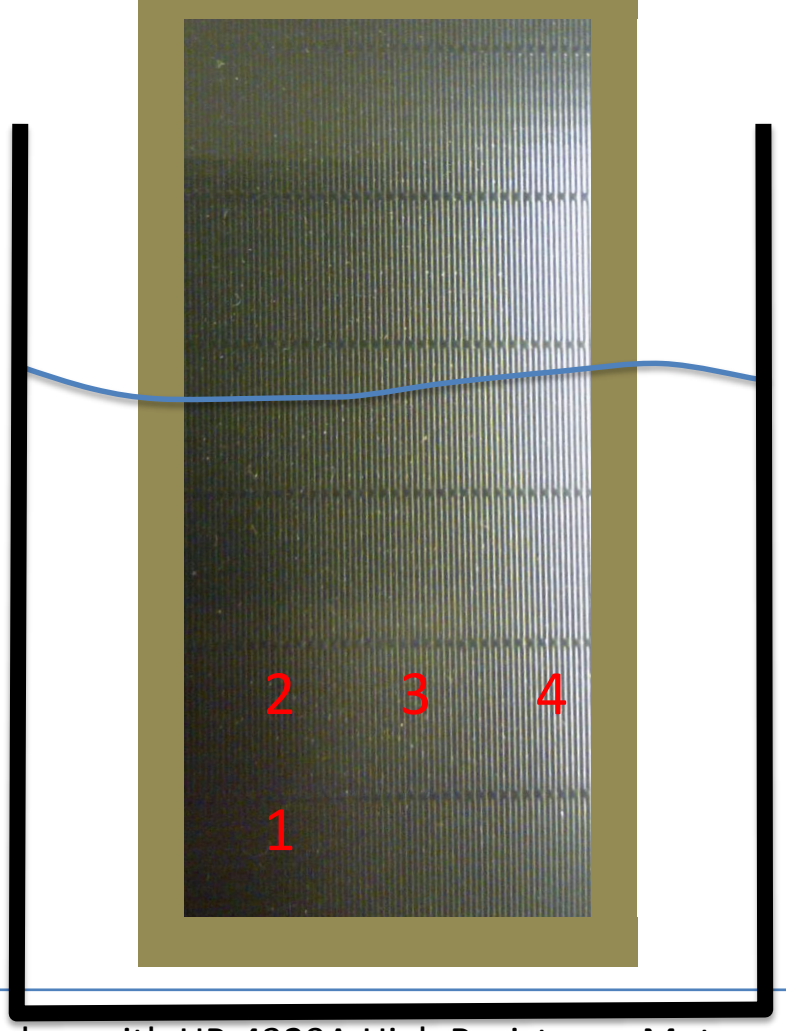

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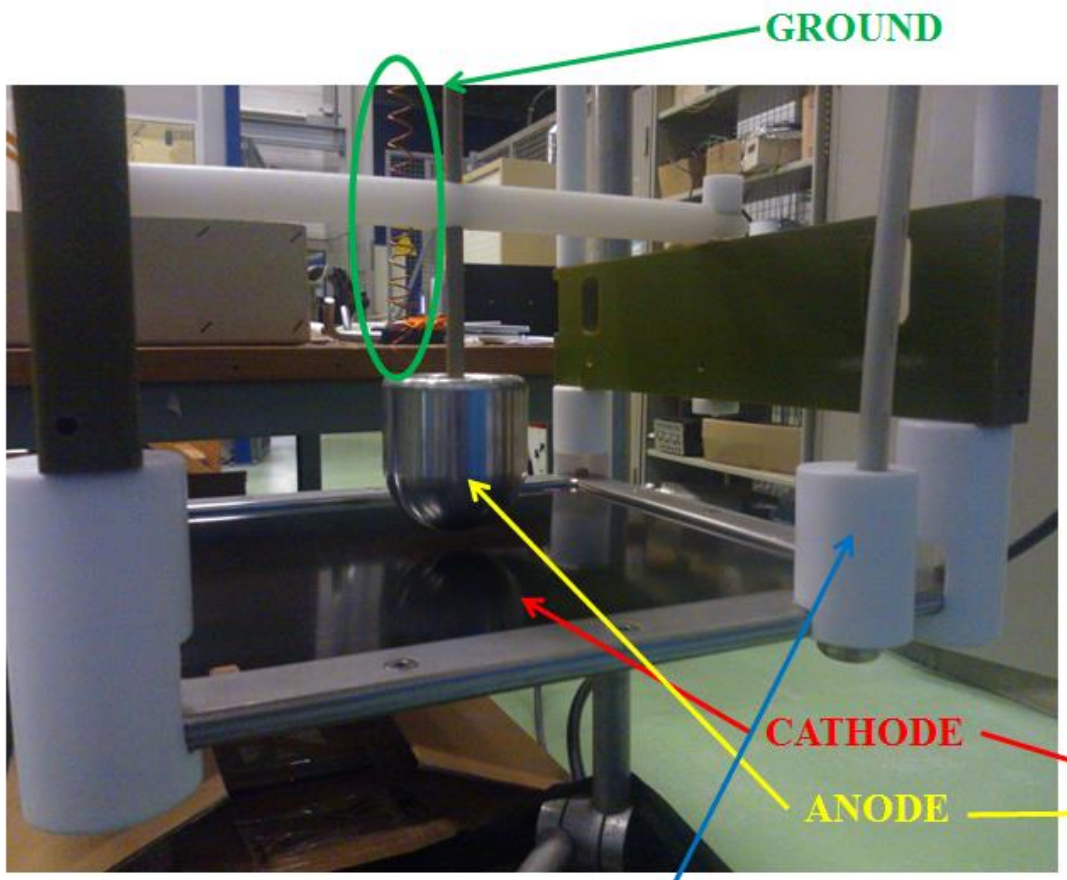
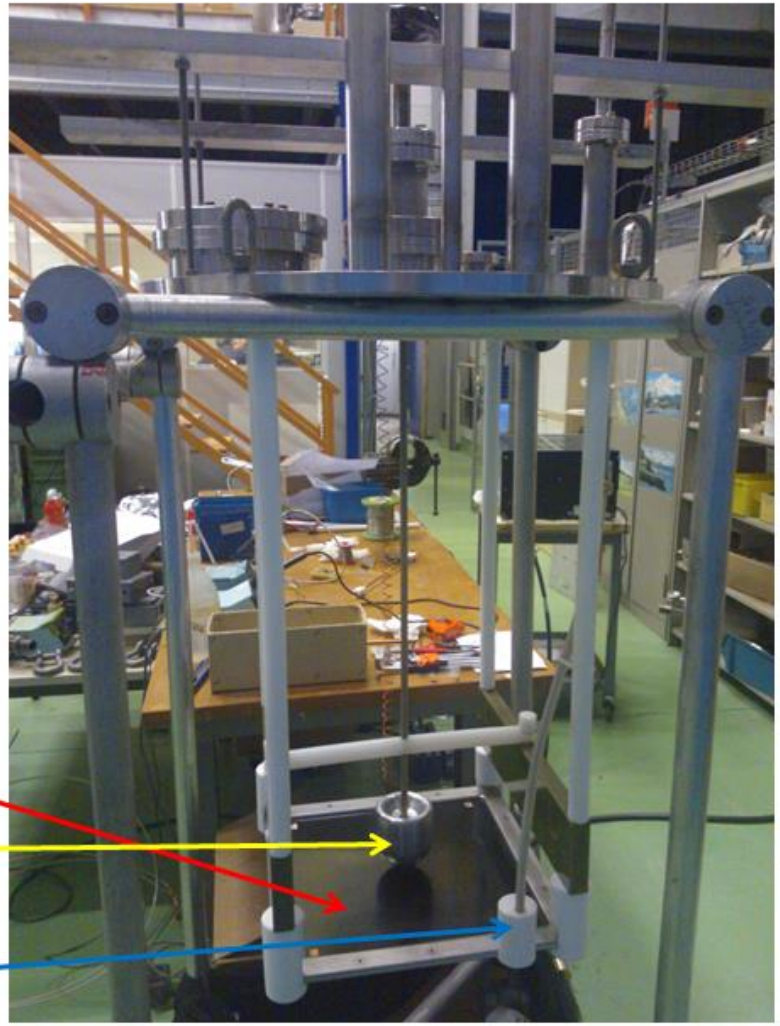
All values are expressed in 10^7 Ohm		

Test setup



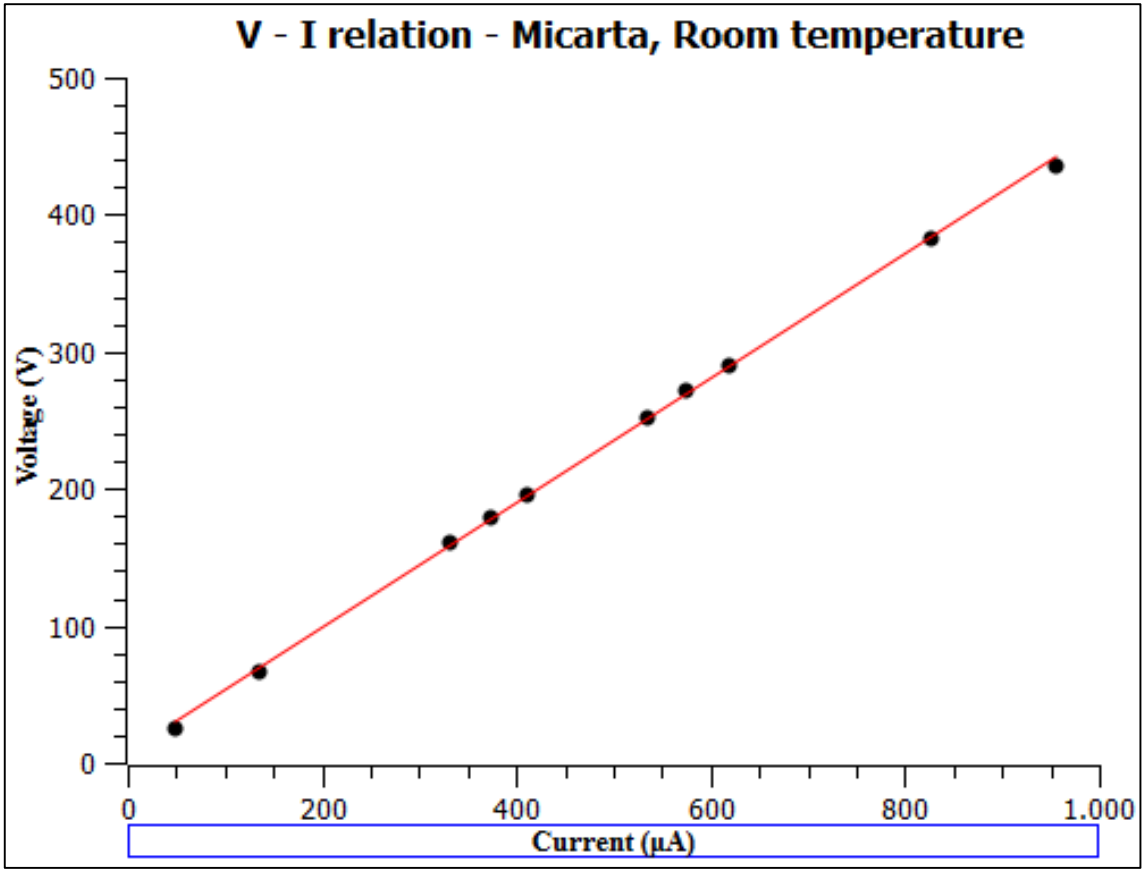


Resistive material is kept in position by SS frame. Connection with a small amount of silver paste. Sustaining structure for cathode plate and anode is in plastics (vetronite, teflon, PEEK).

Set of measurements

- Resistivity measurements on ‘‘ bare’’ material (see previous slides)
- Sparks induction in air
- Sparks induction in LAr
- Resistance measurement with the shown set-up

Micarta - Sparks and R – room temp



Sparks produced at 27.7 kV (1 cm anode-cathode distance) On a second test, sparks at ~ 33 kV/cm. The rate is ~ 1 Hz

Resistance measured by putting in contact anode and cathode (see left plot): good linearity up to 0.5 kV (current out of scale on power supply for higher voltage).
R = 450 kΩ.

Micarta - Sparks and R – LAr temp

- We experienced significant plate bending in LAr: set at 1cm from anode in air, turns out to be at around 2 mm from it in liquid (probably faster shrinking of SS frame).

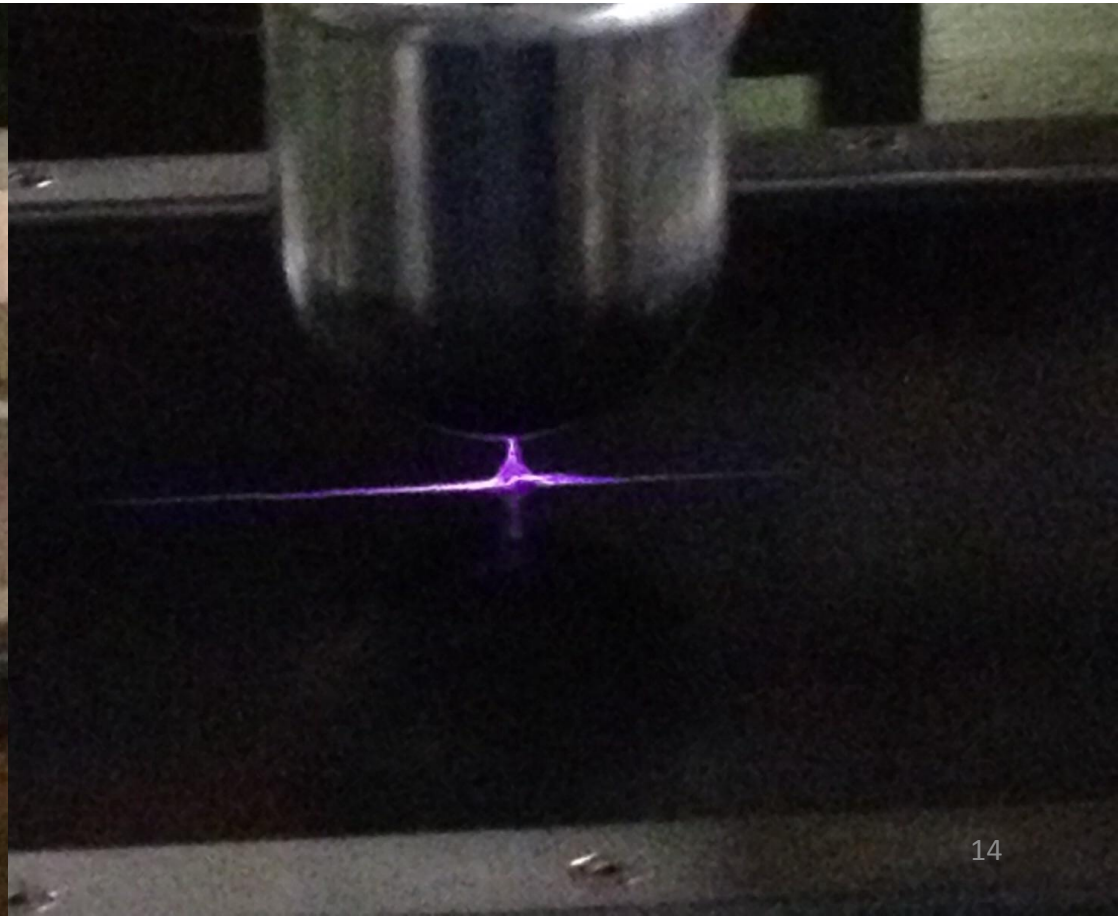
Sparks start at around 60 kV/cm
After sparks start: heating –> production of Ar bubbles. This induces more sparks, even at lower voltage: vicious loop.

- We experience a current flowing between anode and cathode, in liquid (55 kV). Some numbers:

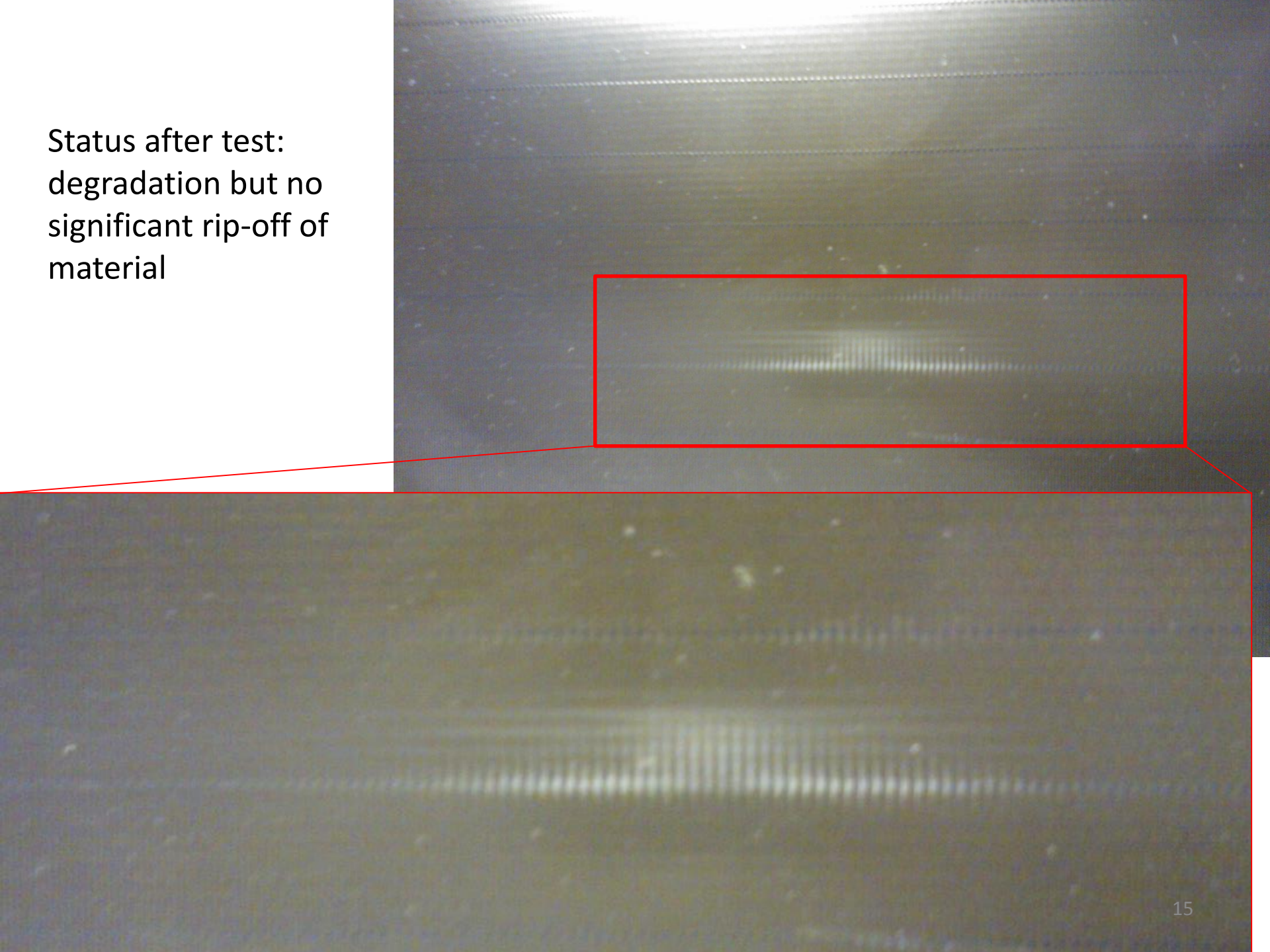
Anode-Cathode distance (mm)	Current (μA)
8	23
9	15
10	11
11	9
….	….
18	1

Ink print – room temperature

Sparks from 42-43 kV/cm, 1-2 Hz rate (depending on current limit)
Spark series alternated to dormant periods
Sparks develop along direction of less resistivity, perpendicular to strip direction

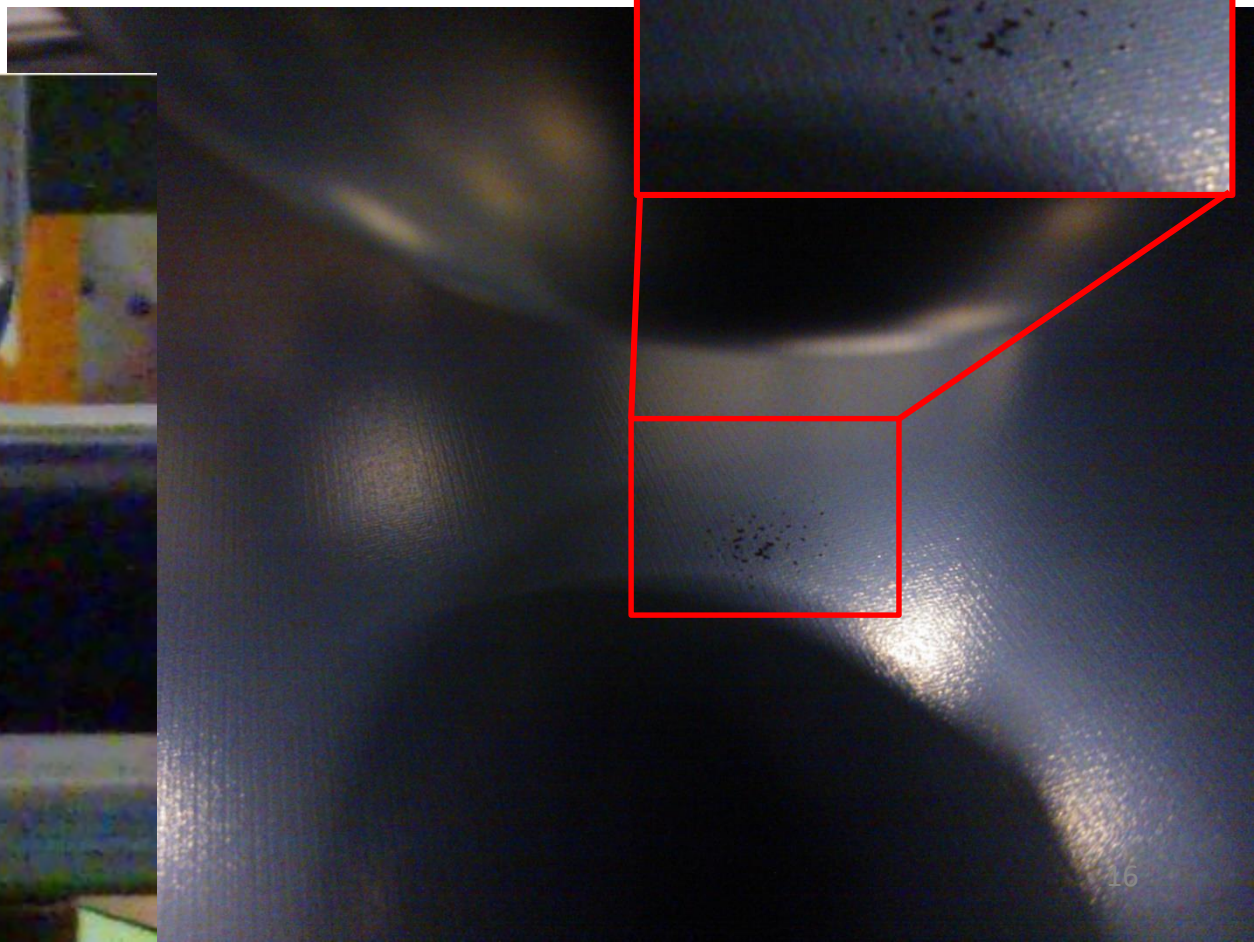


Status after test: degradation but no significant rip-off of material



Resistive kapton – room temperature

Discharges from 45 kV/cm, 1-2 Hz rate (depending on current limit)
Sparks are point-like
‘‘Holes’’ in the material, at the spark position.



Next steps

- Evaluation of behaviour in liquid of Ink print and resistive kapton. Complete ongoign measurements with Micarta.
  - Does the damage produced in air have some effect (lower local resistivity)?

- V – I curve in air and liquid for Ink print and Kapton

- Check of set-up, to avoid bending in liquid

- Operational test with the FLIC set-up ?