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（试验机构 Institution ： DGA)

INVESTIGATION REPORT

A319 cockpit windshield de-icing system

N° 19-DGATA-MTI-P1400227009008-F-A

DIRECTION GÉNÉRALE DE L'ARMEMENT

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A319 cockpit windshield de-icing system

INVESTIGATION REPORT

N° 19-DGATA-MTI-P1400227009008-F-A

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Abstract :

This document presents the results of the investigations conducted on the power braids from the cockpit windshield de-icing system of the Sichuan Airlines A319 n° B-6419 following the loss of the windshield during the 3U8633 flight of May 14th 2018.

Different tests were performed on the power braids:

- tensile test with or without electric current,
- serial arc test in air or in solution,
- parallel arc test in air or in solution,
- immersion in acid solutions,
- heating in furnace at different temperatures.

Scanning electron microscopy examination, energy-dispersive X-ray spectroscopy, thermogravimetric and chemical analysis and ion-exchange chromatography were performed in order to determine the different chemical elements released during the electric tests of the braids and to observe the aspect of the samples after the tests.

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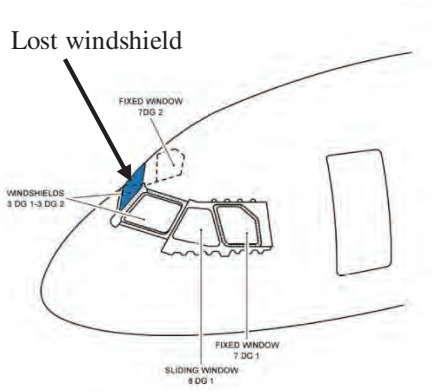
1. INTRODUCTION

On May 14th 2018, a Sichuan Airlines Airbus A319 n° B-6419 lost its cockpit windshield during the flight 3U8633. The windshield has cracked and was sucked out of the cockpit at 32,000 feet.



DGA Aeronautical Systems was enquired to perform investigations on the braids that bring power to the windshield de-icing system. The system is described in Figure 1.

2. REVISIONS RECORD

Version	Date	Revisions
F-A	Approval date	Initial document

3. INVESTIGATIONS OBJECTIVES

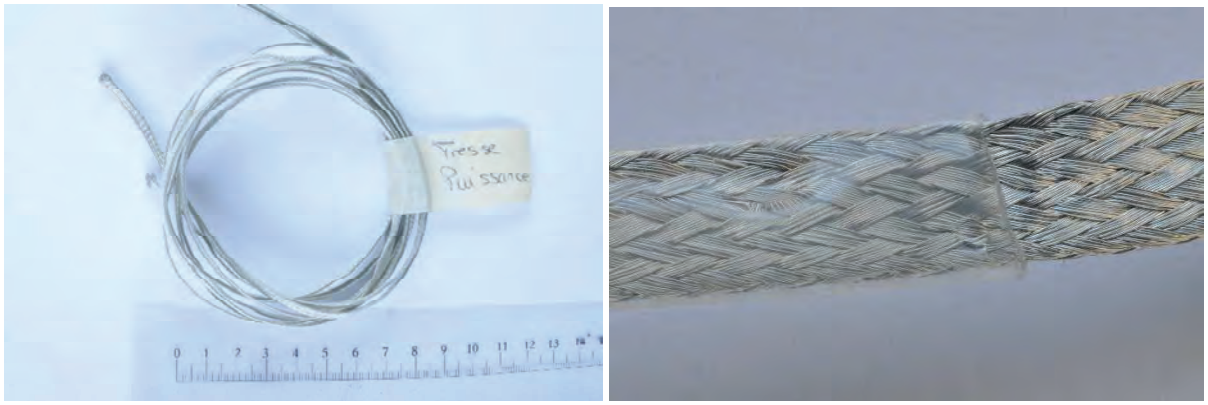
The investigations are performed in order to determine:

- if the degradation of the polymer braid sleeve and the polysulfide seal could create fluorine and sulfur ions leading to the acidification of the braid environment,
- the minimum section of the braid required to withstand the electric current delivered by the power supply of the de-icing system,
- the aspect of a braid subject to mechanical failure or thermal electric failure,
- if the heating of the braid could lead to the increase the copper wire grain size,

- if a serial arc in the power braid or a parallel arc between the power braid and the Zbar could occur.

4. DESCRIPTION OF THE TEST SPECIMEN

The power braid is constituted of 320 copper wires coated with tin, wrapped in a polymer sleeve.



Pictures of the power braid

5. ANALYSIS METHODS

The micrographic examination performed on the wires to determine the copper grain size was performed after immersion in a chemical etching solution composed of:

- 25 mL of NH_4OH,
- 50 mL of $(NH_4)_2S_2O_8$ at 2.5%,
- 25 mL of distilled water.

All the grain sizes values are given on an indicative basis for comparison between the different samples.

6. RESULTS

6.1. THERMOGRAVIMETRIC AND CHEMICAL ANALYSIS

The braid sleeve and the polysulfur seal PR 1829 were subject to thermogravimetric and chemical analysis. The results show that the braid sleeve is composed of polyvinylidene fluoride (PVDF). Its thermal degradation starts at 350°C and ends at 480°C. All the carbon is vaporized.

Some scientific publications show that thermal degradation of PVDF can lead to the creation of hydrofluoric acid and acetic acid.

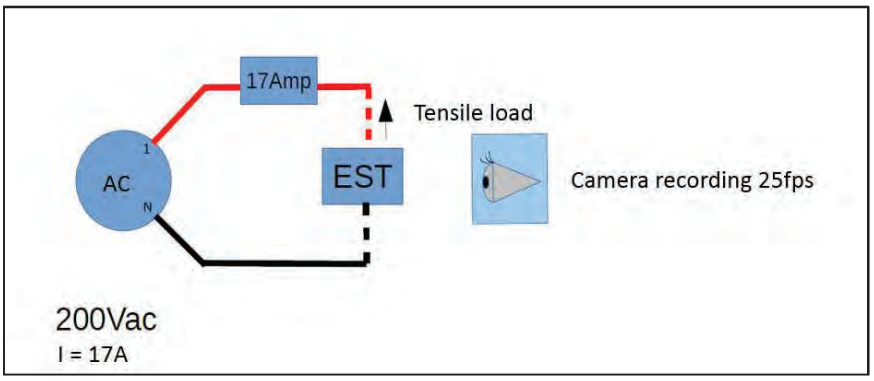
The PR 1829 is a polythioether polymer that contains sulfur. Its thermal degradation starts at 250°C and ends at 375°C. It leads to the creation of calcium carbonate $CaCO_3$.

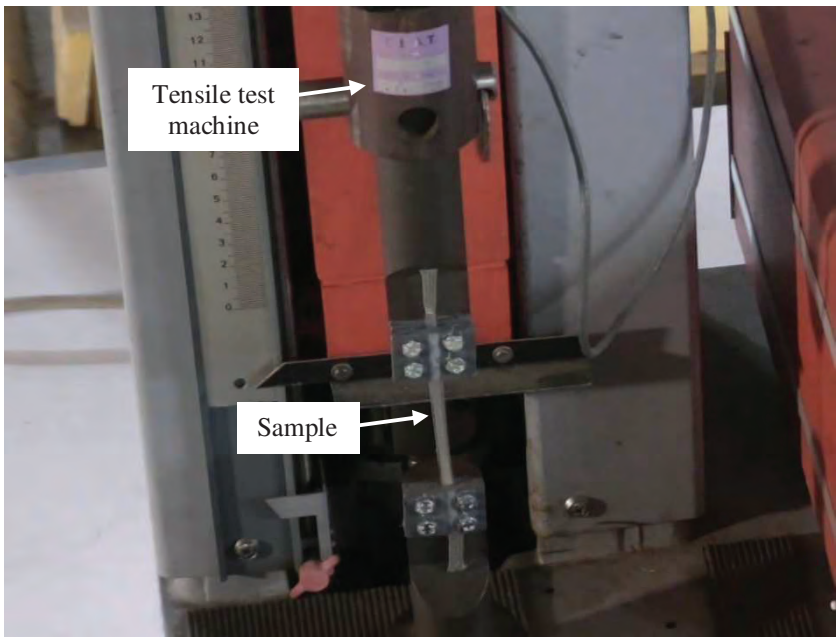
6.2. TENSILE TEST

6.2.1. EXPERIMENT

The power braid was subjected to a controlled tension until failure. The tension was applied to the braid at a speed of 1mm/min.

2 tests were performed: one without electric current (sample T1), one with electric current of 200VAC, 17A (sample T3).





Experimental setup

Both samples broke at a tensile load of about 30kg.

On the test with electric current T3, a serial arc occurred at failure (Figure 2). On the broken sample T3, some wires exhibit a melted aspect due to heating of the copper induced by electric current (Figure 3).

6.2.2. FRACTOGRAPHIC EXAMINATION

The T1 sample failure zone exhibits some dimples representative of a ductile fracture. Some cracks are visible on the tin coating close and far from the failure zone (Figure 4).

On T3 sample, some wires have a fracture surface similar to the T1 sample, and the other ones are melted (Figure 5). A chemical analysis by EDS shows that the melted surface is composed of copper and tin from the coating. The presence of oxygen indicates that some oxide has formed during the process.

6.2.3. MICROGRAPHIC EXAMINATION

The copper grain size was measured on the wires in two locations, close to the failure zone and far from it in an unaffected zone.

Sample	T1	T3
Grain size in failure zone	3.4µm	3.3µm
Grain size in unaffected zone	3.8µm	2.8µm

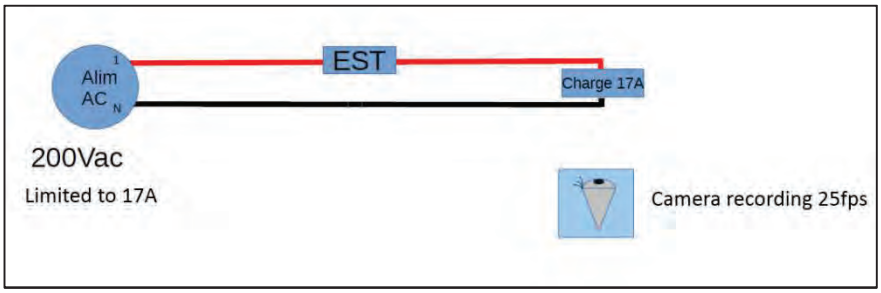
On the T1 sample, no significant difference in grain size is visible between the failure zone and the unaffected zone (Figure 6).

The T3 sample exhibits the same results, as the grain size is similar in both zones (Figure 7). It seems that the heating due to one serial arc is not long enough to allow grain growth.

6.3. SERIAL ARC TEST IN AIR

6.3.1. EXPERIMENT

The power braid was cut in order to let a few percent of its section intact (6%, 4% or 2% of section remaining). The two extremities of the wires that had been cut were bent apart in order not to be in contact. The sleeve was not removed. An electric current was applied to the braid (200VAC, 17A). The test was performed until serial arcing occurred. If nothing happened after 1 hour, the test was stopped.



Experimental setup

3 samples were tested:

- SS1 sample: 6% of section remaining,
- SS2 sample: 4% of section remaining,
- SS4 sample: 2% of section remaining.

During the test with SS1 sample, no arcing occurred. A slight dark coloration of the remaining wires is visible (Figure 8).

The SS2 sample exhibits an important dark coloration but no arcing happened (Figure 9).

With the SS4 sample, the power braid started to glow red until a serial arc occurred (Figure 10). Some wires exhibit a melted aspect due to the serial arc (Figure 11).

6.3.2. FRACTOGRAPHIC EXAMINATION

The chemical analyses performed on the heated wires of samples SS1 and SS2 reveal that they are still covered with tin, but the oxygen percentage has increased, meaning that some oxide was formed on the surface (Figures 12 and 13).

The SS4 sample is characterized by melted wires (Figure 14). Some fluorine is visible on the wires, probably a consequence of the thermal degradation of the PVDF sleeve of the braid during heating or arcing.

6.3.3. MICROGRAPHIC EXAMINATION

The grain size measurements show no significant growth happened during the test on SS1 and SS2 samples, with values about 3µm (Figures 15 and 16). However, some wires on the SS4 sample exhibit an important increase in grain size with a value of 12µm (Figure 17). It is correct to conclude that, depending on the remaining section of the braid, the Joule effect could be important enough to heat the wires in which the current goes through and provoke an increase of grain size.

Sample	SS1	SS2	SS4
Grain size in failure/heated zone	3.0µm	4.0µm	12.6µm
Grain size in unaffected zone	3.0µm	3.0µm	3.1µm

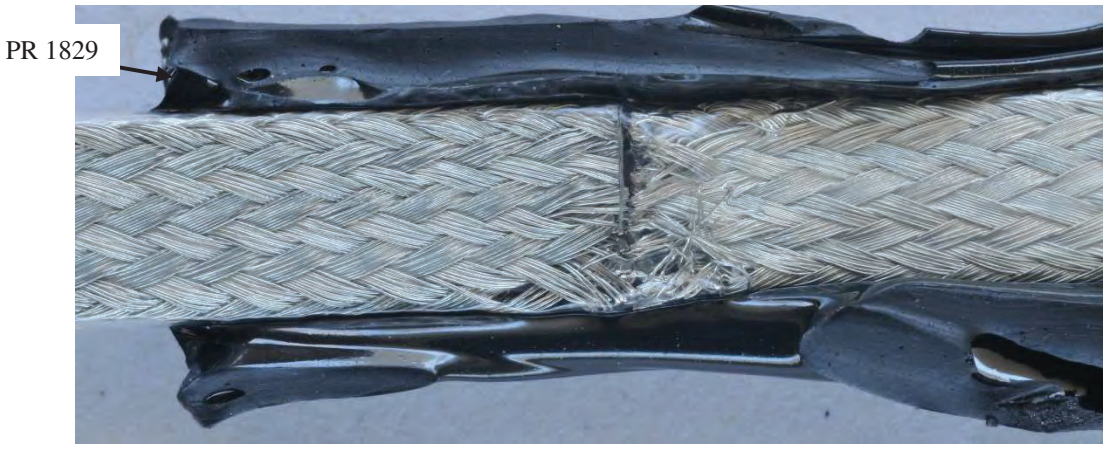
6.4. SERIAL ARC TEST IN SOLUTION

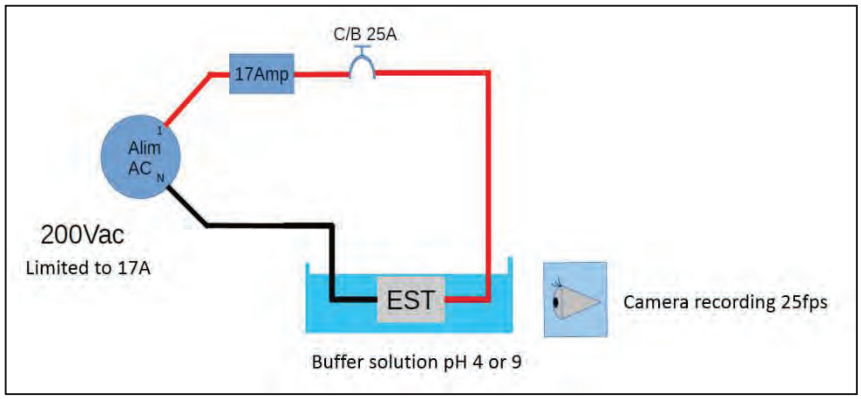
6.4.1. EXPERIMENT

The power braid is cut in order to let a few percent of its section intact (6%, 4% or 2% of section remaining). A cut is then made on the sleeve to allow liquid to get into the sleeve and come in contact with the wires. The sample is immerged into a buffer solution (pH 4 or 9). An electric current is applied to the braid (200VAC, 17A). The test is conducted for a few hours until serial arcing occurs. If nothing happens after 5 hours, the test is stopped. pH measurements were performed at the end of the test, and 1 mL of solution was taken to be analyzed by ion-exchange chromatography.

2 kinds of samples are tested:

- the braid with its sleeve only,
- the braid with its sleeve coated on one of its sides with PR1829 (see following picture).





Experimental setup

The pH 4 solution is made of :

- 50 mL of potassium hydrogen phthalate (0.1 mol/L),
- 50 mL of distilled water.

The pH 9 solution is made of :

- 50 mL of Borax (0.025 mol/L),
- 4.6 mL of HCl (0.1 mol/L),
- 45.4 mL of distilled water.

Before any test:

- the two solutions were analyzed by ion-exchange chromatography (Figures 18 and 19). The pH 4 solution shows a large peak at 7 minutes which cannot be attributed to any particular chemical species because of its large width. The pH 9 solution exhibits an important peak at 4 min which corresponds to the chlorine ion,
- pieces of power braid were immerged for a few hours into the pH 4 and pH 9 solutions. The solutions were then analyzed by ion-exchange chromatography (Figures 20 and 21). The results show that in the pH 4 solution, a small quantity of fluorine is released, probably from the sleeve. In pH 9 solution, no element is released in significant quantity.

The following chart presents the results of the different tests performed and a description of the samples. On some samples, the cut wires were bent apart in order not to be in contact. The results of the ion-exchange chromatography analyses made on the solution in which the samples were immersed are presented in figures 28 to 32.

Test n°	Remaining section of braid	PR1829	Type of sample	Observations	pH	Acidification of the solution	Release of Fluorine/Sulfur
HS1	6%	No	Cut wires in contact	No heating of the solution	4	No	No
HS2	2%	No	Cut wires in contact	Gas formation, heating of solution	4	No	No
HS3	2%	No	Cut wires not in contact	Gas formation, heating of solution, serial arcs occurring	4	No	Fluorine (33 mg/L) Maybe sulfur
HS4	4%	No	Cut wires in contact	Light heating of the solution	9	Light acidification (pH 8.90 to 8.74)	No
HS5	2%	Yes	Cut wires in contact	Gas formation, heating of solution, serial arcs occurring	9	Light acidification (pH 8.90 to 8.77)	No
HS6	2%	Yes	Cut wires not in contact	Gas formation, heating of solution	4	No	Fluorine (80 mg/L)

The results show that only tests performed with a remaining section of the braid of 2% had led to the creation of a serial arc. On tests with 4% of section remaining, a heating of the solution and some gas formation were observed, probably water steam.

The acidification of the solution was observed with tests conducted in the pH 9 solution. Fluorine was released during the tests with the samples HS3 and HS6, and potentially sulfur during HS3, but the exact quantity cannot be determined.

All the arcs observed lasted less than half a second.

6.4.2. FRACTOGRAPHIC EXAMINATION

The fractographic examinations are presented on figures 34 to 38.

Globally, all the samples exhibit traces of oxygen, probably as oxide. Elements such as fluorine and sulfur are not detected in significant quantities.

6.4.3. MICROGRAPHIC EXAMINATION

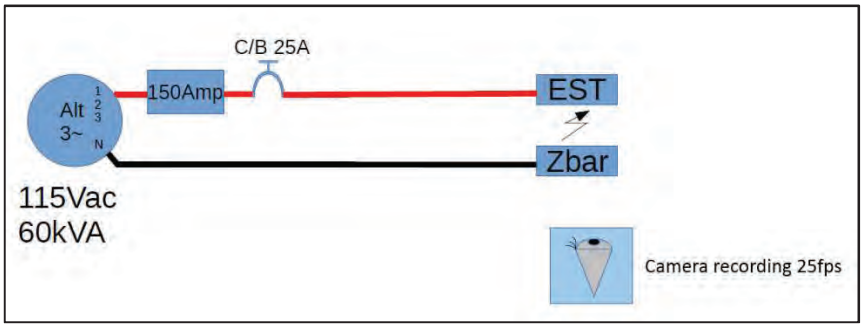
The grain size measurements performed on the different samples show that a slight growth has occurred on HS2 and HS3 samples. The growth was very important on the HS5 sample (Figures 39 to 44).

Sample	HS1	HS2	HS3	HS4	HS5	HS6
Grain size in failure/heated zone	2.3µm	5.2µm	5.2µm	2.9µm	11.6µm	3.8µm
Grain size in unaffected zone	1.9µm	2.7µm	3.0µm	2.6µm	4.0µm	4.0µm

6.5. PARALLEL ARC TEST IN AIR

6.5.1. EXPERIMENT

The power braid (sample SP1) and the Zbar are set facing each other. The electric current is limited to 150A (limit value of possible current on aircraft). The tension is applied to the setup described here after. The braid is then drawn closer to the Zbar until parallel arcing occurs (Figure 45). The test is then stopped.



Experimental setup

The intensity going through the braid during parallel arcing was measured at 123A.

6.5.2. FRACTOGRAPHIC EXAMINATION

The figure 46 shows that the braid surface which was in contact with the Zbar is defined by a melted aspect, mainly composed of copper. Almost no oxide has formed during the arc.

6.5.3. MICROGRAPHIC EXAMINATION

The grain size measurements show that a slight growth of the copper grains has occurred (Figure 47).

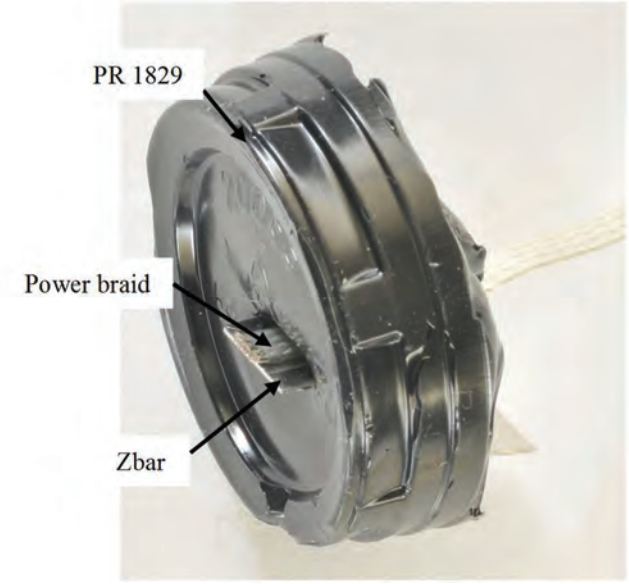
Sample	SP1
Grain size in failure/heated zone	4.7µm
Grain size in unaffected zone	3.5µm

6.6. PARALLEL ARC TEST IN SOLUTION

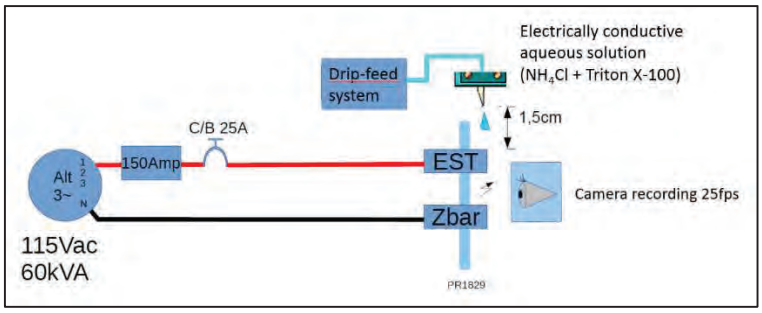
6.6.1. EXPERIMENT

The power braid and the Zbar are wrapped into PR1829 as shown on the following picture (sample HP2). An electric current limited to 150A is applied to the braid. Electrically conductive solution is then poured by a drip-feed system on the surface of the Zbar and the braid, allowing

parallel arcing to occur when a drop comes in contact with both braid and Zbar (Figure 48). The test is conducted for 5 hours.



Sample used for parallel arc test



Experimental setup

The electrically conductive solution is composed of 2% of NH_4Cl and 0.1% of Triton X-100 in distilled water.

The current intensity going through the braid during parallel arcs was measured between 86 and 133A. All the arcs lasted around a few milliseconds.

6.6.2. FRACTOGRAPHIC EXAMINATION

The EDS analysis shows that elements such as oxygen and chlorine are present on the surface of the braid in quantities above 10%, but sulfur and fluorine are found in very small quantities, about 1% (Figure 49). This test is very damaging for the PVDF sleeve and the PR 1829 because of the important number of parallel arcs occurring.

6.6.3. MICROGRAPHIC EXAMINATION

The grain size close to the heated zone is much bigger than in the unaffected zone (Figure 50). It could be explained by the numerous parallel arcs occurring that led to an important increase of the temperature of the braid.

Sample	HP2
Grain size in Failure/heated zone	13.6µm
Grain size in unaffected zone	2.6µm

6.7. IMMERSION IN ACID SOLUTIONS

3 samples of power braid without sleeve were immersed for 72 hours into solutions of bromic acid HBr, hydrofluoric acid HF and sulfuric acid H_2SO_4 at the concentration of 0.1 mol/L. The 3 samples exhibit different colors after immersion (Figure 51).

Samples immersed from left to right in HBr - HF - H_2SO_4

6.7.1. SCANNING ELECTRON MICROSCOPY EXAMINATION

The results show that with the HBr solution, the tin coating is still present but is characterized by corrosion pits (Figure 52).

The sample immersed into HF has almost no tin coating remaining and the copper is corroded (Figure 53).

The impact of H_2SO_4 is minor, considering that the tin coating is still almost intact on the surface of the copper wires (Figure 54).

6.7.2. MICROGRAPHIC EXAMINATION

The micrographic examination results show that with HBr solution, the surface of the copper wires is corroded, but the tin coating is still mainly present (Figure 55).

With the HF solution, almost all the tin coating is dissolved and the copper exhibits a light corrosion.

With the H_2SO_4 solution, the wires and the coating seem unaffected.

6.8. HEATING TEST

3 samples of power braid were heated in a furnace for 30 min at temperatures of 600°C, 800°C or 1000°C. The pictures of the 3 braids are presented here after. They all exhibit a tarnished aspect due to the formation of oxide in surface.

Samples heated from left to right to 600°C – 800°C – 1000°C

6.8.1. SCANNING ELECTRON MICROSCOPY EXAMINATION

Figures 56 to 58 shows the different aspects of oxides existing on the surface of the wires.

At 600°C, the tin coating is still present on all the surface of the wire, as an oxide.

At 800°C, the oxide seems to have coalesce from all the wires, making a homogeneous layer. This oxide does not contain tin, it is only composed of copper and oxygen.

At 1000°C, the oxide seems to have grown thicker than at 800°C.

6.8.2. MICROGRAPHIC EXAMINATION

The braid heated at 600°C is characterized by a grain size of about 11.5µm (Figure 59).

The wires heated at 800°C exhibits a thick layer of oxide surrounding the copper core. The grain size cannot be determined because of the too small size of the remaining copper phase (Figure 60).

The sample heated at 1000°C is only composed of copper oxide (Figure 61).

7. CONCLUSIONS

The tests performed on the power braid show that:

- the thermal degradation of the braid sleeve can lead to the release of fluorine, potentially as hydrofluoric acid,
- the thermal degradation of PR1829 can lead to the release of sulfur,
- the acidification of the environment of the braid has been identified during serial arc testing in immersion,
- the heating of braid because of electric current can lead to copper grain growth and to creation of oxide on the surface of the wires,
- a serial arc can be obtained on a braid with 2% of section remaining,
- fractographic examinations confirm that braids that were subject to serial or parallel arcing exhibit a melted aspect.

- FIGURES -

Figure 1 : Description of the windshield de-icing system

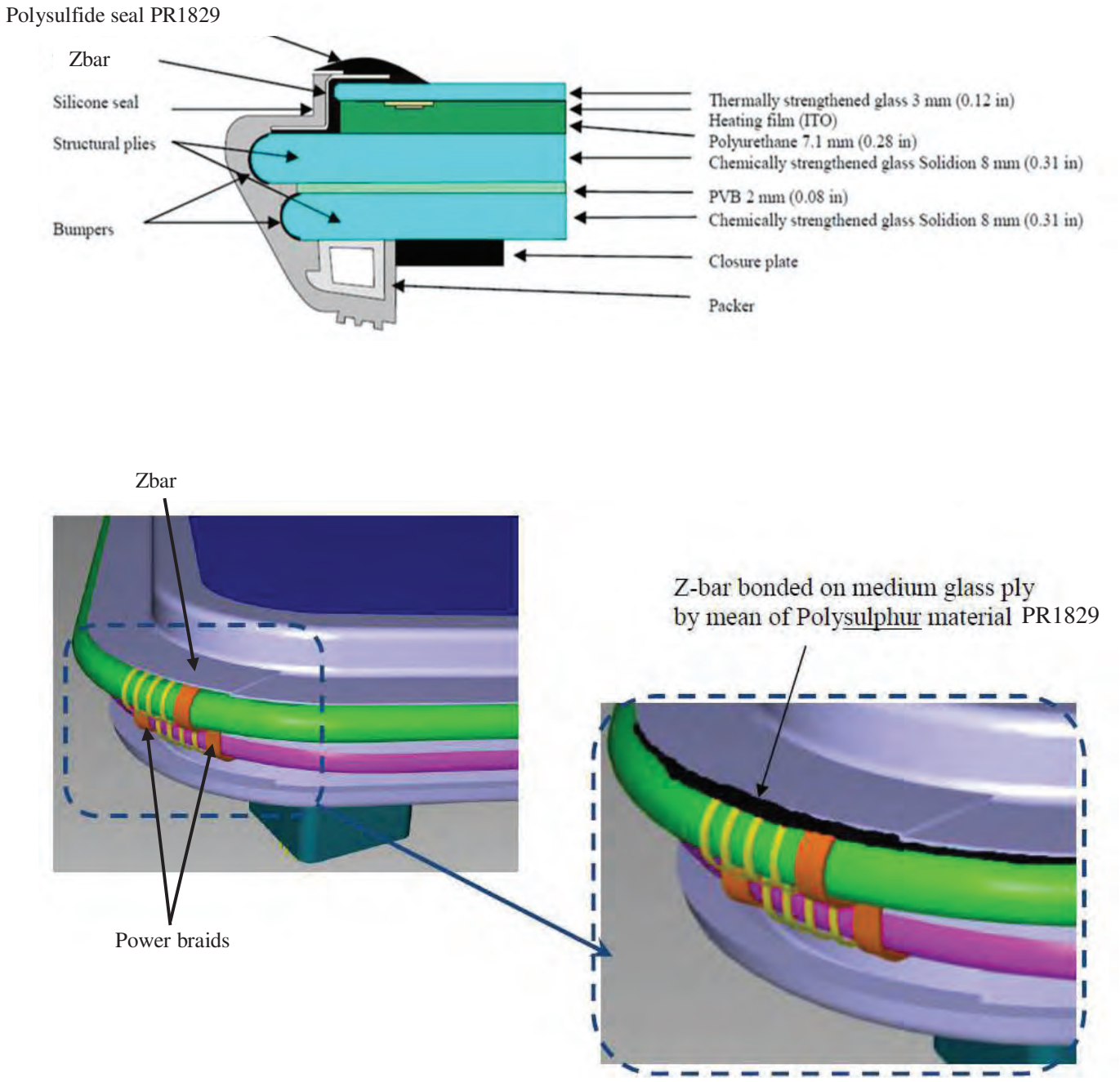
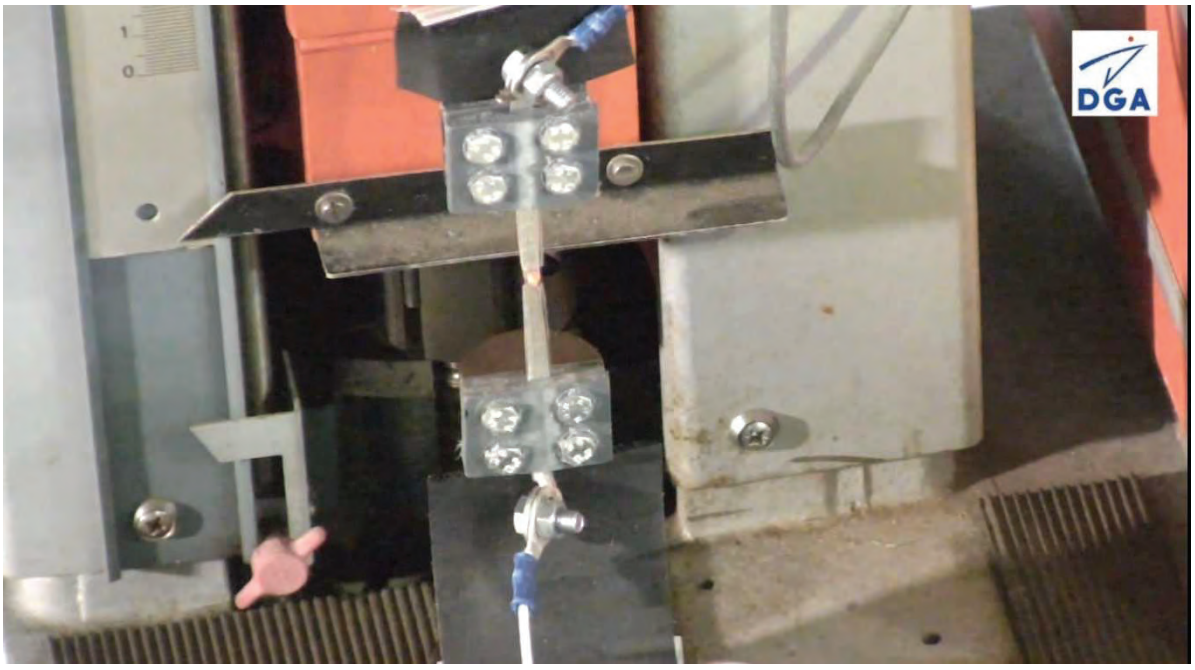
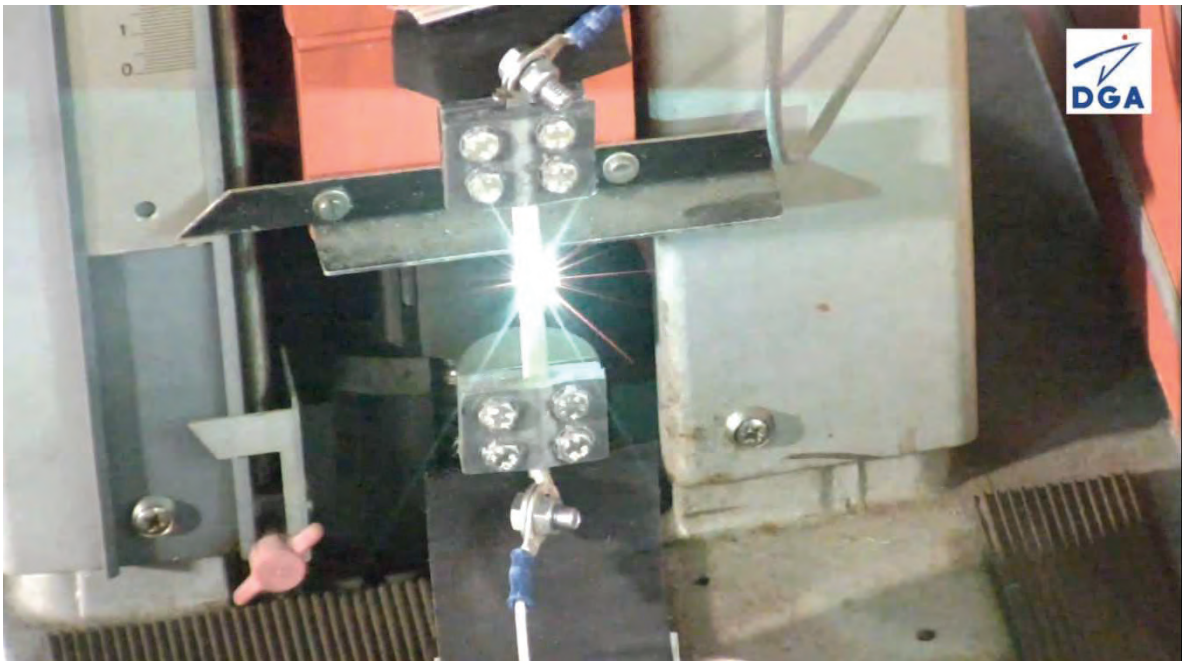


Figure 2 : Tensile test results

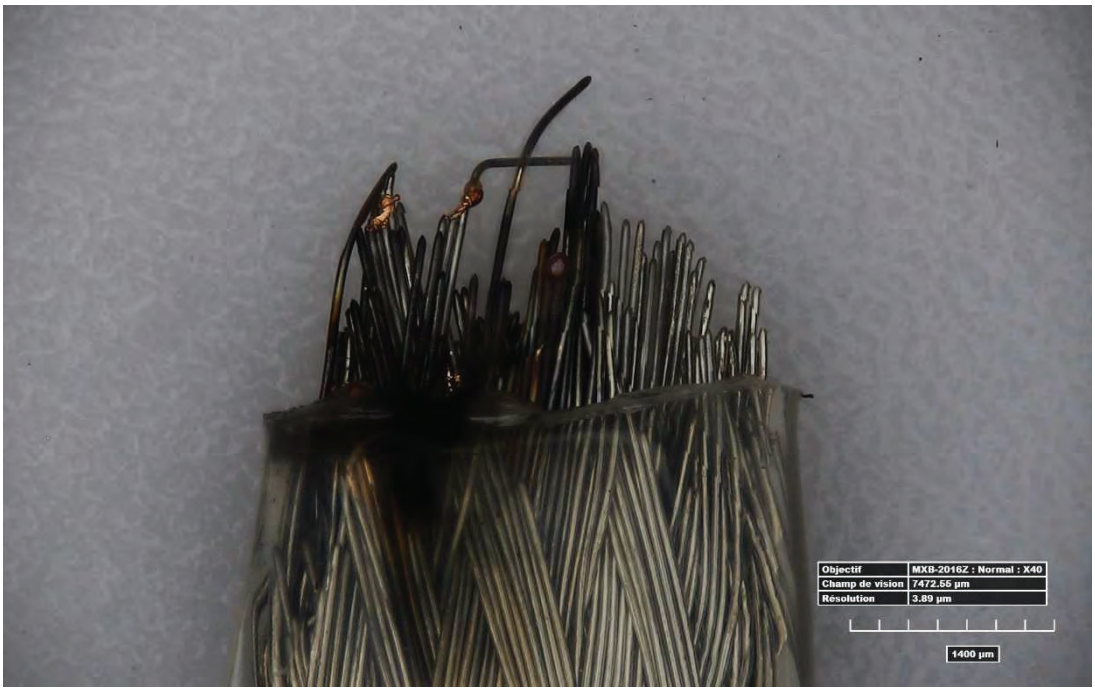


Red glowing of the braid because of electric current – T3 sample



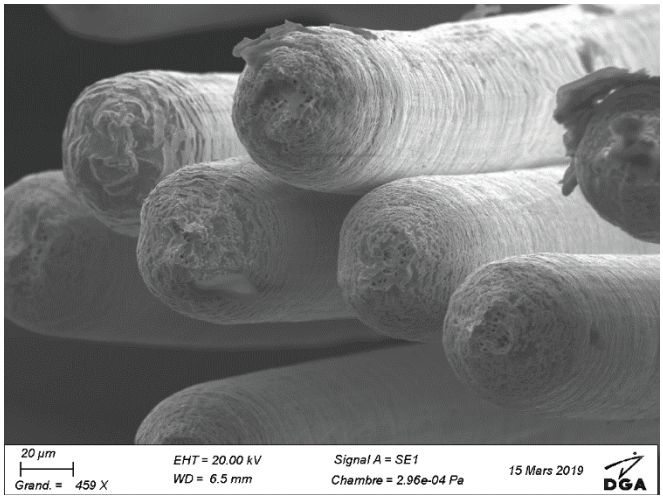
Serial arcing at failure - T3 sample

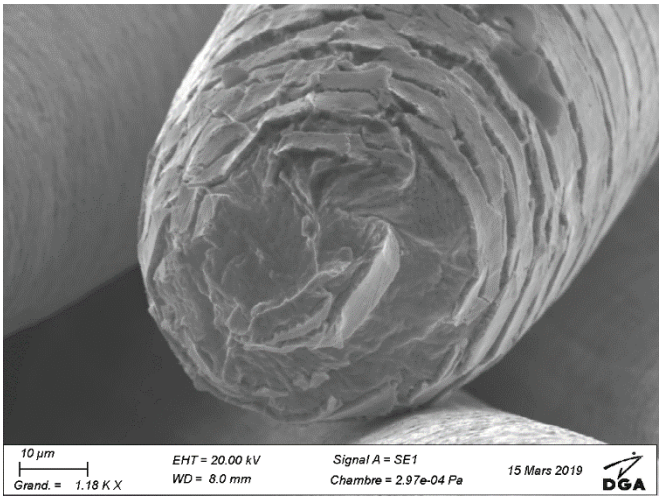
Figure 3 : Tensile test sample T3 pictures

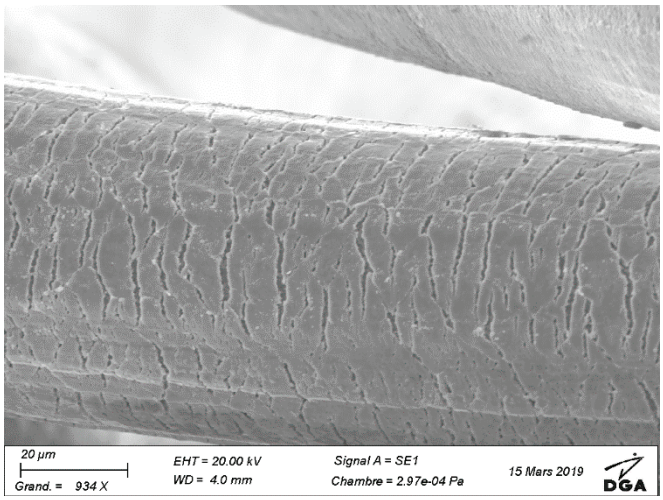


Sample T3 after failure

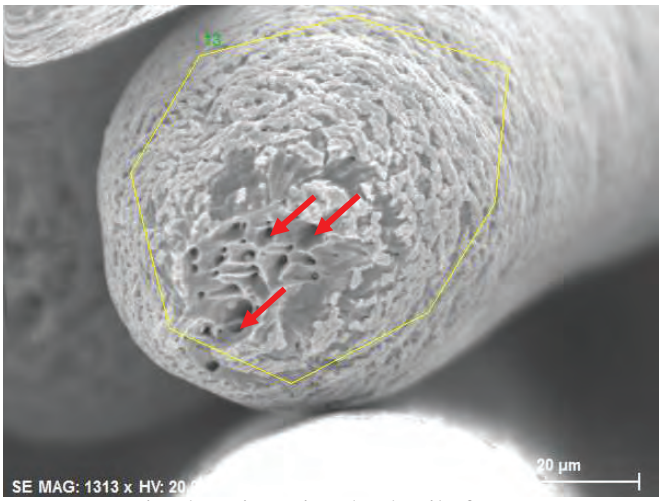
Figure 4 : Tensile test sample T1 – Fractographic examination



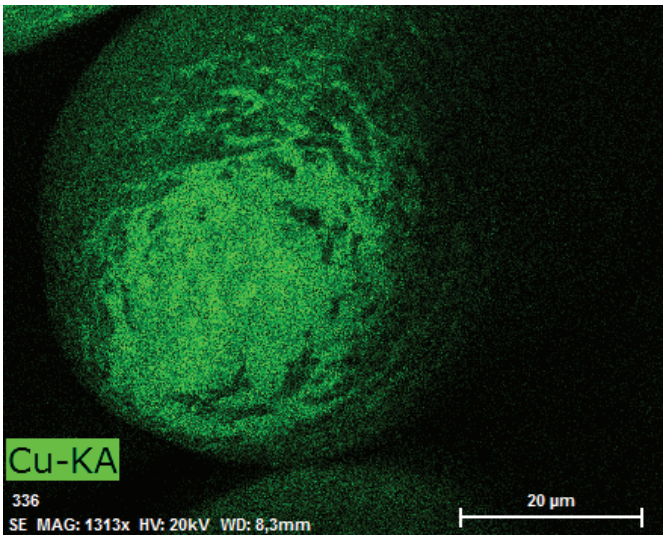




Cracks on tin coating



Dimples witnessing the ductile fracture



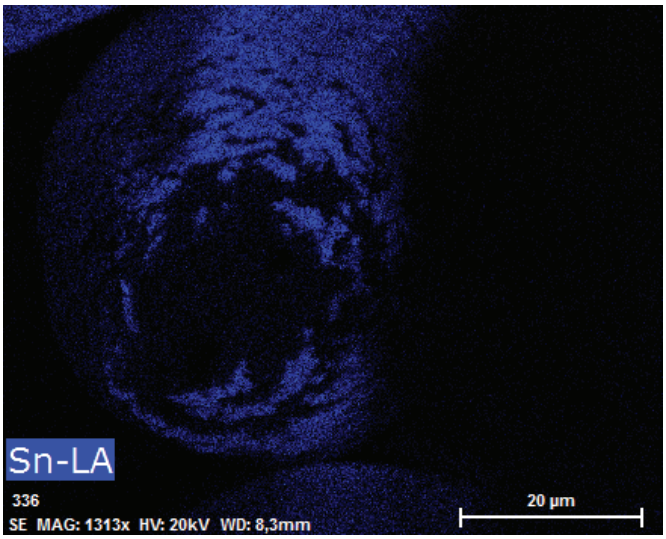
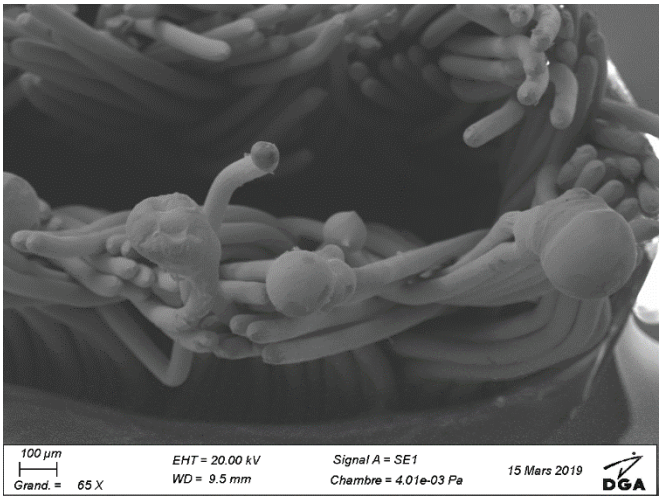
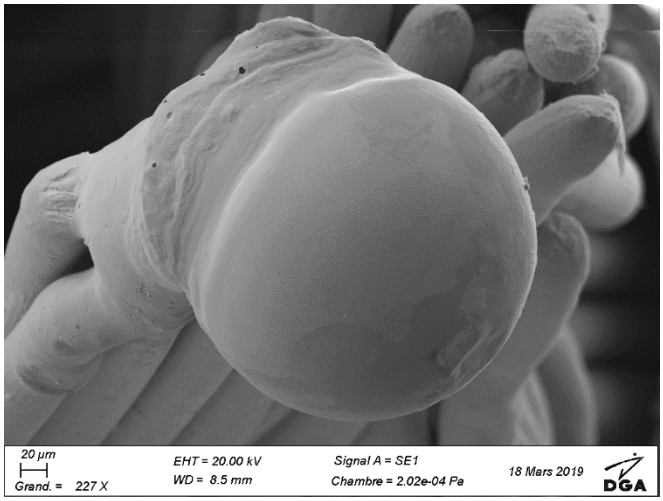
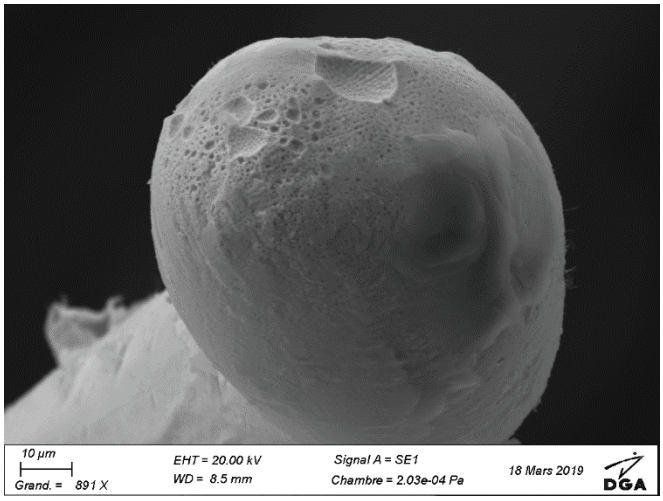
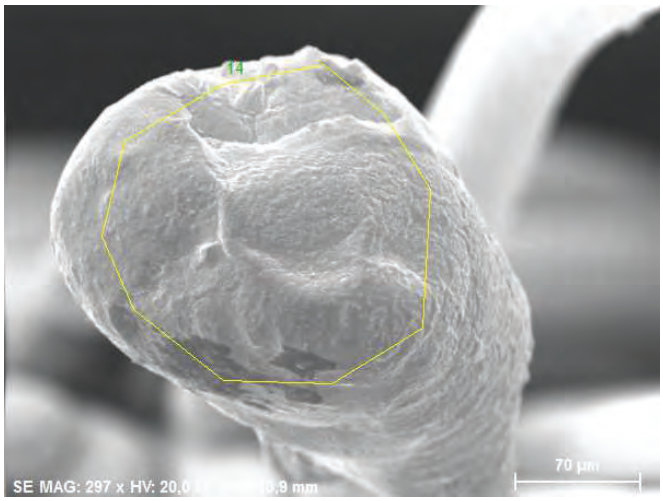


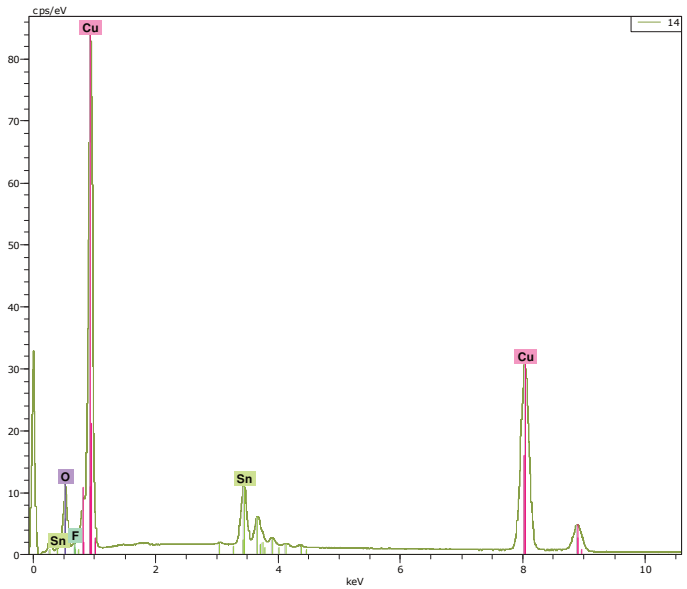
Figure 5 : Tensile test sample T3 – Fractographic examination











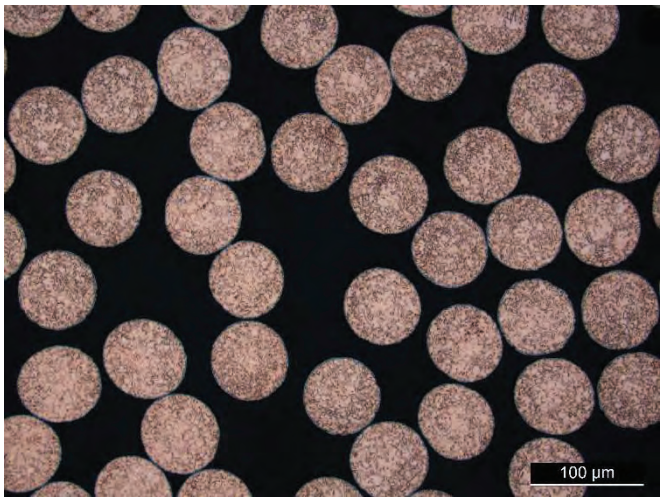
EDS spectrum

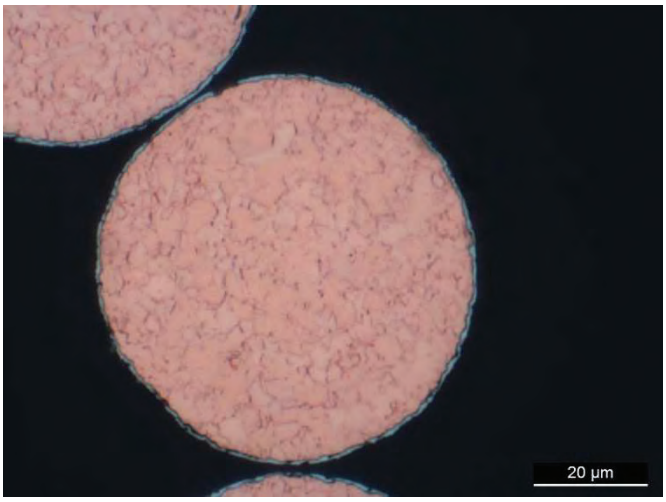
Spectrum: 14

Element	Series	norm. C [wt.%]	Atom. C [at.%]
oxygène	K-series	9,70	31,14
cuivre	K-series	74,39	60,15
étain	L-series	15,12	6,54
fluor	K-series	0,80	2,17
Total:		100,00	100,00

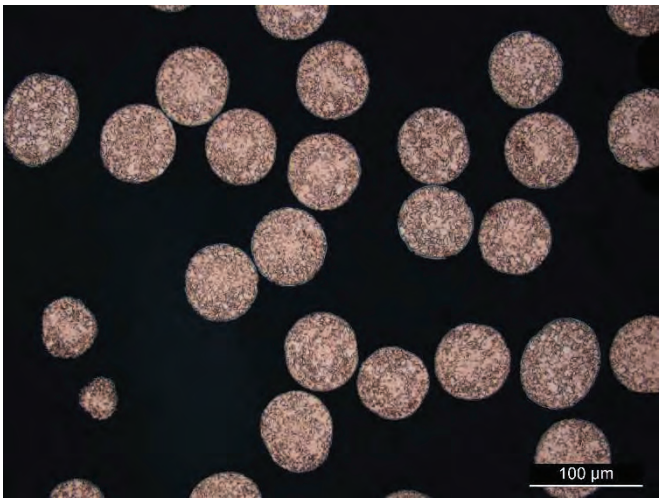
Figure 6 : Tensile test sample T1 – Micrographic examination

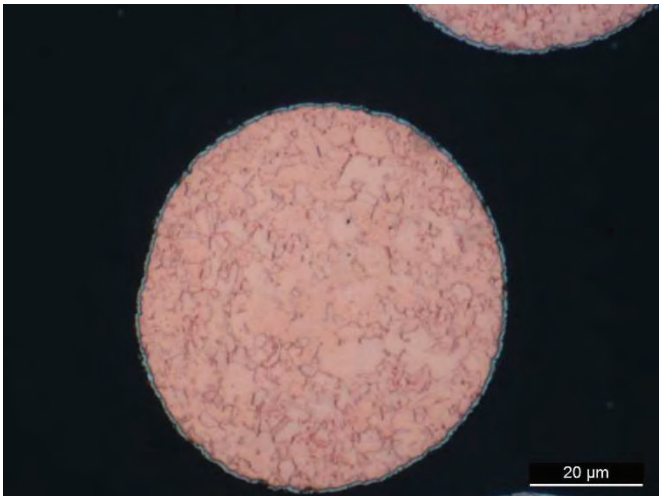
Grain size far from failure zone

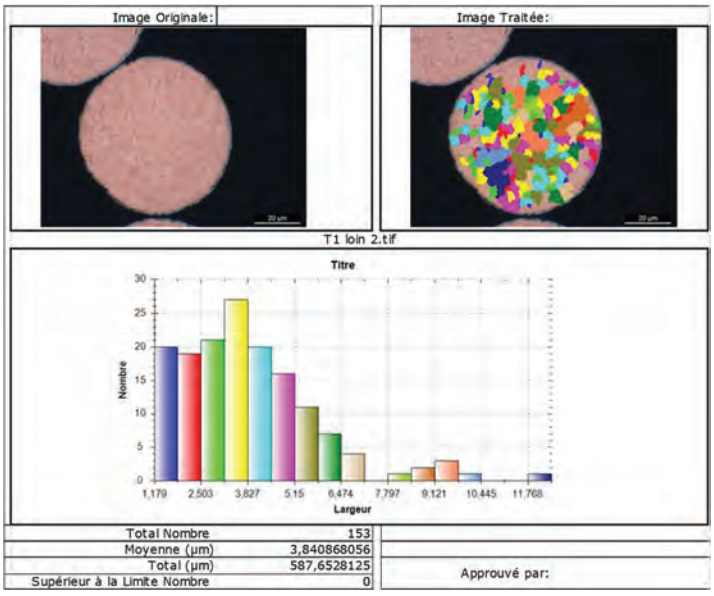




Grain size in failure zone







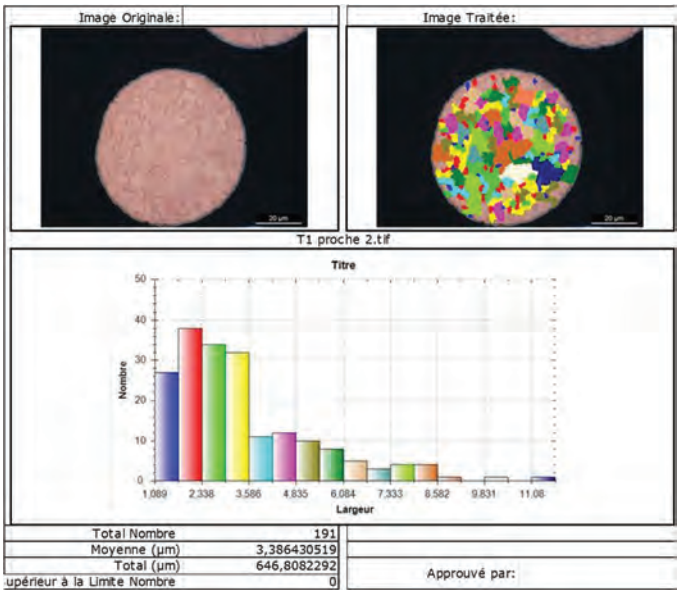
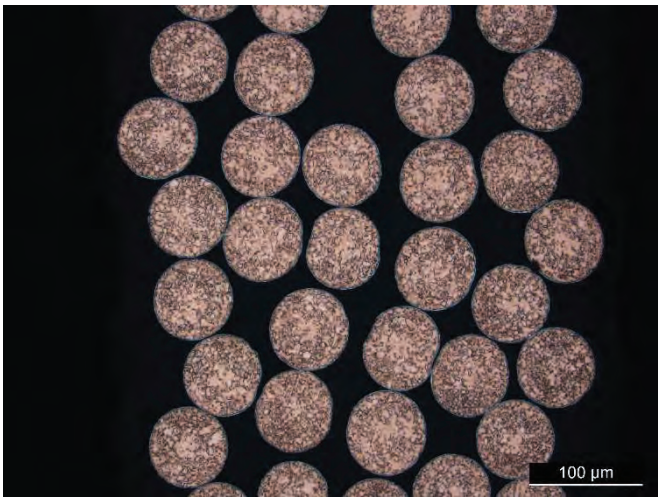
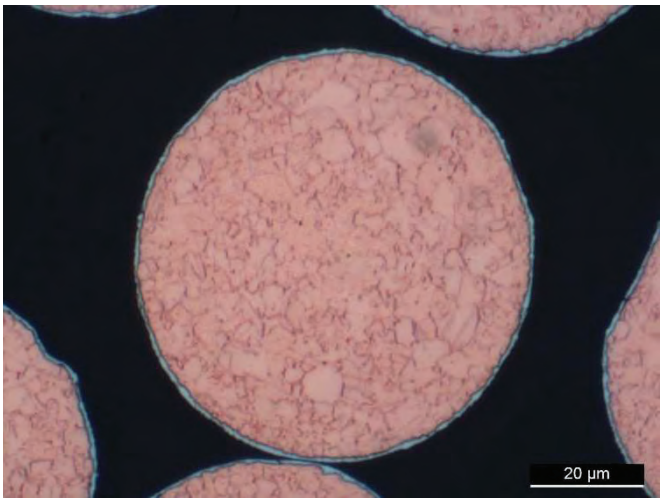
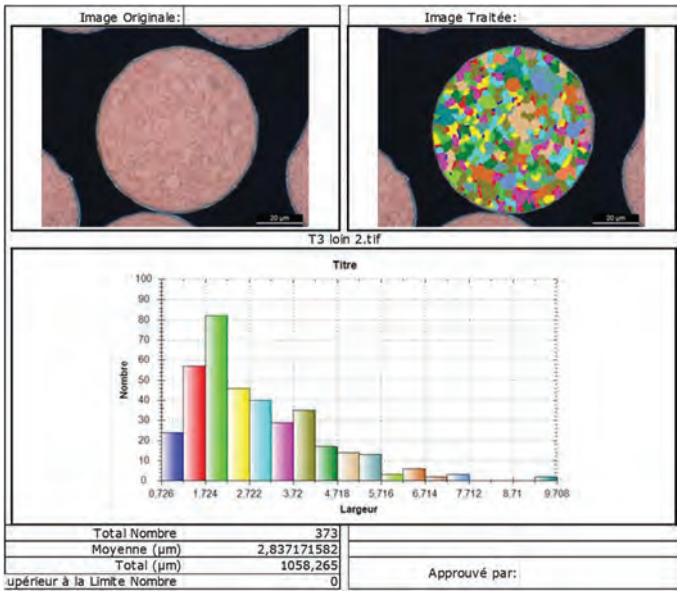


Figure 7 : Tensile test sample T3 – Micrographic examination

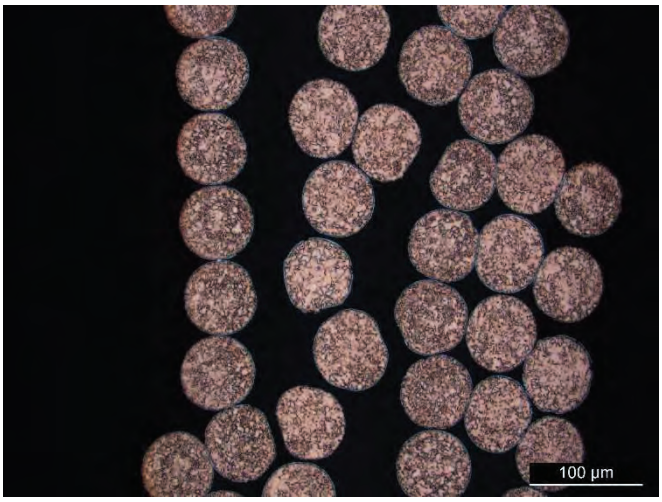
Grain size far from failure zone

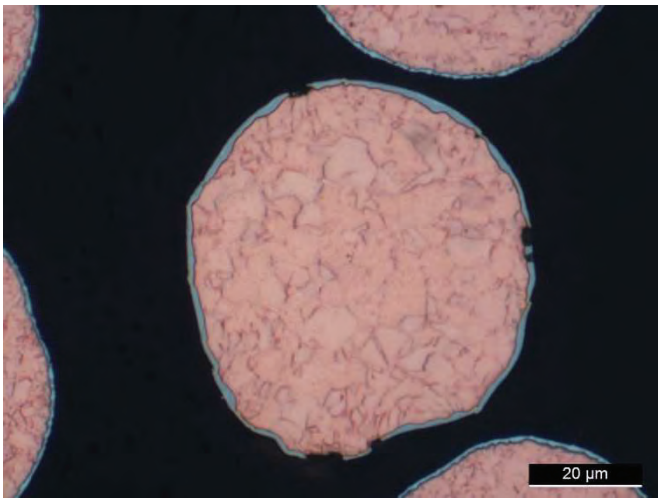






Grain size in failure zone





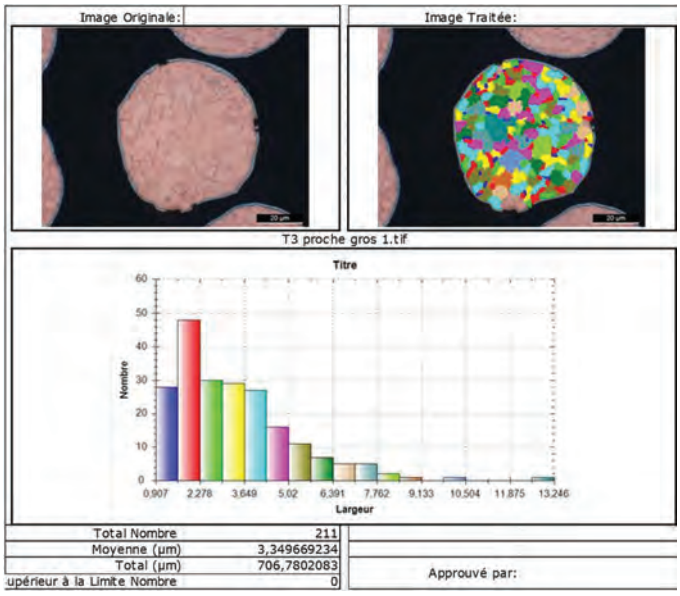
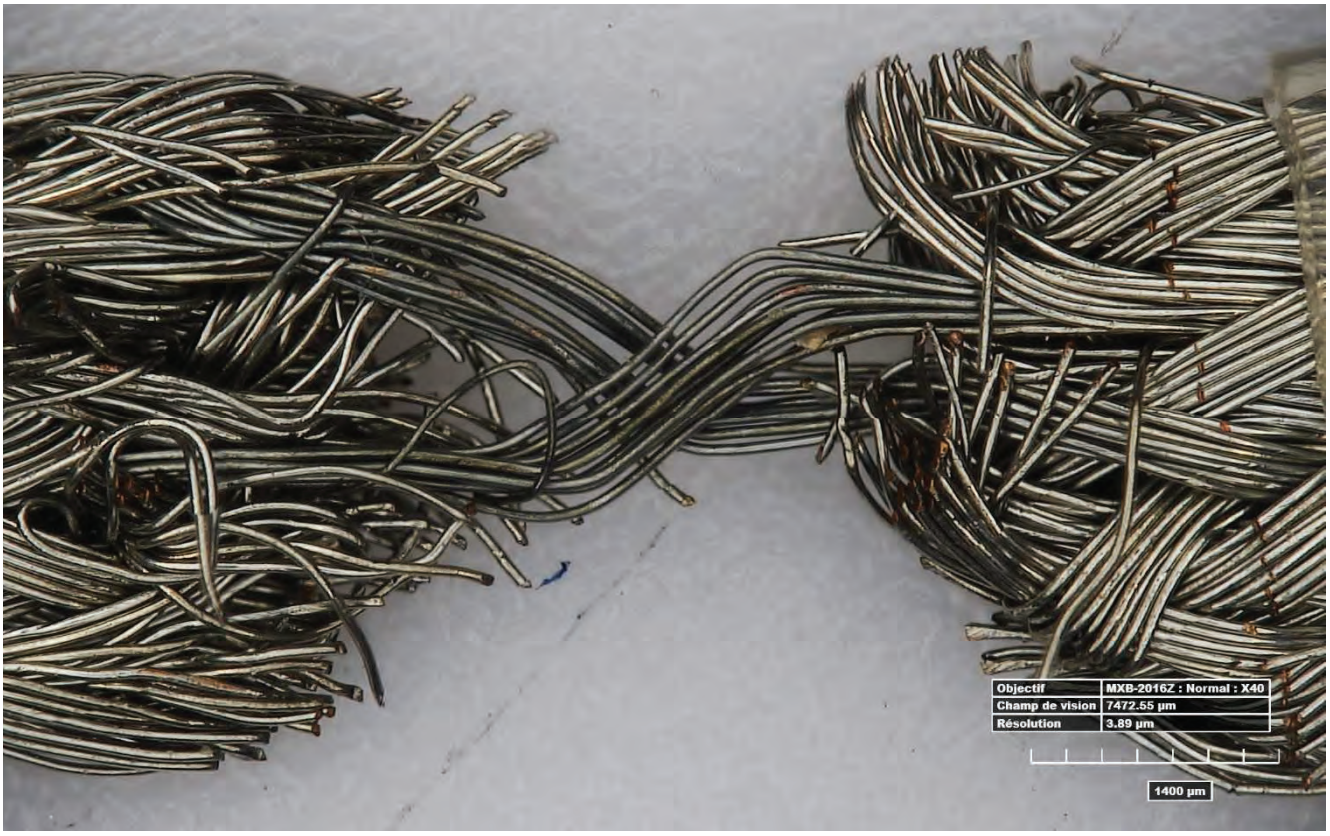


Figure 8 : Serial arc test in air results – SS1 sample 6%

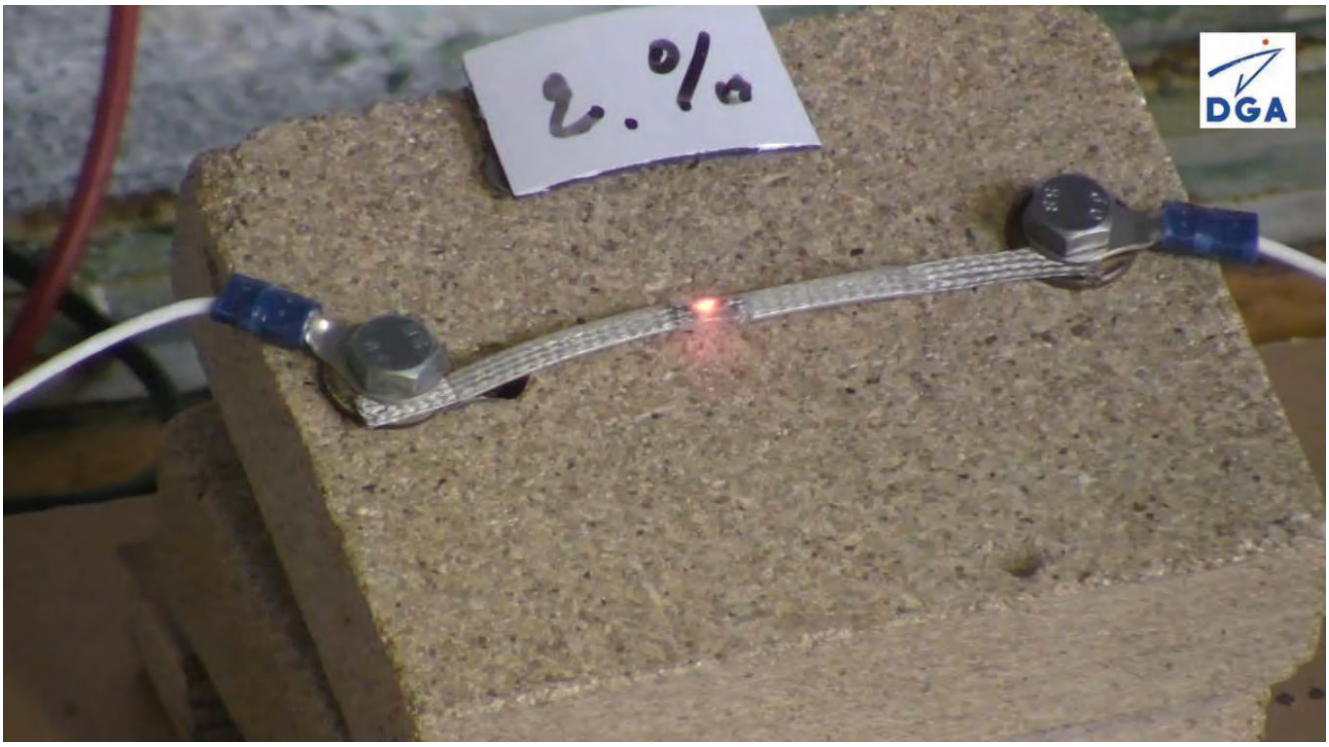


Sample after test – Light coloration of remaining wires

Figure 9 : Serial arc test in air results – SS2 sample 4%

Sample after test – Significant coloration of remaining wires

Figure 10 : Serial arc test in air results – SS4 sample 2%



Red glowing braid just before arcing

Serial arcing

Figure 11 : Serial arc test in air results – SS4 sample 2%

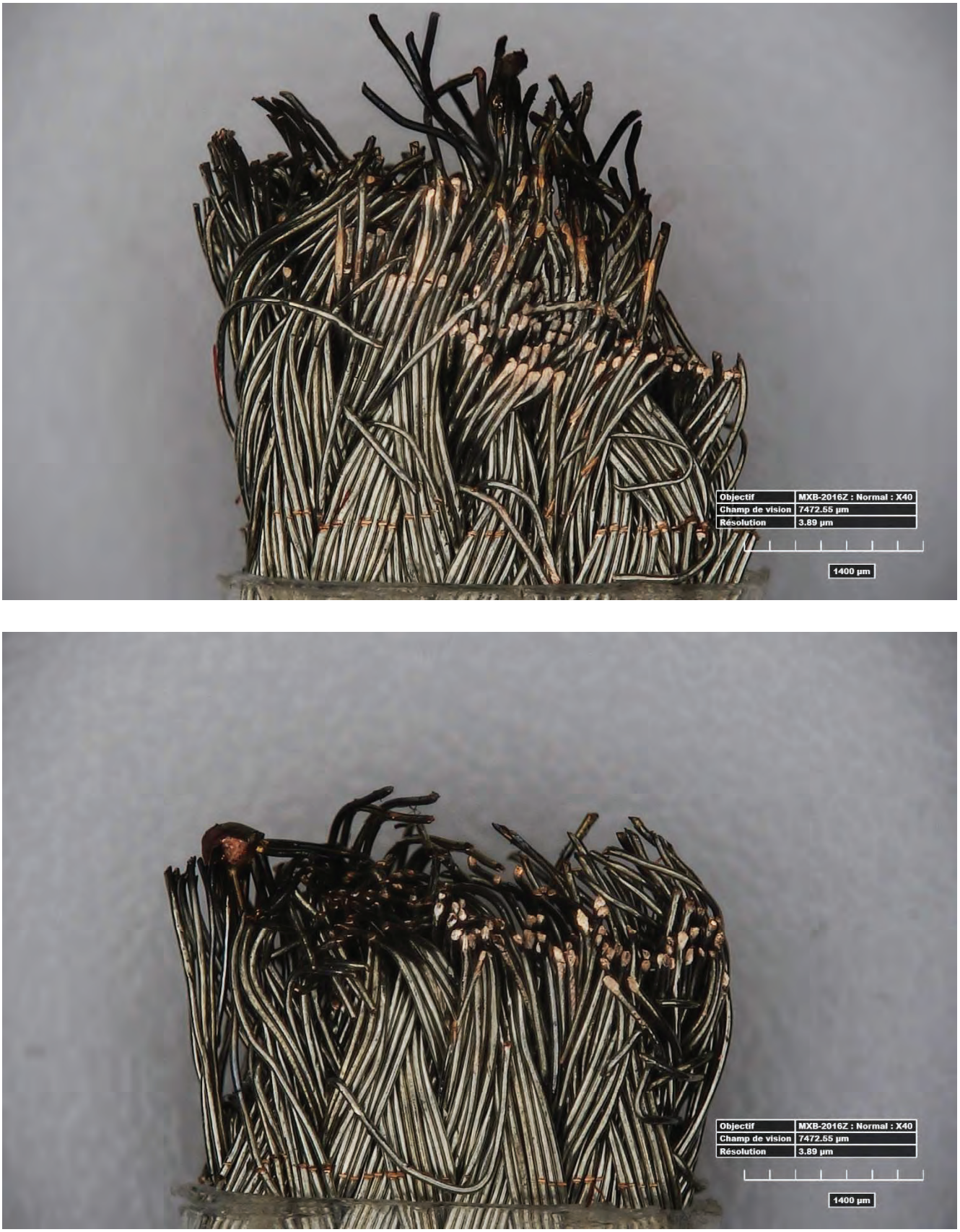
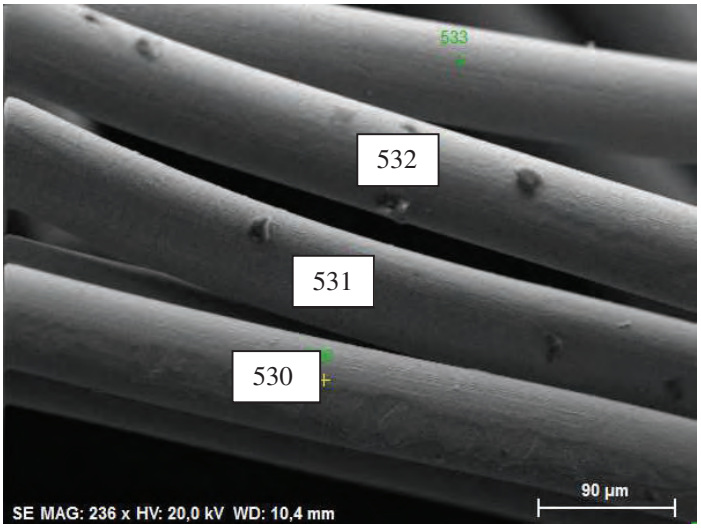
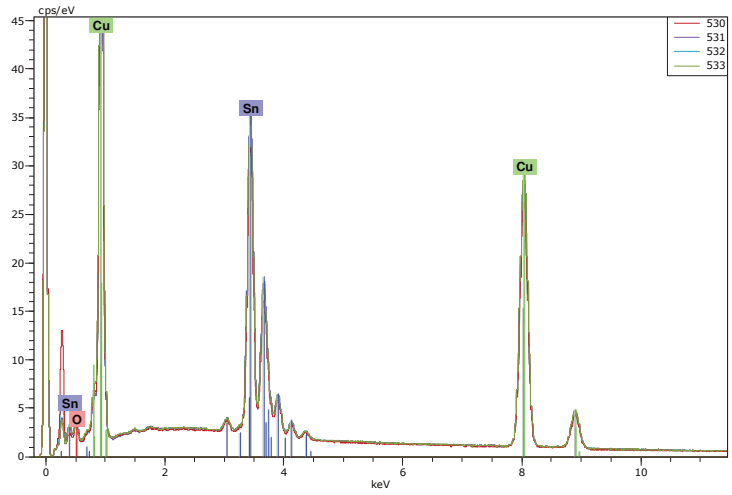


Figure 12 : Serial arc test in air - SS1 sample 6% – Fractographic examination





Spectrum: 530

El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	5998	0,63	0,82	3,91	0,12
Cu	29	K-series	560553	44,95	59,01	70,43	1,23
Sn	50	L-series	743459	30,60	40,17	25,66	0,92
		Total:		76,18	100,00	100,00	

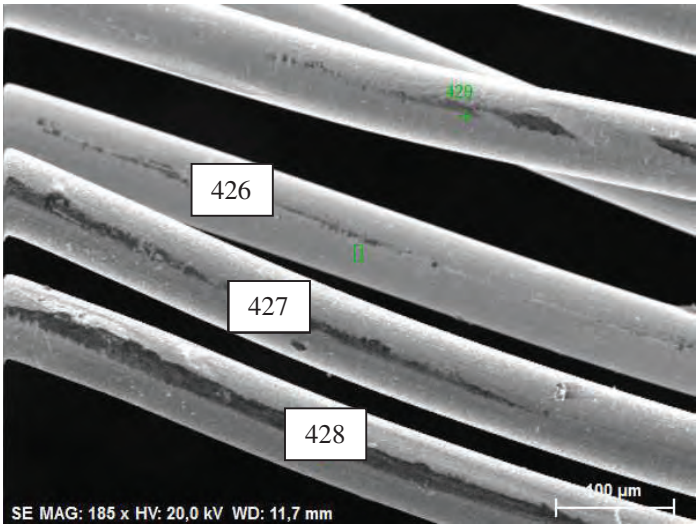
Spectrum: 531

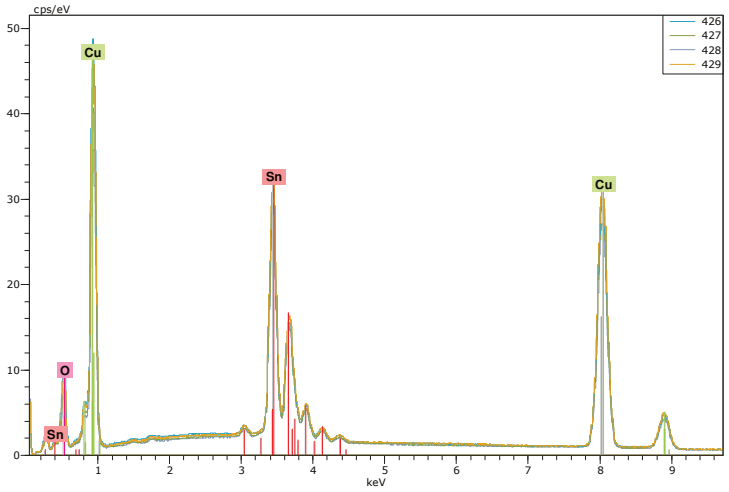
El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	8534	0,88	1,08	5,07	0,15
Cu	29	K-series	602362	47,51	58,47	69,27	1,29
Sn	50	L-series	840314	32,87	40,45	25,66	0,99
		Total:		81,25	100,00	100,00	

Spectrum: 532

El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	12712	1,29	1,64	7,61	0,20
Cu	29	K-series	580924	44,50	56,57	66,21	1,21
Sn	50	L-series	839388	32,87	41,79	26,18	0,99
		Total:		78,66	100,00	100,00	

Figure 13 : Serial arc test in air – SS2 sample 4% – Fractographic examination





Spectrum: 426

El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	78868	7,59	8,42	30,44	0,89
Cu	29	K-series	647116	53,14	58,99	53,69	1,44
Sn	50	L-series	721159	29,36	32,59	15,88	0,89
		Total:		90,09	100,00	100,00	

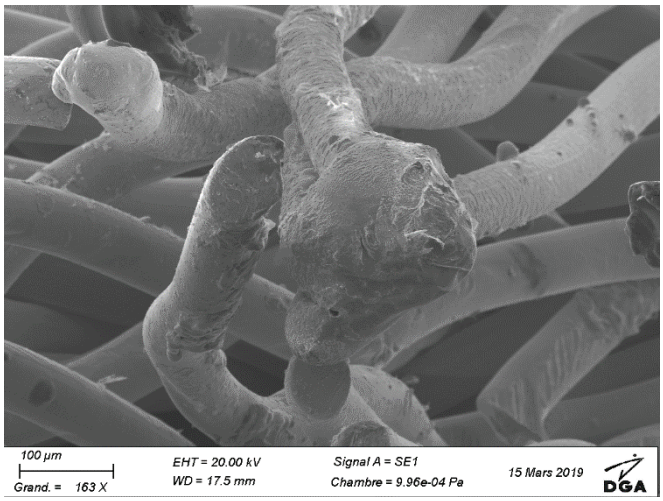
Spectrum: 427

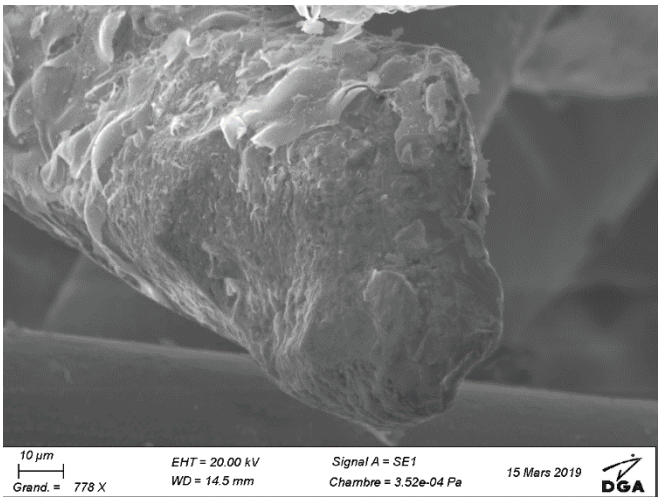
El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	55201	5,90	6,33	24,46	0,71
Cu	29	K-series	627103	55,17	59,25	57,62	1,50
Sn	50	L-series	692673	32,05	34,42	17,92	0,97
		Total:		93,11	100,00	100,00	

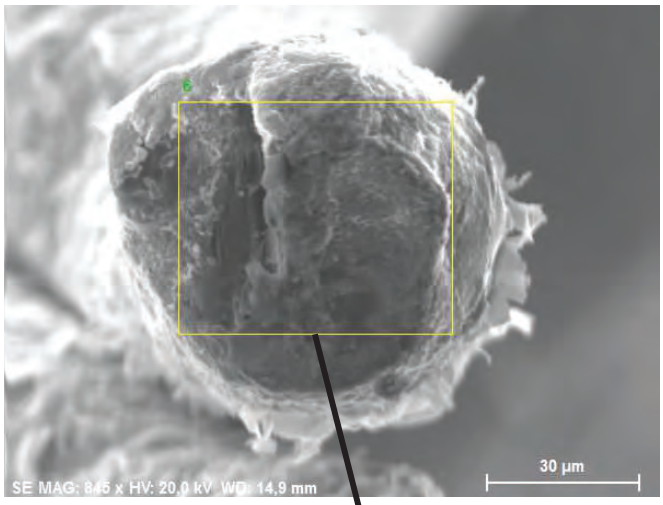
Spectrum: 428

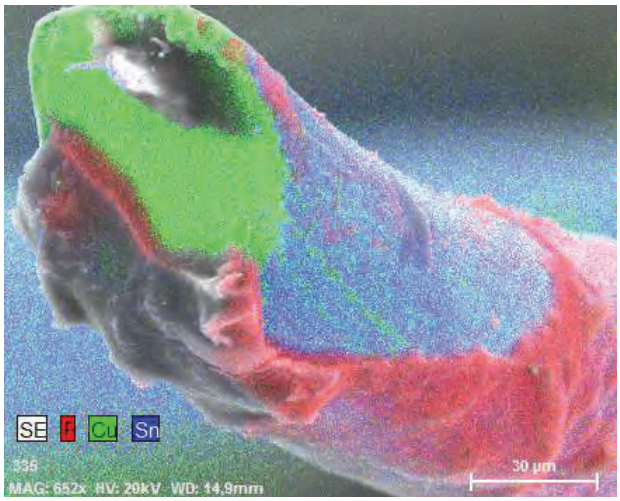
El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
O	8	K-series	48724	5,62	6,01	23,58	0,69
Cu	29	K-series	581270	54,29	58,03	57,38	1,48
Sn	50	L-series	685930	33,64	35,96	19,03	1,01
		Total:		93,55	100,00	100,00	

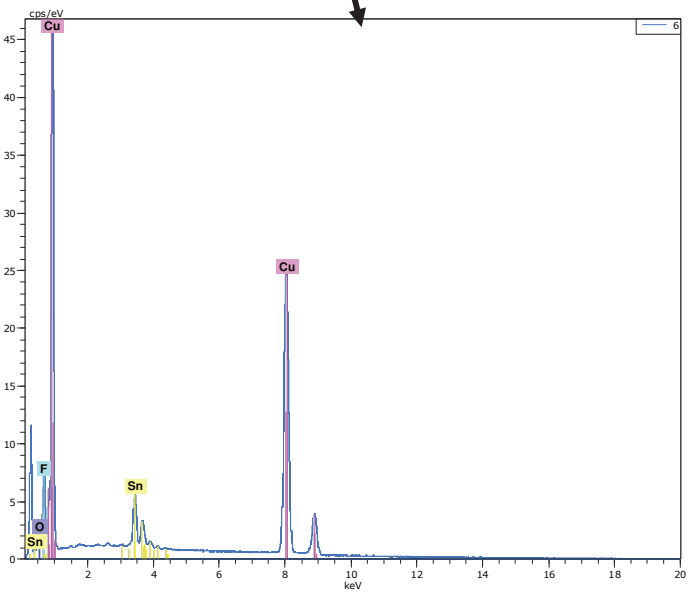
Figure 14 : Serial arc test in air – SS4 sample 2%– Fractographic examination









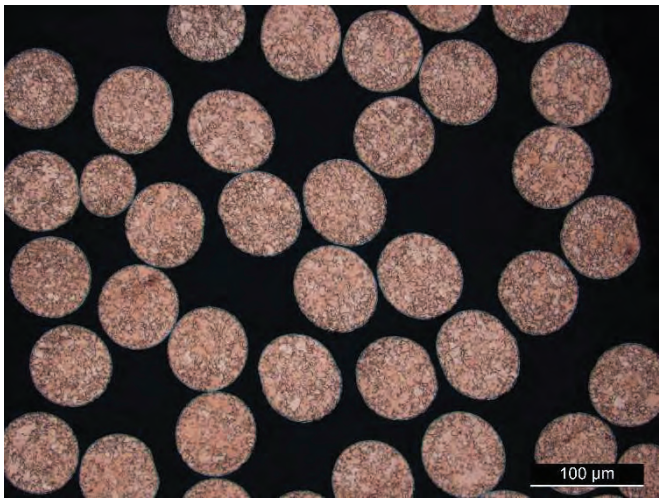


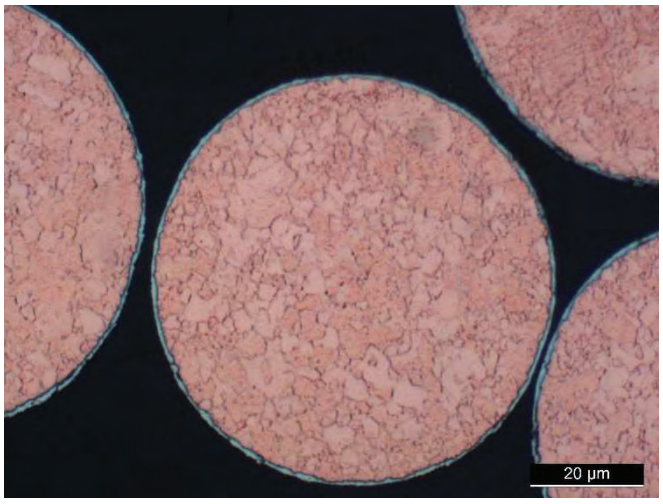
Spectrum: 6

Element	Series	norm. C [wt.%]	Atom. C [at.%]
oxygène	K-series	1,62	5,74
fluor	K-series	5,21	15,55
cuivre	K-series	82,69	73,72
étain	L-series	10,48	5,00
	Total:	100,00	100,00

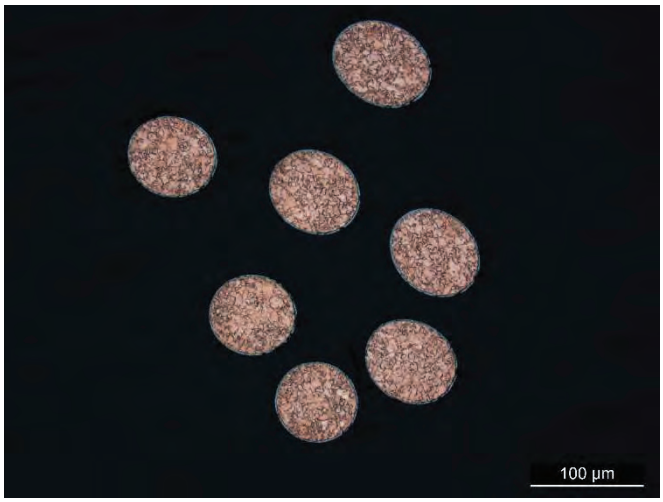
Figure 15 : Serial arc test in air - SS1 sample 6% – Micrographic examination

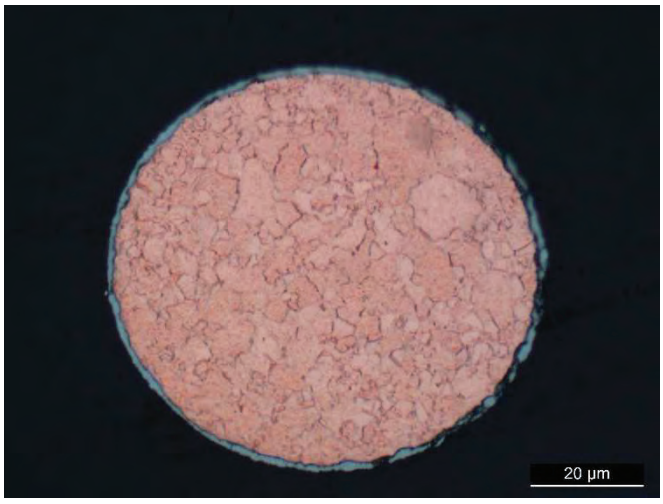
Grain size far from failure zone

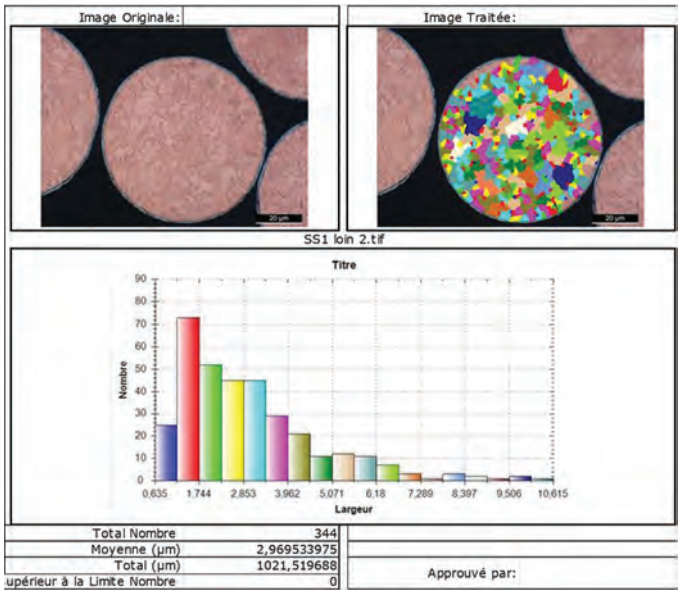




Grain size in failure zone







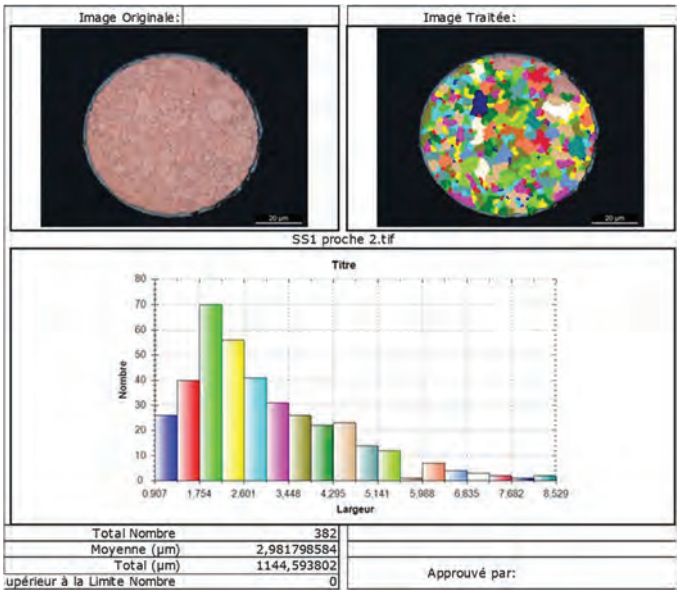
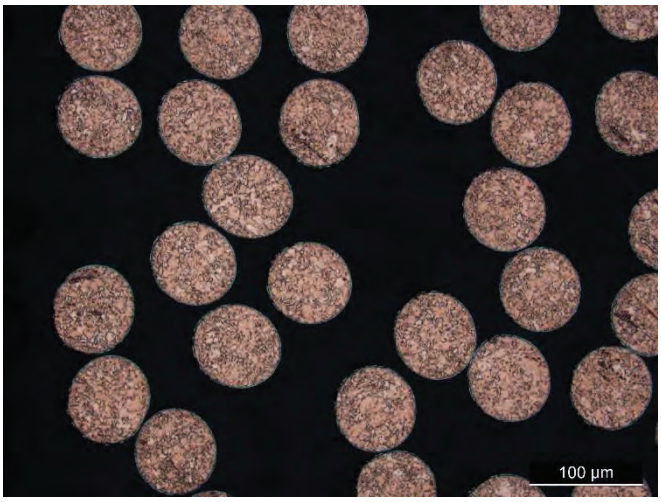
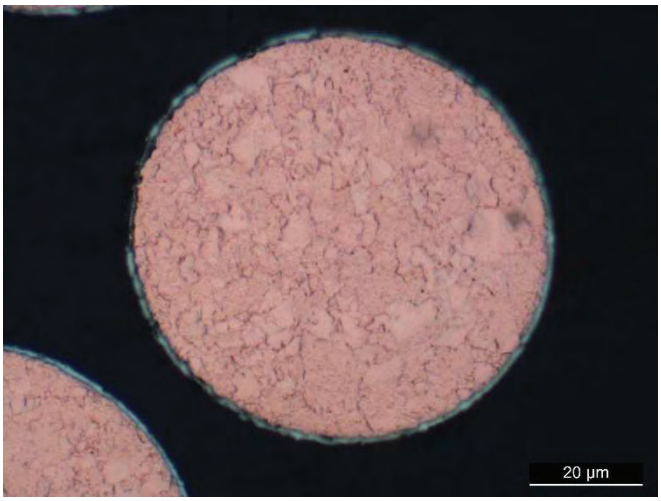
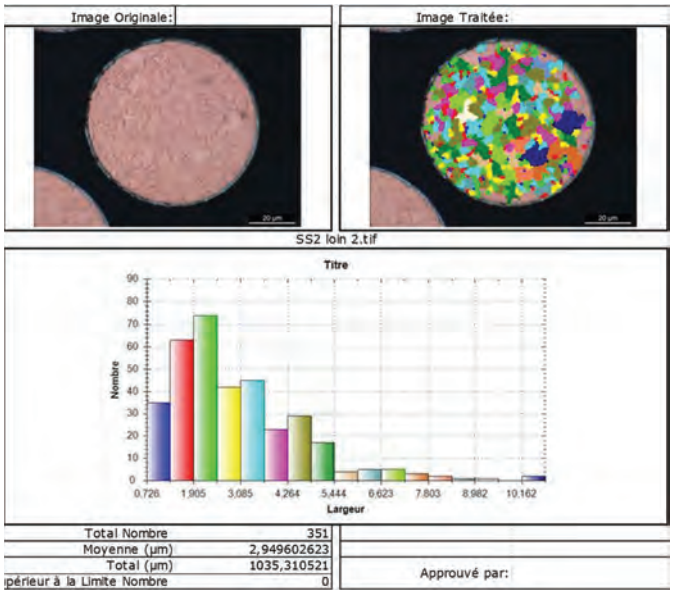


Figure 16 : Serial arc test in air – SS2 sample 4% – Micrographic examination

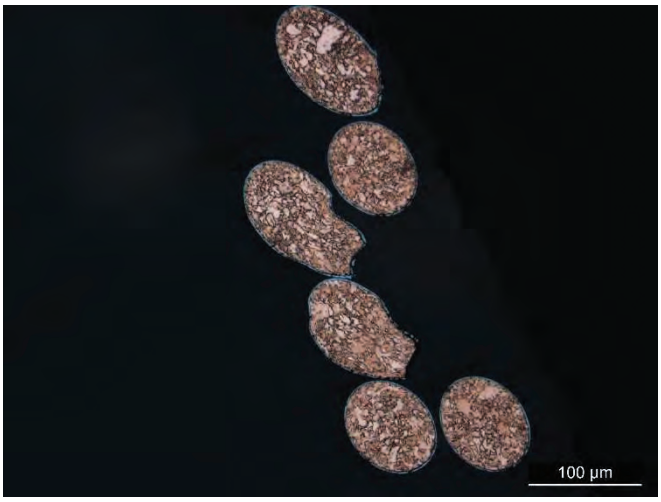
Grain size far from failure zone

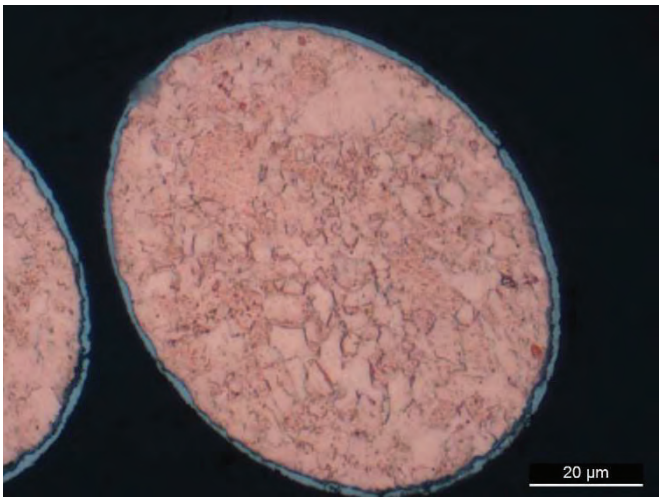






Grain size in failure zone





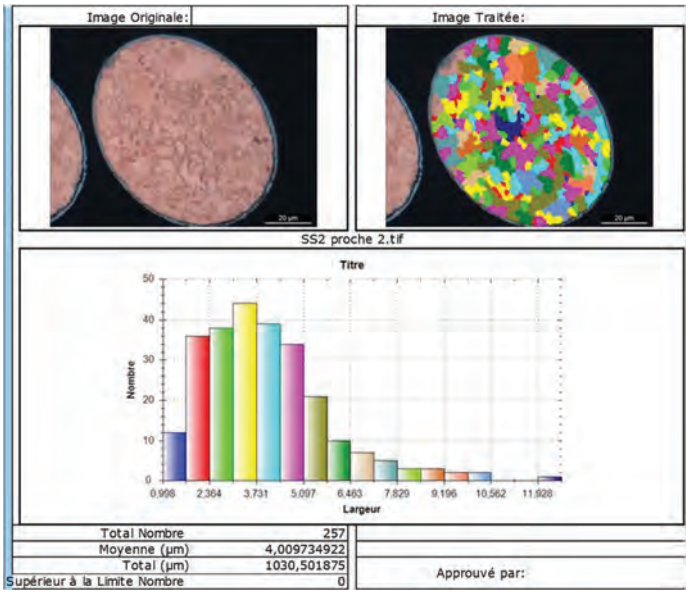
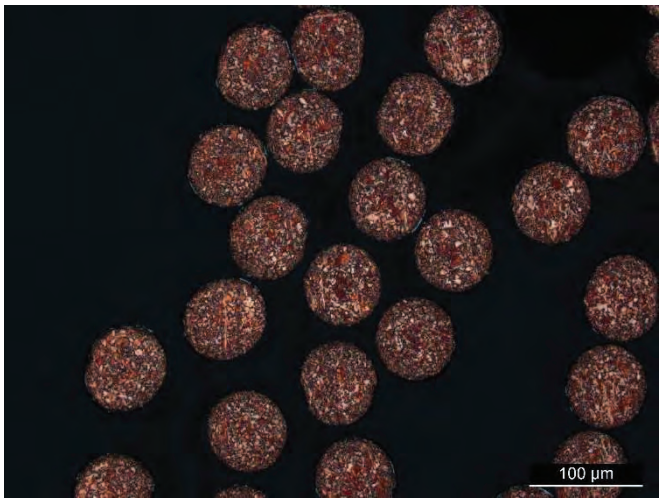
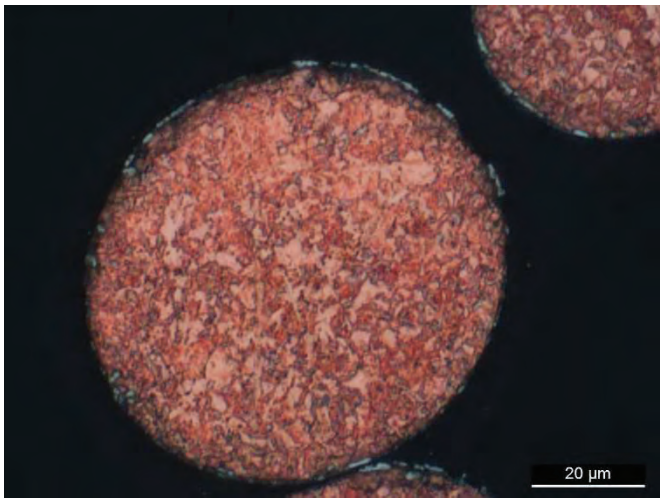
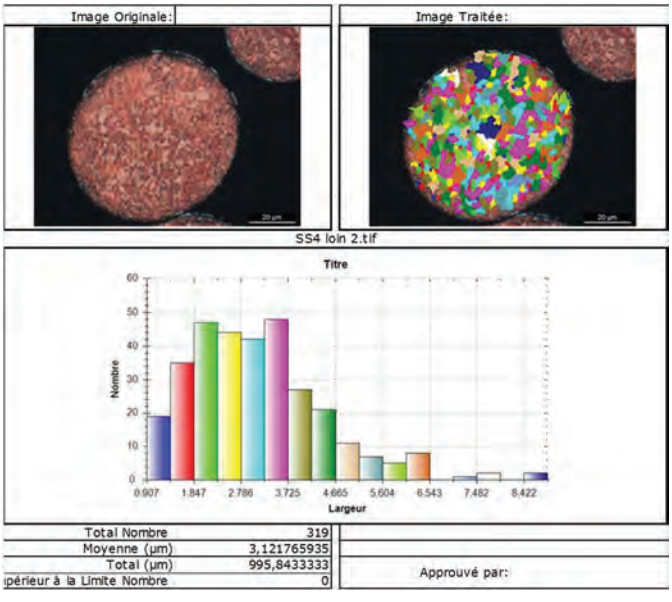


Figure 17 : Serial arc test in air – SS4 sample 2%– Micrographic examination

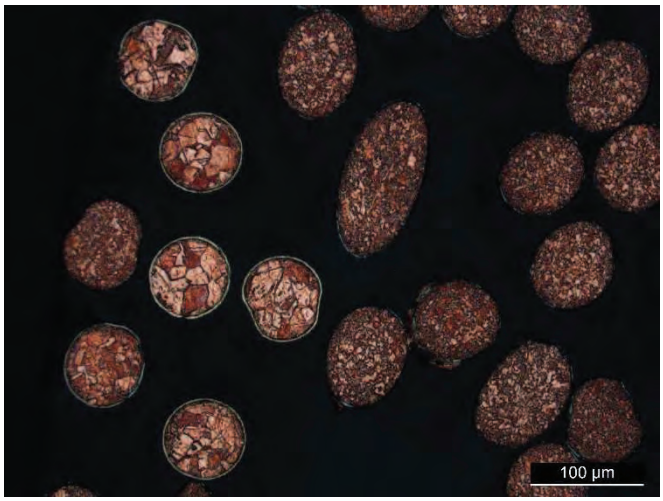
Grain size far from failure zone







Grain size in failure zone



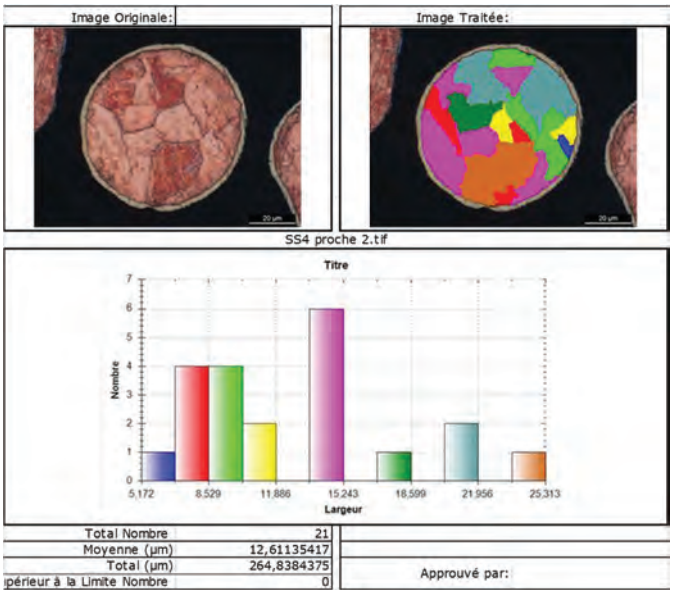


Figure 18 : Ion-exchange chromatography – pH4 solution

310 ExpA319-PH4

Séquence :	ESSAI GRADIENT KOH 2018 05 Septeml	*Volume injecté :*	
Emplacement :	Essais ICS	*Dilution :*	
Sample Type:	Unknown		
Programme :	Gradient_KOH de 1mM à 40mM de 3 à 16min		
Méthode	Anions		
Date d'injection :	29/3/2019 9:57		

N°.	T.Ret. min	Nom du pic	Conc. mg/l	Surface µS*min	Résolution(EU)	Plateaux (EU)	Type de pic
1	1.48	Fluorure	0.3805	0.147	4.17	1598	BMB*
2	2.57		n.a.	0.047	5.93	742	BMB
3	4.47	Bromure	n.a.	0.099	0.99	4553	BMB
4	6.11		2244.9392	273.273	n.a.	64	BMB
Total:			2245.320	273.566	11.09	6957.000	

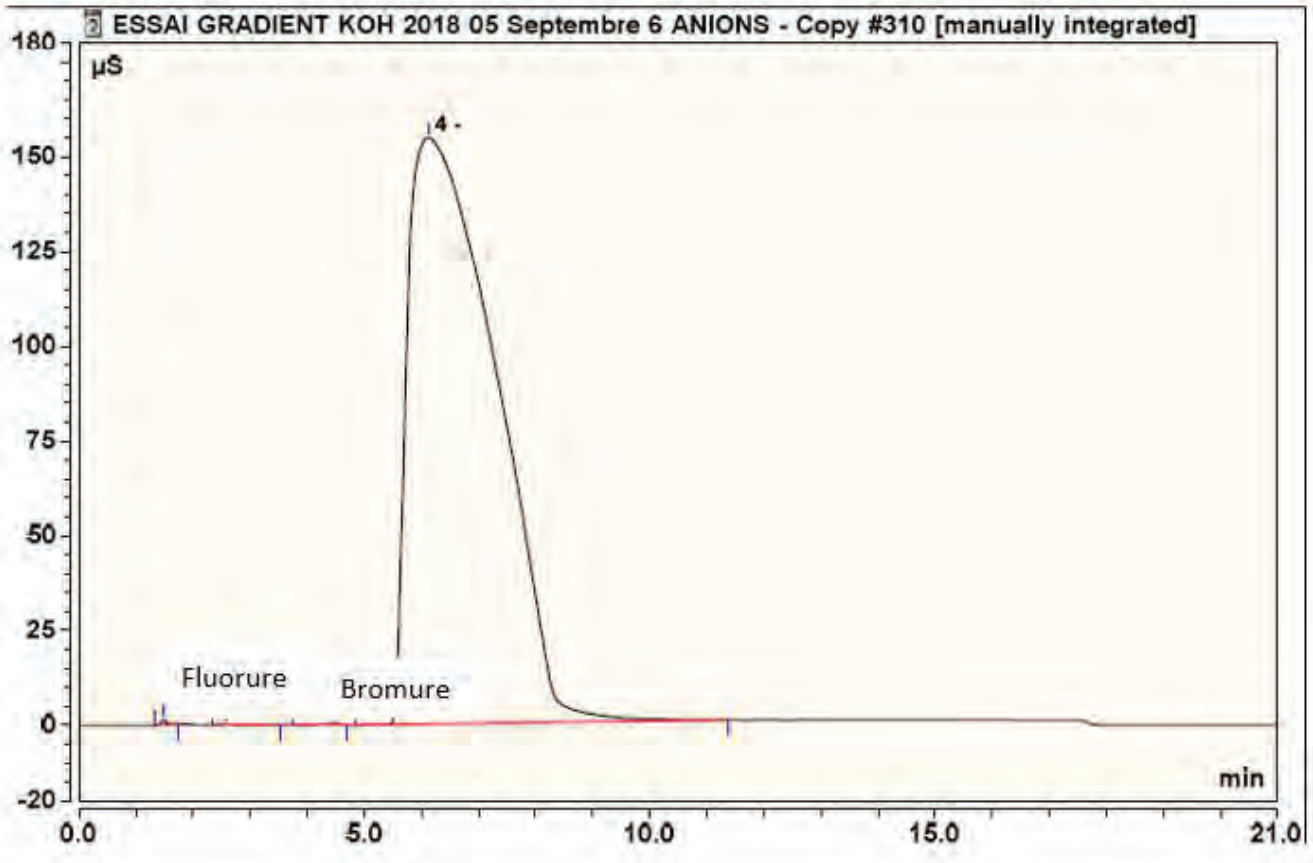


Figure 19 : Ion-exchange chromatography – pH9 solution

315 ExpA319-PH9

Séquence :	ESSAI GRADIENT KOH 2018 05 Septemt	*Volume injecté :*	
Emplacement :	Essais ICS	*Dilution :*	
Sample Type:	Unknown		
Programme :	Gradient_KOH de 1mM à 40mM de 3 à 16min		
Méthode	Anions		
Date d'injection :	29/3/2019 14:27		

N°.	T.Ret. min	Nom du pic	Conc. mg/l	Surface µS*min	Résolution(EU)	Plateaux (EU)	Type de pic
1	1.63	Fluorure	0.0992	0.038	0.75	376	BMB
2	2.10		n.a.	0.018	n.a.	83	BM
3	3.03		n.a.	0.029	n.a.	n.a.	M
4	3.79	Chlorure	159.2574	38.035	4.70	760	MB
5	5.37	Nitrate	0.4779	0.056	3.93	30068	BMB
6	6.70	Sulfate	0.5962	0.073	1.14	2330	BMb
7	7.09		n.a.	0.014	4.35	48459	bMB
8	8.10		n.a.	0.036	0.71	9237	BMb
9	8.26	Phosphate	0.1005	0.014	n.a.	73621	bMB
Total:			160.531	38.313	15.58	164934.000	

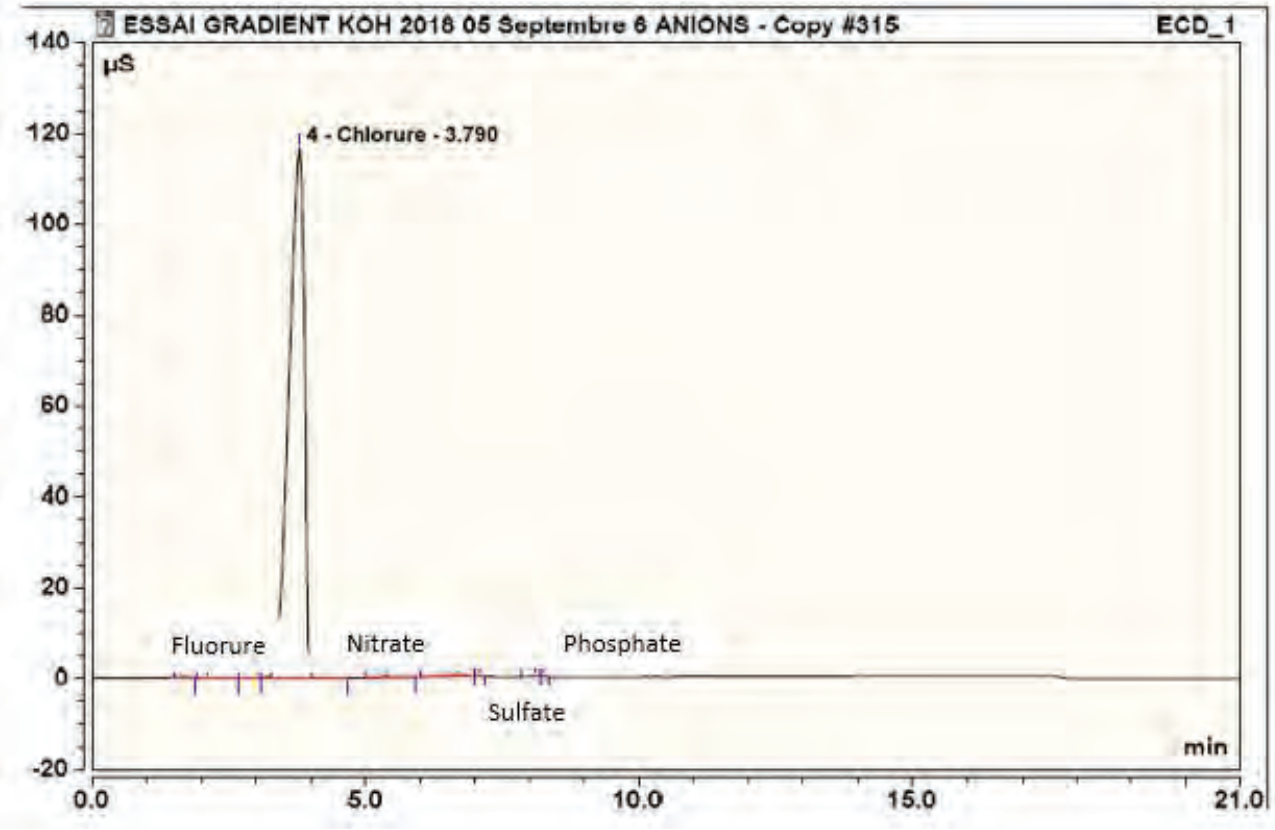


Figure 20 : Ion-exchange chromatography – Immersion in pH4 solution

363 ExpA319-PH4-Immersion

Séquence :	**ESSAI GRADIENT KOH 2018 05 Septemt**	*Volume injecté :*	
Emplacement :	**Essais ICS**	*Dilution :*	
Sample Type:	**Unknown**		
Programme :	**Gradient_KOH de 1mM à 40mM de 3 à 16min**		
Méthode	**Anions**		
Date d'injection :	**3/4/2019 14:01**		

N°.	T.Ret. min	Nom du pic	Conc. mg/l	Surface µS*min	Résolution(EU)	Plateaux (EU)	Type de pic
1	1.54	Fluorure	3.2817	1.266	15.15	888	BMB*
2	4.82	Bromure	n.a.	0.055	0.62	7256	BMB
3	5.93		2993.6838	362.499	3.02	50	BMb
4	11.37		n.a.	0.012	n.a.	38153	bMB
Total:			2996.965	363.832	18.80	46347.000	

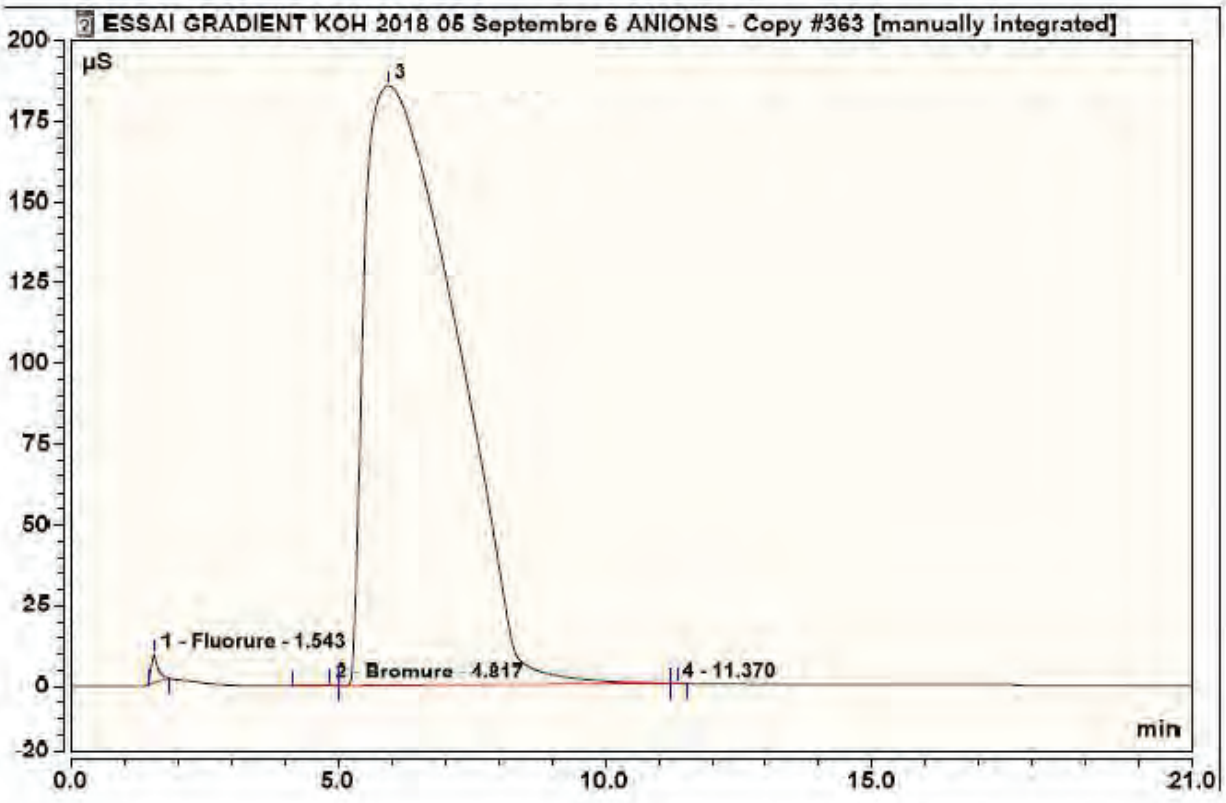


Figure 21 : Ion-exchange chromatography – Immersion in pH9 solution

438 ExpA319-PH9-Immersion

Séquence :	ESSAI GRADIENT KOH 2018 05 Septemt	*Volume injecté :*	
Emplacement :	Essais ICS	*Dilution :*	
Sample Type:	Unknown		
Programme :	Gradient_KOH de 1mM à 40mM de 3 à 16min		
Méthode	Anions		
Date d'injection :	29/4/2019 11:12		

N°.	T.Ret. min	Nom du pic	Conc. mg/l	Surface µS*min	Résolution(EU)	Plateaux (EU)	Type de pic
1	1.50	Fluorure	0.2871	0.111	3.98	139	BMB*
2	3.56	Chlorure	161.1926	38.497	4.72	726	BMB
3	5.18	Nitrate	0.6802	0.079	8.22	17042	BM
4	6.91	Sulfate	1.7095	0.208	2.87	10954	BMB*
5	7.87	Phosphate	0.1234	0.020	n.a.	6022	BMB
Total:			163.993	38.915	19.79	34883.000	

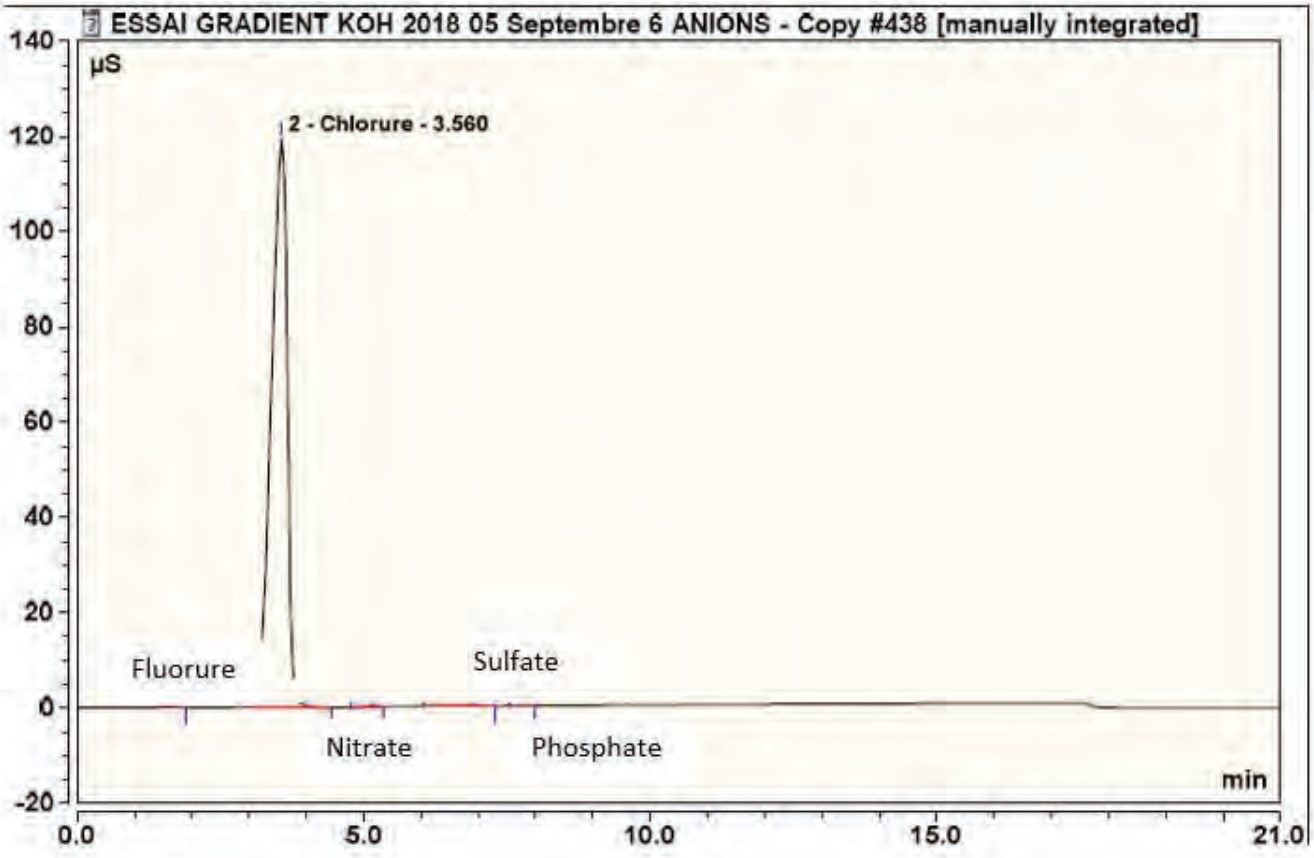
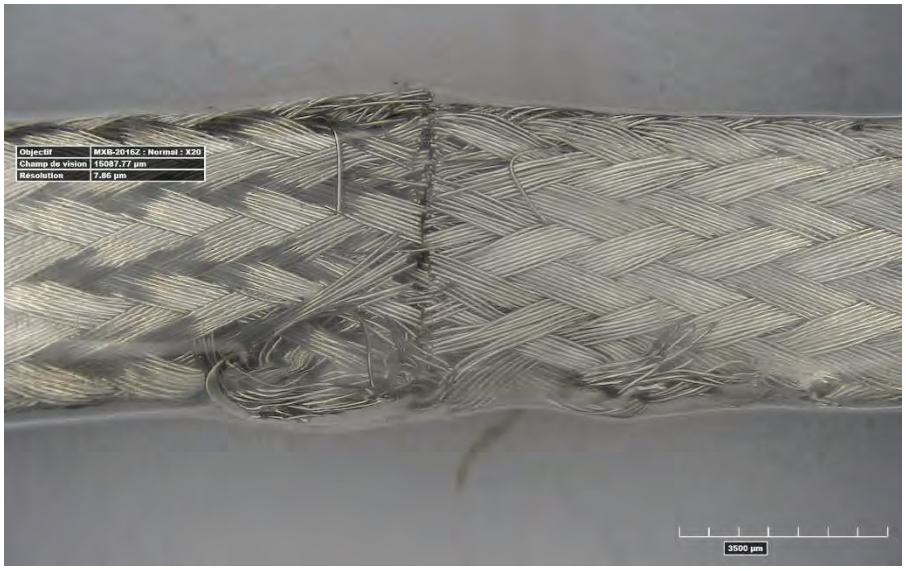


Figure 22 : Serial arc test in solution – HS1 sample 6% in pH4 without PR

The cut wires are in contact



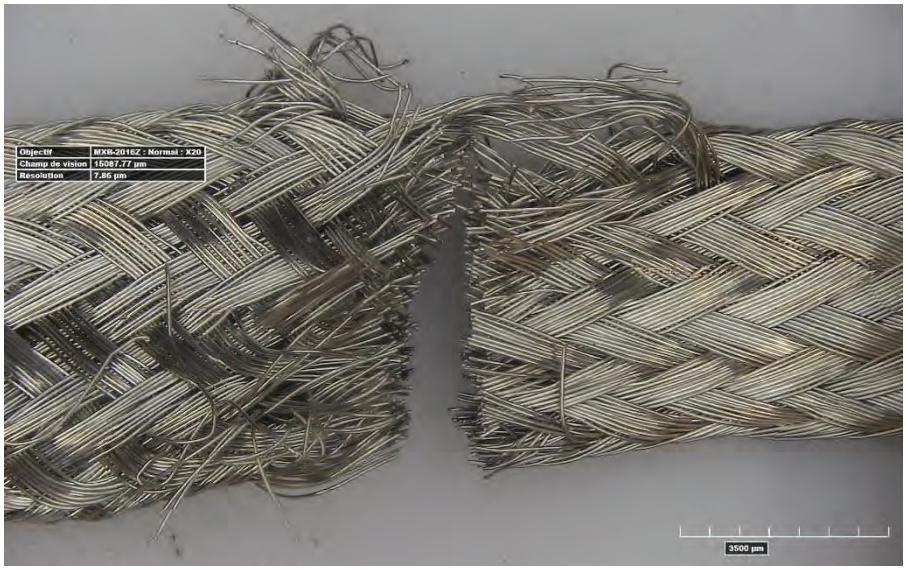
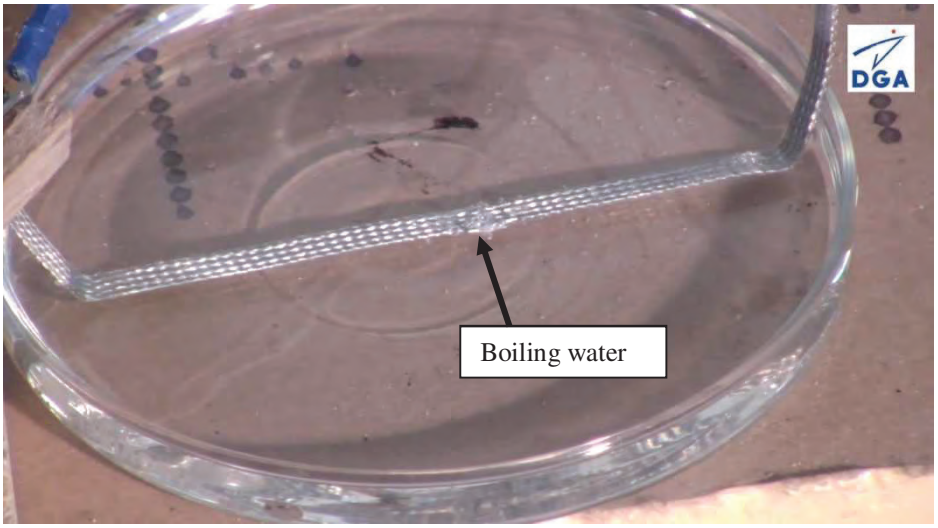


Figure 23 : Serial arc test in solution – HS2 sample 2% in pH4 without PR



The cut wires are in contact

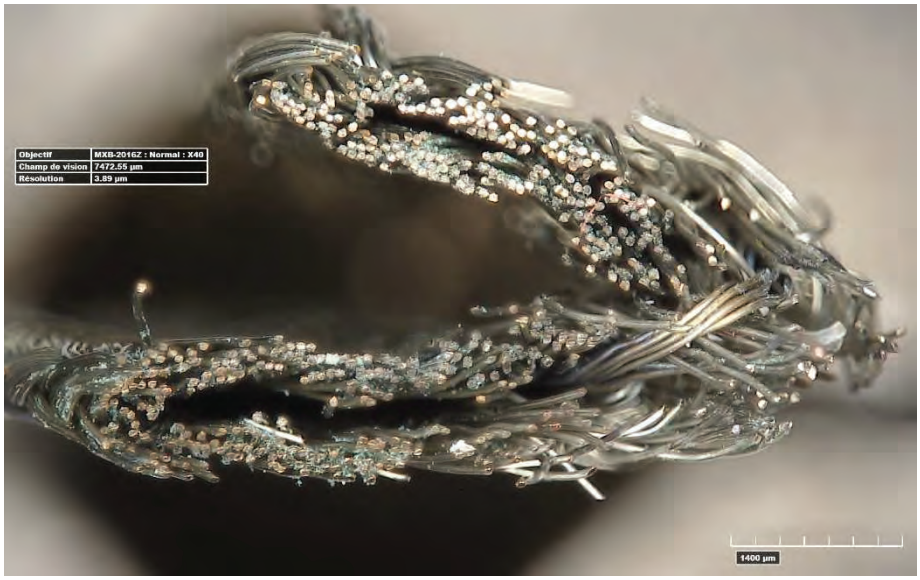
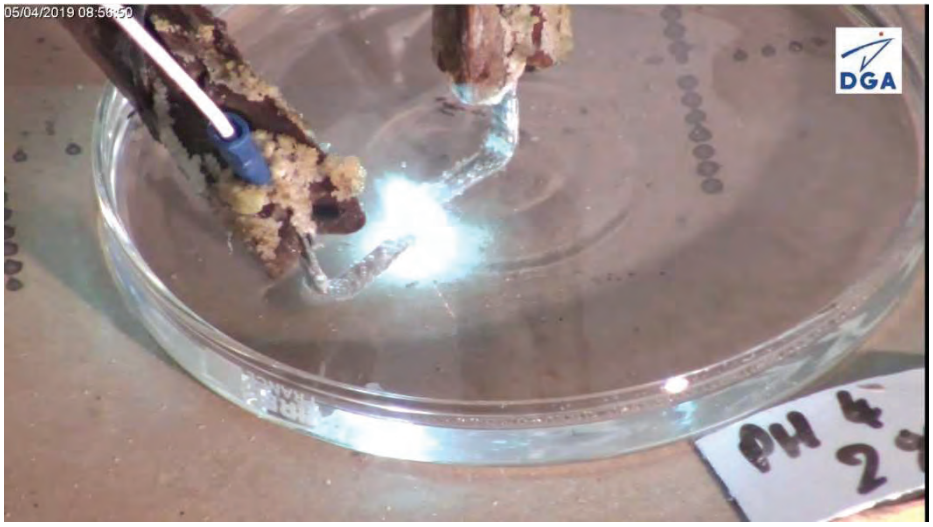


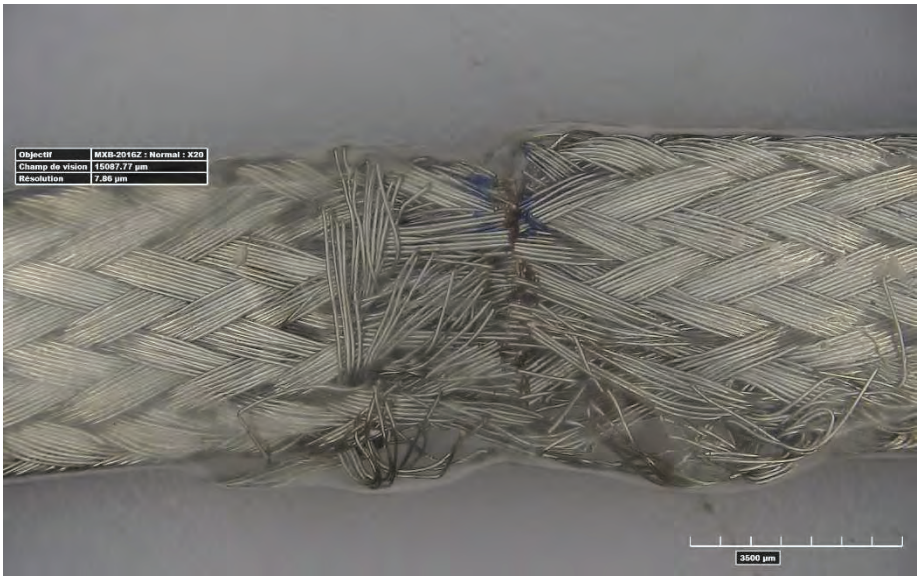
Figure 24 : Serial arc test in solution – HS3 sample 2% in pH4 without PR



The cut wires are not in contact

Figure 25 : Serial arc test in solution – HS4 sample 4% in pH9 without PR

The cut wires are in contact



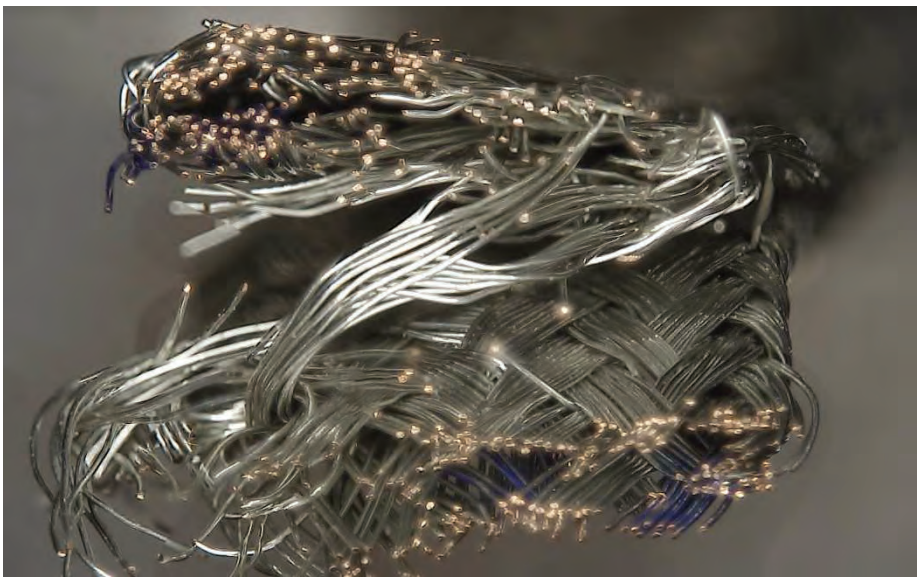


Figure 26 : Serial arc test in solution – HS5 sample 2% in pH9 with PR

The cut wires are in contact

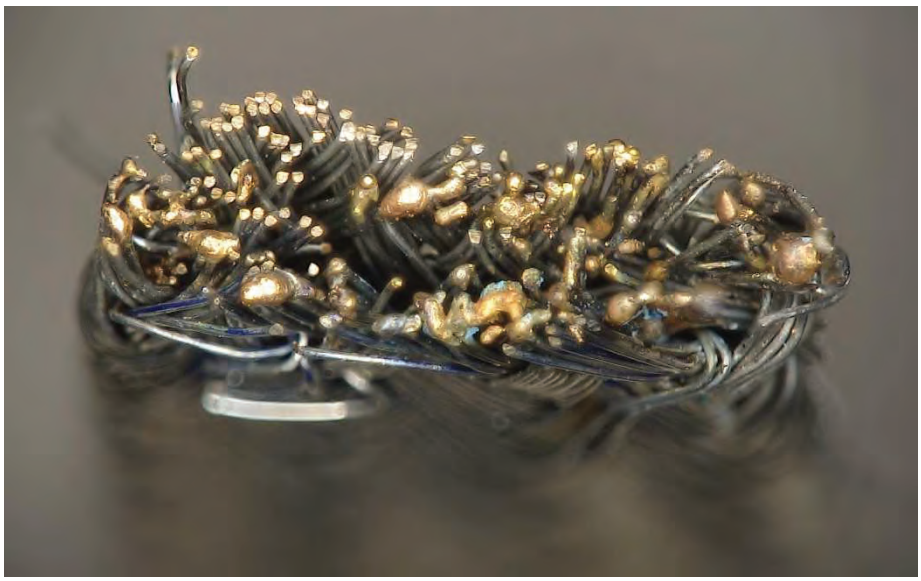
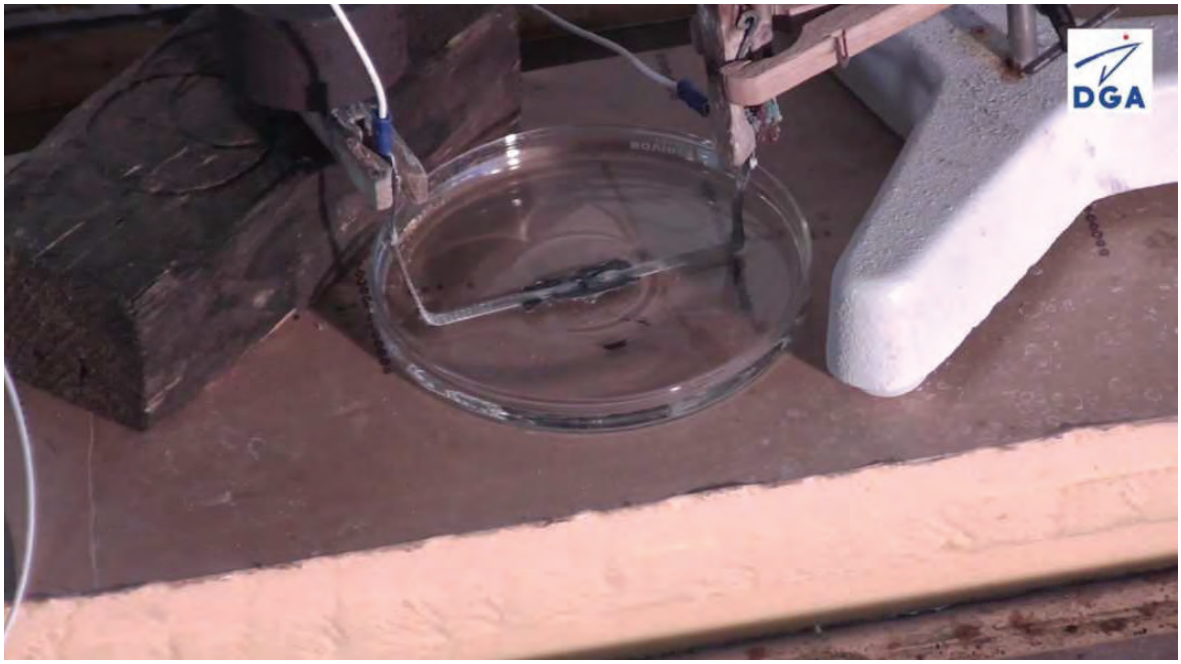


Figure 27 : Serial arc test in solution – HS6 sample 2% in pH4 with PR



The cut wires are not in contact