CNRS *Centre National de la Recherche Scientifique*
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OUTGASSING TEST OF NICKEL PLATED SAMPLES

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Approved by:

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In this note we briefly report the outgassing measurement of nickel plated samples prepared as the blades for the suspensions. The measurement method is described in detail in VACPISA 025.

1 - System performances

The typical base pressure of the test chamber after a baking at 250 ^{0}C for several days is ~10^{-10} mbar, with an outgassing rate of the order of ~10^{-12} mbar l s^{-1} cm^{-2}.

The main components of outgassing after baking are H_2, H_2O, N_2/CO, CO_2. The internal surface of the chamber is 2500 cm^2.

2 - Measurement of the outgassing flow of blades

The experimental samples were two nickel plated pieces made of the same material and prepared according to the recipe of the filter blades. We have been advised that the samples were not originally prepared for a vacuum test, so no particular care had been taken in handling and storing. The samples have been cleaned in various steps: ultrasound bath of UHV soap for 20 minutes; rinsing; ultrasound bath of isopropyl alcohol for 40 minutes; rinsing; four hours baking at 100 ^{0}C in air. The exposed surface was 210.7 cm^2.

We monitored the evolution of outgassing (time is measured from beginning of the test through the whole paper):

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
2.5	25	3.7×10^{-6}	2.3×10^{-6}	2.8×10^{-5}
25.8	30	1.3×10^{-7}	3.3×10^{-8}	1.9×10^{-6}
46.6	28	8.2×10^{-8}	3.3×10^{-8}	9.8×10^{-7}
122.8	29	3.0×10^{-8}	1.2×10^{-8}	3.6×10^{-7}
147	25	2.7×10^{-8}	1.2×10^{-8}	3.0×10^{-7}

The mass spectrum after some days pumping at room temperature is shown in Fig. 1.

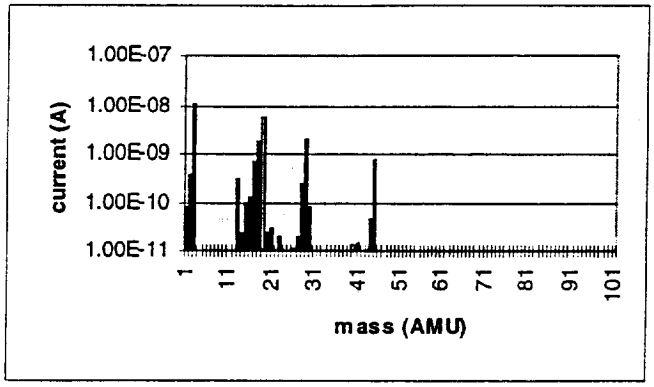

Fig. 1 Outgassing spectrum after some days pumping at room temperature

We set temperature at 50 ^{0}C for 216 hours and monitored the evolution of outgassing:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
147.3	36	6.0×10^{-8}	1.4×10^{-8}	9.2×10^{-7}
147.4	57	1.7×10^{-6}	1.3×10^{-7}	3.1×10^{-5}
148.1	58	3.2×10^{-7}	7.0×10^{-8}	5.0×10^{-6}
148.5	52	2.8×10^{-7}	8.2×10^{-8}	4.0×10^{-6}
172.1	52	8.9×10^{-8}	2.3×10^{-8}	1.3×10^{-6}
197.2	49	6.5×10^{-8}	2.8×10^{-8}	7.4×10^{-7}
218.9	53	5.1×10^{-8}	1.3×10^{-8}	7.6×10^{-7}
291.2	51	3.1×10^{-8}	1.1×10^{-8}	4.0×10^{-7}
315.5	51	2.5×10^{-8}	1.0×10^{-8}	3.0×10^{-7}
338	49	2.9×10^{-8}	1.3×10^{-8}	3.2×10^{-7}
362.8	50	1.8×10^{-8}	8.9×10^{-9}	1.8×10^{-7}

The mass spectrum measured at beginning of heating at 50 ^{o}C is shown in Fig. 2. Another spectrum measured after several tens hours heating at 50 ^{o}C is shown in Fig. 3.

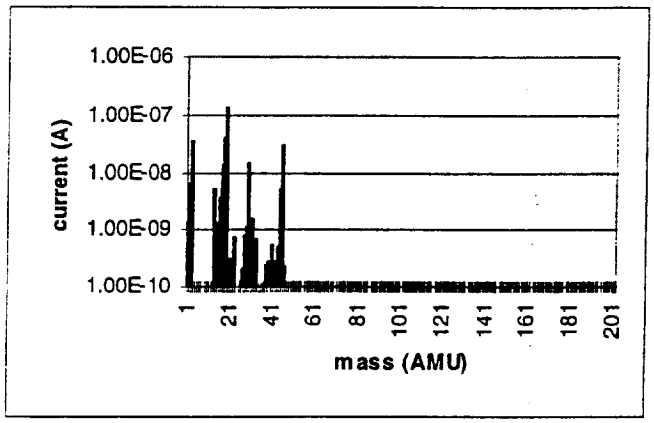

Fig. 2 Outgassing spectrum at beginning of heating at 50 °C

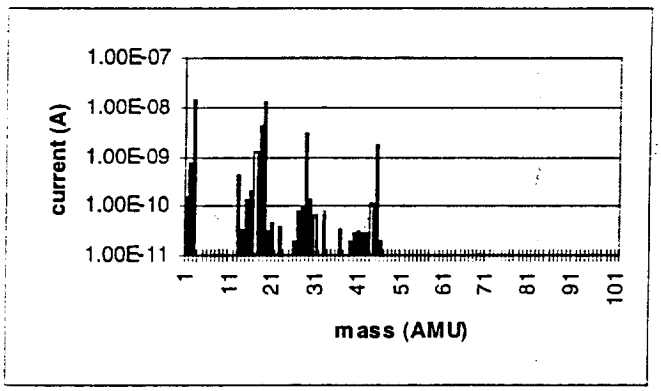

Fig. 3 Outgassing spectrum after several tens hours at 50 °C

In both cases a peak at 43 AMU, typical of organic contamination was observed.

We set temperature at 100 °C for 166 hours and monitored the evolution of outgassing:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
363.2	58	5.5×10^{-8}	1.3×10^{-8}	8.4×10^{-7}
363.3	109	4.8×10^{-6}	3.9×10^{-7}	8.8×10^{-5}
363.8	102	1.3×10^{-6}	1.9×10^{-7}	2.2×10^{-5}
364.4	110	1.6×10^{-6}	2.6×10^{-7}	2.7×10^{-5}
389	108	1.5×10^{-7}	3.4×10^{-8}	2.3×10^{-6}
458.3	107	7.3×10^{-8}	1.9×10^{-8}	1.1×10^{-6}
459.75	117	5.3×10^{-8}	1.6×10^{-8}	7.4×10^{-7}
507.3	104	4.2×10^{-8}	1.4×10^{-8}	5.6×10^{-7}

533.2	102	5.3×10^{-8}	3.0×10^{-8}	4.6×10^{-7}
549.8	100	3.0×10^{-8}	1.0×10^{-8}	4.0×10^{-7}

The mass spectra measured at beginning of heating and after several hours at 100 °C are shown in Fig. 4 and Fig. 5.

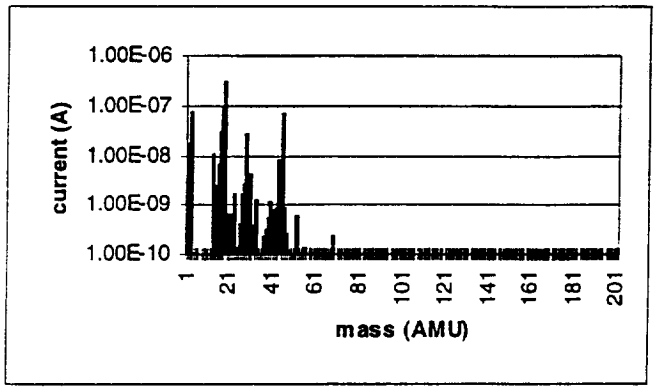

Fig. 4 Outgassing spectrum at beginning of heating at 100 °C

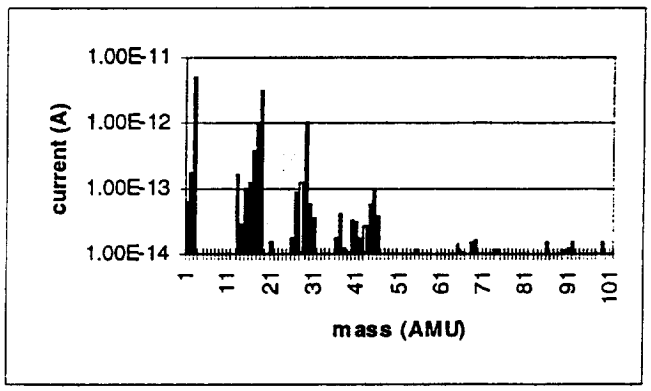

Fig. 5 Outgassing spectrum after several tens hours at 100 °C

Some fragments, beyond the one at 43 AMU, appeared.

We set temperature at 150 °C for 124 hours and monitored the evolution of outgassing:

t(h)	T(°C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
549.9	107	4.6×10^{-8}	1.1×10^{-8}	7.0×10^{-7}
550	135	8.2×10^{-7}	7.1×10^{-8}	1.5×10^{-5}
550.1	156	2.1×10^{-6}	1.8×10^{-7}	3.8×10^{-5}

550.5	147	7.9×10^{-7}	1.1×10^{-7}	1.4×10^{-5}
556.1	144	4.4×10^{-7}	1.3×10^{-7}	6.2×10^{-6}
628.2	142	1.6×10^{-7}	3.3×10^{-8}	2.5×10^{-6}
652.6	158	9.2×10^{-8}	2.9×10^{-8}	1.3×10^{-6}
673.8	145	5.4×10^{-8}	1.8×10^{-8}	7.2×10^{-7}

The mass spectra measured at beginning of heating and after several hours at 150 °C are shown in Fig. 6 and Fig. 7.

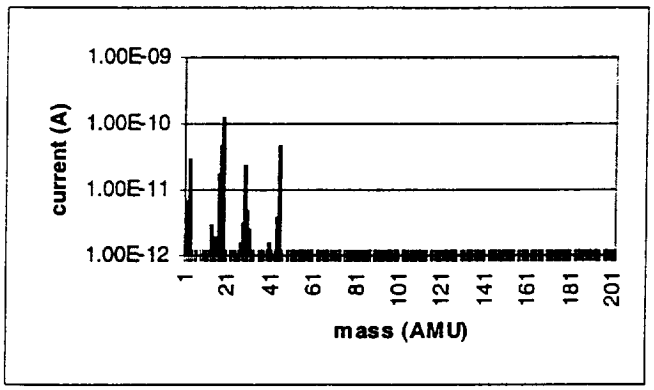

Fig. 6 Outgassing spectrum at beginning of heating at 150 °C

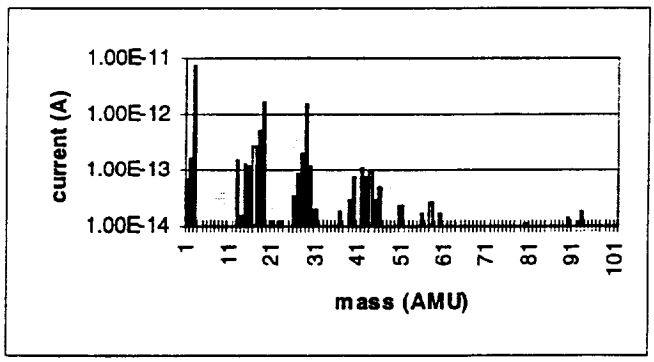

Fig. 7 Outgassing spectrum after several tens hours at 150 °C

The pattern is similar to the one measured at 100 °C.

We switched off the heating and we measured:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
715.3	31	8.7×10^{-9}	4.5×10^{-9}	8.4×10^{-8}

The mass spectrum measured after the thermal cycle is shown in Fig. 8.

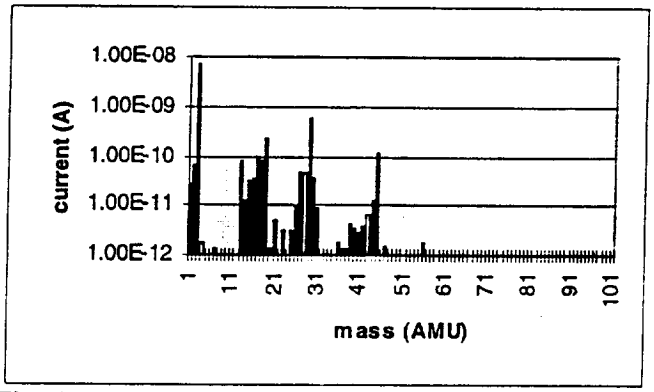

Fig. 8 Outgassing spectrum after the thermal cycle

The outgassing rate evolution is summarized in Fig. 9.

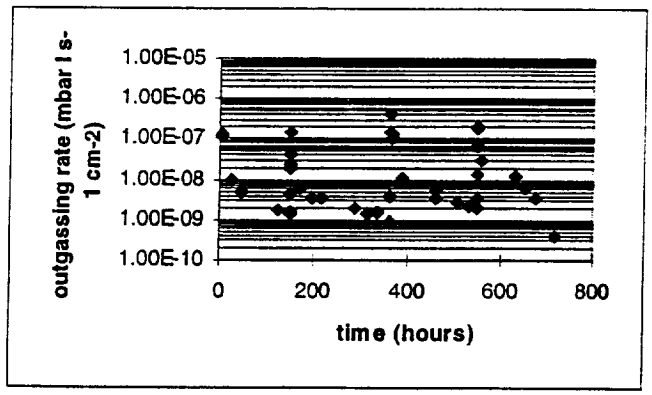

Fig. 9 Time evolution of the outgassing rate

3 - Discussion

The average outgassing rate of the nickel plated steel measured after some days pumping at room temperature was of the order of was ~$2x10^{-9}$ mbar l s^{-1} cm^{-2}, at least one order of magnitude above the value of unbaked metals, but in agreement with literature data for nickel plated parts. The outgassing rate after vacuum baking was ~$4x10^{-10}$ mbar l s^{-1} cm^{-2}, comparable with the outgassing rate of unbaked metals, but at least one order of magnitude above the value for baked metals. Several mechanisms could be responsible for this result and the presence of the observed fragments:

- the samples have been handled with no particular precautions and have been stored in not ideal conditions before the test, so the cleaning could not be very effective
- some compounds used during the plating process cold have been not completely removed before plating, such as nickel chloride hexahydrate and citric acid.

The fact that the fragments appear even at moderate temperatures should be taken into account. We strongly suggest a further test with proper samples.