

# Contamination Produced by Vacuum Outgassing of Kapton Acrylic Adhesive Tape

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3M® 1205 Kapton® film tape with acrylic adhesive is widely used in space technology for vacuum applications. The present work describes the outgassing properties of this tape adhesive, studied by a modified outgassing system. The system is equipped with a quartz crystal microbalance, which is used as one of the collectors. The hot module, containing tape samples with the adhesive facing the cold module, was operated at 40–125°C. The cold module was kept at 5°C and the quartz crystal microbalance was operated at 5 and 15°C. Kinetic parameters of the outgassing and reevaporation processes were derived using an analytical model. The main acrylic contaminant was most likely methyl-methacrylate monomer. An interesting finding was the slow reevaporation rate at 5°C that was highly increased at 15°C. This may lead to the formation of a detrimental contamination layer on nearby surfaces at typical satellite temperatures. As an example, the operation of moving electrical contacts such as slip rings may be degraded due to their increased electrical resistance.

## Nomenclature

$M_q(t)$	=	total quartz crystal microbalance mass
$m_q$	=	mass of the outgassing products deposited on the quartz crystal microbalance
$m_s$	=	mass of the outgassing material within the sample
$m_s(0)$	=	contamination potential
$t$	=	outgassing time
$t_1$	=	end of the outgassing stage
$t_2$	=	end of the quartz crystal microbalance reevaporation stage
$\tau_q$	=	characteristic reevaporation time constant from the quartz crystal microbalance
$\tau_s$	=	characteristic outgassing time constant from the sample
$1/\alpha$	=	view factor from the sample to the quartz crystal microbalance

## I. Introduction

KAPTON® polyimide is extensively used in the space industry due to its superior thermo-optical and mechanical properties, its high stability under UV and ionizing radiations, and its very low outgassing potential. The 3M® 1205 Kapton tape with acrylic adhesive is used in many satellites. This tape is commonly used as a thin-layer insulating material and its outgassing properties were tested according to the American Society for Testing and Materials (ASTM) E595 test [1,2]. NASA materials outgassing database provides several collectable volatile condensable material (CVCM) values ranging from 0.07 to 0.35%, thus approving some batches for space applications and rejecting others according to the CVCM  $\leq 0.1\%$  criterion [3]. It is known, however, that even if a material is approved according to its outgassing properties, it does not completely exclude the possibility of contamination in its vicinity. ESA reported about problems in a satellite related to increased resistivity of relay contacts, which resulted in the deterioration in the performance of switch-relay contacts [4]. It was

suspected that condensation of a contaminant was responsible for this behavior [5], because the contacts were in close vicinity to two foil adhesive materials: polyester adhesive and acrylic adhesive. Several preliminary tests were performed, indicating that outgassed molecules from such tapes can cause appreciable increase in the resistance (several ohms) of precious metal-alloy relay contacts (slip rings). These tests also indicated that polyimide tapes with acrylic adhesive caused bigger changes in resistances than polyimide tapes with polyester adhesive. However, the tests were carried out in air and at an elevated temperature (70°C) and, as such, are not representative of the actual satellite environment in which one could expect enhanced outgassing and contamination due to the vacuum environment and the lower condensation temperatures.

The aim of this work is to evaluate the outgassing properties of the 3M 1205 Kapton acrylic adhesive tape to assess the threats induced by its use in spacecraft. The outgassing experiments were performed in high vacuum at a temperature range of 40 to 125°C using various collectors, such as a quartz crystal microbalance (QCM), a germanium plate, and a metallic surface, all held at 5°C. The reevaporation of the outgassing products was also investigated using the QCM in a temperature range similar to that present at inner surfaces of satellites (i.e., between 5 and 15°C). The chemical nature of the outgassing products was studied by infrared (IR) spectroscopy. Changes in the electrical resistivity resulting from the contamination of the metallic substrate were observed, a behavior closely related to the switch-relay deterioration and its interpretation [4,5].

## II. Experimental

Measurements of the outgassing kinetics were performed in the ASTM E595-compliant outgassing system. The system was modified by replacing one of the collectors by a gold-coated 15-MHz QCM crystal having the sensitivity of  $6.21 \times 10^{-10}$  g/Hz (QCM Research, model MK10). The detailed description of the QCM apparatus was published elsewhere [6]. The Kapton tape with acrylic adhesive was placed in the outgassing system's hot module in such a way that the adhesive side faced the cold module (see Fig. 1). The latter contained the QCM crystal and two additional collectors maintained at low temperatures: namely, a Ge plate and a metallic disc. The Ge plate served for IR spectroscopy measurements of contaminants following outgassing of the tape, and the metallic disc was used to study changes in surface electrical conductivity due to deposition of organic contaminants. The vacuum system, using a dry turbomolecular pump, ensured a base pressure lower than  $1 \times 10^{-7}$  torr.

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The following experimental procedures were applied. In the first experiment, the sample (Kapton tape) was heated to 125°C and the QCM was maintained at 5°C. Having such a temperature regime, the outgassing properties were determined in the conditions defined by ASTM E595 with respect to the total mass loss [2]. However, the collector plates were kept at a lower temperature, representing inner satellite temperatures at which moving contact mechanisms are generally installed, whereas the ASTM E595 standard requires the collector temperature of 25°C [2].

To study the kinetics of the outgassing processes, a more sophisticated procedure was used:

1) There was a stepwise increase of the sample temperature from 40 to 80°C, keeping the QCM, the Ge plate, and the metallic disc at a constant temperature (5°C).

2) The sample was cooled to room temperature while still maintaining the QCM temperature at 5°C, so that reevaporation kinetics of the contaminants were monitored.

3) The QCM temperature was increased to 15°C, studying the effect of the temperature on the reevaporation kinetics of contaminants.

It should be noted that the pressure inside the vacuum chamber was always maintained below  $5 \times 10^{-6}$  torr. In all the experiments, the area of the Kapton tape facing the QCM was identical and equal to 2 cm<sup>2</sup>, and so the mass of the acrylic adhesive was also the same.

The chemical nature of the contaminants was studied by performing transmission mode Fourier transform IR (FTIR) measurements of the Ge plate at the end of the preceding outgassing processes.

The surface electrical resistivity measurements of the metallic disc were carried out by using the experimental setup shown in Fig. 2. The measuring system consisted of an aluminum block that served as an anode on which a stainless steel ball (cathode) was placed. Both were connected to a voltage source through a digital voltmeter. The metallic disc, either clean or contaminated with the outgassing products of the acrylic adhesive, was positioned between the electrodes. A load was applied to the cathode to ensure a constant contact pressure. The current was measured as a function of the applied voltage.

### III. Results and Discussion

#### A. Monitoring the Contaminant Mass by the QCM

Contaminants originated from the outgassing experiments were clearly visually observed on the cold collectors (Ge and metallic plates) as yellow-colored spots, with the thickness of the contamination layer increased toward the center of the plates. The photograph of the contaminated Ge plate and the optical microscope image of the metallic disc surface after the outgassing experiments are shown in Figs. 3a and 3b, respectively.

Figure 4 shows the in situ QCM mass accumulation as a function of the outgassing time for the Kapton tape with acrylic adhesive heated to 125°C. It can be observed that most of the outgassing potential is exhausted after only 7–8 h of heating. As seen from the graph, the mass of the outgassing products deposited on the QCM is  $\sim 12 \mu\text{g}$ . To relate this value to CVCM, the total mass of the acrylic adhesive facing the QCM was determined by weighing the Kapton tape with and without adhesive; the adhesive was removed by immersion of the tape into a commercial stick remover (manufactured by HG, The Netherlands) which does not attack Kapton. The estimated mass of the adhesive was 8.3 mg, which gives a CVCM value of 0.14%. Considering the QCM active area of 0.3 cm<sup>2</sup>, the comparison with a standard outgassing system collector (area of 8 cm<sup>2</sup>) reveals a much higher potential QCM value according to the ASTM E595 standard. One should note, however, that the ASTM E595 defines the sample temperature as 125°C and the collector temperature as 25°C, whereas in our case, the temperature of the collectors was significantly lower (5°C), leading to their higher degree of contamination accumulation.

Figure 5 shows the data from the second outgassing experiment, which represents the kinetics of the outgassing and reevaporation processes. In the first part of the experiment, the sample was stepwise

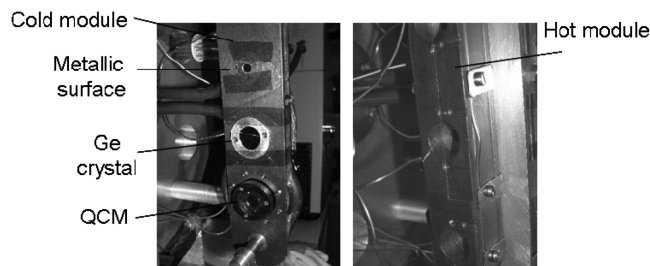


Fig. 1 Outgassing system modules with the Kapton tape attached. The acrylic adhesive faces the collectors.

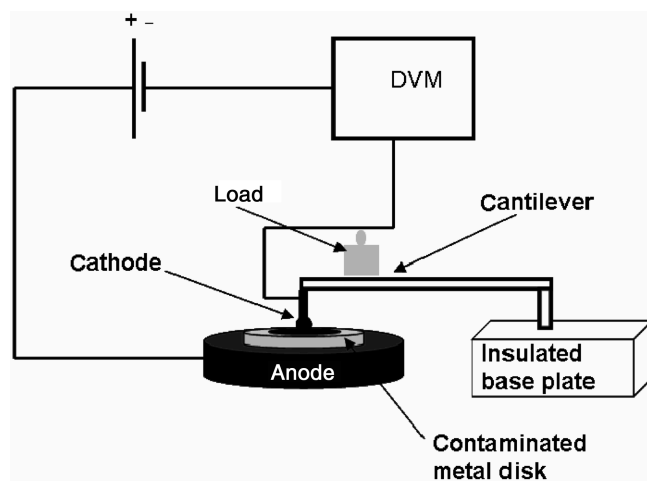


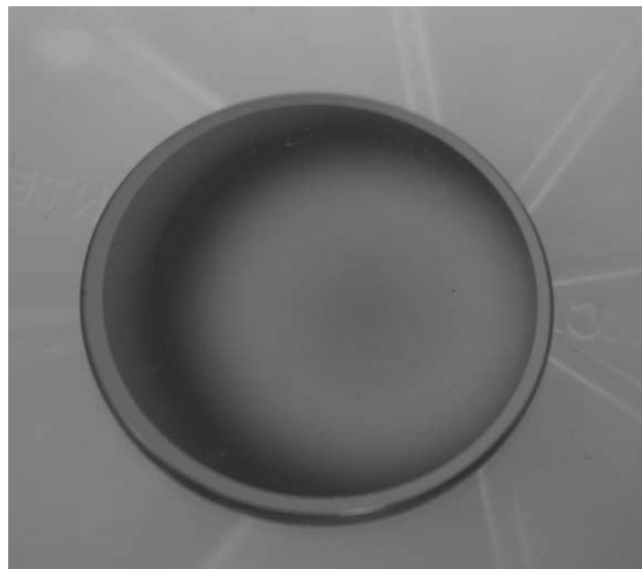
Fig. 2 Schematic design of the surface conductivity measurement setup (DVM denotes the digital voltmeter).

heated to 40, 60, and 80°C, and the QCM and other collectors were kept at a temperature of 5°C (end of this stage at the time  $t = t_1$  is marked by an arrow). It can be observed that at 40°C there was low but measurable outgassing:  $\sim 0.1 \mu\text{g}$  after a few hours; an additional  $\sim 0.25 \mu\text{g}$  of contaminants adsorbed on the QCM when the sample was heated to 60°C for 19 h; the mass of contaminants following heating of the tape to 80°C for 23 h was  $2.3 \mu\text{g}$ ; and the outgassing was interrupted when the desorption rate was far from reaching the plateau. The mass of the adhesive facing the QCM was the same as before (8.3 mg). The kinetics of the outgassing will be elaborated in more detail next.

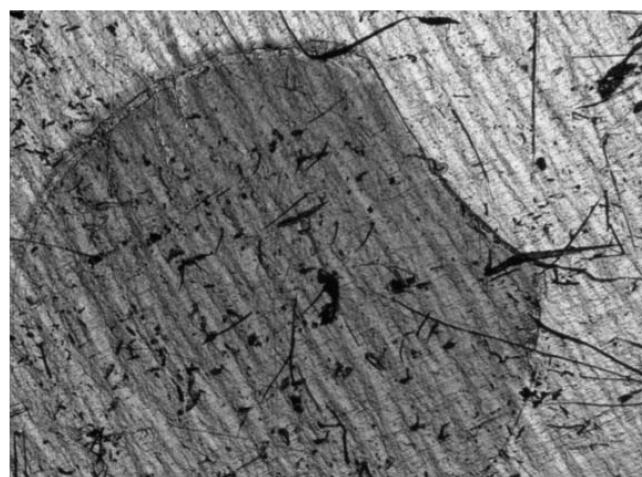
At the second part of the experiment [at the times  $t > t_1$  (see Fig. 5)], the sample (Kapton tape) was cooled down, whereas the QCM was still kept at 5°C. It can be observed that the accumulated QCM mass remains nearly constant over  $\sim 35$  h. Finally, at the time  $t = t_2$ , the temperature of the QCM was raised to 15°C. This moderate temperature change had a significant effect on the mass of the QCM, resulting in a steep decrease due to desorption of the contaminants. The QCM desorption rate was about  $6 \times 10^{-4} \mu\text{g/h}$  at 5°C, whereas at 15°C it was 28 times higher, about  $1.7 \times 10^{-2} \mu\text{g/h}$ , in the linear part of the curve. The kinetics of the reevaporation process will be discussed later in this section.

#### B. Chemical Identification of Contaminants by FTIR

To identify the chemical nature of the outgassing products and to relate it to the studied material (acrylic adhesive), FTIR measurements of the contaminants were performed. For this purpose, FTIR spectra were taken for the Ge plate, which served as one of the collectors in the outgassing system, after each of the two outgassing experiments. It should be noted that identical spectral line shapes were measured for the contaminated Ge plates after high-temperature outgassing (125°C: first experiment) and after the lower-temperature processes (second experiment), with only the intensity being lower for the latter case. This is due to a somewhat lower mass



a)



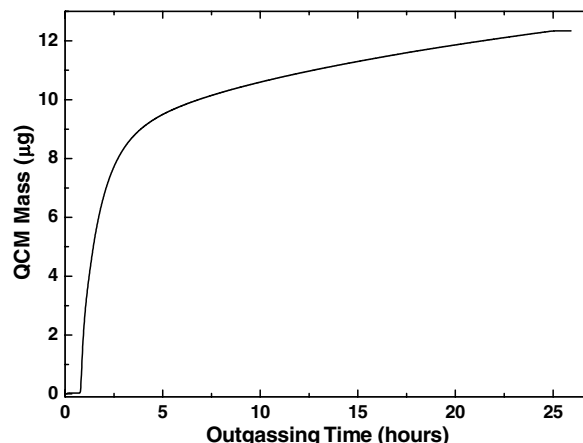
b)

**Fig. 3** Contamination layer: a) photograph on the Ge plate and b) optical microscope image ( $\times 20$ ) on the metallic disk surface.

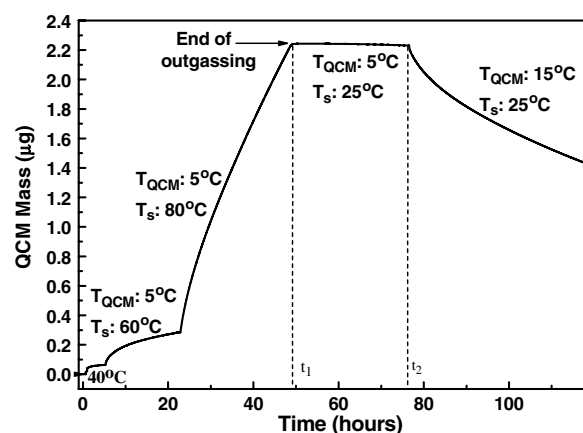
of contaminants adsorbed on the collector, as evident from the preceding QCM data. Therefore, only the FTIR spectrum of the high-temperature outgassing experiment is shown in Fig. 6a. For comparison, Fig. 6b shows a typical FTIR spectrum of the methyl-methacrylate monomer [7]. Note that unlike the QCM, the two collectors (the Ge and metallic plates) were kept at  $5^{\circ}\text{C}$  for the total duration of the experiment until venting the system and were not heated to  $15^{\circ}\text{C}$  under vacuum conditions.

The identification of the outgassing products is complicated due to a probable mixture of different components. According to the assignment of the absorption bands, it may be concluded that the outgassing products contain acrylic monomers, such as acrylic acid and alkyl acrylates. It should be emphasized that in both spectra, the  $\text{C}=\text{C}$  double-bond stretching around  $1635\text{ cm}^{-1}$  was found. This band should not exist in the polymer form of the acrylic adhesive. This is indicative of the low-molecular-weight nature of the outgassing products.

Both spectra contain some common absorption bands due to the  $-\text{CH}_2$  vibrations in alkyl groups around  $2955\text{ cm}^{-1}$ . Other common features are observed at  $\sim 1700\text{--}1730\text{ cm}^{-1}$  for the stretching vibration of the carbonyl group. However, in the spectrum of the outgassing products, both  $1730$  and  $1705\text{ cm}^{-1}$  bands were observed. The lower-wave-number band is most likely associated with the absorption by the acrylic acid carboxyl group. The presence



**Fig. 4** QCM mass accumulation for the outgassing of the Kapton adhesive acrylic tape held at  $125^{\circ}\text{C}$  and the QCM crystal held at  $5^{\circ}\text{C}$ .



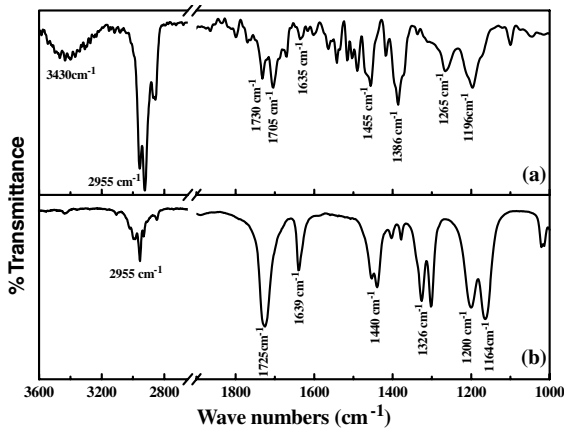
**Fig. 5** Outgassing of the Kapton acrylic adhesive tape at different temperatures and reevaporation of its outgassing products from the QCM.

of an acrylic acid is also consistent with a strong absorption band around  $3430\text{ cm}^{-1}$ , which is generally assignable to the  $-\text{OH}$  stretching vibrations. Absorptions between  $1200$  and  $1260\text{ cm}^{-1}$  are attributed to  $-\text{C}(\text{O})-\text{O}$  and  $-\text{CO}$  stretching bands. Peaks around  $1455$  and  $1386\text{ cm}^{-1}$  may correspond to  $-\text{CCH}_3$  asymmetric and symmetric deformations, respectively. This may be indicative of the methyl-methacrylate monomer of the corresponding polymer.

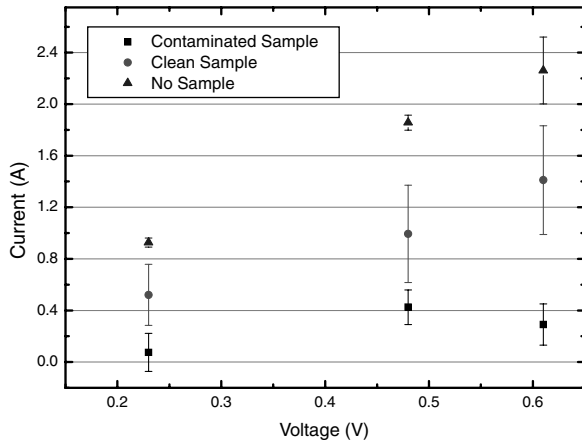
Outgassing products may also contain other hydrocarbon contaminants, based on relatively high intensity of the  $-\text{CH}$  absorption band around  $2955\text{ cm}^{-1}$ .

### C. Surface Conductivity Measurements

To prove that adsorption of the outgassing products of the acrylic adhesive can be responsible for the reduction of electrical conductivity of metal contacts, the following experiment was conducted. Surface conductivity measurements of the metallic disc that was held at  $5^{\circ}\text{C}$  during the outgassing experiments were carried out. Such measurements were quite difficult to perform because the contamination layer was so thin and soft that it was impossible to determine surface conductivity by the common four-point probe: the electrodes penetrated the contamination layer, contacting the metal substrate. Therefore, another approach was used, as shown in Fig. 2. A metallic ball-shaped cathode was placed on the contaminated metallic disc by applying constant force on the cathode. The measurements were carried out in three steps. First, the current-vs.-applied voltage measurements were taken, because the system consists of the stainless steel (cathode) and the aluminum block (anode) only; the metallic disc collector was not attached to the anode



**Fig. 6** FTIR spectra of a) the outgassing products of the Kapton adhesive tape (1205, 3M) and b) typical spectrum of methyl-methacrylate monomer.



**Fig. 7** Surface electrical conductivity measurements: internal current-voltage properties of the system (triangles), reference clean metallic surface (circles), and contaminated metallic surface (squares).

at this point. In such a way, the internal resistivity of the system (electrodes, connectors, electronic devices, etc.) was determined. Then a clean metallic disk was attached and the current was recorded for the same series of applied voltages. Finally, the clean metallic disk was replaced by the contaminated one, and the final set of current-voltage values was measured. These types of measurements were repeated many times with a large deviation in the measured current values. This is attributed to the nonuniformity in the applied contact force and the sensitivity of the measuring system to local minor changes in contaminant thickness and content. However, the qualitative relation between the current-voltage data for the three measured setups was always maintained.

In Fig. 7 the current-voltage data measured for the three preceding setups are shown. The current values are averaged over ten measurements. The calculated internal resistance of the system and resistances of the clean and contaminated metallic discs are summarized in Table 1. It can be concluded from this figure that formation of the contamination layer on the metallic surface indeed results in substantial increase of the resistivity by several ohms with

nonlinear current-voltage dependence. Again, the indicated resistance values are not quantitatively precise, but rather represent the qualitative change in the electrical properties following the contamination of the metal by outgassing products of the acrylic adhesive. Similar contact resistance increase was observed when air outgassing experiments were performed [5].

#### D. Kinetics of Contaminant Outgassing and Reevaporation

The identity between the FTIR spectra measured for the contaminants deposited at 80 and 125°C suggests that there is no apparent difference between the outgassing products of the acrylic adhesive desorbing at these temperatures. From the comparison of the FTIR spectra of the reference methyl methacrylate and the contamination layer on Ge, we can assume that the contaminated layer contains the acrylic component of the adhesive as well as some other hydrocarbon components.

The kinetic data and outgassing potentials of the acrylic adhesive Kapton tape were analyzed using an analytical model previously developed by Soreq Nuclear Research Center [6]. This model considers the outgassing and reevaporation processes to follow a first-order kinetics. The experimental data were fitted to the following equations [6]. At any time  $t$  of the outgassing phase ( $t < t_1$ ),

$$m_q(t) = \frac{m_s(0)\alpha\tau_q}{\tau_s - \tau_q} (e^{-t/\tau_s} - e^{-t/\tau_q}) \quad (1)$$

where  $m_q$  is the mass of the outgassing products deposited on the QCM;  $m_s$  is the mass of the outgassing material within the sample;  $1/\alpha$  is the view factor from the sample to the QCM ( $\alpha = 0.045$  for our system geometry [6]); and  $\tau_s$  and  $\tau_q$  are the characteristic outgassing and reevaporation time constants from the sample and the QCM, respectively (they define the time after which the mass of the outgassing materials reduces by  $1/e$ ). At times  $t > t_1$  (reevaporation stages), the mass of the QCM is defined by Eq. (2):

$$m_q = m_q(t_1)e^{-(t-t_1)/\tau_q} \quad (2)$$

where  $t_1$  is replaced by  $t_2$  for the QCM reevaporation stage at 15°C ( $t > t_2$ ). The outgassing products consist of a variety of molecular species, each with its own set of contamination potential  $m_s(0)$  and time constants  $\tau_s$  and  $\tau_q$ . Arbitrarily dividing the spectrum of the outgassing products into  $n$  distinct species, Eqs. (1) and (2) are generalized by Eq. (3):

$$M_q(t) = \sum_{i=1}^n m_{q,i}(t) \quad (3)$$

The preceding model allows estimating of the accumulated masses of acrylic adhesive contaminants and the characteristic time constants at different outgassing and reevaporation temperatures. One then can use these data at specific spacecraft conditions.

Practically, it may be assumed that the outgassing products of the studied sample consist of the acrylic component and light species, such as water vapor and adsorbed atmospheric gases. The outgassing process of these light components involves fast desorption from the sample toward the QCM according to the system's geometrical view factor  $\alpha$  and then migration from the QCM stimulated by the continuous pumping of the system. We can assume, therefore, that at sufficiently long outgassing times and relatively high outgassing temperatures (60°C and above), the measured mass of the QCM mostly represents its contamination by the acrylic component only. As seen from Fig. 5, the reevaporation rate of the contamination layer from the QCM at 5°C is practically zero, and so we may assume that the time constant  $\tau_q$  tends to infinity at this QCM temperature and  $\tau_s \ll \tau_q$ , thus simplifying Eq. (1) (the latter assumption is valid for the light outgassing components as well). By dividing the outgassing products into two main groups (light fragments, denoted as component 1, and the acrylic adhesive, denoted as component 2) and fitting the experimental curves in Figs. 4 and 5 (outgassing stage,  $t < t_1$ ) to Eqs. (1) and (3), the characteristic time constants and QCM

**Table 1** Surface resistances as obtained from the current-voltage measurements

Material	Average resistance, $\Omega$
Without sample	0.3
Metallic surface (clean)	0.5
Metallic surface (contaminated)	2.1

**Table 2** Kinetic parameters of the outgassing phase

Temperature, °C	Component	$\tau_s$ , h	$M_q$ , $\mu\text{g}$
40	1	0.25	0.15
	2	80.2	
60	1	1.6	0.7
	2	66.7	
80	1	0.5	3.2
	2	29.0	
125	1	0.8	12.5
	2	11	

masses extrapolated to long outgassing times (greater than 100 h) are estimated for each outgassing temperature and shown in Table 2.

It should be noted that outgassing at 125°C was performed in a single step, which explains the higher outgassing time constant for the light components. The light components outgassing at this temperature may also include some weakly bound fragments of the acrylic adhesive as compared with the bulk material. It is clear from the preceding data that long outgassing times are required for acrylic adhesive outgassing at  $T \leq 80^\circ\text{C}$ . The Kapton tape heated to 80°C produces  $\sim 3 \mu\text{g}$  of contaminants on the QCM, as shown in Table 2. The actual contamination mass values shown in Table 2 may be an order of magnitude higher when deposited on the satellite parts, due to view factors substantially higher than that of the QCM. The multilayer contamination coatings can be adsorbed on the sensitive parts such as moving contacts or optical devices.

Turning now to the kinetics of the reevaporation process at 15°C, the appropriate data are fitted to Eq. (2). Good fitting is not accepted until a two-component emission process is assumed with  $\tau_{q,1}$  and  $\tau_{q,2}$  equal to 3 and 110 h, respectively. As was already explained, FTIR data suggest that the contamination layer on the QCM contains the acrylic fragments along with some other hydrocarbon contaminants. The two components outgassing from the QCM at 15°C with different desorption rates either represent two different chemical components of the contamination layer or they can be related to a surface layer having a higher desorption rate than that of bulk layers. It can be seen from the reevaporation data that an increase of the QCM temperature by only 10°C removes considerable parts of the contaminants, albeit the time required for outgassing of the microgram quantities of the contaminants is still very long.

It can therefore be deduced that this Kapton acrylic adhesive tape should not be used as a free-standing film. It should be used with caution, even when only the tape edges are exposed. Its use may require additional treatments such as baking of the interior before the launch and an option to heat nearby sensitive parts during the mission, to remove possible contamination layers. The last method can be the most effective, because the contaminants reevaporation process at 15°C is orders of magnitude faster than at 5°C. The best

results will be obtained if heating of the sensitive parts is performed on a frequent and regular basis to avoid formation of a thick contamination layer that will considerably prolong the time required for its removal.

#### IV. Conclusions

In summary, although space-used, the 3M® 1205 Kapton® acrylic adhesive tape has been found to be extremely volatile, bearing in mind the negligible adhesive material weight. It may be assumed that outgassing of the acrylic component occurs even at temperatures lower than 40°C, albeit at a very low rate. Its outgassing products remain practically adsorbed at 5°C: the actual temperature in many satellite inner surfaces. This behavior results in the contamination of satellite parts in its proximity and potential deterioration of their performances. We proved that formation of such a contamination layer significantly reduces the electrical conductivity of a metallic surface and can be responsible for the degradation of devices based on moving contacts (e.g., slip rings). Thus, though, in general, the mass of the exposed acrylic component is very low, its outgassing products can present a considerable threat to sensitive satellite components.

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