



**OUTGASSING BEHAVIOR OF  
NANOCOMPOSITE MICROMETEORITE  
DEBRIS IMPACT SENSORS**

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# *Abstract*

Nowadays, space industry is evolving fast and ensuring the performance of the space vehicles or satellites is really relevant, not just to save money by saving in maintenance, but to improve the functionality and performance of such vehicles and other applications. In this sense, material selection becomes key; outgassing phenomena is one of the main phenomena that has to be taken into account, and therefore some research about the topic has been carried to better understand what materials are more likely to suffer it in a greater amount. It was concluded that the materials with worse outgassing properties are polymers, i.e. polymeric composites, adhesives, thermal greases, etc. A detailed description about the experimental test to determine those properties is explained on this paper, including some indications on how to perform named test.

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# Glossary

Symbol	Description	Units
CVCM	Collected Volatile Condensable Material	%
TML	Total Mass Loss	%
WVR	Water Vapor Regained	%
RML	Recovered Mass Loss	%
$m_i$	Initial mass	<i>g</i>
$m_f$	Final mass	<i>g</i>
$q_{out}$	Specific outgassing rate after t hours	$Pa \cdot L \cdot cm^{-2} \cdot s^{-1}$
$q_{1h}$	Specific outgassing rate after 1 hour	$Pa \cdot L \cdot cm^{-2}$
$t^\alpha$	Hours of evacuation	h
$Q_{out}$	Total gas flow	$Pa \cdot L \cdot s^{-1}$
V	Void volume in the chamber	$cm^3$
$dp/dt$	Pressure increase rate	<i>L/s</i>
$A_s$	Surface area of the sample	$cm^2$
$A_a$	Surface area of the apparatus	$cm^2$
$q_s$	Specific outgassing rate of the sample	$Pa \cdot L \cdot cm^{-2} \cdot s^{-1}$
$q_a$	Specific outgassing rate of the apparatus	$Pa \cdot L \cdot cm^{-2} \cdot s^{-1}$
<i>S</i>	Pumping speed at exit from the chamber	<i>m/s</i>
p	Chamber pressure	<i>Pa</i>

Many environmental constraints are surrounding the materials selection for design of spacecraft structures. The chosen materials must be lightweight to maximize the allowable payload mass while being stiff enough to maintain precision alignment of instruments under severe orbital environmental conditions. Therefore, selecting an optimum material from a wide range of available candidate materials for a specific application would be a difficult task. Specially, knowing that space environmental aspects (i.e. over 21 different phenomena) can affect both mechanical properties of materials and also performance of spacecraft devices. To deal with such a complicated problem, one would need to employ appropriate tools.



Figure 1.1: Inflatable structure to be sent to the space, where it is going to be subjected to space environmental aspects, being outgassing one of them.

Material selection is highly affected by the vacuum conditions. The outgassing deforms the structure of spacecraft, causing contamination and failure of dimensional stability. Thus, outgassing is a severe vacuum effect, and it is basically the release of gaseous species from the material sample under high vacuum conditions. To avoid bad performance of the parts conforming the aerospace vehicles, engineers have to take this phenomenon into account when deciding the materials to be used. To qualify the materials for space use one has to follow specific tests and the materials have to meet the standards.

The outgassing phenomena, the material selection, the standard test and the per-

formance of specific material families are going to be covered this text, and tests will be carried for nanocomposite material samples made of carbon nanotubes and epoxy polymer matrix in order to determine their reliability to be used in space vehicles without losing properties due to the vacuum conditions. The major concern is the performance of the polymer and the epoxy, since this kind of materials are more susceptible to be affected by the vacuum conditions and have high outgassing properties. The apparatus to do the test it is been designed by another student at Embry-Riddle Aeronautical University and fabricated for this purposed, and the vacuum facilities in the College of Arts and Sciences (COAS) will be employed for the experimentation performance. Once the test is done the results will be compared with the NASA's material ougassing database and standards to conclude on the material's stability level.

A thorough understanding of the outgassing behavior of materials under vacuum conditions, specially the material that is going to be tested, will be key in order to design an efficient vehicle, and that is the major challenge of the project.

## *Outgassing and the TML test*

### 2.1 Outgassing phenomenon and the need for tests

The outgassing effect is a phenomenon where a material releases gas that was trapped inside it, usually due to the heat or reduced pressure. It is really important to have this problem into account when designing aerospace vehicles because of two main reasons: the properties of those materials could change because of the mass loss so the performance of the materials would get worse, and the released gas converted to particles could cause problems in parts like solar panels or optic instruments, which need a delicate transparency to operate. Also local clouds may form that could affect some instrument readings.

Outgassing includes desorption, diffusion, vaporization and decomposition. Desorption is the process by which molecules of a volatile substance, that were previously absorbed to the surface of the material, detach themselves and escape into the ambient air. Desorption depends strongly on local temperature. Diffusion happens when gases naturally diffuse from the interior and from the bulk materials and escape due to the pressure difference between the outside environment and the interior of the spacecraft. Thus, this process is determined by pressure variations and temperature has little effect. The last process is decomposition. After long periods of time in extreme vacuum conditions, some materials will undergo chemical decomposition, which can be significant for the overall outgassing. These phenomena can be observed graphically at Figure 1.1 [1].

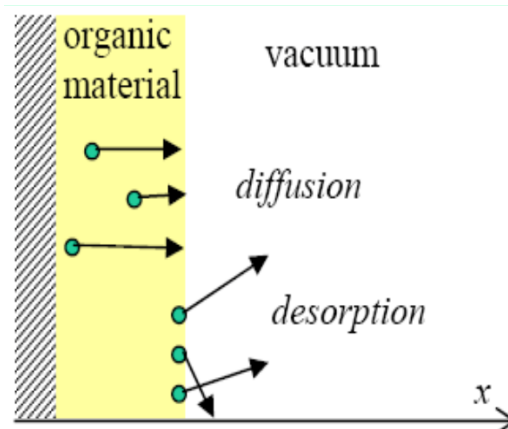


Figure 2.1: Outgassing processes explained graphically [1].

When a material is exposed to vacuum, the gas release or outgassing occurs basically because of three phenomena previously mentioned: diffusion from the material, desorption from the surface of the material and vaporization of the material. Vaporization occurs in all materials, and is characterized by the vapor pressure of the material. If the vapor pressure of the material is higher than the operational pressure, the phenomena should be considered. For example, for a plastic material the vapor pressure is usually between  $10^{-7}$  and  $10^{-5}$  Pa, and considering the standard space pressure condition as  $10^{-3}$  Pa, the vaporization phenomena can be safely neglected when talking about outgassing. Another phenomena to be analysed is the desorption. Gas molecules trapped or bound on the surface are slowly released by this mechanism that could be theoretically interpreted using kinetic or thermodynamic approach, although it is really difficult to obtain accurate results due to complicated molecular behaviors. Empirically, it is been proved than the phenomena is inversely proportional to time [16]. Finally, gas diffusion from the interior of the material is characterized by a concentration gradient. The rich concentration of initially dissolved gas inside the material pushes it to the surface, where it is released from the surface after undergoing a desorption process. This phenomena is the slowest of the three described, and thus is to be considered the controlling factor for the outgassing rate considering long-term uses such as space applications.



Figure 2.2: Lenses and other optical instruments can get contaminated by outgassed material.

Despite the perfected productive processes, there are always trapped light molecules in the materials used for building structures for aerospace vehicles. Under the atmospheric pressure, those molecules stay inside the material, but when these components are under the vacuum conditions of the space environment, nothing can be done to retain them. They tend to escape in a form of a gas, and after that the debris created can end up forming a layer of coating that could cover other surfaces of the vehicles,

sometimes without causing problems and other times with some performance deterioration. That is the case, for example, of the optic instruments (Figure 2.2), solar panels and radiators. Thus, the material stability ranking not only depends on the outgassing test results but also on its relative position with the sensitive elements.

The materials that more likely cause this phenomenon are lubricants and adhesives, but some light metal alloys and composites have to be add to the list too. In order to avoid bad performance of the aerospace vehicles, it is important to follow improved engineering processes and material selections. Consequently, some standard tests have been carried in order to determine what materials fit the necessary conditions to be used in the space environment. There are a few outgassing measurement methods such as the pressure rise method, the throughput method and the total mass loss method [16]. In this study the last is going to be mainly considered.

## 2.2 Test methods and specimens

As it is been said, there are some requirements regarding outgassing stability in order to use a material for a space application. There are two main standard tests used for that purpose. The ESA certified test is the European Corporation for Space Standards "ECSS-Q-ST-70-02", but a material that is going to be used by NASA is required to meet some specifications and this can be tested by performing the American Society for Testing and Materials "ASTM E595-15" standard test. This experiment method covers the technique used to determine the volatile content of materials when exposed to a vacuum environment, and it is the one that is going to be considered for this project's purpose.

It has to be noted that this test is a screening technique for material selection, but it can't be considered to compute or simulate the actual contamination or outgassing in the real space environment, since the conditions could change. Further analysis should be done in order to determine if the material of the component under study will remain stable and the performance satisfactory.

### 2.2.1 ASTM E595-15

This standard test can be used for many types of materials: polymeric, organic and inorganic materials. These include polymers, elastomers, insulations, fabrics and composite materials [2]. The test provides the necessary data that is going to determine the acceptance or rejection of the material to be used at the space. Thus, the test

provides the Collected Volatile Condensable Material (CVCM), the Total Mass Loss (TML), the Water Vapor Regained (WVR) and the Recovered Mass Loss (RML).

- **Total Mass Loss:** The TML is the total mass of material outgassed from a test specimen that is maintained at a specified constant temperature and operating pressure for a specified time. The TML is obtained by measuring the weight of the sample before and after the test and then subtracting the final weight from the initial one, and is expressed as a percentage of the initial mass.

$$\%TML = \left( \frac{m_i - m_f}{m_i} \right) \times 100 \quad (2.1)$$

Where  $m_i$  is the initial specimen mass and  $m_f$  is the final specimen mass.

- **Collected Volatile Condensable Material:** The CVCM is the mass of outgassed substances from a test specimen that condenses on the collector maintained at specific temperature for an specified time. This parameter is as well expressed as a percentage of the initial mass and it can be calculated as the difference between the mass of the collector before and after the test.
- **Water Vapor Regained:** This parameter is the mass of the water vapor regained after the specimen is reconditioned. The WVR is calculated as the difference in the specimen mass after the TML and CVCM have been tested, and after exposure to a 50 % relative humidity at 23°C for 24h. It is also expressed as a percentage of the initial specimen mass.
- **Recovered Mass Loss:** The RML is the difference between the TML and the WVR.

Those values are evaluated under the following conditions: Material sample subjected to 125°C at less than  $7 \cdot 10^{-3} Pa$  or  $10^{-6} torr$  for 24h [3]. The overall mass loss can be separated into two categories: noncondensables and condensables [5]. The condensables are the ones that are capable of condensing in a collector at 25°C.

The reason for using such a high temperature is to obtain an accelerating effect. The test last 24h, but since it is subjected to 125°C that is representative of some hundreds of days in vacuum at room temperature [4]. This way, the test represents the actual outgassing of years of operation. The temperature of the test is recommended to be at least 30°C higher than the expected maximum service temperature of the mission. If expected temperatures exceed 65 to 70°C, the test temperature should be increased.



**Pass/Fail requirements**

The outgassing requirements for the material selection are the ones listed below:

- TML < 1.0 %
- CVCM < 0.10 %

Where the Total Mass Loss (TML) is the Water Vapor Regained plus the Recovered Mass Loss:

$$TML = WVR + RML \tag{2.2}$$

and the CVCM is the quantity of outgassed matter from a test specimen that condenses on the collector [3]. As it is been said, the specimen is held at 125°C, but the collector is at 25°C.

There is one exception for these requirements that allows the material to still be considered to operate on a space environment: if  $TML > 1.0 \%$  and  $CVCM < 0.1 \%$  the material will still be considered if the difference between the TML and the WVR is less than 1 %. This is, if  $RML < 1 \%$ . If the material doesn't meet any of these conditions, the material fails.

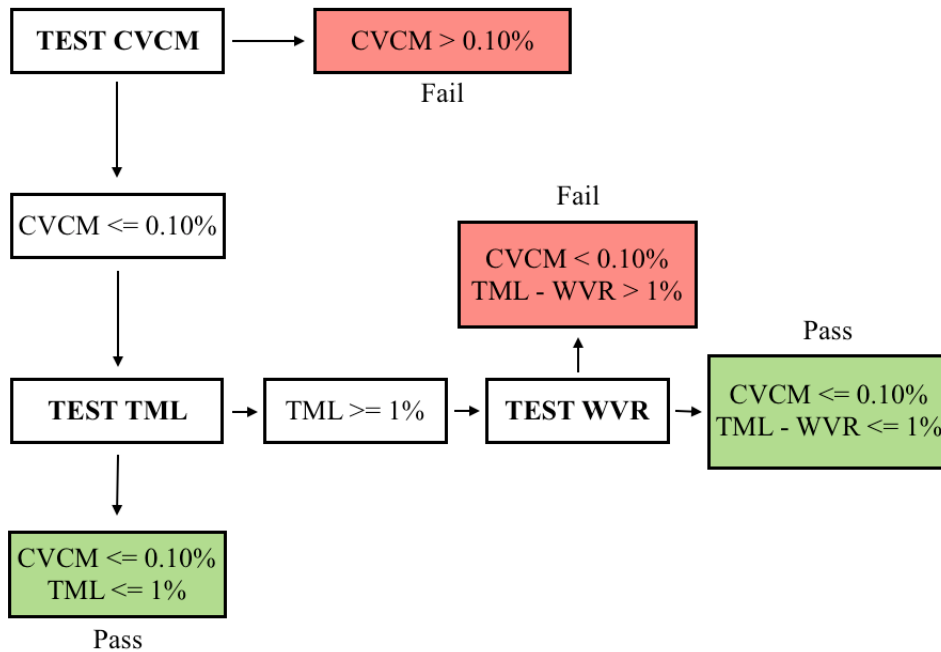


Figure 2.3: Schematic description of the pass/fail requirements.

## 2.2.2 Materials and equipment

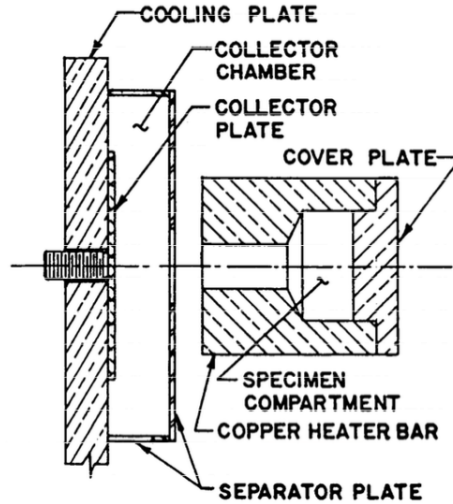


Figure 2.4: Schematic of the test apparatus [2].

The test follows the described procedure: first, the material specimen has to be exposed to 23°C and 50% of relative humidity for 24h inside a weighted container. After this, both the container and the specimen have to be weighted and will be placed in the specimen containers of the copper plate, part of the test apparatus [6]. This copper component will have a heating system to rise the temperature to the one desired. Once the material is set into the copper place, the whole apparatus will be placed into the vacuum chamber and evacuated to a vacuum of at least  $10^{-6}$  torr during 24h. Since the temperature is raised, this causes vapor to stream from the specimen place, and a portion of this vapor will pass into the collector chamber in which the condensable mass loss will be gathered and ready to be weighted.

With this results the data can be calculated. Usually three or more samples of the material will be tested at the same time to obtain average data. Also, some of the holes in the copper plates for the specimens could be leave empty to control the cleaning procedures after each test.

To carry this test procedure, the following materials and equipment are required:

### Micro-VCM apparatus

The device was designed by another student in a previous research and it is the most critical part of the experiment. The apparatus was designed to meet the objectives of the overall outgassing project. Its function is to heat the test specimen in

a copper chamber at 125°C and receive the CVCM on a collector plate at 23°C in a cooler chamber that is 0.03" away. Specific information about design and fabrication is covered in [6].

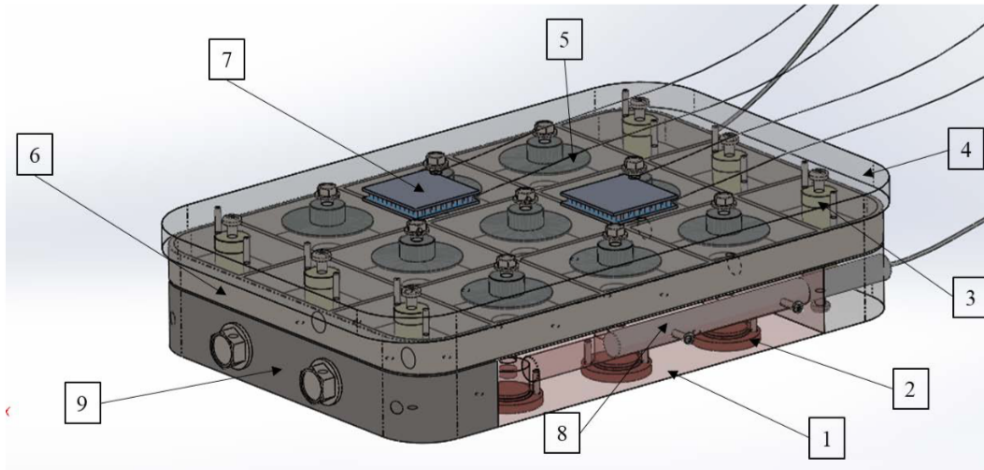


Figure 2.5: CAD model of the final apparatus design [6].

The parts conforming the apparatus are:

1. Heating plate
2. Chamber cover
3. Ceramic standoff
4. Aluminum plate
5. Collector plate
6. Separator
7. Cooling Peltier
8. Cartridge heater
9. Holder

In Figure 2.3 the final apparatus is shown with the peltiers on the surface:

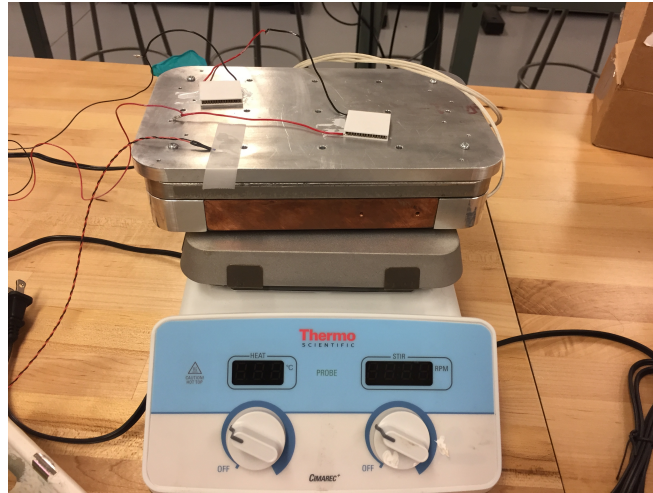


Figure 2.6: Apparatus used for the outgassing tests.

### Vacuum Chamber

The vacuum chamber creates the atmospheric pressure needed for the outgassing test. Embry-Riddle Aeronautical University has two vacuum chambers in the College of Arts and Sciences, the Large Vacuum Chamber and the Micro-plasma Chamber. Ideally the Micro-plasma chamber would be used because its simplicity and ease of access but it was found out that this chamber can't reach the required vacuum pressure for the moment, so the large one will be used finally with the proper set-up modifications until the other is ready to be used.



Figure 2.7: Large Vacuum Chamber at ERAU's COAS Astrophysics laboratory.

### Specimen boats

In order to hold the material specimens to be tested during the experiment, some aluminum dishes will be used.

## 2.3 Outgassing rate

A mathematical model for calculating the outgassing rate of a material is going to be explained. As it is been discussed in a previous section of the document, outgassing phenomena involves vaporization, desorption and diffusion, but since diffusion is the slowest mechanism is the one that is considered as the controlling factor for the outgassing rate of a material.

Although the outgassing topic has been studied in many researches and it is a state-of-the-art subject matter, it is still difficult to exactly define or predict the behaviour of all materials. The microscopically irregular surface of a material, the material properties or the different phenomena involved in the process make this mechanism a really complex one. Thus, it has to be noted that it is recommendable to accompany every theoretical study with some experiment, because there is not a well established theorem to characterize it yet.

Outgassing phenomena can be defined empirically as:

$$q_{out} = \frac{q_{1h}}{t^\alpha} \quad (2.3)$$

where  $q_{out}$  and  $q_{1h}$  are the outgassing rates at  $t$  hours and 1h after the start of the evacuation and  $\alpha$  is the decay exponent which is considered to be between 0.2 and 1.2. Depending on which mechanism, diffusion or desorption, is to be considered to control the outgassing, lower or greater value for alpha will be used respectively. This mathematical model can be apply to both the Total Mass Loss method and the Pressure Rise Method.

When outgassing phenomena is present and a chamber is completely sealed and set at a determinate pressure, the chamber pressure begins to rise due to outgassing. If the pressure is measured before and after, the total gas flow  $Q_{out}$  by outgassing can be expressed as:

$$Q_{out} = V \frac{dp}{dt} \quad (2.4)$$

where  $V$  is the void volume in the chamber. The outgassing occurs in both the material tested and the apparatus and the chamber itself. Thus, this background

should also be considered when describing the outgassing rate. Then, the total gas flow due to outgassing becomes:

$$Q_{out} = A_s q_s + A_a q_a \quad (2.5)$$

where  $A$  is the surface area,  $q$  is the specific outgassing rate and the subscripts  $s$  and  $a$  refer to the sample and the apparatus. If the two equations are combined, the specific outgassing rate can be calculated as:

$$q_s = \frac{V}{A_s} \cdot \frac{dp}{dt} - \frac{A_a}{A_s} \cdot q_a \quad (2.6)$$

Ideally the second term of the equation accounting for the apparatus and chamber effect should be minimized to obtain reliable results for the material to be tested. The way to calculate this is to carry the test without an specimen. If the background outgassing rate accounts for a small portion of the main material outgassing rate, this effect can be ignored.

Some researches have been carried to mathematically determine the diffusive outgassing based on Fick's Law, and some other authors have described the way to consider the surface desorption in the outgassing mathematical model as well, but these studies are beyond the literature review of this project.

# 3

## *Alternative to the TML method: Pressure Rise Method*

In this chapter an alternative method to the Total Mass Loss method is going to be described. The alternative to the Total Mass Loss method is the Pressure Rise Method. The schematic of this method can be observed at Figure 3.1.

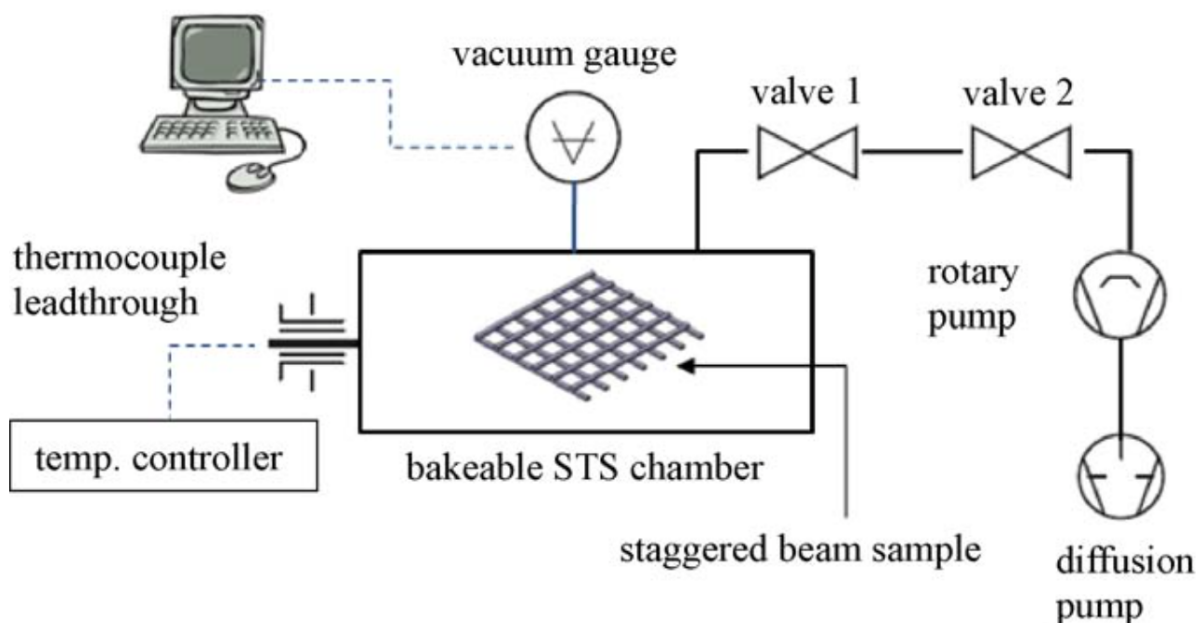


Figure 3.1: Outgassing measurement apparatus using pressure rise method [17].

The experimental setup consists of a bakeable stainless steel chamber equipped with a vacuum gauge, some valves, the thermocouple leadthrough and a gas admittance system. The gauge indicates the pressure of the gas inside the chamber, and depending on the gas used the user will have to apply the proper correction factors. All components should be sealed to avoid leakages. Another important thing that should be done in every outgassing test is to bake and degas the test apparatus several times to minimize the outgassing from the chamber itself, because that could influence the results for the actual outgassing test of the material of interest. In addition, a leak test has to be carried to inspect and ensure that the chamber doesn't have relevant leakages.

All the experiments have to be carried out at 23°C and 50% of humidity room.

The basis of the pressure rise method is the general flow equation:

$$Q = V \cdot \frac{dp}{dt} + Sp \quad (3.1)$$

where  $Q$  is the total outgassing rate in the test chamber,  $V$  is the volume of the chamber,  $dp/dt$  is the pressure rise rate in the chamber,  $p$  is the chamber pressure and  $S$  is the pumping speed at exit from the chamber. In order to carry the test the pumping speed should be zero because the chamber has to remain isolated, so the resulting equation becomes

$$Q = V \cdot \frac{dp}{dt} \quad (3.2)$$

The procedure of the experiment is the following:

1. The valves connected to the chamber have to be shut down to isolate the test chamber and be able to depressurize the system.
2. Place the material sample into the chamber and sealed the chamber off.
3. Evacuate the chamber to a base pressure and, after the valves are closed and the system is isolated, measure the pressure rise over time.
4. With that pressure-time rate and the Equation 3.2, the outgassing rate can be calculated.

As it is been said, it is important to evaluate the background outgassing effect. In order to do that, the user should measure the outgassing rate without the sample in the chamber. If the background outgassing effect is small enough in comparison with the total outgassing rate, the background outgassing effect can be ignored. If not, the empty chamber outgassing contribution has to be subtracted from the total outgassing rate to obtain the material sample outgassing rate.

In addition, the user has to make sure the value of  $S$  (the pumping speed) is zero. Otherwise that would be a violation of the assumption to obtain Equation 3.2.

There are two major types of errors and limitations when measuring outgassing rates. One is due to the inability of the system to produce free outgassing, and the second type is caused by the inaccuracies in the measurements, including instrument errors and other such as the leak measurements of the system, and those errors occur in all the methods used for measuring outgassing rates. However, the pressure rise method for outgassing rate determination is one of the least accurate because there might be an inherent increase in the ambient pressure that could interfere with the results.



## 4.1 Material selection for outgassing

With the pass of the years the materials selection procedures for engineering applications have evolved and improved, and the more mechanical properties are involved in the process, the more reliable is the material choice. However, having into account the space environmental effects is a difficult task still in development. The aim thus is to come up with a material selection procedure that allows the engineer to have into account both mechanical properties and space environmental effects. This will allow the designer to better choose the material that suits the specific characteristics of a mission.

Application of the traditional material selection methods is not much popular in space environment. When designing for space applications it is important to take special things into consideration. There are more than 21 space environmental effects and the importance of each of them depends on the orbit where the spacecraft is going to operate, between others. Research has been made through this project and it is been found that some authors have developed a road map for employing systematic material selection methods in the field [3]. Their procedure consists in engaging the mechanical properties with one of the space environmental conditions in the selection procedures. This way the incapability of some methods in dealing with the space conditions could be overcome. The fact of considering the mechanical properties in conjunction with the space environmental effects will provide more reliable ranking of the candidate materials, and the more effects one takes into account, the better outcome of the selection procedure.

There are lots of environmental factors affecting spacecrafts, but the predominant one for the aim of this project is the vacuum condition meaning the outgassing phenomena. Thus, to simplify the material selection problem, this effect will be considered with the conventional mechanical properties. The introduced material selection methods are the Digital Logic (DL), the Modified Digital Logic (MDL) and the Z-transformation. All of them are quantitative materials selection methods.

### 4.1.1 Digital Logic (DL)

There are a lot of methods which are used for dealing with quantitative and qualitative properties of materials. Ashby [7] was pioneer in this field, and from his study three types of material selection methodology can be identified [8]: searching based on

quantitative analysis (the most famous is the graphical engineering selection method or ranking method, or the material selection charts), the checklist based on expertise capture which are knowledge-based and intelligent data based systems, and the inductive reasoning and analog procedure. However, in recent years the digital quantitative methods have received more attention [9,10]. One of these methods is the Weighted-Properties Method (WPM) introduced by Farag in 1997 [8]. In this method each material property is assigned a certain weight, depending on its importance to the performance of the part in service. The weighted-property value is obtained by multiplying the numerical value of the property by the weighting factor ( $\alpha$ ). Then, all the individual weighted-property values of each material are summed to give a comparative material performance index ( $\gamma$ ). The higher the performance index, the more suitable the material for the application. The drawback of this method is that the relative importance of each property is not clear. Thus, it is an intuitive method not much reliable. To overcome this disadvantage, the Digital Logic method adopts a systematic approach for determining  $\alpha$ . To evaluate the properties, only two of them are considered at a time. By doing this, the more important property or performance goal is given the number 1 and the less important is given 0. Relative weighting factor  $\alpha$  for each property is obtained by dividing the number of positive decisions for each goal into the total number of possible decisions  $N$ . This way, the weighting factor  $\alpha$  for each property is defined between 0 and 1. Then, each property is scaled in a way that its highest numerical value does not exceed 100. One property is considered at a time when the candidate materials are evaluated. The best value on the list will be rated as 100 and the others will be scaled proportionally. To do this dimensionless, the scaling factor  $\beta$  is introduced. When the highest value of a property is of interest, the following equation has to be used:

$$\beta = \frac{\text{Numerical value of the property}}{\text{Maximum value in the list}} \times 100 \quad (4.1)$$

If the lowest property value is seek:

$$\beta = \frac{\text{Minimum value in the list}}{\text{Numerical value of the property}} \times 100 \quad (4.2)$$

Once all properties are scaled, the material performance index  $\gamma$  can be calculated as follows:

$$\gamma = \sum_{i=1}^n \beta_i \alpha_i \quad (4.3)$$

where  $n$  are all the relevant properties [9,10].

---

### 4.1.2 Modified Digital Logic

In the Modified Digital Logic method defined by Manshadi et al. at [10], the property evaluation is changed so that the less important value is assigned a 1 while the more important one is assigned a 3, when two properties are considered at a time. This way, the least important property still remains in the list oppositely to the Digital Logic method, when some times properties were completely eliminated of the list. Also, another advantage is that if two properties have the same importance, they can have the same value (2) assigned. In addition, the linear scaling of the properties by DL method is replaced by two different nonlinear functions to normalize the scaling factor  $\beta$ :

$$\begin{cases} \beta = a_1 \ln(b_1 X + c_1) & \text{for } X_c \neq X_{max}/2 \\ \beta = \frac{200X}{X_{max}} - 100 & \text{for } X_c = X_{max}/2 \end{cases} \quad (4.4)$$

$$\begin{cases} \beta = a_2 \ln(b_2 X + c_2) & \text{for } X_c \neq 2X_{min} \\ \beta = \frac{200X_{min}}{X} - 100 & \text{for } X_c = 2X_{min} \end{cases} \quad (4.5)$$

where  $X$  is the numerical value of the property, and  $a_1$ ,  $a_2$ ,  $b_1$ ,  $b_2$ ,  $c_1$  and  $c_2$  are constants. The first equation is used when higher values are desired and the second when lower value is the primary concern.  $X_c$  is a user defined parameter to assign a critical value to each property. When the value of the property is less than  $X_c$ , the scaled property becomes negative and the performance index is reduced.

This method has some disadvantages too: the dependence on  $X_c$ , some problems with scaling functions, the indefiniteness of some of the constants in some cases, and the unstandardized scaling of the properties. All these problems can affect the ranking of the materials. Therefore, another method is proposed to replace the nonlinear scaling functions in the Modified Digital Logic method.

### 4.1.3 Z-Transformation

Although the MDL method has improved the DL method, it might still need some modifications. The final ranking of the candidate materials should be find more efficiently, and also the methodology is quite complicated and time consuming. Moreover, the fact that it employs nonlinear scaling functions could be avoided in order to make the method simpler. Furthermore, the use of the variable  $X_c$  makes the process user knowledge dependent. Thus, the Z-Transformation method provides a new normalization function that simplifies the calculations, has accordance with statistic normalization and increases the reliability of the MDL method by eliminating the user-depending variable [8]. In this method the same weighting factor  $\alpha$  as for MDL method will be

used because it is able to show a good comparison between material properties and their level of importance.

The Z-value, which would be the equivalent to  $\beta$ , is a dimensionless quantity defined by:

$$Z = \beta = \frac{X - \mu}{\sigma} \quad (4.6)$$

where X is the individual score that is to be standardized,  $\sigma$  is the standard deviation of the population and  $\mu$  is the mean of the population. Those variables are calculated by:

$$\sigma = \sqrt{\frac{1}{N} \sum_{i=1}^N (X_i - \mu)^2} \quad (4.7)$$

$$\mu = \frac{1}{N} \sum_{i=1}^N X_i \quad (4.8)$$

where N is the number of candidate materials. If the lower value of the property is of interest, the function to use will be:

$$Z = \beta = -\frac{X - \mu}{\sigma} \quad (4.9)$$

The overall material performance index is calculated as the previous method, shown in Eq. 3.3.

Once these material selection methods are introduced and after reviewing some study cases, some conclusion can be made. Although the Digital Logic and the Modified Digital Logic are widely used, the Z-transformation method is more likely to obtain better results. This method has its roots in statistics science and it is powerful enough to do the ranking of the candidates for space applications. Besides, when doing material selection for spacecrafts a lot of properties have to be taken into consideration. Introducing the outgassing property in conjunction with other mechanical properties during the selection process provides better results. However, the selection of the properties (environmental and mechanical) considered during the process it is really determining for the resulting ranking, and changing the conditions of the material selection procedure might change the result too.

## 4.2 Polymeric composites

The behavior of a composite material is the result of the combined behavior of the fiber (or reinforcement element), the matrix and the interface fiber-matrix. The proportion of each of the components has to be one such that the individual elements transfer some of their properties to the composite itself without making the final material properties much different than the ones of a material formed exclusively of the matrix constituent. It is important too the compatibility of the fiber and the matrix, meaning the capability and success of the joining between them.

When studying the properties of a composite, it is important to have information of the interactions that happen between the components of the composite material. Thus, the interface plays a significant role. The interface of a composite is defined as the region of the composite where the interactions between the fiber and the matrix are placed. As a consequence, the properties of the material will depend not only on the properties of the matrix and fiber, but on the interactions between both, this is, the interface.

The composite materials can be classified based on the type of matrix and reinforcement. In these section the focus is going to be put on the composites with polymeric matrix and fiber reinforcement. In Figure 3.1 it can be observed the different possible distributions of the fiber in the composites:

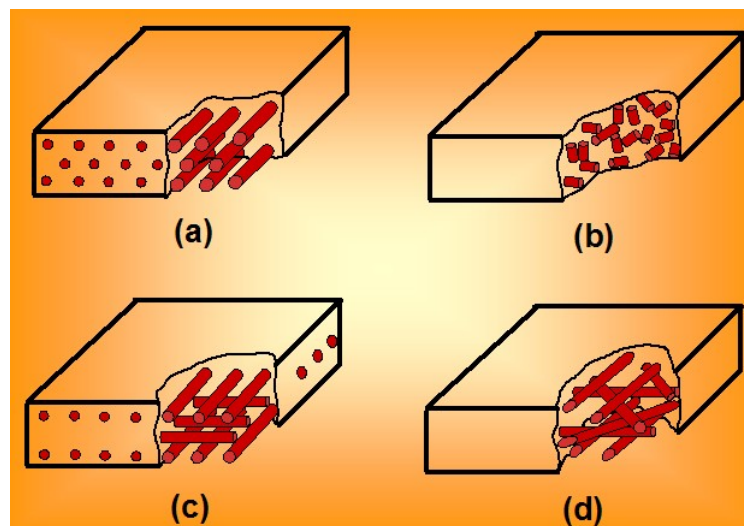


Figure 4.1: Morphology of composites with fibers [11]. a) Continuous unidirectional fibers. b) Discontinuous fibers randomly oriented. c) Orthogonal fibers and d) Multi-layer fibers.

The election of a material for an application usually begins with the matrix choice,

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which has to meet the basic properties needed for the purpose. For that matter, one wants to search materials that present certain conditions regarding axial modulus, axial tensile, Young's modulus, density, axial CTE, thermal conductivity, etc. but in this case the special environmental conditions have to be added to the list (i.e. the outgassing rate). Once the matrix is determined, the reinforcement can be introduced to contribute to the improvement of the properties of the final material. This improvement can be related to some requirements or just to secondary benefits that could decrease the cost of the material.

One of the trends of the composite designs is the substitution of steels and metal alloys for composites of less density that allow the structures to be lighter. Composite materials are widely applied to space structures and systems because of their extraordinary optical, thermal, electrical and mechanical characteristics. In this sense, it appears that composites provide a lot of advantages, and so they do, but space applications are a special case since the materials have to operate for extended periods of time in space. Thus, they will be subjected to a harsh space environment and they could have problems maintaining their outstanding performance.

Regarding the outgassing phenomena, it is not much relevant in metals as it is in polymers, or at least it is easier to be fixed in metallic materials using some treatments, so this tendency to use composite materials have to be done carefully and the matrices used as well as the fibers have to be selected having into account the standards required in order to avoid the degradation of the composite due to the vacuum.

Polymers don't usually conform the common concept of structural materials by themselves because their low rigidity and mechanical strength properties, but when combined with other materials they actually do.

For this matter, the cause of the outgassing problem is the polymeric matrix more than the fiber material. Also other polymeric materials are to be have into account when designing for structural applications on aerospace vehicles, such as sealants, paint finishes, insulating materials or lubricating greases. Thermosetting and thermoplastic materials are widely used in many applications both in the interior and the exterior of a space vehicle because of their excellent electrical properties, their high strength-to-weight ratio and their dimensional stability. Electrical potting compounds, wire insulations and some miscellaneous plastics are some of the examples of this.

Some examples of polymeric materials used for inflatable structures are [15]:

- Membranes and films: polyethylene, polyvinyl fluoride, Mylar, polyvinyl chloride and polybenzimidazoles.

- Foams: polyurethanes and polyethers.
- Fibers: nylon, acrilan and polyamides.
- Coated fabrics: neoprene, butyl, vinyl or Teflon.

### 4.3 Nanomaterials and nanocomposites

During the last two decades, the nanoscience field has experienced a drastic evolution, and its development has been emphasized by the advances and new findings on areas such as biomedicine, electronics or aerospace.

A nanocomposite is a composite where one of the constituent phases has one, two or three dimensions of less than 100 nm. These materials are made of a solid that acts as a matrix and a nano-dimensional dispersed phase inside the matrix. The interaction between the matrix and the fibers result in an improvement of the properties that each material would have by itself. Depending on the matrix material, nanocomposites can be classified by composites with metallic, polymeric or ceramic matrix. A big window of opportunities has opened specially for polymer-nanocomposites to overcome the limitations of traditional micro-composites. On the other side, the nanomaterials used as a reinforcement can be classified in three groups depending on their geometry [18]:

- Nanoparticles: the three dimensions of the material are of nanometric order. They can be also named as nanocrystals or nanograined materials.
- Nanotubes: two of the three dimensions of the particle are in nano range and the third dimension is larger. They are also known as nanofibers or whiskers.
- Nanolaminates: this type of particles have only one dimension in the nanometer scale. They are presented as a laminate with a thickness of a few nanometers, while the length is of hundreds of thousands of nanometers. This kind of particles are also named nanosheets or nanolayers.

This types can be observed in the following figure:

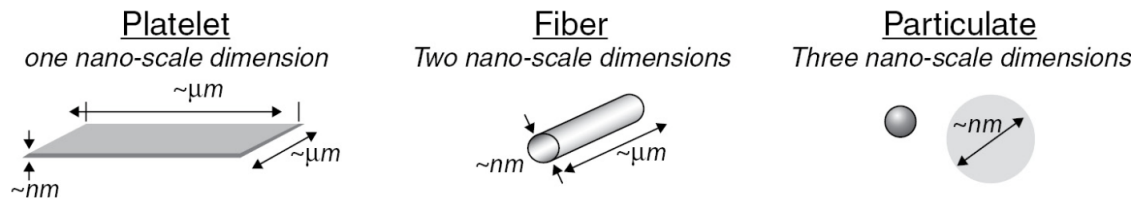


Figure 4.2: Types of nanomaterials depending on the geometry [19]. a) Nanolaminates. b) Nanotubes. c) Nanoparticles.

The nanomaterials can also be classified into three categories depending on their origin or pathway: natural, incident or engineered. The first, naturally formed, occur in the environment (volcanic dust), the incident nanomaterials are the ones resulting from the human action through industrial processes (diesel exhaust), and the engineered ones are obtained from complex processes and, differently from the first two types, these have regular shapes.

The principle advantage of nanocomposites in comparison with the traditional composites is the ability to obtain an improvement of the material properties with a relative small amount of reinforcement material. The reason for this is the high aspect ratio of the nanomaterials, which allows the interfacial area between the matrix and the reinforcement to be larger than the one in normal composites. Thus, the use of nanomaterials reduces weight and cost and simplifies the industrial process of obtaining the material.

For the space industry, the researches on nanocomposite materials are of special importance. Improving the mechanical properties, decreasing the weight of the components and decreasing the cost are always the objectives on sight. As it is been said, there are three types of nanomaterials regarding their matrix: polymeric, ceramic and metallic. For space applications and in order to minimize the weight and improve the mechanical properties, it is clear that the nanocomposites that should be used are the polymeric nanocomposites. However, these new materials also have to be tested for outgassing to be qualified for being used in space applications under harsh environment conditions. Similarly to the ordinary composite materials, the main problem for the outgassing properties come from the matrix, which is usually a polymer. Regarding the reinforcement, the materials commonly used are silicates and metals, specially carbon nanotubes, which outgassing rates are not deeply studied due to the novelty of this material science.



## 4.4 Outgassing of polymeric materials

Quoting Norton at [12] "...all gases, including the rare gases, will permeate all polymers to some degree". When the outgassing is defined as a problem in the space environment, the polymers are the trouble-makers. Being exposed to a space environment will make the composite materials used in the space structural components undergo different conditions such as fatigue cracking, surface erosion, structural modification and mass loss by outgassing, modification of properties by UV radiation, delamination by collisions with micrometeoroids, etc. Again, it is extremely important to consider these phenomena when designing for space applications. In order to do that, the understanding of the space environment is key.

There are two different ways of studying the space conditions and the exposure of the materials to such environment: in-flight experiment and accelerated ground simulated experiment [14]. On one side, the in-flight experiment offers reliable and accurate results about the degradation of the materials in the space because the samples are carried and placed on space stations or spacecrafts, where they can be exposed to the real harsh environment effects. On the other side, the accelerated ground simulated experiment offers the opportunity to carry the experiment several times and have unlimited samples of different materials. Moreover, it is less time consuming and cheaper.

A polymeric composite that is subjected to ultraviolet (UV) radiation in vacuum experience outgassing phenomena, involving the following basic processes [13] (Figure 2.1):

- Desorption of volatile products absorbed or generated on the surface of the material.
- Diffusion and desorption of gaseous substances absorbed by the material or generated in it due to UV radiation.
- Evaporation of the material being the result of UV action.

The influence of the UV radiation on outgassing processes in polymeric composites exceed those induced by exposure to solar radiation. During the outgassing the photo-decomposition of the polymeric components occur, and this is stipulated by a number of features of the photochemical processes that occur in materials under exposure to UV. This can cause surface-condensable volatile products to be moved from the thin layer in the surface of a polymeric composite into the gaseous phase. This is described by mathematical models in some other researches trying to create the model to form the basis for the engineering prediction method of the outgassing phenomena in polymers. The analysis of the results show that the temperature of the material is of a great

influence and also the volume ratio of the polymeric binder/filling agent. However, the molecular weight of the volatile fragments is not well established, and neither is the equilibrium decomposition pressure of most polymers. Thus, experimental studies of the weight loss of polymers in vacuum are required to characterize this phenomenon.

# 5

## *Test setup*

In this section the test setup is going to be explained in detail so the experiment can be carried following the given instructions. At the moment of turning this report in, the plasma chamber hadn't been fixed so for the moment the big chamber in Figure 2.7 is going to be used until further notice.

There were multiple reasons for the unavailability of the plasma chamber; first, it turned out that the material used to build the plasma chamber was also outgassing, so the chamber couldn't reach the desired pressure due to leakage through the material. It was mainly a material selection mistake. Once this was troubleshot, the next problem was to replace that part of the plasma chamber. Said chamber has specific dimensions and it was impossible to find the part from a vendor, so it was concluded that it had to be redesigned and ordered made to measure. The order was placed, the new glass was installed and further test were done to determine if the plasma chamber could be used. To do that the pressure in the chamber was pumped down to  $5 \cdot 10^{-5}$  torr and left for vacuum for 24 hours, but there were still some leakages that couldn't be traced. This is the latest update for the plasma chamber, so another alternative had to be considered. This alternative is the big chamber available at COAS. In order to use that chamber another problem had to be considered: the pump that operates in the chamber can't be contaminated with the outgassed material, so a metallic box was ordered to keep the whole test setup inside and ready to be used.

Outgassing phenomena is present in most of the materials. Therefore, obtaining accurate results when doing outgassing tests is a difficult task. To check the proper performance of the design test setup, a first test of a certain material should be carried. The outgassing information of named material should be known from previous researches, so by doing this the results from our setup and the results from previous researches could be compared and the correct behaviour of the setup could be ensured. Once this is done, other tests of the material of interest can be done.

### 5.1 Apparatus and chamber setup

The explanation for the apparatus setup and the test procedure is explained in detail in the following steps:

1. First of all, the heating plate, which is made of copper, has to be attached to the aluminum holders on the sides.

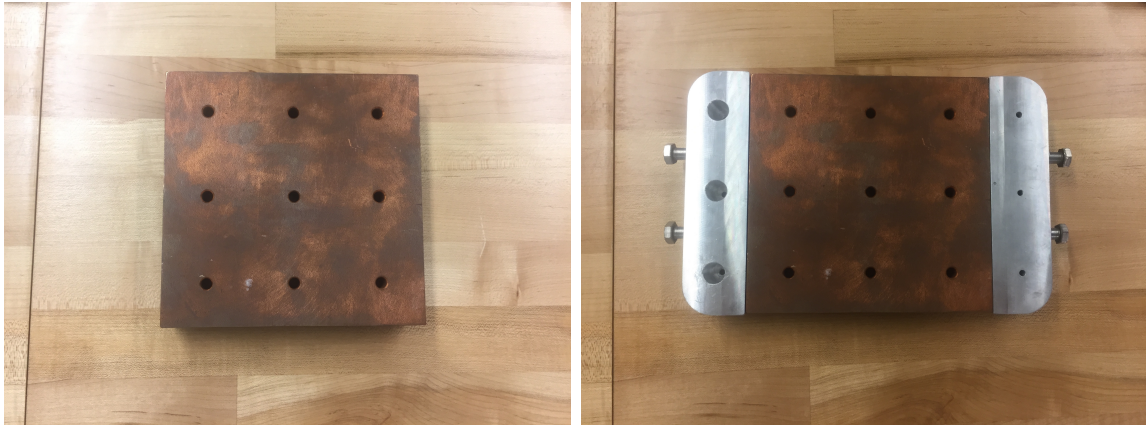


Figure 5.1: Heating plate made out of copper with the aluminum holders.

2. Second, the material specimens have to be placed inside the specimen boats, and the specimen boats inside the heating plate. The holes for the material specimens are in the down side of the heating plate, as shown in Figure 5.2. There are nine specimen boats in total, but some of them should remain empty to control their cleanness when testing.

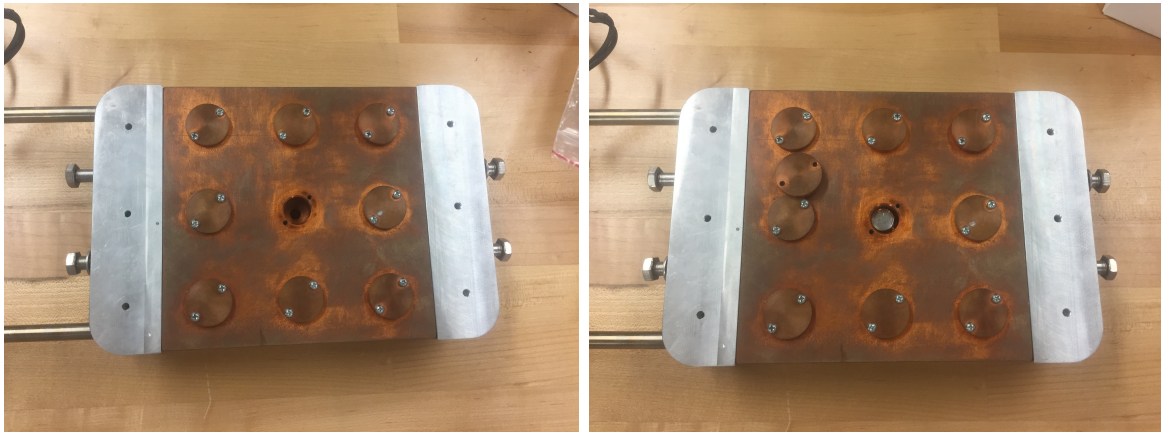


Figure 5.2: Bottom side of the heating plate, without and with the specimen boat.

3. Next, the cartridge heater has to be placed inside the heating plate in the holes and connected to the PID controller. The end of the red wire has to be attached to the heating plate so that the controller can measure the temperature and control it. Further details on the PID controller can be found on the manual [20].



Figure 5.3: Bottom side of the heating plate, without and with the specimen boat.

4. Once the heating side of the apparatus is set, the next step is to set the cooling side, which is made of aluminum. First of all, the collector plates have to be screwed to the cooling plate. It is very important to keep this collectors very cleaned, because there is where the mass loss is going to be kept. The researcher will have to weight the collector plates before and after the test. There are nine collector plates in total.

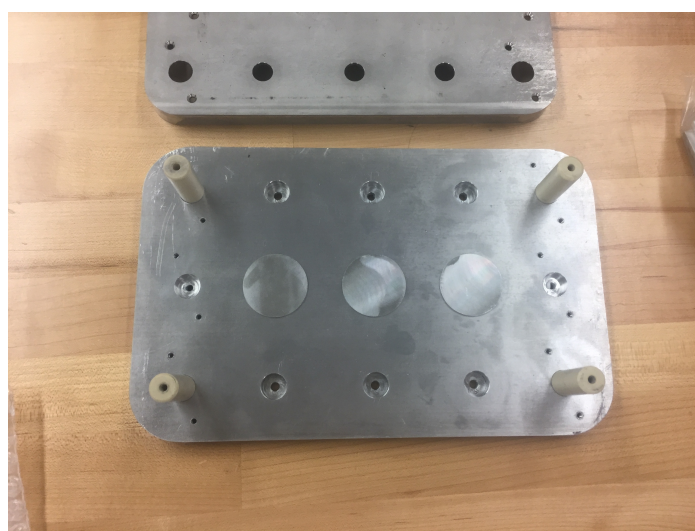


Figure 5.4: Cooling plate with the collector plates.

5. The heating and cooling plate have to be kept separate by a certain distance specified in the standards of the test. Therefore, a separator plate needs to be added to the setup. This separator is made of steel and it has some ceramic standoffs that helps to maintain this certain distance.



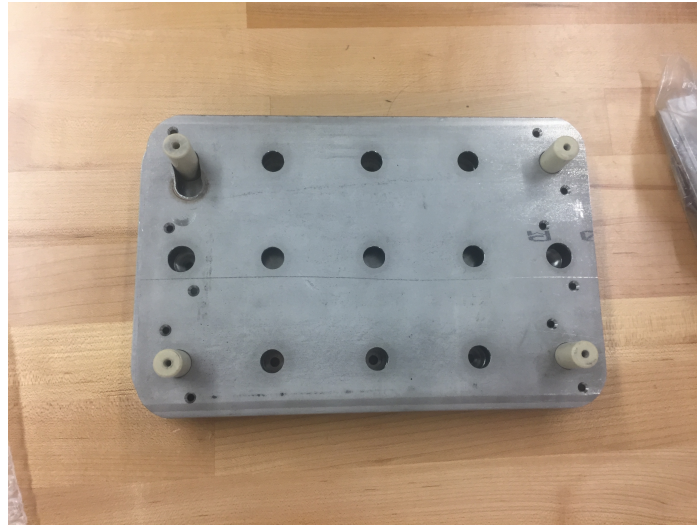


Figure 5.5: Separator plate with the ceramic standoffs.

6. Once the three plates are together, the peltiers have to be placed on top of the cooling plate. For this experiment four peltiers will be connected in series and at the same time connected to another PID controller and the power source. The peltiers have a maximum voltage of 16 V, so the controller makes sure this limit is not reached and also controls that the temperature stays where it needs to be set for the test. In the next Figure there is just two peltiers, but in the final setup there should be at least four. Also the heat generated at the peltiers has to be evacuated to avoid overheating of the components. This can be done with a heat sink made of some kind of metallic material. Moreover, the peltiers should be attached to the cooling plate using some kind of thermal tape to ensure the contact, or thermal epoxy.

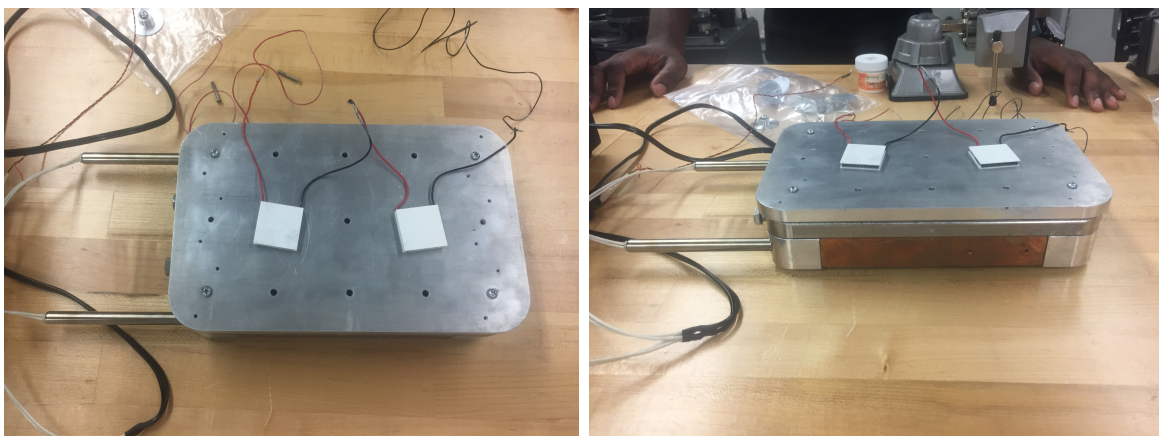


Figure 5.6: Whole test setup with the peltiers on top of the cooling plate and the cartridge heater inside the heating plate.

7. Finally, once the test setup is ready with the material inside the specimen boats, both PID controllers for the heat cartridge and the peltiers are connected and the power source is on, the test can be started. The details on the temperatures, the timing and the test conditions are explained previously in Section 2.2.1.

The final CAD of the apparatus with the four peltiers can be observed in Figure 5.7:

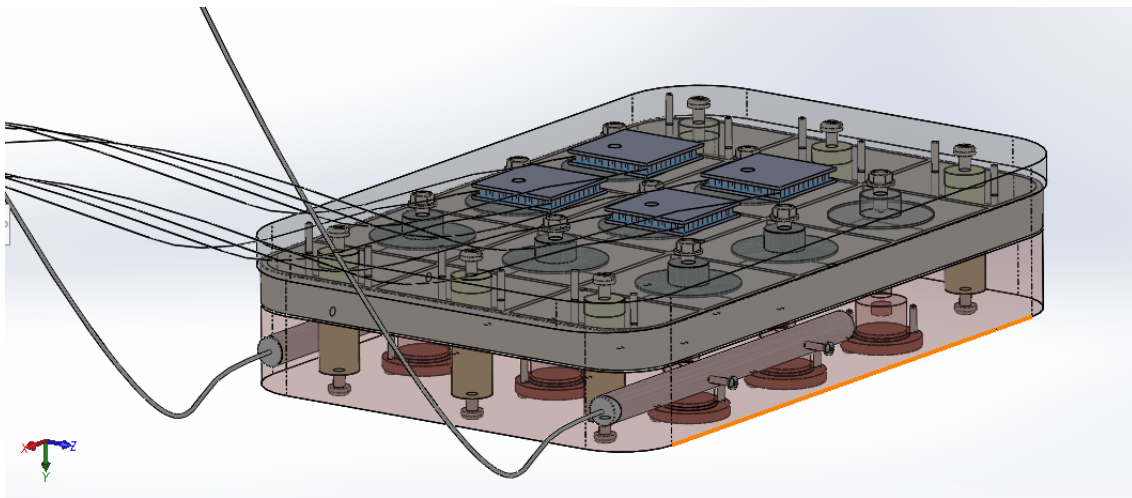


Figure 5.7: Final CAD scheme of the outgassing test apparatus.

## 5.2 Reporting

Some information about the materials tested should be reported before and after the test is completed:

- Name and number of the material, manufacturer and further identifications.
- Summary of the preparation specifications, such as mixing proportions, cure time and temperature, cleaning procedures, etc.
- Number of samples per experiment. Usually three is the common amount.
- Sample configuration: size, shape, etc.
- Statement of the sample test temperature, the collector plate temperature and the test duration.
- Altered conditions or nonstandard conditions if used.
- Initial mass of the samples.

- Mass of the samples as taken from the test chamber.
- Percentage of Total Mass Loss (TML). Take three values for each material and then take the average.
- Percentage of water vapor regained, WVR. Take three values and then make the average.
- Initial mass of the dried collector plates.
- Change of mass of the collector plates.
- Final mass of the collector plates.
- Percentage of CVC. Take three values and then take the average.
- Remarks about any noticeable incident or deviation of the standard conditions.



## *Conclusions and future work*

The main conclusion of this research is the importance of the material selection, both for space applications because of outgassing issues and for apparatus and instrument applications. It turned out that the plasma chamber that was going to be used to carry the outgassing tests was actually outgassing itself, so obviously it couldn't be used. This fact led to a delay of the experimental testing schedule, being impossible to carry any test on any material. From this problem, the future work of this research becomes clear:

- First, the plasma chamber should be fixed and the leakage should be traced and arranged so it can be used. If not, necessary modifications of the test setup should be made in order to use the bigger chamber. However, said chamber is bigger and thus it takes longer to get to the desired pressure, making the experiment slower and not as comfortable as it would be using the plasma chamber.
- Another important thing to be done in future work should be to test for the apparatus. Testing a thermal grease which outgassing properties are already known would be a good way to validate the setup fabricated, calibrate it and make sure it works properly.
- Finally, after the chamber is ready and the apparatus is tested, the next step would be to carry the actual experiments to measure the outgassing properties and behavior of the nanocomposite materials fabricated at Embry-Riddle Aeronautical University, or any other material wanted to be tested. The results will be post-processed and compared with the NASA standards to decide if such materials can be used for space applications.

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