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A chamber for studying planetary environments and its applications to astrobiology

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Abstract
We have built a versatile environmental simulation chamber capable of reproducing atmospheric compositions and surface temperatures for most of the planetary objects. It has been especially developed to make feasible *in situ* irradiation and characterization of the sample under study. The total pressure in the chamber can range from 5 to 5×10^{-9} mbar. The required atmospheric composition is regulated via a residual gas analyser with ca ppm precision. Temperatures can be set from 4 K to 325 K. The sample under study can be irradiated with ion and electron sources, a deuterium ultraviolet (UV) lamp and a noble-gas discharge UV lamp. One of the main technological challenges of this device is to provide the user the possibility of performing ion and electron irradiation at a total pressure of 0.5 mbar. This is attained by means of an efficient differential pumping system. The *in situ* analysis techniques implemented are UV spectroscopy and infrared spectroscopy (IR). This machine is especially suitable for following the chemical changes induced in a particular sample by irradiation in a controlled environment. Therefore, it can be used in different disciplines such as planetary geology, astrobiology, environmental chemistry, materials science and for instrumentation testing.

Keywords: planetology, irradiation, environmental chamber, modelling

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Nowadays, the study of planetary environments of astrobiological interest has become a major challenge. Planetary objects such as Mars and the Europa satellite are among the priority targets for searching for biosignatures in our solar system. This is mainly due to the presence of water in any of their phases during the past or present of their geological histories [1–4]. However, the effect of the environmental parameters at the planet surface, such as radiation, gas pressure or temperature, is critical for both microorganism survival and the preservation of biosignatures [4, 5].

On the other hand, Earth, Mars, Europa, Triton and other bodies in our solar system are continually exposed to the effects of different types of radiation. This radiation results in the breaking and rearrangement of chemical bonds within ices and minerals, causing the destruction of some species and the creation of others (HCO, H_2CO, CH_4, CO_2, etc). Furthermore, many of these species are of biological interest for understanding the origin of life. For example, photochemical reactions, driven by solar UV, are believed to be the principal source of complex organic molecules observed in most planetary and lunar atmospheres [6]. The knowledge of the photochemical routes by which organics are formed in other planetary atmospheres could provide insights into photoproducts that were formed in the atmosphere of the primitive Earth.

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Planetary environmental studies are not only important for understanding the origin of life, but they are also relevant for elucidating the presence of different mineral phases and their association with geological processes. Thus, understanding the role played by different sources of radiation in the chemical process on the planetary surfaces is relevant. Ionic energetic impacts could induce many effects on an icy surface, such as chemical reactions, electrostatic charging, lattice damage, desorption and evaporation, some of which alter the appearance of the surface [7].

Due to the obvious technical limitations for *in situ* planetary exploration, laboratory simulations are one of the most feasible research options to delve further in both planetary science and in a consistent description of the origin of life. Furthermore, new and updated data arrive constantly from space missions. With this aim in mind, we have built a versatile planetary simulation chamber providing computer-controlled gas composition in the atmosphere and sample temperature for most of the solar system planets. Our equipment has been especially developed to make feasible *in situ* irradiation and characterization of the sample. Therefore, it allows for recording chemical changes in a given sample with respect to gas environment, temperature and radiation dose. For this purpose we include irradiation sources such as UV-photons, ions and electrons, and the implementation of analytical techniques like IR and UV spectroscopy.

During the past few years, environmental simulation chambers have been developed as a useful tool for studying geology, astronomy, cosmochemistry and planetology. The majority of these machines consist of an uncomplicated system, which reproduces a particular gas composition and temperature. In this paper, we present a multipurpose machine, which can be used in many different research fields and for studying different kinds of problems. Therefore, a figure of merit of this chamber is its versatility. As will be discussed in the last section of the manuscript, the machine allows for studying processes of different nature related to different areas (planetary geology, astrobiology, environmental chemistry, material science, instrumentation testing). There exist other sophisticated chambers that have included *in situ* analysis techniques for specific problems, such as gas chromatography mass spectrometry (GCMS), quadrupole mass spectrometry (QMS), infrared spectroscopy. Among them, EXOCAM [8] is devoted to the study of physical–chemical interactions between the atmosphere and the surface and subsurface in Mars conditions, SURFRESIDE [9] simulates interstellar and protostellar environments, and the Andromeda [10] planetary simulation chamber is mainly dedicated to simulate conditions on Mars.

2. Technical description

To fully achieve the ambitious objective of versatility we had to overcome some technical difficulties. The most important challenge was to develop an experimental system able to operate over nine orders of magnitude in pressure, i.e., making achievable the study of planetary objects almost without atmosphere (i.e., Europa) and with small atmospheres (i.e., Mars). We have built an ultra-high vacuum (UHV) chamber 500 mm long by 400 mm diameter (total volume of about 62 800 cm^3) with standard CF flanges and fittings (see figure 1). Several flanges have been left available for future instrumentation developments. The pressure is controlled by a Pirani–Penning combined sensor, covering a range from 1000 to 5×10^{-9} mbar. The base pressure of the chamber is in the 10^{-10} mbar range. This warrants the simulation of planets with pressures of 10^{-8} mbar, with a negligible amount of residual gases. On the other hand, it is difficult for an UHV system to work steadily at mbar pressures. To achieve conditions of relative high pressures a feedback-stepped motor valve controls the aperture of a CF-500 turbo molecular pump changing it as the pressure changes (i.e., the aperture of the valve is set as 10% open for Mars conditions). A schematic view of the main chamber is depicted in figure 2.

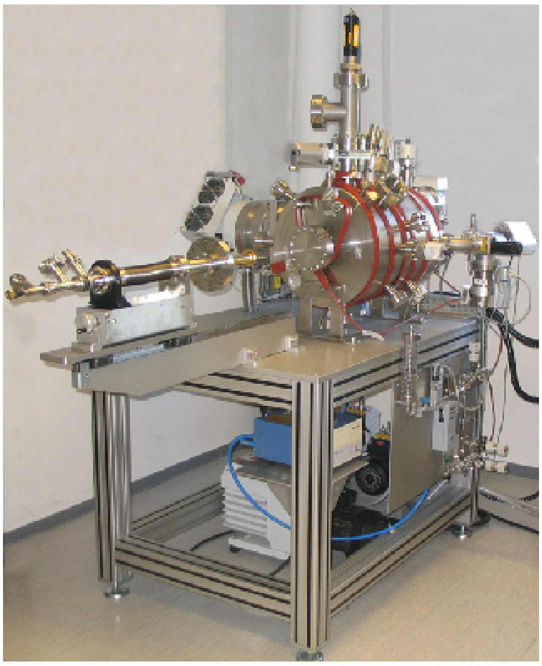

Figure 1. Photograph of the planetary environmental simulation chamber equipment.

To simulate a particular atmosphere, the desired gases are mixed in a manifold to obtain the required proportion, each gas is controlled by individual fluxmeters (dosing valves). Gas composition is constantly measured by a residual gas analyser (RGA) mass spectrometer (quadrupole type); therefore, we can control the desired partial pressure of a particular gas by acting (opening or closing) on its corresponding fluxmeter. To estimate the total concentration of a particular gas, cracking of the molecules, sensitivity factors and mass coincidences have to be taken into account. The relation between the current (amperes) measured in the RGA for a mass and the concentration of a particular gas requires a thorough calibration process. We have calibrated the RGA signal by measuring a known gas mixture. Thus, for our particular system, we found a calibration factor for every mass in the gas mixture, which accounts for the cracking and the specific sensitivity of the gas, and that converts intensity into concentration. The partial pressure of each of the gases in the experimental system can be independently controlled and

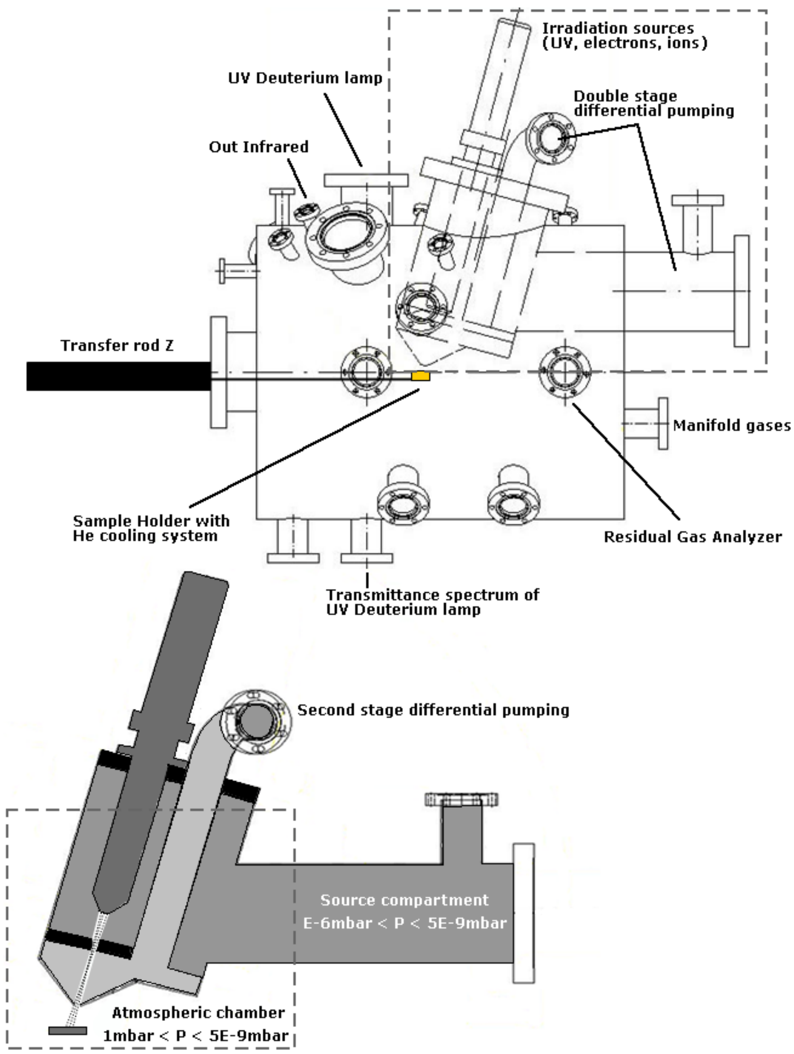

Figure 2. Schematic drawing of the planetary environmental simulation chamber.

varied from 5 to 5 $\times$ 10^{-9} mbar. Thus, the percentage of each gas in the atmosphere is continuously monitored in order to follow possible condensation or desorption processes as a function of time or irradiation. The RGA can work from UHV to atmospheric pressures. This is achieved by an efficient differential pumping system.

A helium cooling system, which is connected to the sample holder through a thermally isolated He-transfer rod, cools down the sample. This system assures that only the sample and sample holder are cooled. The temperature is measured by using two silicon diodes in different parts of the sample holder, and a 50 Ω electrical resistance warms up the sample if the temperature drops below the temperature set-up. With this configuration, the temperature of the sample can be stabilized from 4 to 325 K.

Taking into account that small concentrations of water could be important for many biological processes, a water partial pressure can also be set and regulated in the chamber. Liquid water is introduced in a heater where it is vaporized and sent to a gas line and is dragged to the chamber by the gas mixture. To achieve that, a controlled evaporator mixer has been implemented.

Samples are mounted horizontally in order to allow for the study of low cohesive material. Special care should be taken for this kind of material because the turbo pump can drag it, since 50 μm is a minimum grain size for this kind of sample. Single crystals, soils, rocks and minerals are among samples that can be safely introduced into the simulation chamber and studied. The sample is placed into a removable sample container, which is made of copper with a gold coverage layer to improve the thermal conductivity.

The standard experimental protocol to set specific atmosphere conditions works as follows: the starting point is to pump down the system to 10^{-10} mbar. To obtain this low pressure a standard bake-out procedure should be performed. This low base pressure is important for Europa conditions, but it is not required for simulating planets with greater atmospheres, such as Mars. Once the base pressure has been achieved, the sample is cooled down to He temperatures, and then the heater at the sample holder (connected to a feedback system) is used to keep the temperature at the desired value. This feedback system also allows for setting temperature cycles (day and night or seasonal cycles). When the desired temperature is stable, the next step is to introduce gas composition at the working partial pressure for any atmosphere simulation conditions.

Thus, the sample can be positioned in an environmental chamber where it can be irradiated (see the next section). It is important, therefore, to be able to follow *in situ* the chemical changes induced for either a specific atmosphere, temperature or radiation. One of the most suitable techniques for these kinds of studies is infrared (IR) spectroscopy. Thus, two pairs of symmetric CF windows made of KBr have been designed to allow the IR beam to get in and out of the simulation chamber after scattering at the sample surface. The possible incidence angles are 30° and 45° with respect to the normal sample position. Through the first window the IR beam is focused on the surface by a mirror. Once reflected on the surface, the IR beam is collected through the symmetrical window by an external focussing mirror, which focuses the reflected beam into the IR detector. A deuterium lamp has also been set to perform UV irradiation. The lamp emits in a wavelength range from 200 to 500 nm. The total power consumed by the lamp is 150 W. The UV radiation from the lamp enters the system through a quartz window. Straightaway the UV light finds a beam-splitter, which lets 88% of the radiation pass through. The other 12% is reflected into another quartz window, where a UV detector is placed that permits continuous monitoring of the incoming UV flux. After the beam-splitter we have set a focussing lens to focus the beam on the surface position. The beam-splitter, lens and quartz windows can be isolated from the main chamber by a valve to avoid damage of the optical system when using corrosive gases or water vapour. The UV detector can also be located in a window directly under the sample, in order to measure transmittance through the sample for transparent samples. In this case, the sample holder should be made of CaF_2.

2.1. Environment of the irradiation sources: the source compartment

In order to irradiate with ion or electron guns (sources requiring a lighted filament to irradiate), the pressure should be less than 10^{-6} mbar. For planetary objects with a total pressure of about 10^{-2} mbar, such as Triton, it is not possible, *a priori*, to study the role of irradiation at the surface using this type of instrumentation. In this range of pressures, neither the ion nor the electron gun can be switched on. This problem has been solved in the present equipment by a double-stage differential pumping system (see figure 2). Pumping tubes in every compartment have been designed to maximize pumping speed. First, the atmospheric chamber is connected to the area where the irradiation sources are mounted (sources compartment) by a 2 mm diameter exit pinhole. Between the source compartment and the atmospheric chamber there is a second stage, where an entrance pinhole with variable aperture controls the partial pressure in the radiation compartment. The smaller the entrance pinhole diameter, the higher will be the pressure in the atmospheric chamber, making it possible to study irradiation in planets with relatively high atmospheres. The entrance pinhole can be replaced by unscrewing a small Cu gasket from the irradiation sources flange. We have used gaskets with three different diameters, i.e., 0.5, 2 and 4 mm. We have estimated, and experimentally confirmed, that a pressure of 10^{-2} mbar in the atmospheric chamber, using a 2 mm diameter entrance pinhole, will lead to a total pressure of 10^{-6} mbar in the source compartment. Therefore, we gain about two orders of magnitude in pressure at every stage of the differential pumping system. We have estimated the higher pressure to allow irradiation to be around 0.5 mbar in the atmospheric chamber, which roughly corresponds to 9×10^{-6} mbar in the source compartment. This procedure can also be applied for a UV discharge lamp. This type of instrumentation provides high radiation doses at fixed wavelength. No back draw of the discharge gas to the atmospheric compartment was detected in the RGA. Different wavelengths can be obtained by changing the discharge gas (He, Ne, Ar, etc). We have tested the instrumentation by using He I radiation at 5 mbar (Mars pressure), attaining stable functioning for hours.

Irradiation sources are designed to have the focal distance at the entrance hole, in such a way that most of the radiation could go throughout the double outlet configuration. When the sample has to be irradiated with ions or electrons, we focus the sample (by a linear translation motion) below the exit pinhole. Although the irradiated area is about 2 mm^2, which corresponds roughly to the pinhole diameter, it can be extended by scanning the sample. To simulate the irradiation of atmospheres with a total pressure lower than 10^{-6} mbar, the use of the entrance hole is not necessary, and therefore, the Cu gasket can be unscrewed and removed to get a wider area irradiated with higher doses.

2.2. Simulation of environmental conditions of three different bodies in the solar system

We will next describe the experimental protocols to reproduce the environmental conditions of three different bodies in the solar system. The three chosen planetary objects have a wide range of atmospheric pressures and surface temperatures.

2.2.1. Mars. The exploration of Mars, starting in the 1970s, has revealed some of the atmospheric and surface properties of this planet [11, 12], for instance their major chemical constituents and the UV radiation environment [13], which are important constraints for life. To simulate Martian conditions, 7 mbar is used as the average atmospheric pressure of the planet, temperature cycles ranging from 150 to 280 K can be programmed (this could be particularly interesting to simulate seasonal cycles). To reach these values, we have to first set the partial pressures of the gases known for the Mars atmosphere (i.e., about 95% CO_2, 2.7% N_2, 1.6% Ar and 0.6% H_2O), with a total pressure of 7 mbar. In order to have this pressure, we have to close the valve of the main turbo pump up to 90%, and to stop the entire differential pumping at the source compartment. This prevents the use of the irradiation sources in a Martian atmosphere. In any case, it is known that ions and electrons from cosmic rays have a small action on the Martian surface. However, the effect of the ionizing radiation could be simulated working at 0.5 mbar instead of 7 mbar and keeping the concentration of all Martian gases in the atmosphere constant. As previously described, at 0.5 mbar, irradiation sources can be switched on.

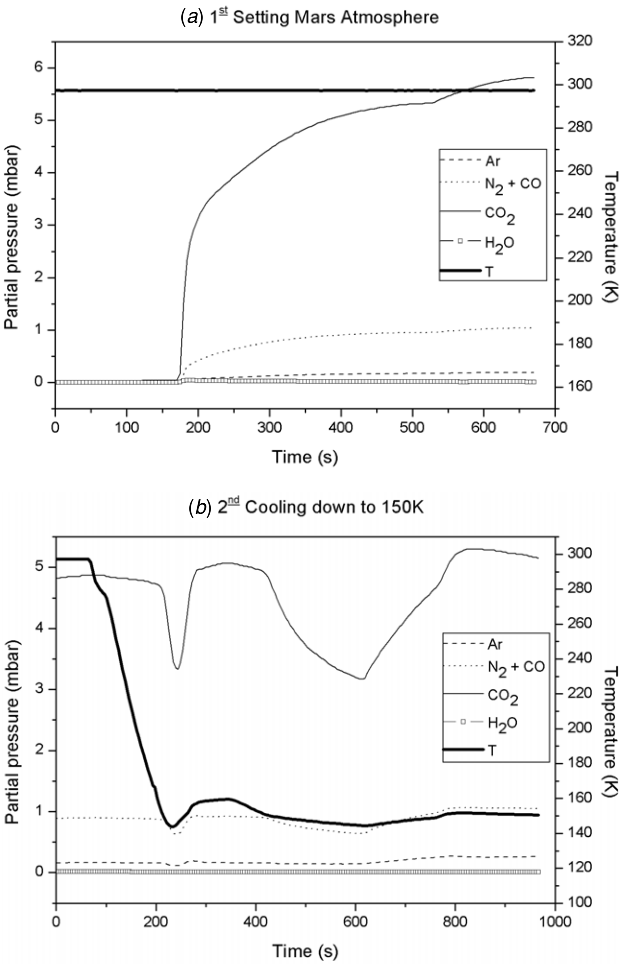

Figure 3. Mars surface environment. (*a*) Evolution of the partial pressure of the different gases in the Mars atmosphere. Stable concentrations are attained after 5 min. (*b*) Evolution of the partial pressure of the different gases in the Mars atmosphere as the temperature is decreased to 150 K.

The process of setting the desired atmosphere under Martian conditions takes about 5 min at room temperature. The result is shown in figure 3(*a*). Once the partial pressure of all the gases is stabilized, we cool down the surface to the desired temperature, in our example case to 150 K. We get a stable temperature after 15 min, as is shown in figure 3(*b*). The fluctuations in the partial pressure of the gases are due to the heating stage, which induces adsorption and desorption of the molecules around the sample holder.

Attenuation of electronic and ionic irradiation depends on the electron and ion mean-free path, which is a function of the total gas pressure of the atmosphere and the energy of the incident particle. Minimum irradiation has been found for particle energies of about 100–200 eV. In the case of Mars, the presence of atmospheric gases absorbs the incoming radiation and a small number of electrons and ions arrive at the surface. This scenario is different when we talk about UV radiation. In this case the attenuation strongly depends on the atmospheric composition, the radiation wavelength and total pressure. We have recorded *in situ* absorption curves both for deuterium radiation in the range of 200–400 nm, and strong monochromatic He I radiation at 58 nm.

2.2.2. Europa. The satellite of Jupiter, Europa, is an interesting planetary object from both geological and astrobiological points of view. Its most attractive characteristic is the possible presence of an ocean in its interior. Europa is a promising object for harbouring subsurface habitable environments, which could include non-photosynthetic and extreme low-temperature organisms. The potential water ocean has converted Europa to a priority one objective for the NASA astrobiological exploration. Voyager and Galileo missions have obtained some data about the physics, chemistry and geology of this satellite, including the global temperature distribution [14] and the high radiation environment at its surface [15]. In addition, observations from Earth have determined the existence of a low-pressure atmosphere [16].

In order to study the surface of the satellite Europa, the base pressure of the chamber should be reduced as much as possible. As we have used CF standard flanges for the vacuum, these low pressures can be attained after baking out the whole machine at 420 K. This procedure is well known in surface physics ultra-high vacuum systems. The residual pressure is in the 10^{-10} mbar range, and mainly composed of water and hydrogen molecules. We have simulated critical conditions of Europa's atmosphere (50 K surface temperature, partial pressure of oxygen 10^{-8} mbar) to test the equipment under the most adverse conditions.

The experimental protocol consists of

1. Cooling the sample to the minimum temperature of the surface (50 K). To get this temperature stable on the sample takes about 20 min (see figure 4(*a*)). In this process many of the residual gases condense and the total pressure of the atmospheric chamber decreases to 10^{-10} mbar (see figure 4(*a*)).
2. Oxygen is then dosed until the required pressure is measured in the mass spectrometer. The oxygen concentration remains very stable at the desired pressure (see figure 4(*b*)).

The radiation environment is reproduced by means of UV, ion and electron sources. Due to the low total pressure (10^{-8} mbar) on Europa, electronic and ionic irradiation play an important role in the surface chemistry. Therefore, this seems to be an important parameter to take into account for describing the geological process of the satellite. We have been able to irradiate with electrons a dose of about 5 μA cm^{-2}, and with ions about 10 μA cm^{-2}. The user can set the electron and ion energy ranging from 0.1 to 5 kV.

2.2.3. Triton. Voyager 2 spacecraft has unveiled the current activity of the Neptunian satellite, Triton. Geological processes like cryovolcanism occur in this extremely cold environment, in which even nitrogen is seasonally solid. Interactions between the atmosphere and the surface have been described, such as geysers ejecting gases. Once in the atmosphere, some materials are photolytically destroyed [17].

Triton's conditions have also been simulated on this machine as one technically extreme environment because of the circumstance of relatively low pressure (10^{-2} mbar, from 93% N_2, 4% CO and 3% CH_4) and very low temperatures. Although Triton does not have any evident interest for astrobiology yet, it deserves attention from a geological point

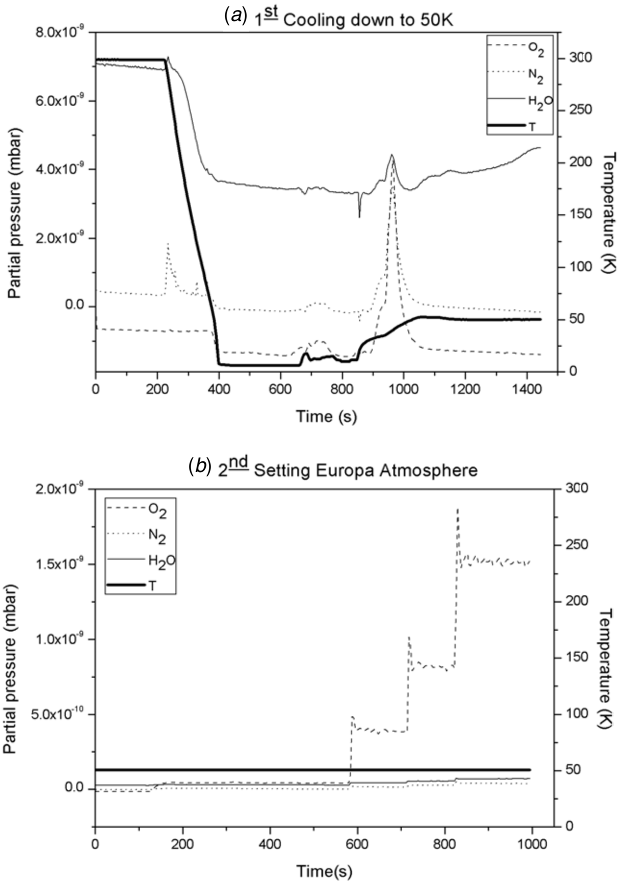

Figure 4. Europa surface environment. (*a*) Evolution of the partial pressure of the different residual gases of the atmospheric chamber as the temperature is decreased to 50 K. (*b*) Evolution of the partial pressure of the gases to attain Europa atmosphere.

of view. To reproduce Triton's atmosphere we set first the gas composition, which is stable after 5 min. The partial pressure of the gases in Triton's atmosphere is represented in figure 5. Then, we decrease the temperature down to 38 K. Note that this temperature is close to the critical point of the gases of the Triton atmosphere; therefore, a slightly lower temperature makes CO, CH_4 and N_2 condense on the sample surface (from time 500 s to 1000 s in figure 5). As a consequence, extreme control of the surface temperature should be attained to have stable atmospheric conditions for Triton. We were able to reach stable Triton conditions after 15 min.

Triton presents a pressure lower than Mars and higher than Europa; therefore electronic and ionic irradiation is strongly reduced by absorption in the atmosphere but could not be neglected. After electronic and ionic irradiation with the described simulation chamber, we have detected electron doses of about 0.1 μA cm^{-2}, and ion doses of about 1 μA cm^{-2}. Also UV irradiation deserves special interest, and both the UV-deuterium lamp and the noble gas discharge lamp are available in the experimental chamber.

2.3. *Summary of the technical specifications*

- Total pressure ranges from 5 mbar to 5 $\times$ 10^{-9} mbar. Partial pressure of the gases can be set with this precision.

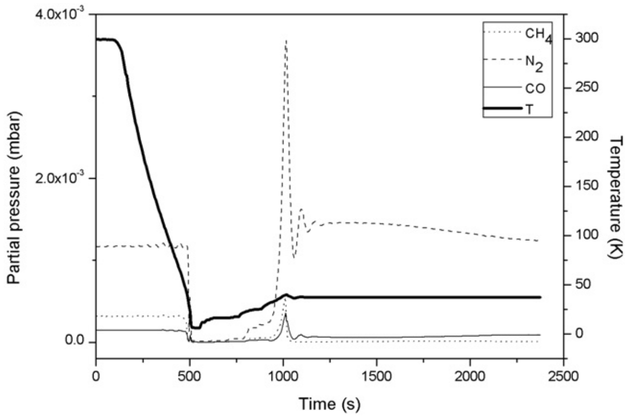

Figure 5. Triton surface environment: evolution of the partial pressure of the different gases of the Triton atmosphere as the temperature is decreased to 38 K.

- Temperature ranges from 4 K to 325 K.
- Gas composition is regulated via a residual gas analyser with ca ppm precision.
- Sample size ranges from 5 to 35 mm (other sizes of sample container are available for specific developments).
- Available irradiation sources: up to 5 kV ions (ions), 5 kV electrons, deuterium UV lamp and noble-gas discharge UV.
- Techniques for *in situ* anlaysis: UV spectroscopy, infrared spectroscopy.

3. Applications

Many disciplines could profit from using this kind of experimental simulation chamber. All scientific disciplines requiring a well-controlled atmospheric environment, which are interested in checking induced chemical processes, are among the potential beneficiaries of this machine. Our original motivation was related to planetology and astrobiology, although we have also found interesting applications in the domain of biology (to know, for instance, about the resistance of extremofile bacteria, or genes expressed in a particular environment), materials space science (resistance of different materials to radiation) and Earth-atmospheric (terrestrial) processes (environment protection).

Instruments and engineering technology for space applications could take advantage of our new space environment simulation chamber. Therefore, new developed technology (materials, gas and pressure sensors) could be calibrated and functionally checked under controlled conditions. Technology belonging, for instance, to the International Space Station (ISS) could be tested under space simulation conditions. Photochemistry of Earth atmospheres could also be an interesting field to work on.

Some other applications come from the field of astrobiology. Asteroids, meteorites and comets attract researchers' attention as they are primitive residues of the formation of the solar system; the simulation of the most primitive solar system could help in understanding many unknown facts about the origin of life. Examining other parts of the solar system, we may deduce what the early Earth might have been like.

A large and wide range of applications have been enumerated in order to provide an understanding about the potential of the simulation chamber. The flexibility of this experimental set-up makes many different types of experiments feasible, both from the geological and astrobiological points of view. In particular, physico-chemical changes induced in astromaterials and in microorganisms embedded in them, and the behaviour of bio-products in extreme planetary environments would be among the more suitable investigations.

4. Summary

We have described a new and unique instrument able to simulate any planetary atmosphere with a ppm gas composition control, a temperature range between 4 K and 325 K, and a pressure range from 5 mbar to 5×10^{-9} mbar. The wide range of pressure, temperature and gas composition conditions gives an idea of the versatility of the system. Irradiation sources and *in situ* analytical techniques expand the powerful characteristics of this planetary environment simulation chamber.

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