

Developing an Environmental Chamber to Simulate Conditions at the South Pole of Mars

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Abstract

Carbon dioxide (CO_2) is the main constituent of the Martian atmosphere, and about one third of it is annually deposited from the polar regions down to ~ 50 degrees latitude in each hemisphere, forming seasonal polar caps (Leighton & Murray, 1966; Tillman et al., 1993). This cycling of CO_2 is connected to water and dust cycles, and is a major driving force for the weather on the entire planet. The polar regions are also the areas with the most current surface activity on the planet due to seasonal changes related to the CO_2 cycle. Many polar geomorphological features such as spots, fans, and spiders are related to or formed directly by CO_2 processes, and in many cases, the occurrence of these features and mechanisms of their formation are closely linked to the behavior of CO_2 ice under different environmental conditions (Kieffer et al., 2006; Piqueux et al., 2003). Numerous orbital missions returned data on the large-scale temperature, albedo and the spectral signatures of these CO_2 ice deposits. However, there are not sufficient laboratory studies to explain the deposition conditions for these various ice textures and their role in the formation of a number of surface features. Previous experiments observed several different CO_2 ice textures formed under Martian temperatures, but questions about the CO_2 processes remain. Creating an experimental setup where more experiments on CO_2 ice could be conducted was an important step to continue investigations into polar CO_2 processes.

We have created and extensively tested an experimental setup to study the texture and photometric properties of CO_2 ice under environmental conditions representative of polar areas on Mars. In this thesis we describe the environmental chamber and spectroscopy systems and present some results obtained during experiments conducted to date. These results agree with important findings of previous experiments reported in literature. We observed two temperature dependent textures of CO_2 ice that formed by direct deposition:

polycrystalline slab ice and prismatic crystal frost as reported by Portyankina et al. (2019). Sintering of a small-grained frost into a slab, and fracturing due to thermal cycling were recorded similarly to experiments by Grisolle et al. (2014) and Philippe et al. (2015). We report one instance of breaking of a slab ice due to a rapid sublimation on a warmed substrate, which has not been previously reported. This is similar to a process that likely forms jets: geyser-like eruptions of sublimed CO₂ gas under the transparent ice on the south polar seasonal ice cap on Mars.

Additionally, reflectance spectra of the deposited ice have been measured in the near infrared range (1-2.5 microns) from a nadir direction at zero phase angle. Relative albedos of the slab ice, frost, and fractured slab are calculated from images taken by an infrared camera. Higher relative albedos are recorded for directly deposited frost and for slab ice that was fractured due to substrate heating. Lower albedos were measured for the clear slab ice.

We recreated the results of previous authors in our experimental setup and expanded on some of the previous experiments. Our ongoing and future work aims to measure reflectance from different geometries and to constrain the conditions that cause breaking of the slab ice by underlying gas pressure. Using mars regolith analogue substrate to investigate the effects of substrate on ice deposition and test the jet formation model would also be of interest in the future.

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Introduction

1.1 Present climate and Martian atmosphere

Mars and Earth share many similarities in terms of their atmospheric processes and climates. Both planets have solid rocky surfaces surrounded by substantial atmospheres, similar rotation rates and inclinations, which causes similar seasons (Read et al., 2015). Seasons on Mars are, however, longer because the orbital period of Mars is about twice as long as the year on Earth. There are also major differences between the two planets that affect the climate. Mars is further away from the sun, meaning that it receives on average only 43% of the sunlight that the Earth receives. The planet has no oceans, its atmosphere is about 150 times thinner than the atmosphere of Earth, and consists of drastically different constituents (Leovy, 2001). The average atmospheric pressure on Mars is about 6 mbar, and CO₂ makes up around 96% of its volume. Nitrogen and Argon each constitute close to 2%; H₂O, CO, O₂ together with some additional trace gases make up the rest (Owen, 1992). There is very little water in the atmosphere, its average partial pressure is 10^{-3} mbar, which is 10^4 times less than on Earth (Jakosky & Phillips, 2001).

The average global annual temperature is approximately 220 K but can reach above 273 K in the equatorial region or on steep slopes in the higher latitudes during peak insolation. Even at the highest temperatures, liquid water is considered to be unstable for extended periods of time due to the low atmospheric pressure. Liquid water can only exist as a transient phase and would evaporate quickly and freeze out in the cold high latitudes (Jakosky & Phillips, 2001).

During the winter in the polar regions, temperature can fall to ~ 145 K, at which the CO_2 from the atmosphere condenses directly onto the surface, forming seasonal polar ice caps (Leighton & Murray, 1966). About 30% of the atmospheric mass is deposited on the northern and southern polar caps each winter, resulting in significant drops of the atmospheric pressure globally. These yearly fluctuations have been first recorded by Viking landers (Hess et al., 1980; Tillman et al., 1993). The landers recorded a pressure minimum around $\text{Ls}=150$ (Ls denotes solar longitude, see section 1.1.1 for explanation), during the southern winter, after which the pressure rises while the southern seasonal cap sublimates re-supplying the CO_2 back into the atmosphere. Another smaller drop occurs around $\text{Ls}=340$ during the northern winter due to the formation of the northern seasonal polar cap. This drop is smaller because the northern winter is much shorter than the winter in the south as a result of the high eccentricity of the Martian orbit (Hourdin et al., 1993). The CO_2 cycle influences the climate on Mars, as it creates an atmospheric circulation known as the "condensation flow" over the entire planet. The flow transports heat, momentum, and mass from the cap undergoing sublimation to the opposite pole where the cap is forming. Seasonal cycles of H_2O and dust are also connected with the CO_2 cycle (Forget, 1998).

There are also many unique phenomena on the polar caps themselves that have no analogues on Earth due to the nature of carbon dioxide. They include features such as dark fan deposits that likely form in geyser-like eruptions of sublimed CO_2 gas every spring on the south polar seasonal cap. The fans disappear, blending into the background soil when the ice is gone in summer leaving gas-carved channel structures called spiders (Kieffer et al., 2006; Piqueux et al., 2003).

In order to better understand all of the described phenomena, it is valuable to understand the processes governing the seasonal caps, the properties of the CO_2 ice that constitutes them, the unique regions within the caps, and their interannual variability.

1.1.1 Solar Longitude and seasons on Mars

Mars experiences seasons due to the $\sim 25^\circ$ tilt of its rotation axis. We use the term solar longitude (Ls) as a way to specify the date on Mars in the context of seasons. Solar longitude represents the Mars-Sun angle, measured from the Northern Hemisphere spring

equinox where $L_s=0$. $L_s=90$ then corresponds to northern summer solstice, just as $L_s=180$ marks the northern autumn equinox and $L_s=270$ the northern winter solstice (Forget, 2008). For the southern hemisphere $L_s=180$ marks the spring equinox and $L_s=0$ marks the fall equinox (Figure 1.1).

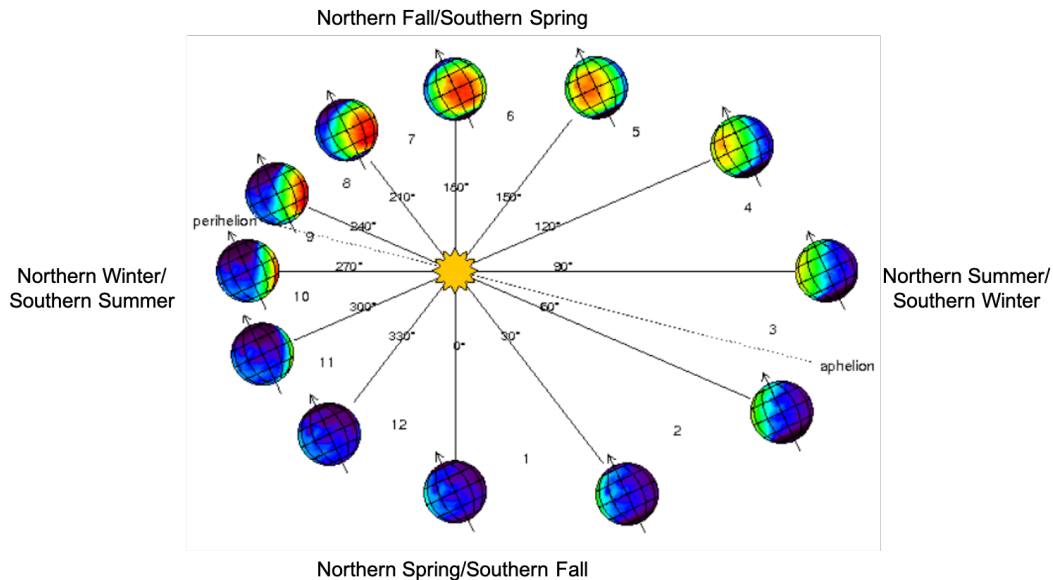


Figure 1.1: Seasons on Mars and solar longitude. The L_s angles represent the Sun-Mars angle where $L_s=0$ is the northern spring equinox. The figure also shows the eccentricity of Martian orbit that causes longer winters in the Southern Hemisphere (Forget, 2008).

1.2 Polar ice deposits and recent climate history

Several different polar ice deposits are present on Mars, which formed over a different range of timescales, and preserve climate records of these different periods in recent Martian history. The largest of these are the north and south polar layered deposits (NPLD and SPLD) (Figure 1.2), which reach up to 3-4 km in thickness, consist of water ice and dust layers that are 10^7 to 10^9 years old. The units of NPLD and SPLD likely formed due to the changing obliquity cycles that occur on the time scales of 10^5 to 10^6 years, as well as changes of other orbital components such as the orbital eccentricity and season of perihelion, and therefore can reveal the climatic history over the past hundreds of thousands to several million years (Jakosky et al., 1995; Levrard et al., 2007).

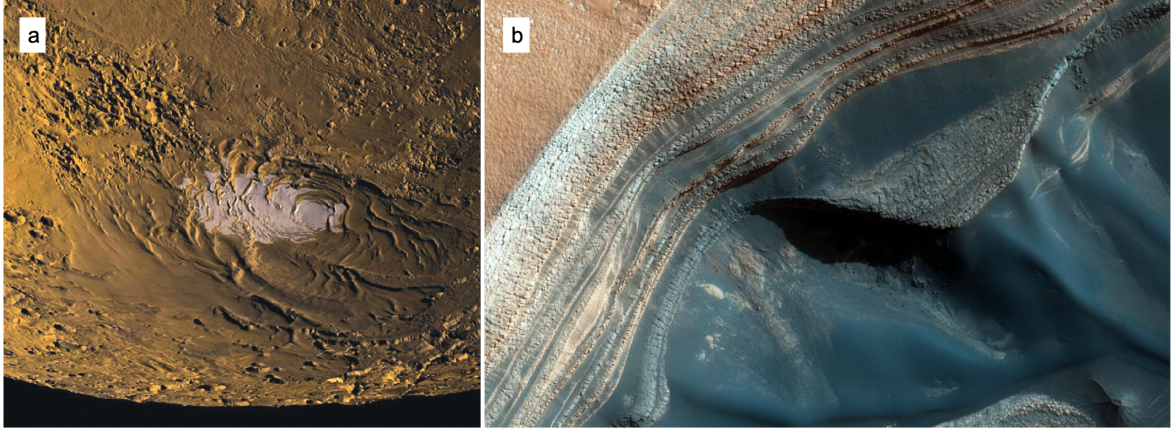


Figure 1.2: a) Mars Orbiter Laser Altimeter shaded relief mosaic combined with color from Mars Global Surveyor imaging of Mars' south polar region showing the residual ice cap (in white) within the smooth SPLD that overlie the cratered southern highlands. (NASA/MOLA Science Team); b) High Resolution Imaging Experiment (HiRISE) image showing an edge and layers of the NPLD (NASA/JPL-Caltech/Univ. of Arizona).

The residual ice caps are several meters thick and are younger, about several hundred years old, which makes them indicators of present and recent climate variability. The north polar residual cap (NPRC) consists of darker large-grained water ice while the south polar residual cap (SPRC) is made up of high-albedo CO_2 ice (Byrne, 2009; Kieffer, 1979). Massive CO_2 ice deposits up to 1 km in thickness have been detected within the SPLD using the SHARAD (Shallow Radar) instrument (Phillips et al., 2011). This deposit likely formed during the changing obliquity cycles of Mars in the past 500 thousand years, and if sublimated would double the current atmospheric pressure (Bierson et al., 2016; Buhler et al., 2020).

Finally, there are the northern and southern seasonal ice caps introduced in the previous section, which cover the poles in winter and fully sublimate every summer. These are important due to their connection to yearly weather on the planet as they drive the seasonal changes in atmospheric circulation (Byrne, 2009). Our main focus will be on the seasonal CO_2 ice in the following sections.

1.2.1 South seasonal CO₂ ice cap and notable regions

The seasonal cycling of the CO₂ from the ice caps to the atmosphere drives planetary atmospheric circulation and weather patterns (Calvin et al., 2017). Therefore, the deposition and retreat of these caps is extensively studied using remote sensing instruments to better understand these processes.

Polar areas of Mars start experiencing CO₂ condensation temperatures during the fall season. The caps start depositing at the poles and continue to extend toward the equator reaching down to 50 degrees latitude. The condensation of CO₂ ice follows the Clausius-Clapeyron equation, which states that the condensation temperature depends on the atmospheric pressure (Giuranna et al., 2007). Since pressure on Mars differs depending on elevation, the condensation temperature will vary at different elevations too. This will affect the distribution and longevity of seasonal ice at certain regions of the polar caps. Surface albedo is another important factor that affects sublimation of the seasonal ice by controlling surface temperature. Areas with higher albedo stay colder due to reflecting more solar radiation and preserve their seasonal ice cover for longer periods. These differences in ice cover can be most clearly seen on the southern seasonal cap, which exhibits a wide range of albedos that affect the rates of the cap's retreat, making it more asymmetric than on the north pole (Kieffer et al., 2000).

There are several regions on the south seasonal cap that exhibit similar albedo patterns every year (Figure 1.3). An area called the Cryptic region is the darkest part of the seasonal cap, while the mountains of Mitchel are the brightest (Kieffer et al., 2000). The south polar residual ice cap has a high surface albedo as well. Most authors consider it to be the main reason for its persistence during the summer (Kieffer, 1979; Thomas et al., 2005). Since the south polar cap exhibits such a wide variety of albedos that are often related to the texture and grain size of CO₂ ice, we briefly summarize the distinct stages it goes through every year, from its maximum extent around Ls=90 to the end of recession at Ls=325, as observed from remote sensing data. The most comprehensive datasets were provided by instruments such as the Thermal Emission Spectrometer (TES) on board Mars Global Surveyor (MGS), OMEGA (Observatoire pour la Mine´ralogie, l'Eau, les Glaces et l'Activite) on the Mars

Express spacecraft, the Compact Reconnaissance Imaging Spectrometer for Mars (CRISM) and Mars Color Imager (MARCI) on the Mars Reconnaissance Orbiter (Brown et al., 2010; Kieffer et al., 2000; Langevin et al., 2006).

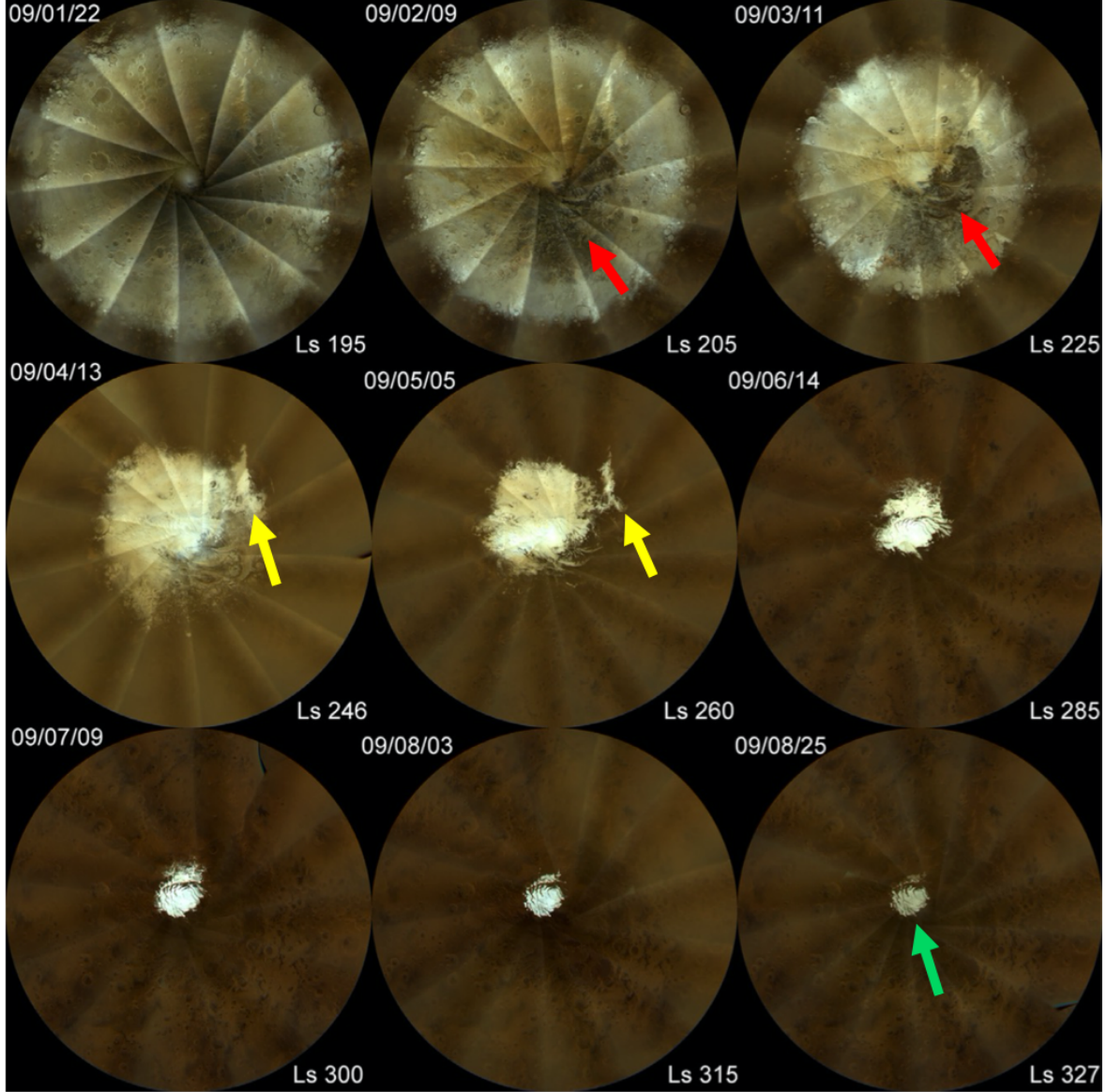


Figure 1.3: Mars Color Imager (MARCI) mosaics of the south seasonal ice cap from early spring till mid summer. The development of the dark cryptic region can be seen at Ls=205 as shown by red arrows. The ice retreats earlier in the cryptic region hemisphere, while brighter areas stay covered in ice longer. Yellow arrows show Mountains of Mitchel, one of the bright regions persisting till after Ls=260 as a separated ice-covered region. By Ls=327 all seasonal ice has sublimated and only the residual cap (green arrow) remains (Calvin et al., 2017).

In winter, the southern seasonal cap consists of relatively evenly distributed CO₂ ice and has a low albedo that is close to the albedo of the ice-free soil. This can be achieved by a layer of polycrystalline slab ice, through which the light can penetrate down to the underlying surface. This is supported by the large path lengths (up to tens of centimeters) inferred from spectral data that dates back to the Mariner 7 mission and has since been confirmed by more recent missions (Calvin & Martin, 1994). Large path lengths would mean that the ice is made up of large crystals where light can travel a long distance before it encounters a grain boundary or any contaminant particle where it can be scattered.

As the sun rises over the horizon in spring, the polar cap gets more illuminated and becomes brighter than its surroundings. It can reach albedos of up to 0.6. Some areas such as the perennial cap and an area in the western hemisphere called the Mountains of Mitchel start to get even brighter than the rest of the seasonal ice. Spectra from these areas show that the grain sizes here are significantly smaller (on the order of millimeters) than seen in the winter (Langevin et al., 2006). The Mountains of Mitchel tend to persist longer into the summer than the darker parts of the cap (Kieffer et al., 2000).

At about the same time in spring by about Ls=224, the cryptic region with an unusually low albedo of 0.25 develops in the eastern part of the ice cap (60 E to 220 E). Observations from TES showed that the surface on the Cryptic region stays below CO₂ sublimation temperature of 150 K long after the surface darkens up until Ls=260, which means that the surface has to be covered by CO₂ ice. Kieffer et al. (2000) suggested that the low albedo can be explained by translucent CO₂ slab, through which the underlying dark soil was visible. Data from the OMEGA spectrometer later showed that the low albedo of the Cryptic region in spring results from extensive dust contamination close to the surface (Langevin et al., 2006). This dark albedo causes an enhanced seasonal ice retreat of the eastern hemisphere of the cap: the cap shrinks faster on the cryptic hemisphere than on the opposite (non-cryptic) hemisphere. The Cryptic region also hosts a number of interesting features such as dark spots, fans, and spiders that are explained in the following section.

1.2.2 Polar Surface Processes: Spots, Jets and Spiders

Polar caps are some of the most active regions on the planet today, and the cryptic region on the south polar cap hosts a number of unique features termed as spots, fans, and spiders that occur every year (Figure 1.4). The processes that form them have no analogues on Earth and are closely linked to the properties of the seasonal CO₂ ice.

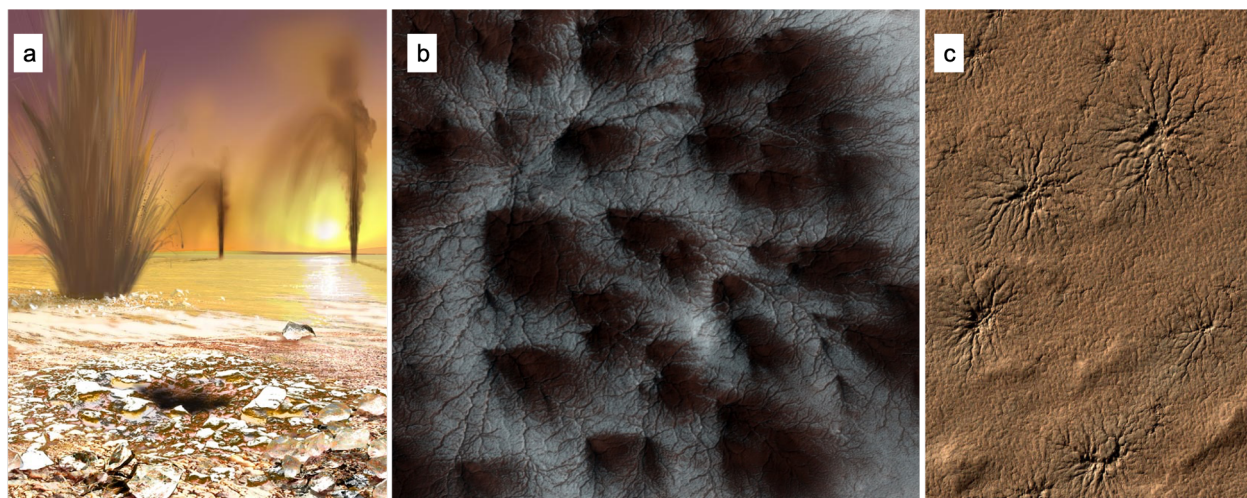


Figure 1.4: a) Artist's rendition of CO₂ jets erupting from slab ice (Ron Miller/NASA/JPL); b) HiRISE image of fan deposits. Underlying channels can be seen through the ice and dust; c) HiRISE image of spiders in summer when the ice has sublimated and no fans can be seen standing out from the background.

Fans and spots are surface deposits of sediments that are darker than the underlying icy surface. They were first thought to be patches of defrosted ground, but TES measurements showed that the temperatures of the spots (<153 K) were very close to CO₂ ice temperatures. Ice-free soil would have been significantly warmer (235 K). Kieffer et al. (2006) therefore concluded that the spots are likely made of a very thin (about 1 mm) layer of sediments that lie on top of the CO₂ ice, remaining in close thermal conductive contact with it. Fans appear more elongated than spots and are likely the cases when sediments have been carried by surface winds and deposited along a dominant wind direction (Aye et al., 2019). These surface features were found to occur in close connection to spiders also more generally termed araneiform features (Piqueux et al., 2003), which are radial dendritic troughs that widen as they converge towards the center (Hansen et al., 2010).

Kieffer et al. (2006) proposed a mechanism of formation for these features that is dependent on CO₂ ice properties, specifically, that such ice is transparent in visible wavelengths but not in infrared. This setup, whereby incoming visible light is absorbed by the regolith and reemitted in infrared (IR) can trap incoming radiation and create a solid state greenhouse effect. Figure 1.5 shows a schematic of the model, also known as the Kieffer model. The first step of the model involves cleaning of the dusty seasonal ice to achieve a transparent slab in late winter or very early spring. This occurs when the dust grains within the ice are heated by solar radiation (or alternative means) and the CO₂ ice around them sublimates forming a gas bubble. The bubbles close to the surface can rupture, while the deeper grains sink to the bottom of the ice layer due to gravity, leaving a transparent and clean slab of ice. Sunlight is then free to penetrate the clear slab ice to heat the substrate underneath. The ice sublimates from the bottom, while the CO₂ gas is trapped under the slab. The gas can accumulate and travel in channels underneath the ice, eroding the subsurface channels further, and once it reaches a critical pressure, breaks through the overlying slab ice, propelling sand and silt-sized sediments onto the surface forming a dark spot. Surface

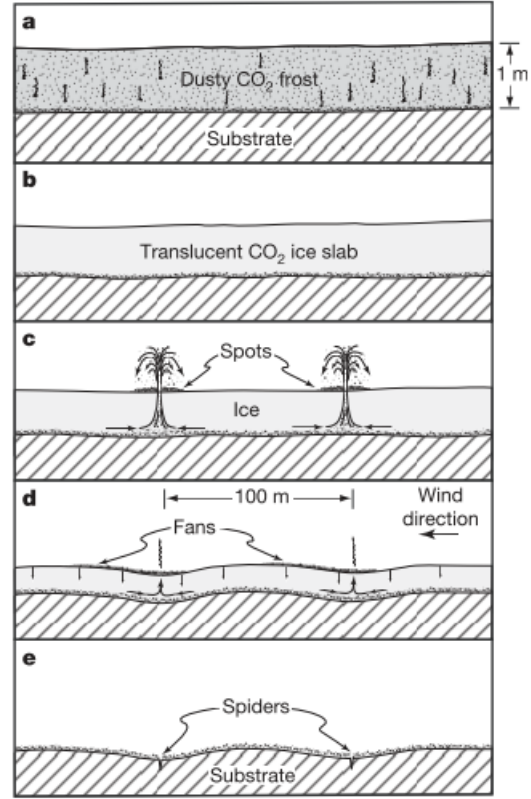


Figure 1.5: Formation of spots, fans, and spiders (Kieffer et al., 2006). a) a 1-meter-thick layer of dusty ice undergoes a "dust-cleaning" process, where warm dust particles sink to the bottom of the ice by creating CO₂ gas bubbles; b) dust-cleaning results in a translucent CO₂ ice overlying the soil; c) CO₂ ice sublimates from the bottom until the trapped gas erupts through the ice, bringing sediments onto the surface; d) winds carry the sediments away, creating fan deposits; e) when all ice sublimates the substrate is left with the channels (called spiders) where gas had travelled under the slab ice.

winds can carry the airborne sediments to form a fan deposit. Once the ice sublimates, the channels where the gas travelled, known as spiders, are revealed on the ice-free surface. The fans and spots typically blend in with the surrounding ice-free soil due to having a similar albedo. This model is supported by many remote sensing observations, such as the dust contaminated spectra of the cryptic terrain measured by OMEGA (Kieffer et al., 2006). Modelling of dust cleaning and rupturing of slab ice also show that this model is consistent with the timing of observations of fan activity (Portyankina et al., 2010).

1.2.3 Deposition modes and grain size of CO₂ ice

Differences in the albedo and emissivity between the different parts of the seasonal caps are often attributed to the deposition modes of CO₂ ice. Long path lengths measured in TES and other spectra (Calvin, 1990; Kieffer et al., 2000) support the presence of polycrystalline slab ice on the seasonal caps. (Kieffer, 2003) proposed that this slab likely forms by direct deposition from the atmosphere onto the surface by radiative cooling. This happens because CO₂ is the main constituent of the Martian atmosphere and there is no non-condensing gas that could provide a thermal gradient for conductive cooling. A crystal growing upward from a surface would not continue growing since condensation on its tip is limited by conductive heat loss. In the case of Mars’s CO₂ atmosphere, it would be more thermodynamically efficient to form smooth polycrystalline ice with high emissivity where the dominant radiative loss comes from a bulk material.

Climate modelling, observations of areas with low infrared brightness temperatures likely caused by CO₂ ice clouds, and observations of CO₂ clouds by the Mars Climate Sounder (MCS) all point to some degree of snowfall contributing to the formation of the seasonal caps (Forget et al., 1998; Hayne et al., 2012; Titus et al., 2001). Climate models that incorporate the regional topography also suggest that areas that receive larger amounts of snowfall coincide with areas of lower emissivity and higher albedo. In other words, larger amount of snowfall in some areas creates fine-grained deposits of CO₂ ice that have high albedo (Colaprete et al., 2005; Gary-Bicas et al., 2020).

Microphysical models allow the formation of polycrystalline ice from smaller grained CO₂ ice that might have fallen as snow. CO₂ ice slab can form by pressureless sintering

(Eluszkiewicz, 1993) and sealing off of pore spaces between crystals by vapor transport known as the Kelvin effect (Eluszkiewicz et al., 2005). These processes together with direct deposition can form slab ice on large areas of the seasonal caps.

The metamorphism of CO₂ frost into a polycrystalline layer is an important process, which has been observed in our laboratory experiments and by other authors (Grisolle et al., 2014; Portyankina et al., 2019). Frost metamorphism is driven by a process known as sintering, which generally refers to compacting or forming a solid mass of material by heat or pressure but without liquefaction (Lu & Ishiyama, 2015). Sintering of CO₂ frost occurs because fine-grained porous frost is not in the state of minimum surface energy. It will tend to minimize the surface and grain boundary energy through grain growth and densification. Sintering of granular material occurs by growth of "necks" between adjacent particles and various diffusion and vapor transport processes can supply material for this growth (Eluszkiewicz, 1993; Maeno & Ebinuma, 1983). We will report our observations of sintering of fine grained CO₂ frost and "healing" of fractures on polycrystalline CO₂ ice by sintering in Chapter 3.

1.3 Previous laboratory studies of CO₂ ice

Various laboratory investigations of CO₂ ice have been conducted in previous work that revealed useful information about its spectral and physical properties. Optical constants, reflection and transmission spectra of CO₂ ice have been extensively measured in the 70s and 80s and are summarized in a compilation by Warren (1986). More recent experiments with a focus on CO₂ ice in the context of Mars have been conducted as well. Portyankina et al. (2019) have studied various textures of CO₂ ice under varied environmental conditions. They report several textures including slab ice and triangular crystals among others (Figure 1.6).

Different modes of CO₂ slab formation, including CO₂ snow metamorphism and direct deposition with and without non-condensable gases have been explored in experimental work by Grisolle et al. (2014). They show that fine grained porous CO₂ snow anneals into a slab over time (about a month), and that direct deposition results in a very clear CO₂ slab.

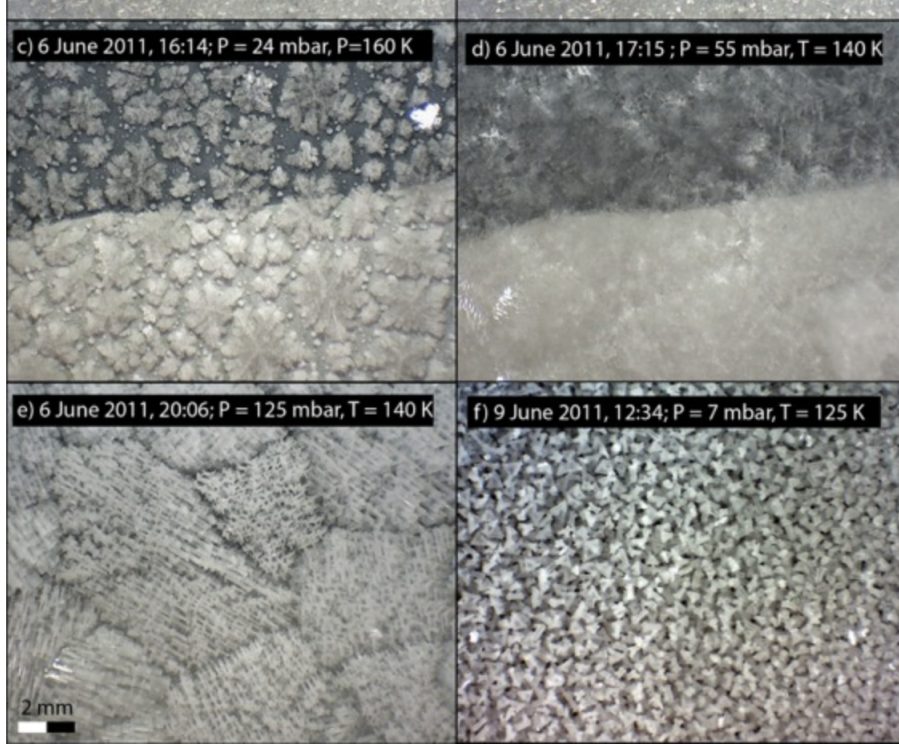


Figure 1.6: *CO₂ ice textures observed by (Portyankina et al., 2019) at different pressure and temperature regimes. Panel c shows CO₂ flakes that over time grow into a translucent slab shown on image in d. Panel e shows oriented crystals, and panel f triangular frost.*

Thermal cracking of the slab ice, and its influence on the albedo were tested in laboratory studies by Philippe et al. (2015). They find that thermal fracturing can be responsible for the abrupt albedo increase of the south seasonal cap that occurs in early spring.

McKeown et al. (2017) have conducted experiments with sublimating CO₂ blocks on regolith, and shown that pits and spider like channels can form by CO₂ sublimation. Kaufmann and Hagermann (2017) have studied dust ejection from CO₂ ice blocks to recreate the geyser-like ice ruptures that form fans in the cryptic terrain. Chinnery et al. (2017) studied the penetration of solar radiation through CO₂ snow. Kaufmann et al. (2020) measured the hardness and yield strength of CO₂ ice under Martian temperatures.

There are several laboratory facilities that have been created to study planetary volatiles including CO₂ ice, contributing to the studies of these materials and processes. Notably, the Simulation Chamber for Imaging the Temporal Evolution of Analogue Samples (SCITEAS) (Pommerol et al., 2015) and CarboN-IR environmental cell together with the spectrogonio

radiometer for the study of the bidirectional reflectance and polarization functions of planetary surfaces (Brissaud et al., 2004) were created to conduct similar experiments as our set up and are being used to study CO₂ ice processes among others. Our aim was to expand some of their capabilities in our setup to continue and improve laboratory investigations of CO₂ ice.

1.4 Reflectance quantities and definitions

We use the terms such as reflectance, albedo, and reflectance factor to explain the quantities we measure in the lab. This section aims to clarify the definitions of the terms and our use of the terminology. Reflectance is most generally defined as the fraction of incident light scattered or reflected by a material. There are different kinds of reflectance depending on the degree of collimation of the source light and the light picked up by a detector. In remote sensing we commonly use bidirectional reflectance, since in many cases it approximates the conditions quite well. For example, the narrow angle subtended by sunlight can be considered directional, and the detector on board of a spacecraft picks up light within a narrow angle (Hapke, 2012). Albedo is translated from latin as "whiteness", and (similarly to reflectance) represents the fraction of incident light that the surface reflects (Coakley, 2003). There are also different types of albedos. Lambert albedo is often used in planetary science. It corresponds to the ratio of the measured radiance to the the incoming solar flux per unit surface, which in turn is proportional to the cosine of the incidence angle (Langevin et al., 2007). Many laboratory measurements of surface reflectance are reported in units of reflectance factor (RF). The reflectance factor of a certain surface is defined as the ratio of the reflectance of the surface to the reflectance of the perfectly diffuse surface (also called a Lambertian surface) under the same illumination and measurement conditions (Hapke, 2012).

In our experiments we use a near infrared reflectance spectrometer, which is calibrated using a diffuse reflectance standard that approximates a Lambertian surface. Since we measure reflectance relative to this surface, we report our values as the reflectance factor.

We will use the term "relative albedo" when we report results of our calculations of surface

brightness imaged with an infrared camera. In this case we also use the same reflectance standard for reference, and use a different term (relative albedo vs. RF) to distinguish the reflectance quantities we measure using two different methods.

1.5 Goals and brief summary of the work

Numerous orbital missions returned data on the large-scale temperature, albedo and the spectral signatures of Martian CO₂ ice deposits. However, there are not sufficient laboratory studies to explain the deposition conditions for these various ice textures and their role in the formation of a number of surface features. Previous experiments observed several different ice textures while they formed CO₂ ice under Martian temperatures. However, they were limited because the pressures, time scales, illumination, and the "warm sky" conditions were starkly different from Martian polar environment (Portyankina et al., 2019). Many of the experiments mentioned in Section 1.3 (e.g. Kaufmann and Hagermann (2017) and McKeown et al. (2017)) used commercially prepared CO₂ ice that might not be the best analogue to the ice that forms on Mars because of contained contaminants.

Our work aimed to expand upon previous experiments and enable future laboratory work to study CO₂ ice. The main goals were to create an experimental setup that can simulate the Martian polar environment, test its capabilities, and conduct experiments to study the deposition and the properties of CO₂ ice. We mainly focus on the textures of CO₂ ice, their corresponding albedos and reflectance spectra, and how these properties change with small variations in environmental conditions.

Within the scope of this work, we completed the setup, recreated the conditions of Mars polar surface in an environmental chamber and deposited CO₂ ice directly from the chamber's CO₂ atmosphere. We conducted experiments to study the texture of the ice on macroscopic and microscopic scales, and recorded its reflectance spectra in near-infrared wavelengths (1-2.5 micrometers). We report three distinct textures: transparent slab ice, columnar/triangular crystal frost and fine-grained "blanket" frost, and the environmental conditions under which these textures form. We confirm that in the conditions most appropriate for Mars poles, slab ice or polycrystalline transparent ice remains the most consistently

forming texture. We observed sintering of fine-grained frost into a pre-existing slab ice as well as fracturing of slab ice due to thermal gradients. We report one instance of ice breaking at a pre-existing fracture due to basal sublimation on a heated substrate, a similar process to the formation of jets on the Cryptic Terrain. We also report relative albedos from infrared images for different textures of CO₂ ice.

The next chapter describes the experimental setup and its capabilities in more detail, and suggests further possible experiments. The results chapter presents observed textures of CO₂ ice, their formation conditions, reflectances and relative infrared albedos. We discuss our obtained results and interpret them in Chapter 4, and summarize this work in Chapter 5.

Methods

2.1 Experimental setup

As an important part of my master's research, I installed and calibrated each piece of equipment and developed the procedures that we use in this lab. The experiments are conducted in a vacuum chamber where we approximate conditions typical for the polar areas of Mars. The main two parameters - pressure and temperature are monitored and controlled using a LabView Software designed for the system and written in-house. We collect imagery and spectral data using digital microscopes, a standard web-camera, an infrared camera, and a near-infrared spectrometer. Figure 2.1 shows the entire setup outside the chamber and the following sections describe the components in more detail.

2.1.1 Vacuum chamber and peripherals

The setup consists of a meter-long and 70 cm diameter cylindrical stainless steel vacuum chamber (Figure 2.2). It features three viewing ports: one on the top window, one on the large door, and one on the smaller reach-in port on the opposite end. A vacuum port with a valve connects the chamber to the rotary vane vacuum pump. There are also ports for two pressure sensors, liquid nitrogen (LN_2) lines supplying the cooling systems, and one gas feedthrough. Another port is used for thermocouple wires, and a 15 pin D-Sub is used to transmit data and electricity. We can collect data from and control the camera and digital microscopes using two USB ports, while two fiber-optic feedthroughs with sub miniature A (SMA) 905 connectors allow us to collect spectral data from the chamber.

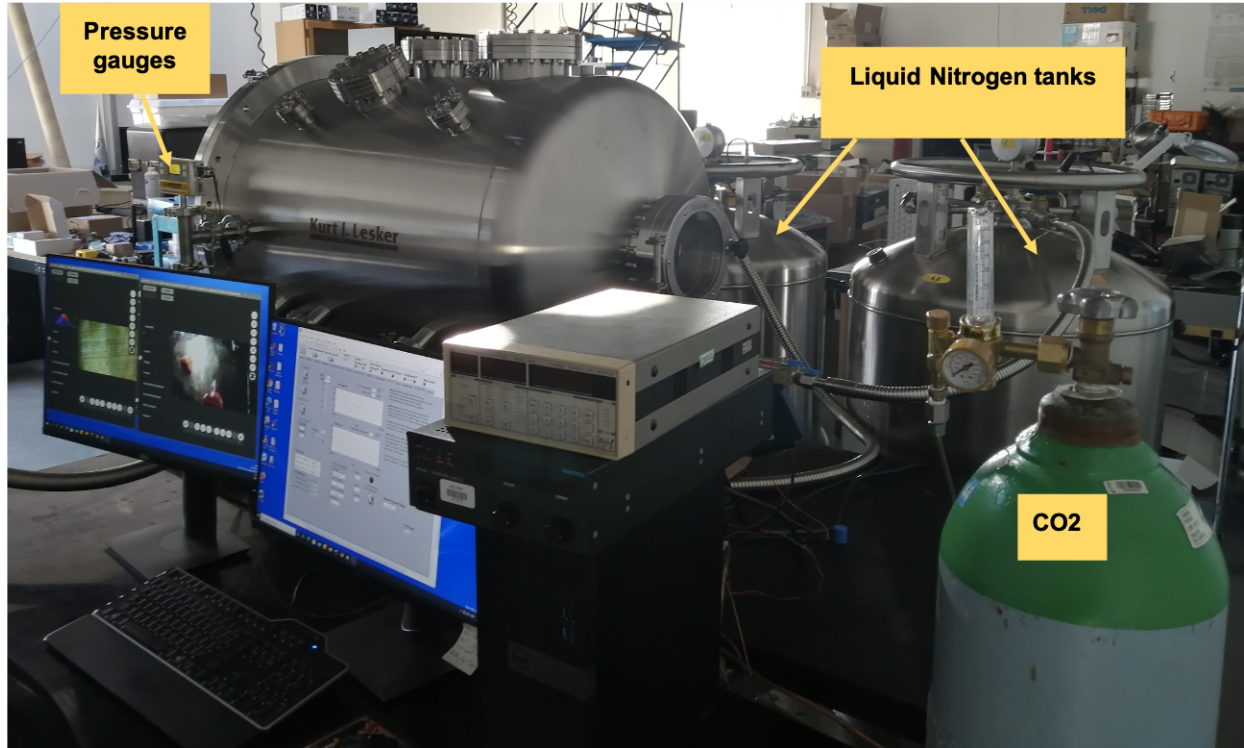


Figure 2.1: Large-scale view of the components of the experimental setup

The cooling system consists of a platen, a cylindrical shroud, and a cold plate, each cooled by a separate liquid nitrogen loop (Figure 2.3 a). The shroud provides a "cold sky" environment which helps to cool the sample plate more effectively. The cooled platen and shroud also lower the temperature of the atmosphere in the chamber to reduce the temperature gradient in the sample. The shroud is coated with high-emissivity paint in order to approximate a black-body, which drastically reduces unwanted reflections during the collection of imagery and spectral data. Additionally, it improves heat transfer from the chamber to the liquid nitrogen circulating in the shroud. We ensure that the platen and shroud are only cooled to a temperature just above the condensation point of CO_2 in order to prevent the deposition of ice on them. Since the CO_2 ice will preferentially deposit on the coldest parts of the chamber, having a too cold shroud or platen can slow down the growth of ice on the sample plate where we can observe it. We insulate copper pipes that carry LN_2 to the plate for the same reason in order to avoid growing ice on the pipes. The ice can also grow thick enough on the shroud to fall onto the sample plate during an experiment, compromising our observations.

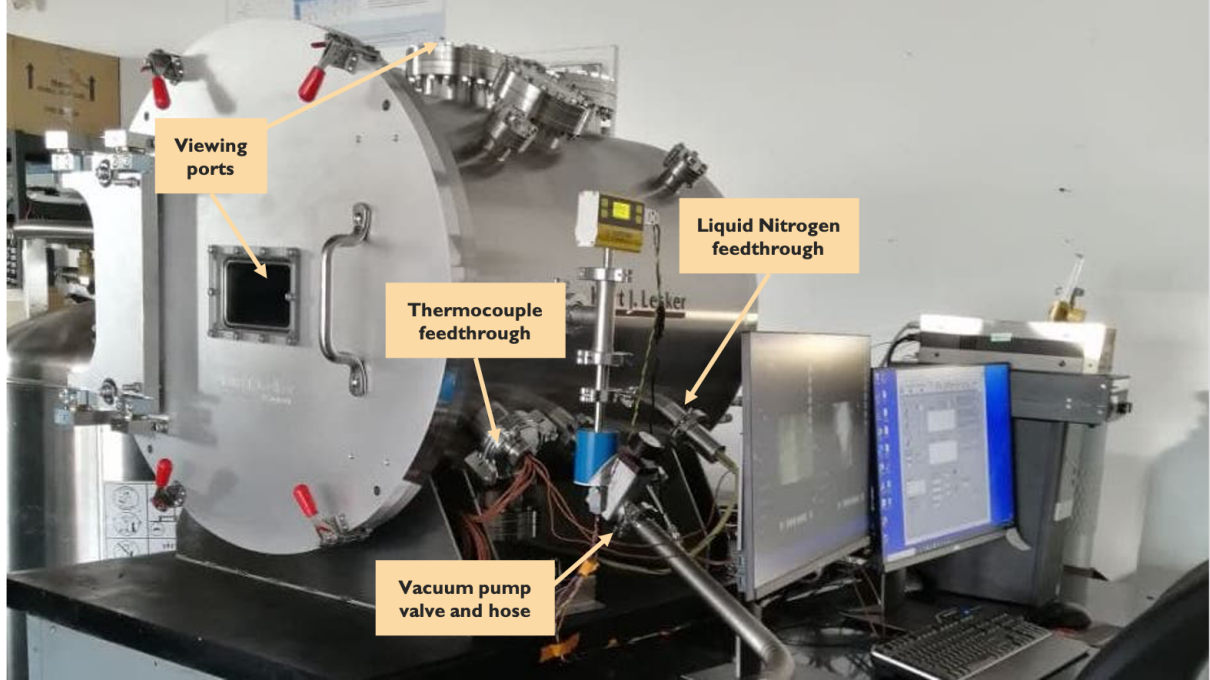


Figure 2.2: Vacuum chamber and the control computer. The ports for thermocouple wires, liquid nitrogen line to the cold plate, and the vacuum pump can be seen.

The cold plate (Figure 2.3b) is made of aluminium about 10 cm by 15 cm in size with copper pipes running through for liquid nitrogen flow. It is mounted on top of the platen, which also holds our imaging equipment. A one-inch thick copper sample plate is placed on top of the cold plate and the CO₂ ice is deposited directly on its surface. In most experiments we used a non-coated copper plate, however, after discovering that its strong specular reflectance affected our measured reflectance of the ice, we used a copper plate coated with black aerosol paint for some of the later experiments.

A thin polyimide flexible electric resistance heater is placed between the sample plate and the cold plate, and is used for achieving more uniform and stable temperature of the sample plate and finer temperature control. It has been added to the temperature control system because the LN₂ system on its own is inaccurate and slow to respond. By cooling the sample plate with liquid nitrogen slightly beyond our desired temperature, and then heating it back up to the desired temperature using the heater, we can significantly improve the temperature precision, stability, and uniformity. This is because the electric heater has a rapid response time and can switch on and off every second, with variable Wattage.

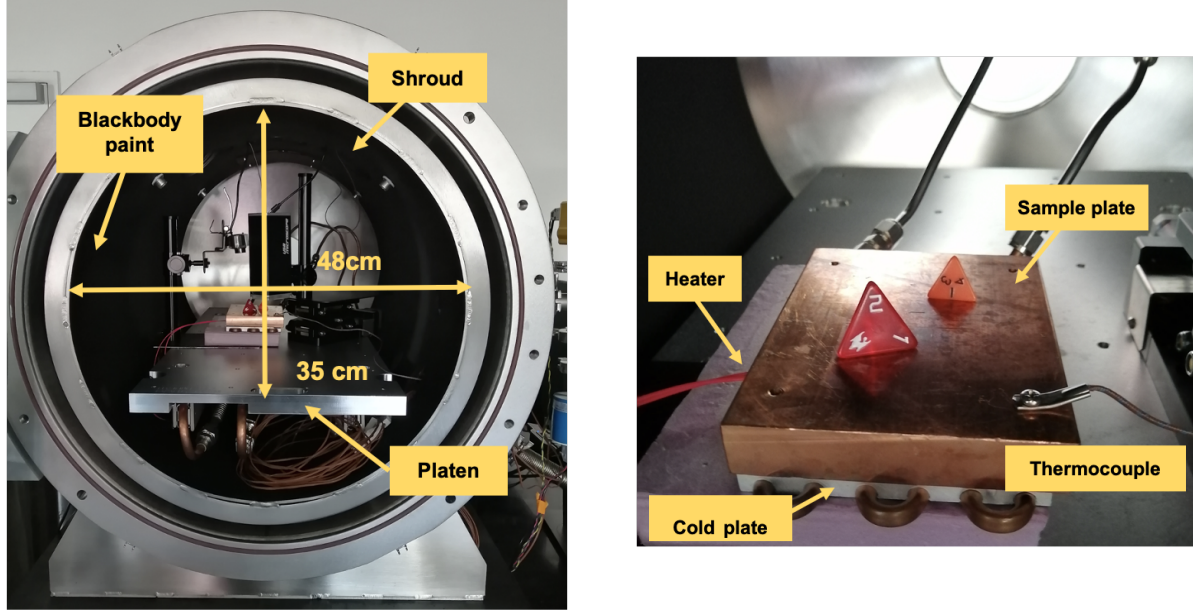


Figure 2.3: a) View inside the vacuum chamber; b) Close-up view of the cold plate and sample plate.

Temperature is monitored by 9 thermocouples fixed on multiple sites on the sample plate, the platen, and the shroud. A thermocouple hanging from the camera stand records the temperature of the atmosphere above the sample plate. The signal from the thermocouples is transferred to the thermocouple reader (12-channel SR360 by Stanford Research Systems), which is connected to the lab computer for logging. The software for the chamber environment control then receives the temperature readings, and using a proportional–integral–derivative (PID) controller algorithm, adjusts the liquid nitrogen flow by opening and closing solenoid valves attached to LN₂ dewars. If the temperature of the sample plate needs to be increased, the software adjusts the power going from the power supply to the heater accordingly.

The pressure system is controlled by the same software based on a PID algorithm developed in our lab. The analogue signals from two pressure sensors are converted to digital form using a digital acquisition device (DAQ MODULE USB 6001 from National Instruments), and sent to the lab computer and logged. To reach a desired pressure, the control software uses these readings and sends commands to an Alicat mass flow controller that adjusts the inflow of CO₂ gas into the chamber.

We use two pressure gauges: a Superbee pirani gauge during the evacuation of the

chamber by the rotary vane vacuum pump and a Kurt Lesker capacitance manometer during the experimental runs. The capacitance manometer is gas independent, so it can measure the accurate pressure of the CO_2 atmosphere we create in the chamber and has a range between 0.02 to 200 Torr. The Superbee gauge is not gas independent and is calibrated for N_2 . This has the benefit of becoming a leak detection system. When pumping down, the two gauges will have the same reading, but when CO_2 is introduced, the Superbee deviates strongly. If there is a LN_2 leak or a leak from the atmosphere, especially when CO_2 is depositing as ice, then the two gauges will read pressures that are closer together than they should be.

A detailed schematic of all system components and controls is shown in 2.4.

2.1.2 Imaging and spectroscopy

The imaging components used in the chamber are shown in Figure 2.5 a. We observe the deposition of CO_2 ice on the sample plate using a standard web camera mounted inside the chamber. Growth of the ice crystals is recorded using two digital microscopes: one with 2 Megapixel resolution for intermediate magnification (approximately 10 times), and another with 5 Megapixel for higher magnification (about 40-60 times). Both microscopes are equipped with light-emitting diodes (LEDs) that illuminate imaged areas. The microscope with the higher magnification can be placed onto an electric elevator stage that can lift the microscope higher as the ice quickly grows thicker, in order to keep the sample surface in focus over a longer time. Additionally, we use a 2 megapixel stereo camera that takes two images: one in visible range and another in near infrared (700-1100 nm). The IR images are especially useful since they do not depend on the external lighting conditions and are free of glare from the metal sample plate when it is illuminated by the microscope LEDs.

Two tetrahedral dice are placed on the sample plate to serve as a scale on the images and to help determine the approximate ice thickness. The smaller orange die has an edge length of 2 cm, and one side has markings for every 5 mm. The larger red die has 2.5 cm edges.

The spectra are collected with a near infrared spectrometer AvaSpec-NIR256-2.5-HSC-EVO from Avantes. The light source we use is AvaLight-HAL-S-Mini equipped with a 10 Watt Tungsten halogen lamp (Figure 2.5 b). The spectrometer covers a spectral range between 1000-2500 nm with a minimum resolution of 12.9 nm. To measure the spectra

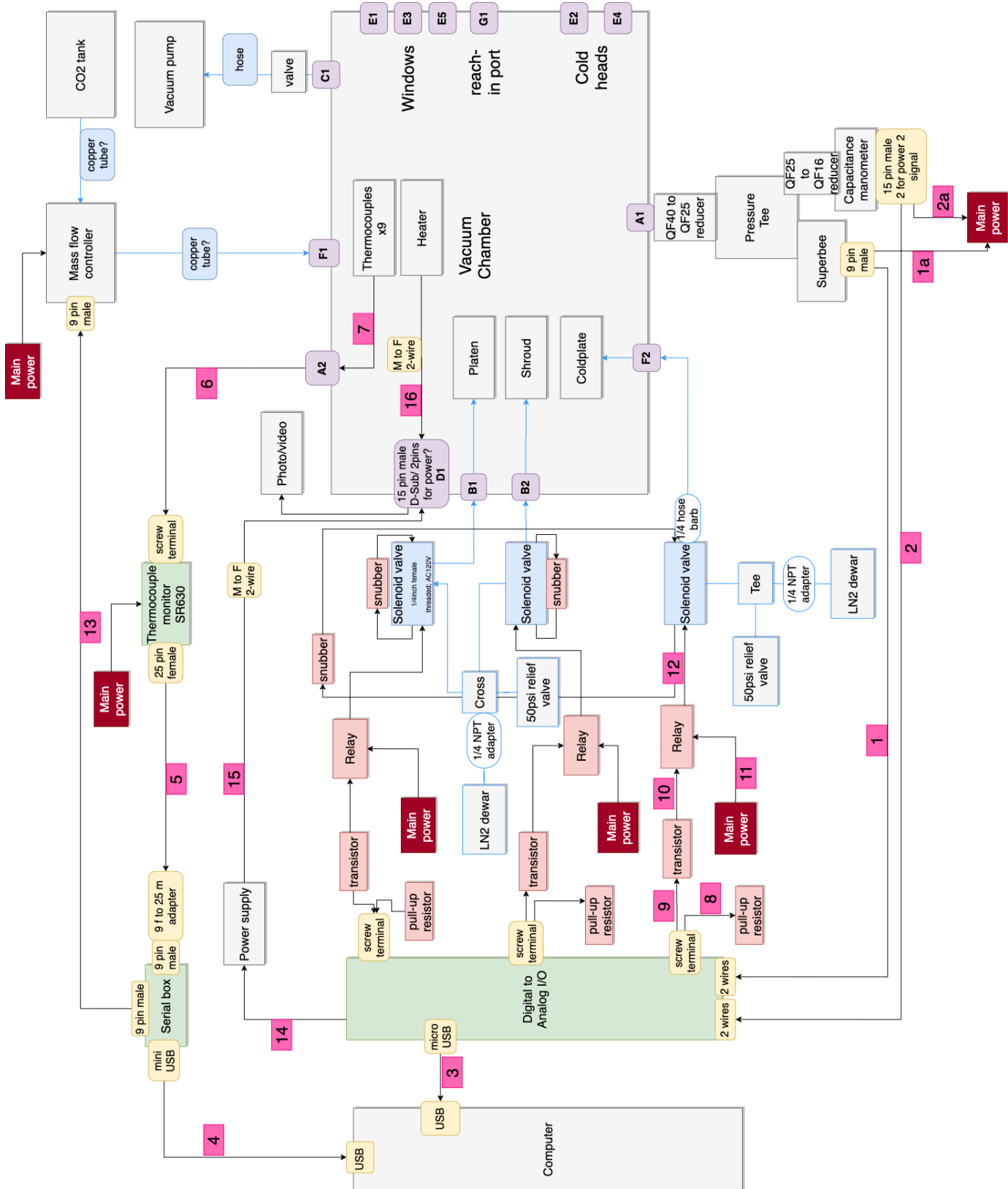


Figure 2.4: A diagram of all vacuum chamber components and controls.

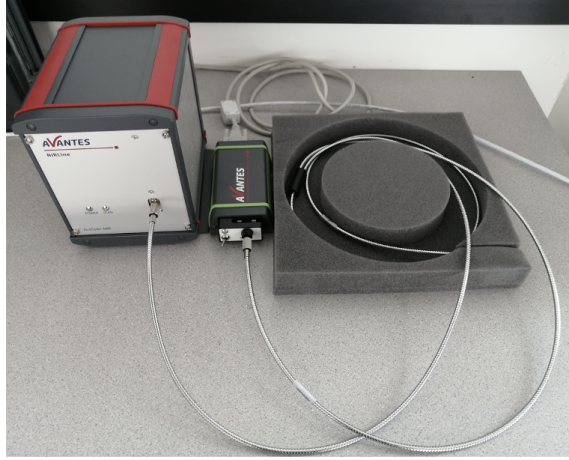
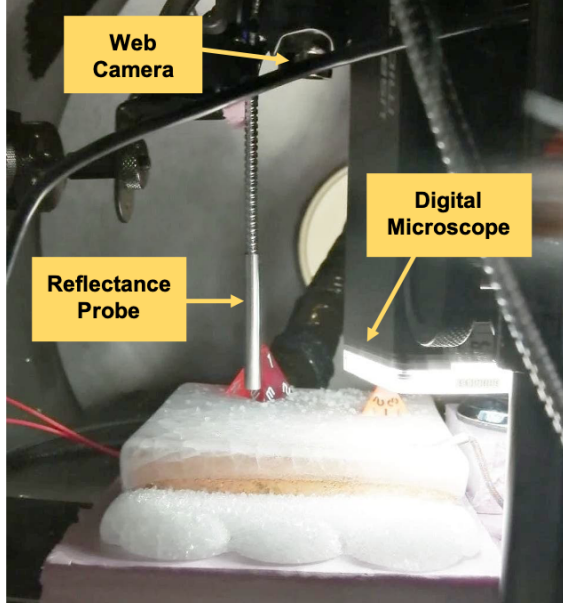


Figure 2.5: a) Components of the imaging system; b) NIR spectrometer and light source.

directly from the sample surface we use custom-made low-temperature fiber-optic cables and a reflectance probe. These cables connect to the light source and the spectrometer on the outside of the chamber via feedthroughs with SMA connectors.

The reflectance probe is used for measurements perpendicular to the ice surface: with zero phase angle, and zero emission and incidence angles. We can achieve this geometry since the detector fiber is located in the center of the probe tip and is surrounded by six 400 micron illumination fibers. The surface illumination and sensed area is relatively small and is less than 1 cm in diameter.

2.2 Experimental procedures

This section describes detailed procedures followed in a typical experimental run for the purpose of documenting these for future users of the setup and reproducibility. The preparation includes checking and adjusting the focus of the camera and digital microscopes, and adjusting the position of the reflectance probe. This is an essential step, since after the evacuation of the chamber, we no longer have direct access to this equipment, and can only control data acquisition. One of the challenges at this step is adjusting the focus on the

cameras since they become slightly out of focus when the air from the chamber is evacuated. We try to "over focus" slightly on the sample plate during the preparation step, which tends to improve the image quality during the experiment.

The evacuation of the chamber takes approximately thirty minutes after the pump is turned on, and we bring down the pressure from atmospheric 760 Torr to about 0.5 Torr within this time. Most of the moisture is expected to evaporate and leave the chamber at this stage, but to ensure dry conditions, we fill the vacuum chamber with CO₂ gas. We let CO₂ into the chamber until the pressure reaches 100 Torr, then pump out the gas till it is below 1 Torr. Stopping at higher pressures will allow ice to accumulate during the cooling cycle before the desired temperature conditions of the experiments are met.

The next step in the process is cooling. We first start cooling the shroud, as it requires more time due to the larger area the liquid nitrogen loop has to cover. We cool the platen to about 233 K at the beginning and its temperature remains relatively stable within a few degrees throughout the experiment. The cold plate cooling can be started after the shroud reaches temperatures around 253 K. After the latest upgrades, our system can maintain a constant temperature of the shroud and ensure that it will not cool to the point of significant deposition of CO₂ ice. The time delay between the start of the three cooling loops is mainly for the purpose of saving liquid nitrogen. To make sure the ice does not grow on the shroud, we set the shroud temperature to around 170 to 190 K. When the temperature of the shroud is lower, CO₂ ice is deposited on the shroud and can grow thick and heavy enough to break-off and fall onto the sample plate, compromising the experiment. In experiments with the use of shroud cooling to 190 K, the air temperature above the cold plate reaches below 213 K. Different parts of the shroud cool at different rates and have different temperatures. However, we are mainly concerned with keeping the temperature of the cold plate and "air" temperature stable because these have more direct influence on the deposition of ice. The cold plate temperature is usually set between 160 to 140 K around which we tend to observe CO₂ ice deposition. Once all the cooling elements reach the desired temperatures, they can be kept stable using our LabView software. An example of temperatures measured at different parts in the vacuum chamber during one experiment is shown in Figure 2.6 a. Small peaks in the cold plate temperatures show when we intentionally heated the plate to cause

fracturing. The "air" stays stable around 220 K despite larger fluctuations in the shroud temperatures.

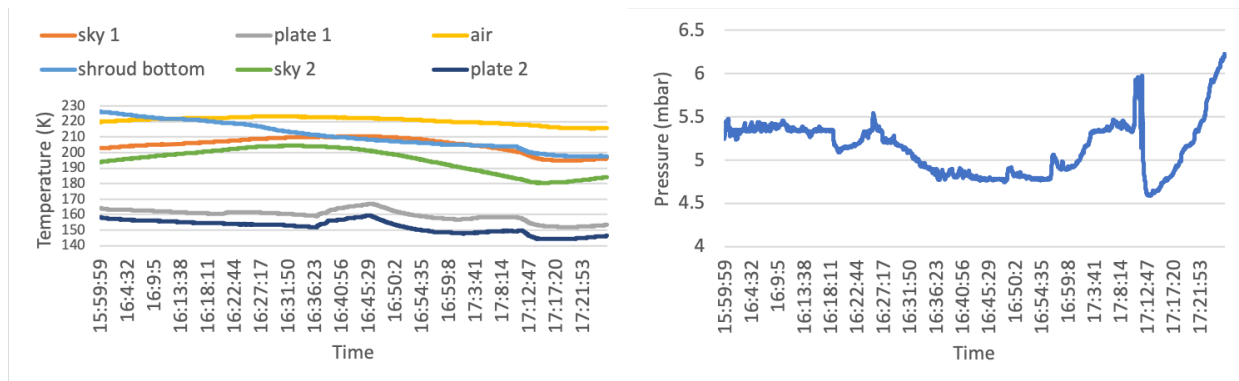


Figure 2.6: a) example of temperature data at different parts of the chamber from one of the experiments. Sky 1 and 2 are readings from two thermocouples on different locations on the shroud, Plate 1 and 2 are from the opposite ends of the cold plate; b) pressure data recorded from the same experiment.

We start the CO_2 gas input shortly before the desired sample plate temperature is reached. This is done because earlier input of the gas results in significant deposition onto the copper pipes carrying LN_2 . If however, we start the gas input after the CO_2 freezing temperature is reached, we start to deposit ice at a lower atmospheric pressure than the 5-7 mbar representative of Martian poles. Our pressure system runs in units of Torr, which have a conversion factor of 1.33322 to mbar. Most commonly, the experiments were conducted with pressures set and kept constant at 5-6 Torr (~ 6 -8 mbar). The gas input by the mass flow controller is able to keep up with the freeze-up rate and keep pressure variations to less than 1 Torr. Figure 2.6 b shows an example of recorded pressure throughout an experiment starting from the deposition of the ice. The pressure is stable around 5 mbar, but slightly larger jumps tend to occur when the temperature in the chamber warms and CO_2 is added into the chamber's atmosphere due to sublimation.

The cameras that image the growth of ice on the sample plate are controlled using an open-source digiCamControl software. We can control the web camera and two digital microscopes simultaneously in the same software. One of the digital microscopes - ViTiny UM12 - can be controlled with the included product software, which allows for manual and

automatic focusing. We take images of the sample plate surface at one-second intervals. A python code records images from the stereo camera at given intervals, and control of the remaining cameras and microscopes is going to be integrated to this software for convenience. Two Spectralon reflectance standards with 99% and 40% reflectance are placed in the stereo camera's field of view to serve as references for albedo calculation from the IR images.

To collect spectral data, we use the proprietary software provided by the spectrometer manufacturer AvaSoft 8. We calibrate the spectrometer before the chamber door is closed, and take a reading of the copper plate before ice deposition begins. We use the autoconfigure integration time option, which selects the optimal integration time for present conditions such as illumination and distance from the sample. We save "dark data" with the light source off, and a reference spectrum with the light source on for given integration time. The software automatically corrects all spectra that follow the calibration process, and the signal we record during the experiment represents the spectra of the CO₂ growing on the sample plate.

2.3 Data analysis

The data products we get from experiments include real-size and magnified sample images taken at one second intervals, log files from the chamber control software, and spectra.

For ease of use and correlation of images to the time-logged environmental data and spectra, we add time stamps to the images using a short python code. We then combine the images into a video file for ease of observation using an open-source command-line video making tool FFmpeg (Developers, 2020).

We use Microsoft Excel to extract and plot the pressure and temperature data from the log files. We also extract excel files containing the spectra from the AvaSoft 8 software for further corrections and creating graphs.

2.3.1 Relative albedo extraction from infrared images

In addition to recording reflectance spectra, we extract albedo values from IR images taken by the stereo camera. The image field of view contains the sample plate surface and two reflectance standard pucks (Figure 2.7). Before the start of the experiment we take a

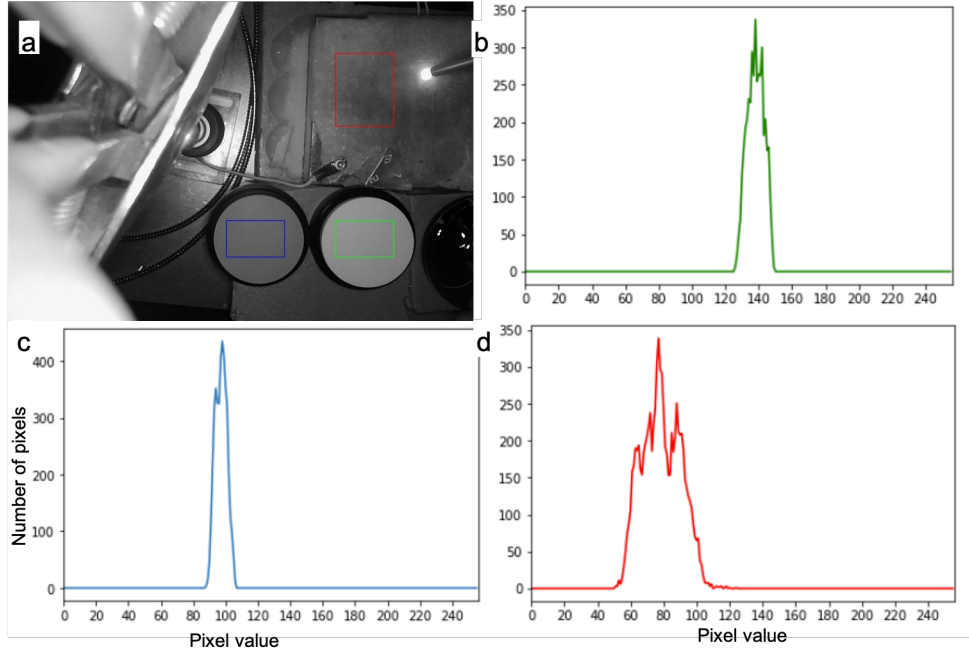


Figure 2.7: IR image from stereo camera with extracted brightness value histograms of regions of interest. The colors of the graphs correspond to the colors of the squares defining the areas of interest.

reference "white" image of a white sheet of paper, in order to correct for the illumination irregularities on the image. The center of the image receives more light than the edges, and dividing by the "white" image corrects for this effect. We take images of the CO₂ ice growth during the experiments, and calculate the albedo values of the ice relative to the spectralon standards. The code for extracting brightness values was created by Jamie Isen. It uses the OpenCV python library (Bradski, 2000) designed for computer vision applications.

The IR image that appears in grey-scale is first converted from an RGB (Red, Green, Blue) image into an HSV (Hue, Saturation, Value) color model and split into the three components. We use the value component, which represents the intensity of light or brightness of the image. We select areas of interest on the ice and on the reference standards, and extract the histograms of these areas. Since these areas are usually of uniform brightness, the histograms have one strong peak around one value. Since we know the nominal albedos of the reference standards (99% and 40%), their brightness values on the IR image, and the brightness values of the ice, we can calculate the albedo of the ice. We report these values

in the next chapter.

2.4 Capabilities of the planetary environment chamber

Completion and testing of the setup was a major part of this work, and the experiments reported here represent only a small share of a wide range of possible studies that our system enables. Individual components of the experimental setup are described in the methods chapter in detail, while this section emphasizes further capabilities and potential use in other experiments.

The planetary environment chamber supports a controlled low pressure and low temperature environment suitable for simulating conditions common on a number of planetary bodies. Our focus has been on the Martian polar processes involving CO₂ ice, hence the setup has been adjusted and extensively tested for pressures ranging between 4-10 mbar and temperatures between 140-200 K. The latest software improvements were successful in reducing the pressure fluctuations to less than 1 Torr and temperature fluctuations to 1-5 K (depending on the part of the cooling system), which is extremely stable for a system of this size. This makes the system well-suited for studying CO₂ ice deposited and kept under Martian polar conditions. Some potential areas of future investigations that our setup in its current configuration can be used for include interactions of CO₂ ice with dust during its deposition and sublimation, as well as the photometric behaviour of these dust-ice mixtures. There is also a possibility to investigate spectral responses of different amounts of water ice contamination in the CO₂ ice, which is commonly expected from remotely sensed data (Forget et al., 1995; Warren et al., 1990). Other experiments that require stable Martian atmosphere but higher surface temperatures approximating mid- or equatorial latitudes of Mars can also be accommodated.

Another notable feature of the environmental chamber is its size compared to other systems used for similar experiments. It provides an advantage for experiments where a direct observation of the sample is required, since we can place measurement instruments directly inside the chamber. The dimensions of these instruments are, however, limited to 35 cm in height and about 90 cm in length, hence extremely precise goniometer systems which

tend to have much larger arm lengths would need to access the sample through the chamber windows. The vacuum chamber features three 15 cm - diameter ports on the top positioned at 90 and ± 30 degrees (Figure 2.8) that can be used for spectroscopy and photometric measurements with the use of appropriate transmission windows.

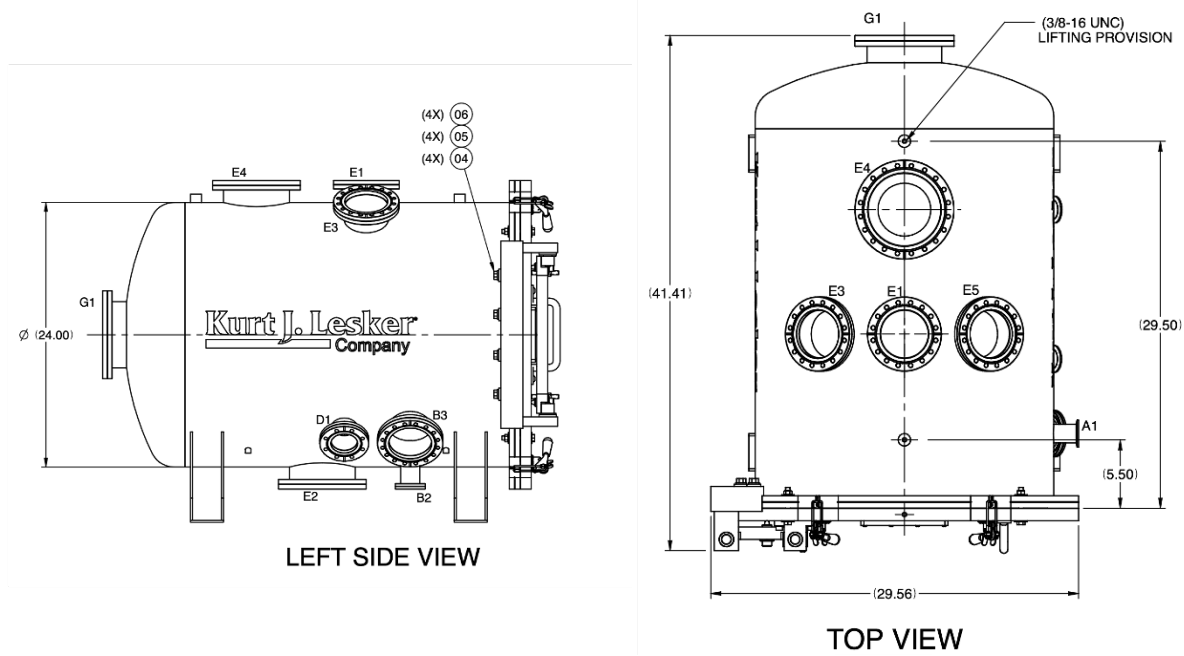


Figure 2.8: Drawings of the vacuum chamber showing a view of the left side and the top of the vacuum chamber. Port E1 features a window, ports E3 and E5 are currently closed but can be used as additional windows. Ports E2 and E4 are reserved for future cold heads. Distances shown in inches.

The experimental setup also allows to change the composition of the gases in the chamber atmosphere by replacing the CO₂ tank with a tank of a required mixture of gases. One example where this might be useful is for experiments where non-condensable gases contained in small amounts in the Martian atmosphere need to be included in the simulated atmosphere.

Apart from Mars, the experimental setup with appropriate modifications and upgrades holds further potential for simulating other, colder planetary bodies with thinner atmospheres and ice-covered surfaces, such as icy satellites, Pluto and other dwarf planets. The vacuum chamber is designed to be able to hold pressures lower than those on Mars if our current seals are upgraded. The temperatures in our chamber can reach as low as 100 K using

liquid nitrogen cooling; however to reach even lower temperatures there is a possibility for installing cold heads on two of the currently unused chamber ports (Figure 2.8). In all of these varying conditions and atmospheric compositions, the set up can provide spectrophotometric measurements of ice samples in the near-infrared (1-2.5 micrometers) range at zero phase angle, with potentially expanding into UV-Visible or to longer near-IR wavelengths. We are also working on a construction of a small goniometer to measure spectral reflectance from a wider range of geometries.

Results

3.1 Textures of CO₂ ice under Martian conditions

We have repeatedly recorded the deposition of CO₂ ice in multiple experimental runs and report on the similarities and differences in textures between the runs, and relate some of these to the environmental conditions where possible. More than twenty experiments of increasing complexity have been conducted since the initial assembly of the setup in parallel with additions and modifications to the system. A table with an overview of each experiment is included in the Appendix. The average duration of the experiment from the start of ice deposition usually was around 1.5 to 2 hours (5-6 hours including cooling). During the majority of the experiments the thickness of the ice was up to 5 mm, and during one of the runs the ice was allowed to accumulate till it reached approximately 1.5 cm in thickness.

The most commonly observed texture was a polycrystalline translucent slab, which is expected to form on Martian poles. The deposition of the slab on the sample plate starts from widely-spaced nucleation centres, and individual crystals rapidly grow up to about 5-7 mm in size until they reach the neighboring crystals. Over time they merge together, and most of the crystal boundaries get smoothed out, creating a single polycrystalline layer (Figure 3.1). This merging process lasts from about 20 to 30 minutes, after which the ice layer mainly grows vertically, while the copper sample plate remains visible through the translucent ice layer. The environmental parameters during the formation of the polycrystalline CO₂ ice were closest to the polar Martian environment. Throughout the experiment described above, pressures were kept between 5 and 9 mbar, and the sample plate temperature was within

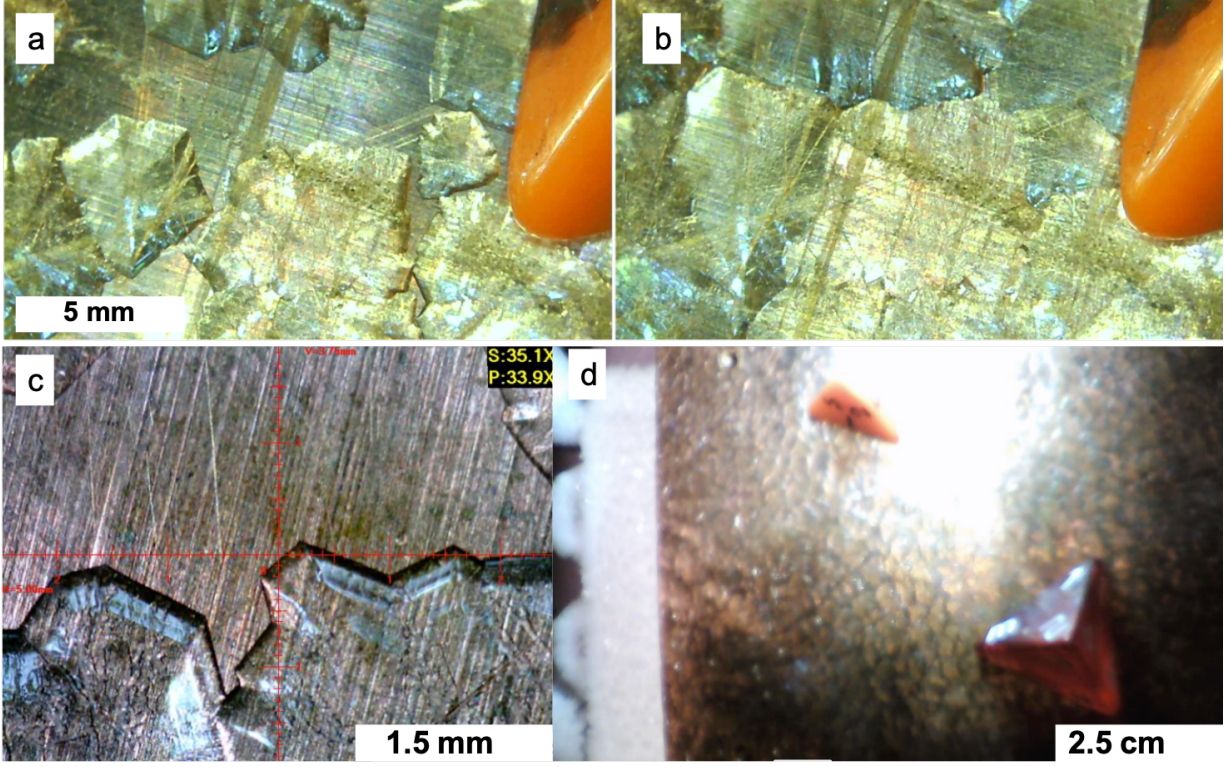


Figure 3.1: Development of the polycrystalline CO_2 ice slab from individual crystals. Orange die for scale; 5 mm from the tip to the black marking. Image b is taken 5 minutes after image a; c) transparent crystal with 35 times magnification; d) web camera view of the beginning of slab ice deposition. Two dice for scale are described in Chapter 2. Scale bar shown in white.

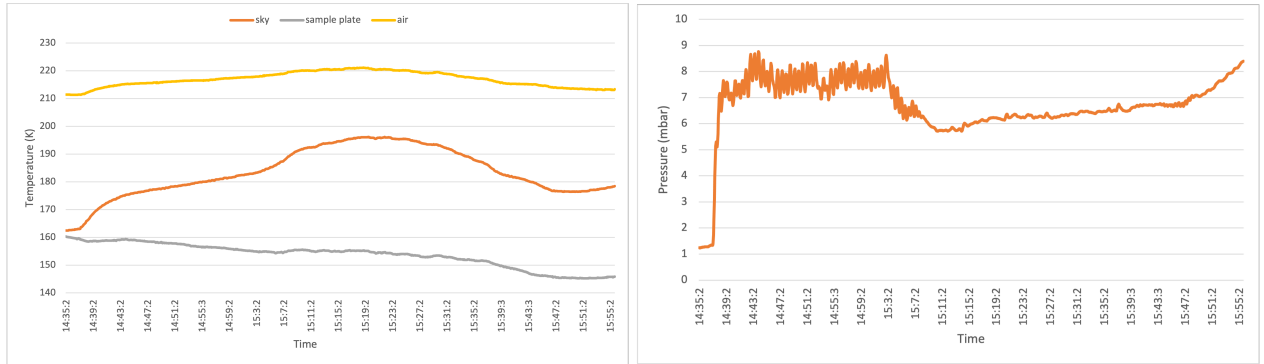


Figure 3.2: a) Temperature of the sample plate, the shroud and atmosphere during the experimental run shown in 3.1; b) Pressure during the same experimental run. First crystals appear at 14:38.

145 to 160 K (Figure 3.2). The cooling shroud was kept between 160 and 195 K and the air measured by the thermocouple above the sample plate was between 212 and 220 K.

The variations in the pressure and temperature here are larger than what our chamber currently supports, since the experiments for investigating texture were conducted before software control for the shroud and platen cooling were introduced, and the shroud flow was controlled manually.

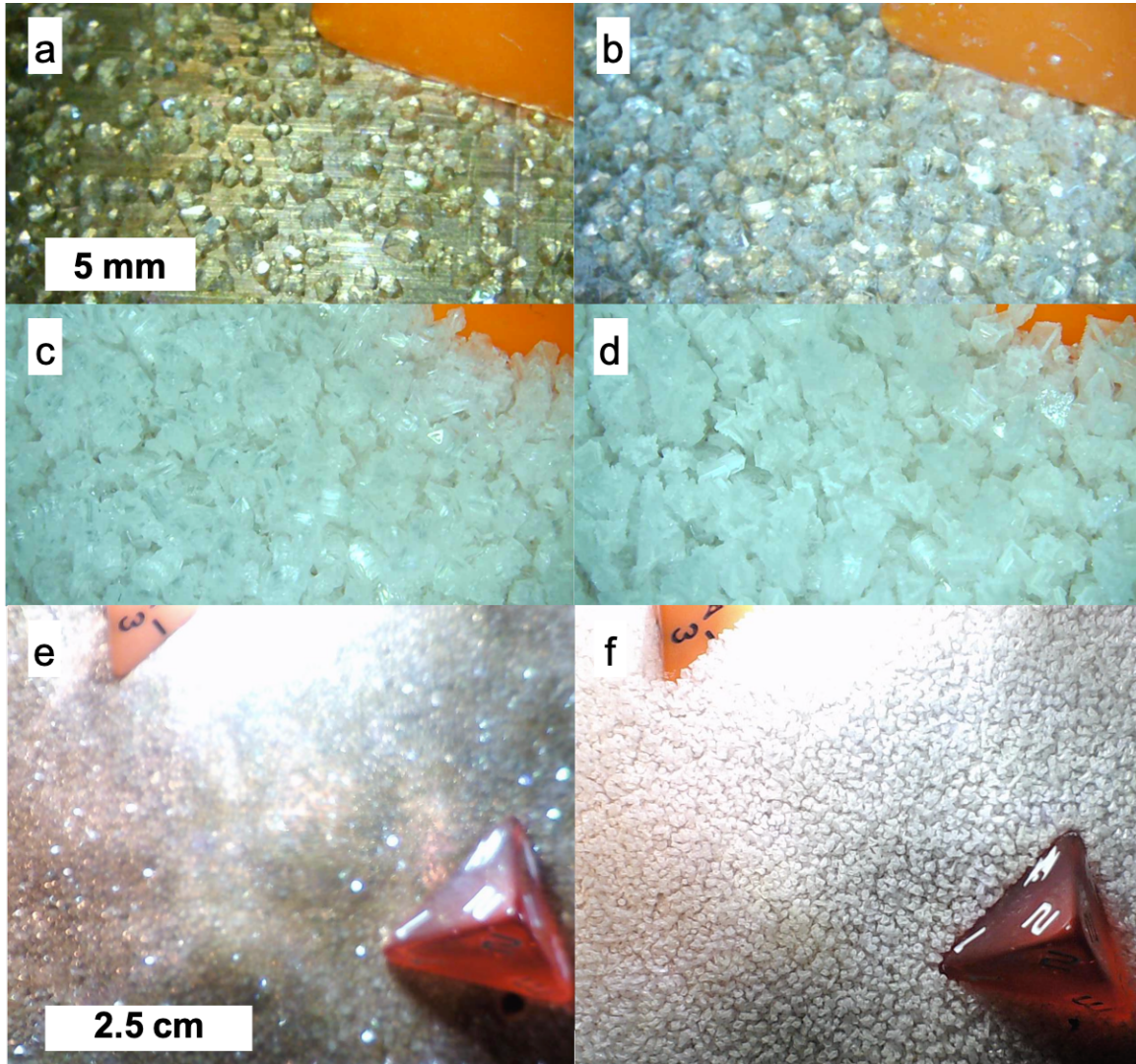


Figure 3.3: Development of a CO_2 frost. Images a,b,c,d are each taken 5-10 minutes apart with about 20x magnification. Images e and f are not magnified and show the beginning and the final stages of frost growth respectively.

Another texture notably different from the transparent slab was observed during the

experiments when the sample plate temperature dropped below 140 K. On a large-scale view, this CO_2 ice appears "frosty", is not translucent, and visually has a much higher albedo than the slab ice (Figure 3.3). Magnified images show the individual crystals that resemble rectangular and triangular prisms in shape, are smaller (up to about 1 mm) and more reflective compared to the crystals that form the polycrystalline layer (Figure 3.3 b, c).

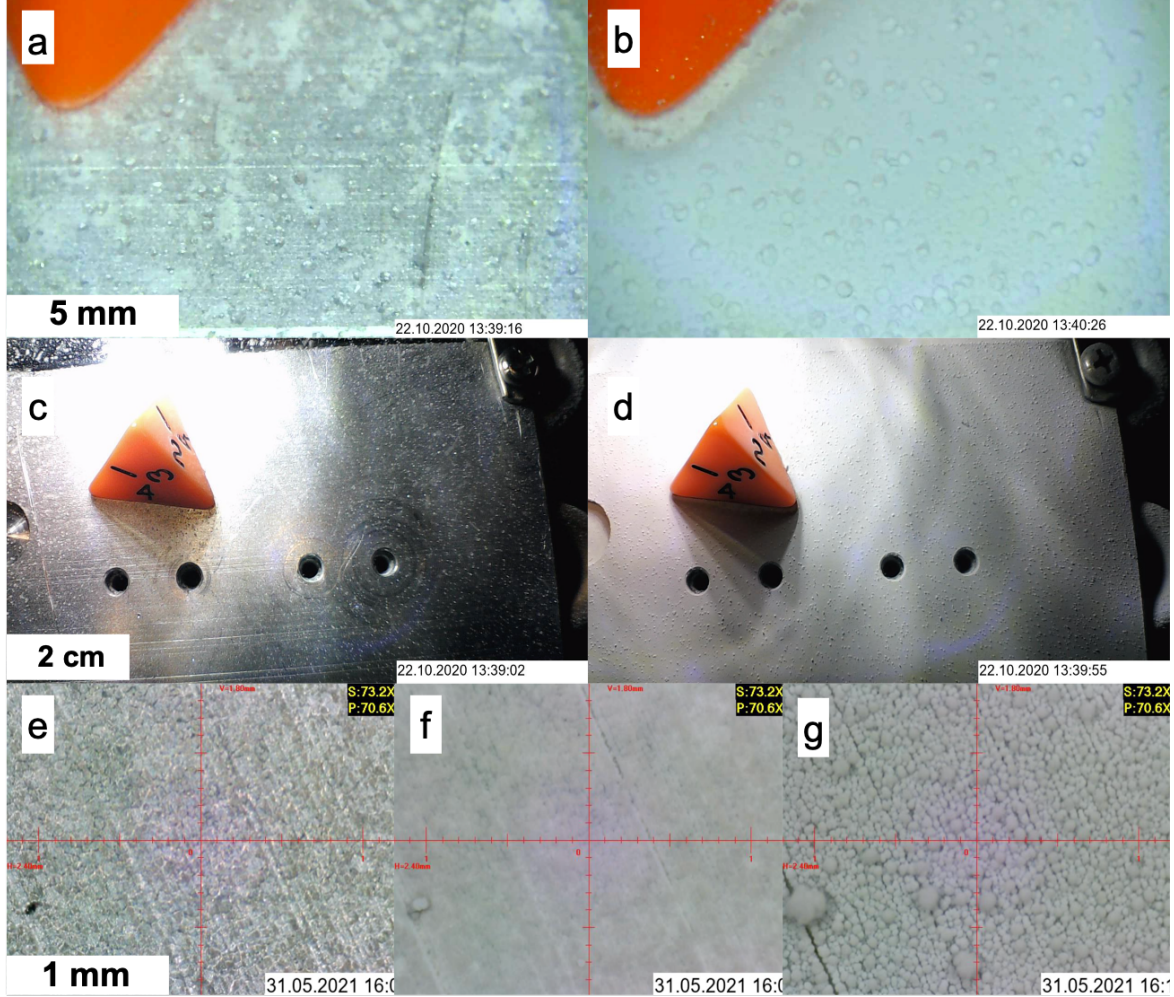


Figure 3.4: Formation of a fine-grained "blanketing" CO_2 frost. a and b are ~ 20 times magnified and taken about 1 minute apart. Aluminium cold plate is exposed in a and c, while in b and d white fine grained frost covers the cold plate. Images e, f, g are taken with 70 times magnification, and show ice-free surface, start of fine-grained ice formation and continued growth of sphere-like crystals.

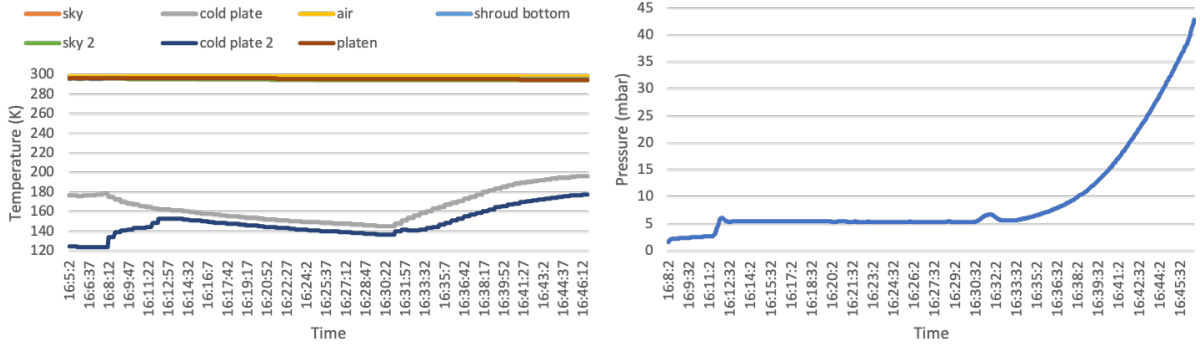


Figure 3.5: Temperature and pressure during the deposition of "blanket" frost. Crystals first appear at 16:09

A third type of texture we observed was a fine-grained "blanketing frost". Figure 3.4 (panels a, b) shows the formation of this texture imaged with ~ 20 times magnification. There are some larger transparent crystal nuclei that start to form, but in several seconds the entire surface gets covered in white frost whose individual crystals were not visible at this scale. Images from the web-camera on larger scale in panels c and d show this "blanketing" frost as well. When observed with a higher resolution microscope under ~ 70 times magnification (Figure 3.4 panels e, f, g) we see that the frost consists of spherical ice crystals most of which are smaller than 0.5 mm. The crystals appear arranged in rows likely due to the linear grooves of the aluminium cold plate. This texture only formed when the ice was deposited directly onto the aluminium cold plate, without the copper sample plate that cools more slowly and uniformly. This may hint at the influence of the substrate on the resulting textures of ice and further investigation is needed.

Most experiments with the formation of blanketing fine-grained frost have been conducted before data logging capability was added, temperature and pressure data was logged only for one experimental run. At the start of nucleation, the cold plate was 124 K at the LN_2 inlet and 172 K at the opposite end of the plate while pressure was at 2.5 mbar (Figure 3.5).

Notably, the shroud and platen were not cooled in experiments when this texture was observed. The importance of the shroud being off is not well understood, but theoretically, a warm sky will result in a larger thermal gradient within the CO_2 ice layer, and that should affect crystal growth.

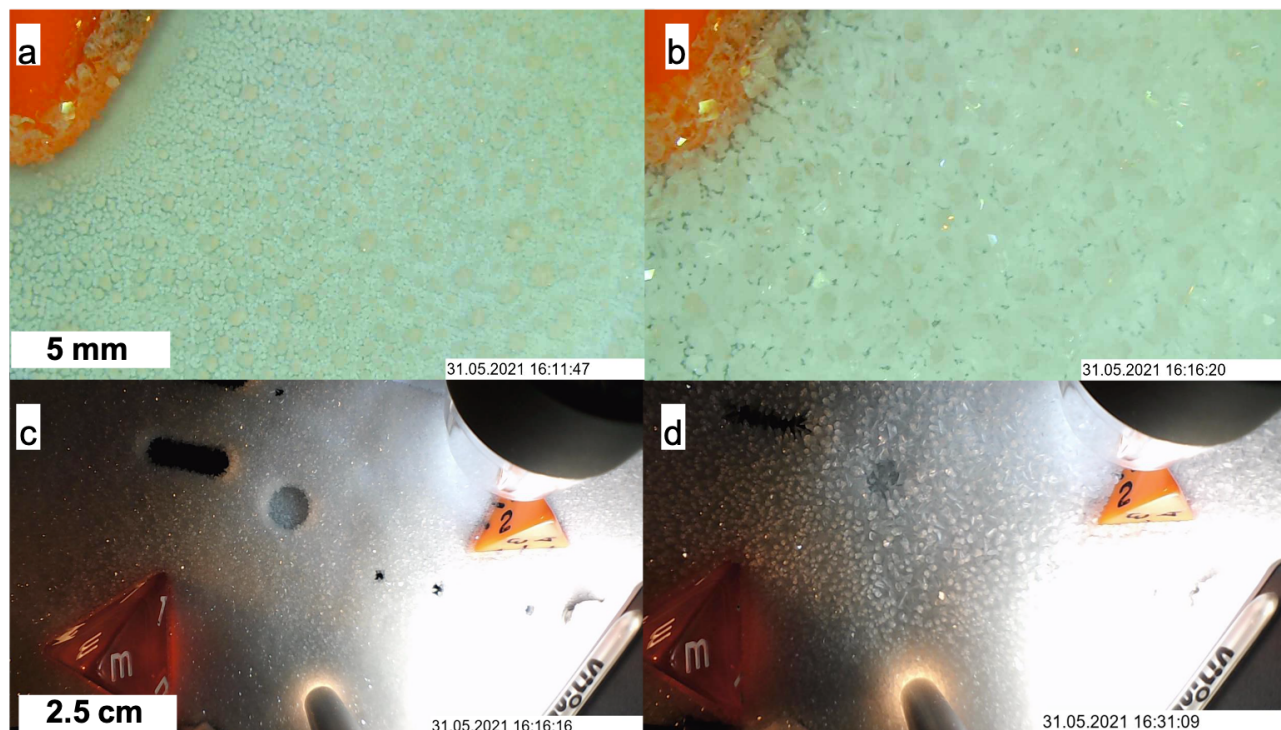


Figure 3.6: Later stages of "blanket" frost transitioning into angular prismatic crystals (a, b microscope images, c and d images from web camera). Each image pair is taken about five minutes apart.

Later during the ice deposition grains continue to grow larger and become visible even with smaller magnification (3.6 a, b). At first, the grains are more rounded, but with time grow more angular and similar to the triangular and rectangular prisms or frost described above, both on small and large scale (Figure 3.6).

Figure 3.7 shows an overview of the average pressure and minimum temperature recorded during the experiments for each observed texture. We can not make a clear correlation of the textures with pressure within our tested range (1 to 23 mbar). Blanket frost might appear to form at pressures below 5 mbar, however, both red points on the graph representing this texture are from one experiment and only show two different temperatures on the different ends of the cold plate. More experiments are needed to confirm whether this texture is associated with lower pressure.

In terms of temperature, slab ice and frost appear to form under distinct regimes. The boundary between the two textures lies between 130 and 140 K, with slab ice forming at

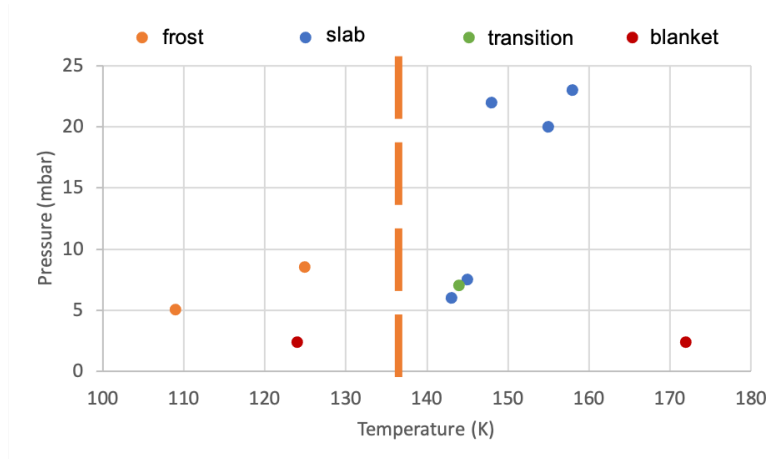


Figure 3.7: Temperatures and pressures at the start of deposition for three different CO₂ ice textures. "Transition" represents a case with slab-like crystals at the start and frost formation at the end of the experiment as temperature dropped.

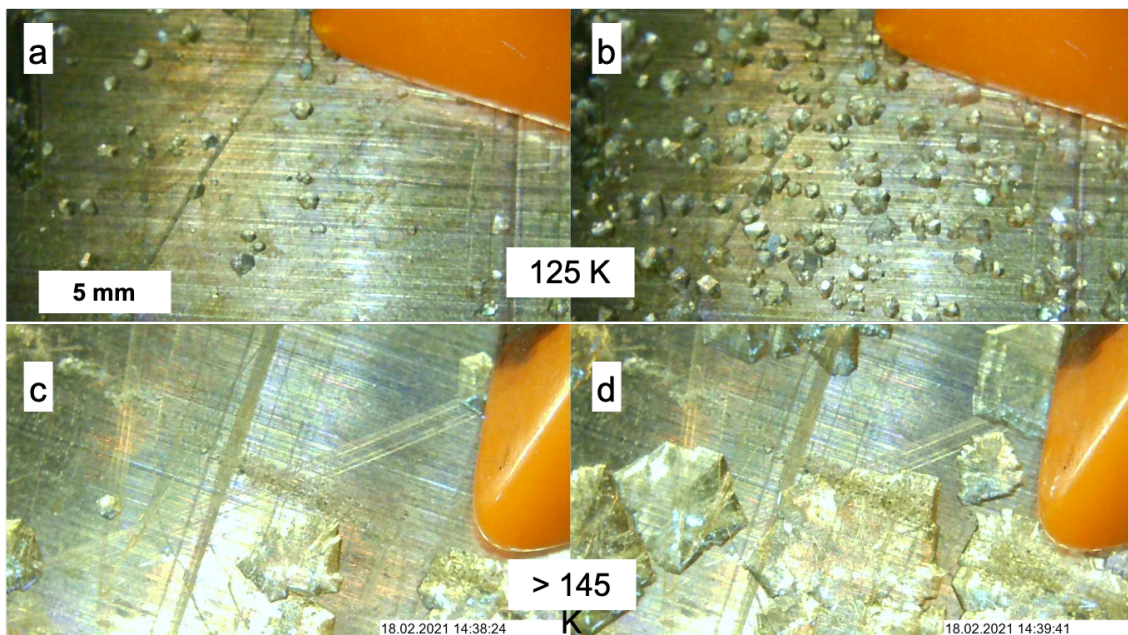


Figure 3.8: Nucleation for CO₂ frost (a and b) and slab ice (c and d). Each image pair is taken 1-2 minutes apart.

temperatures warmer than 140 K and frost forming below 130 K. Temperatures at the south pole of Mars can reach as low as 135 K and experiments in this narrower temperature zone may be useful to identify if forming the finer-grained frost by direct deposition is likely to occur on Mars.

There are also differences in the number and frequency of nucleation sites between different textures. Usually in experiments when slab ice was observed we note fewer and larger crystals, and no new nucleation sites appear after the initial few crystals form. In experiments where the final texture was of CO₂ "frost" we note more frequent and numerous nucleation sites, which continue to form in large numbers until the entire sample plate is covered by ice (Figure 3.8).

3.1.1 Sintering of CO₂ frost

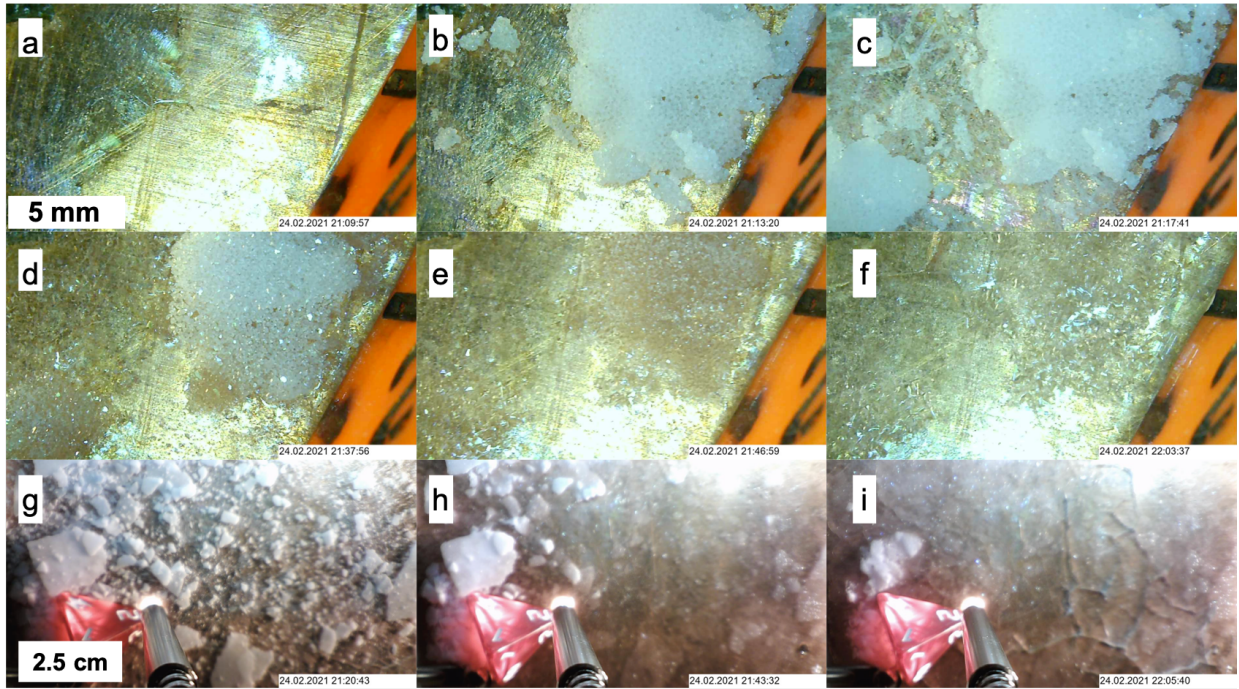


Figure 3.9: Sequence of microscope images showing CO₂ frost sintering into an underlying transparent slab layer (a to f). Images from web camera showing the same experiment (g, h, i).

During one of the experiments, CO₂ frost deposited onto the cold shroud, and grew thick enough to detach and fall onto the sample plate. A thin layer of transparent CO₂ ice had already grown on the sample plate, and the fallen frost was incorporated into the slab by sintering (Figure 3.9). A fraction of the frost likely sublimated before it could sinter into the slab since the cooling was paused for about 5 minutes and temperatures in the chamber

were rising. However, sintering of a large amount of the frost can be seen on the images. Some patches of finer grained areas remained visible after the frost was incorporated into the polycrystalline layer (Figure 3.9 g, h, i).

3.2 NIR reflectance spectra of CO₂ ice

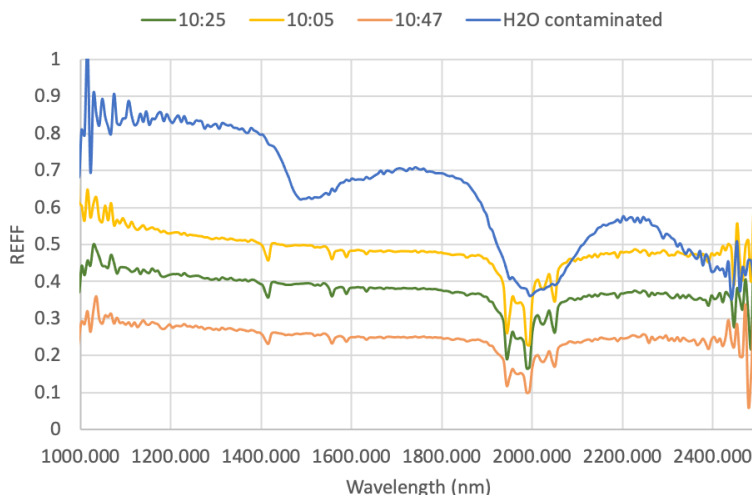


Figure 3.10: Sample spectra recorded for CO₂ slab ice at shown times. Blue line shows spectral measurement with chamber door open, with strong water absorption bands. Reflectance values of these spectra are not comparable to the pure CO₂ spectra plotted, as they were taken during separate experiments with different conditions.

We have measured reflectance spectra for the different textures described above, and report them in this section. Figure 3.10 shows an example of spectra recorded during an experiment when transparent CO₂ ice was deposited. All of our measured spectra show typical CO₂ ice absorption bands at 1400 nm, 1600 nm and 2000 nm. Weaker bands around 2200 nm were not observed, likely due to reduced transmission of our fiber optic components in this range. There are no broad absorption features around 1500 nm and 2000 nm characteristic of water ice, which confirms that the CO₂ ice is not contaminated with water ice. For comparison, an example spectra (shown in blue) was taken when the chamber was open and moisture from the air could condense on the previously deposited CO₂ ice.

It is clear that H_2O absorption bands in this case are dominating and dampening the CO_2 bands.

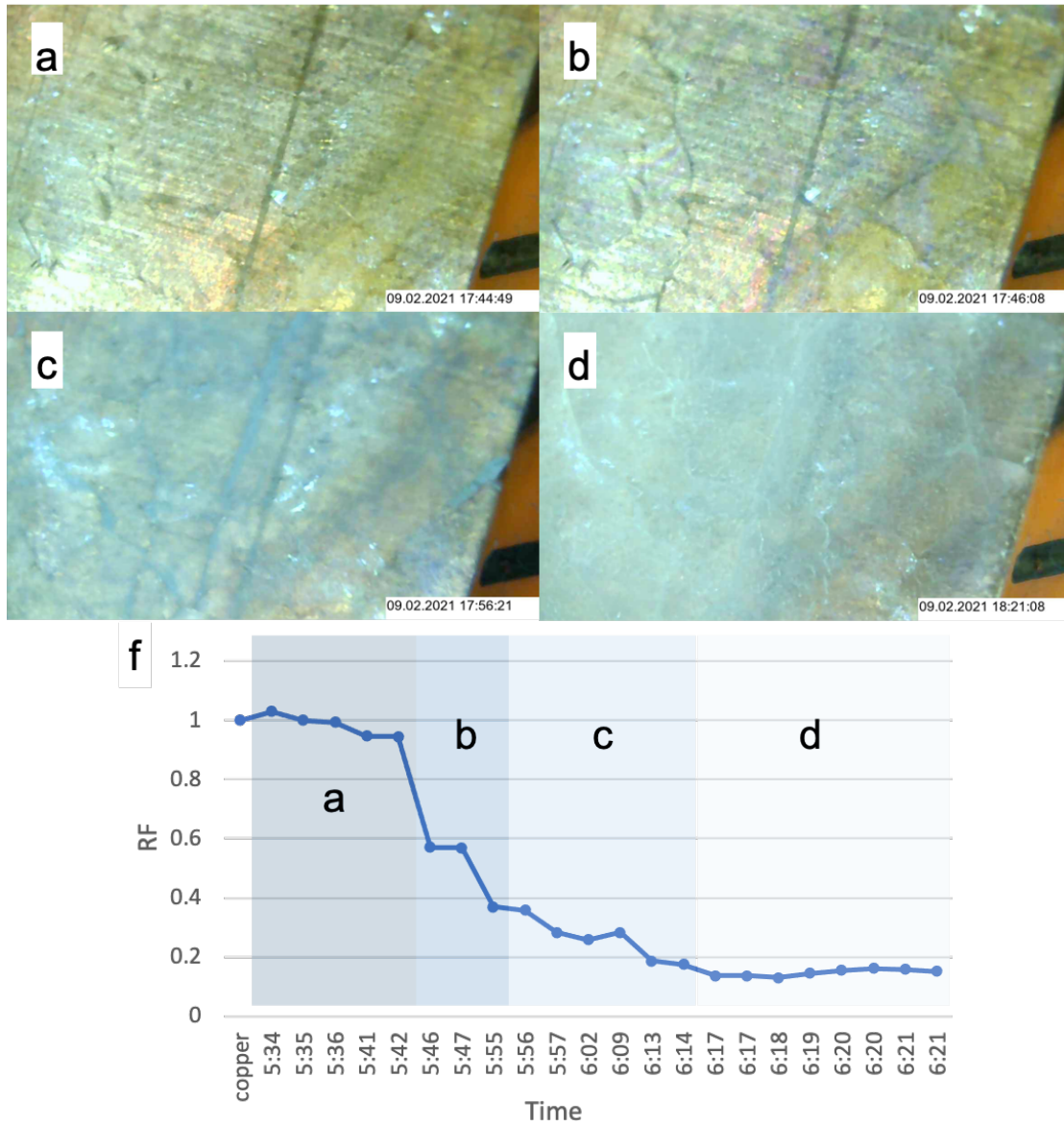


Figure 3.11: Reflectance factor of slab ice at 1700 nm plotted over time the measurement was taken (f). Highlighted boxes outline four stages in the changes of reflectance. Panels a to d are example microscope images for each of the four stages marked respectively.

To report reflectance spectra we use the reflectance factor (RF) introduced in Chapter 1, and we use the two terms interchangeably. Observing the changes in reflectance throughout the deposition of the ice over the entire wavelength range quickly becomes complicated,

since a single experiment usually involves more than twenty measurements. For simplicity we use the reflectance factor at 1700 nm, a point of the spectral range free of CO₂ absorption features and relatively unaffected by noise.

A pure slab of CO₂ ice deposited on the copper sample plate had high RF values between 1 and 0.8. As the ice grew thicker and fractured, the reflectance dropped significantly. Figure 3.11 shows four stages in the development of the slab in terms of its measured reflectance. Stage a shows the high RF (>0.9) of the unfractured slab, stage b shows minor fracturing of the ice with RF falling to ~ 0.4 . Fractures become more prominent in stage c, the ice becomes noticeably less translucent and RF drops to 0.3-0.25. In the final stage d, the ice has visibly lost transparency and shows RF values as low as 0.13. This trend where more transparent ice appears more reflective than the fractured slab is the opposite of what would be expected. Visually and from relative IR albedos that will be described in Section 2.4, fractured ice should appear more reflective than the transparent slab. Our recorded values result from the very high reflectance of the copper plate where the ice is deposited. The transparent layer of ice allows more light from the copper plate reach the detector and we record high RF values. When the ice cracks, the fractures significantly reduce its transparency, blocking the reflections from the metal plate, and we measure low RF. This trend is the same in all experiments as the ice in all of them is grown on highly reflective metal plates.

The development of reflectance values for the fine-grained CO₂ frost texture grown on an aluminium plate are similarly shown in Figure 3.12. In this experiment no slab ice deposited and the first measured spectra were of the "blanketing" fine grained ice. The RF values are low (~ 0.2) from the beginning of ice growth in stage a. The blanket frost significantly reduces the reflectance from the aluminium plate right from the start of deposition. The reflectance continues to decrease as larger crystals develop, and the ice grows into a thick layer of frost made up of prismatic crystals. In stage d, the RF drops to as low as 0.06.

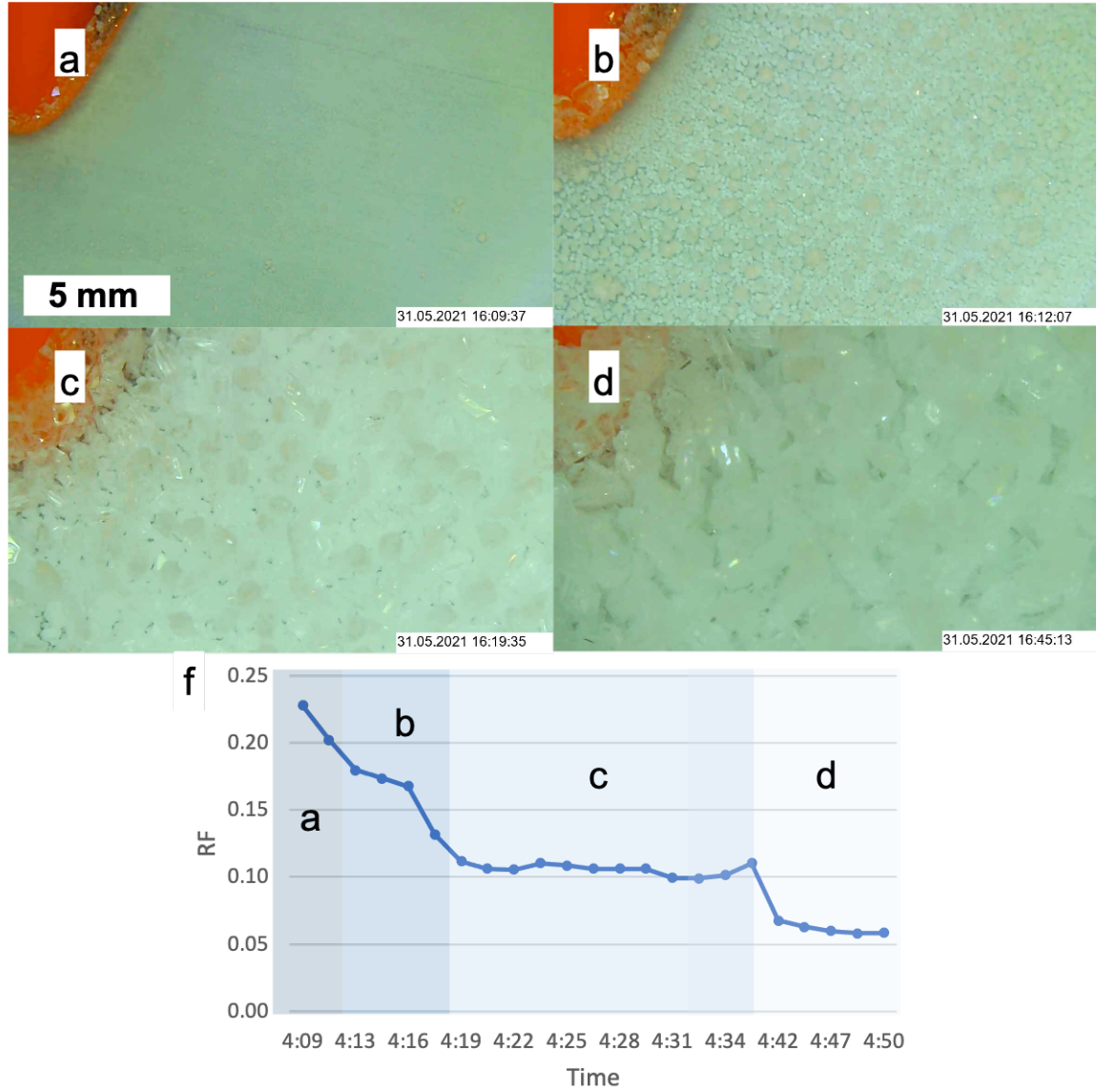


Figure 3.12: Reflectance factor of frost at 1700 nm plotted over time the measurement was taken (f). Highlighted boxes outline four stages in the changes of reflectance. Panels a to d are example microscope images for each of the four stages marked respectively.

3.3 Fracturing due to thermal cycling and spectral response

Fractures occurred during every experiment when transparent CO₂ ice formed. During the experiments with "cold sky" conditions we usually observe a fully transparent slab ice

for the first 10 to 20 minutes of deposition, whereas in some of the "warm sky" conditions the ice appeared less clear from the start. After reaching several millimeters in thickness, the slab ice started fracturing including the cases when the shroud was cooled. Fractures continued forming as the cold plate temperature continued falling. They tended to be up to 2-3 centimeters in length and can be seen on web camera images (Figure 3.13). At first, most of the fractures "heal" within seconds, while new ones form and heal again. The healing of the fractures is driven by sintering processes described in Section 1.2.3. After about 40 minutes of deposition, healing stops and the ice surface is covered in fractures that significantly reduce the transparency of the ice.

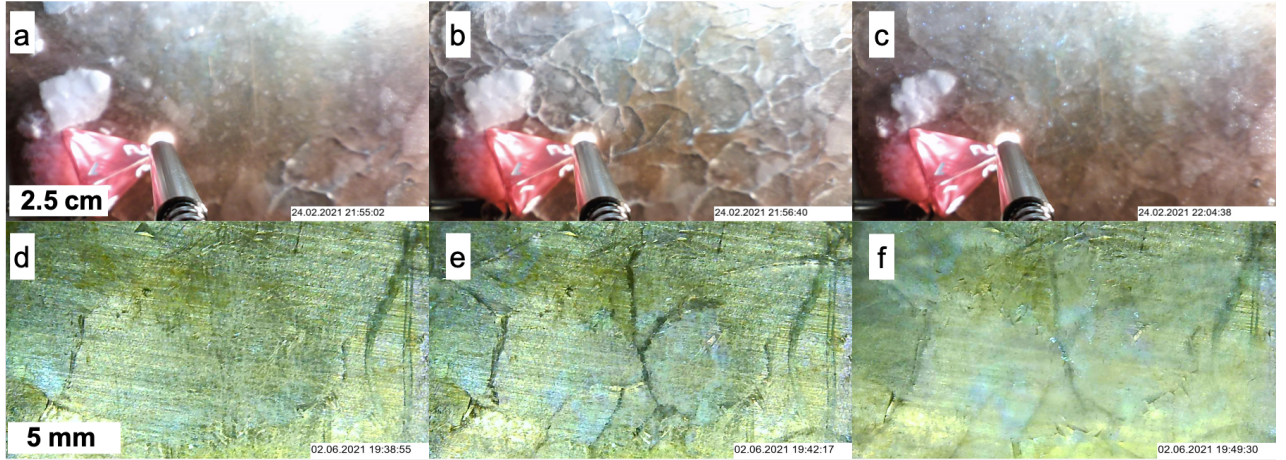


Figure 3.13: Fractures forming within slab ice and "healing" within minutes. Time interval between images is 1-10 minutes.

Another type of fracturing we observed was induced by a rapid warming of the sample plate using the electric heater. We conducted two experiments when the sample plate was rapidly heated by 5 to 7 K. Our goal was to simulate the early spring conditions on the polar caps when the sun rises above the horizon and warms the ice. Images from one of them are shown in Figure 3.14. Panels a and d show the transparent ice before the warming, the temperature at this time was 165 K. Panels b and e show intense fracturing of the ice, and the fractures here are much smaller and denser than the regular fractures described above. Temperature at this time was only one degree higher, 166 K and continued rising up to 169 K. The heater was then switched off and temperature of the plate was allowed to cool back to 165 K, when the fine fractures fully "heal" and the ice regained its former transparency.

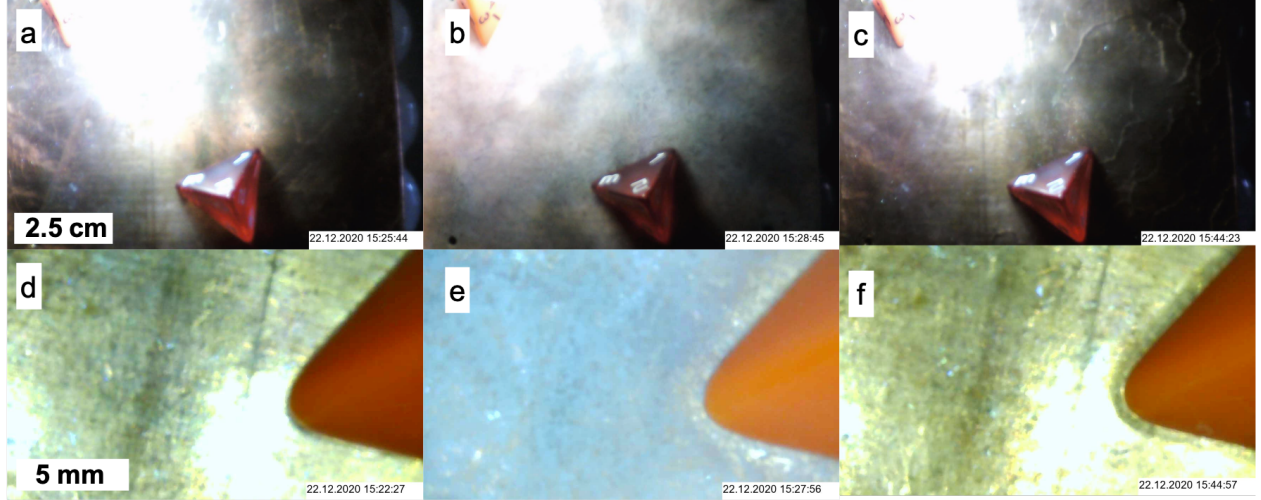


Figure 3.14: Sequence showing fine fractures forming due to heating of the sample plate on large and small scale (a to b and d to e). Fractures later heal fully due to substrate temperature dropping back to original values (b to c and e to f).

Pressure during this experiment was around 26 mbar.

The second "warming" experiment was conducted in order to investigate the effects of this fracturing on the reflectance of the ice. This time, larger fractures had already been present before the rapid warming of the sample plate (Figure 3.15 panels c and f). However, the warming created additional finer fractures and exaggerated the pre-existing ones (panels d and g). Panel g also shows a ruptured fracture that exposed the sample plate underneath. The temperature during this time jumped from 157 K to 164 K (panel b highlighted by a blue box), after which the heater was turned off. We were not able to decrease the temperature back to the original value due to a pressure drop in the LN₂ tank, which could not provide the required LN₂ flow for rapid cooling. Most of the large fractures healed after the heater was off, the rupture in the ice was gradually covered by ice, and the ice surface turned smooth, returning some of the transparency. This entire process is reflected in the RF values shown in panel a. The initial partly fractured ice had RF values close to 0.35; warming induced fractures caused a drop in reflectance, which dropped to 0.2 (highlighted in blue). At the latest stage the healing of the fractures caused the RF to increase to 0.3 (highlighted in orange).

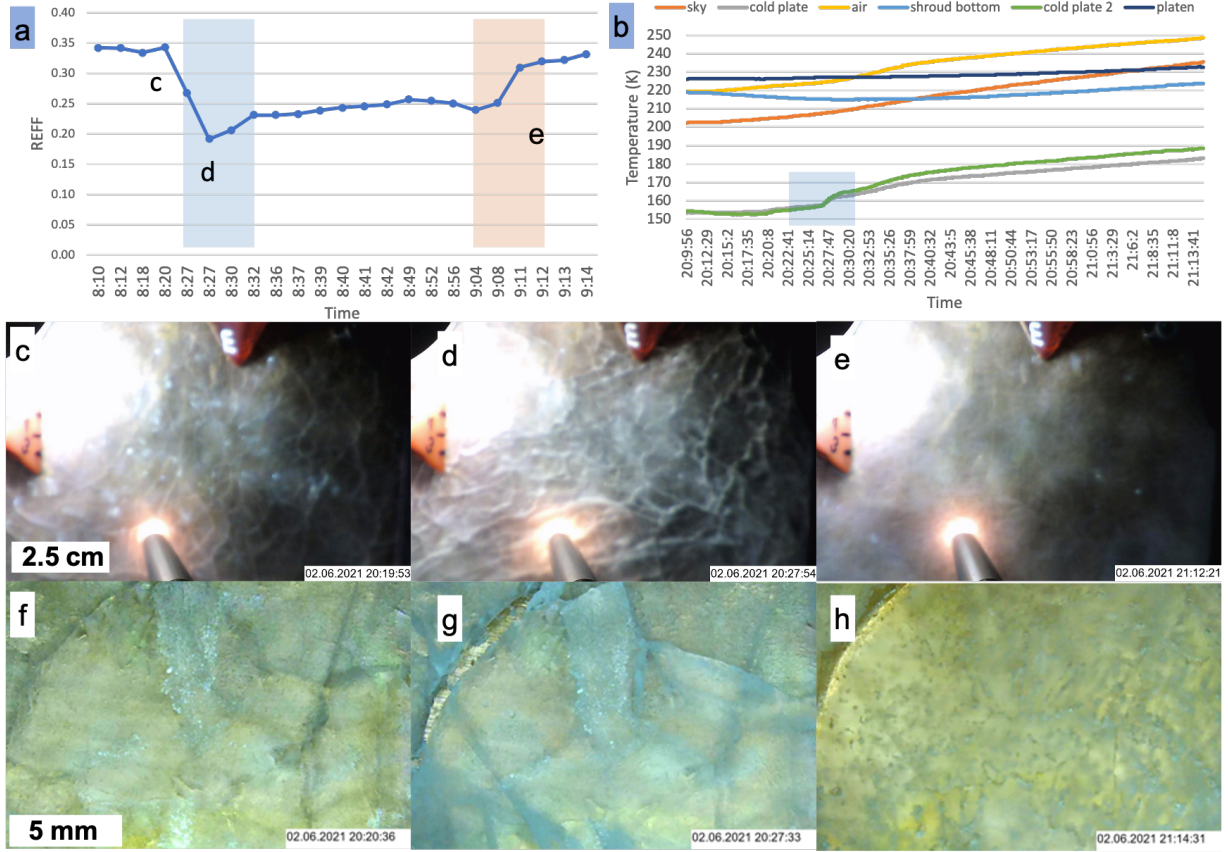


Figure 3.15: a) RF of slab ice undergoing fracturing over time. Blue box shows reflectance drop due to fracturing, orange box - recovering reflectance as fractures heal; b) temperature of the different parts in the vacuum chamber during the fracturing experiment. Blue box shows temperature jump caused by heater; c, d, e, f, g, h - images of fractures forming and healing due to thermal cycling.

3.4 Relative albedo measurements of CO₂ ice from infrared images

Relative albedos for different textures have been calculated from the value component of images taken by an infrared camera. Figure 3.16 shows the images used for calculations, with red squares outlining the areas within the ice samples where brightness data was extracted from. We compare the albedo of transparent polycrystalline ice and heavily fractured slab ice deposited on copper substrate during the same experiment (panels a and b). Fractures in this case increased the albedo of the ice from 0.3 to 0.42. The sampled area is a mix of

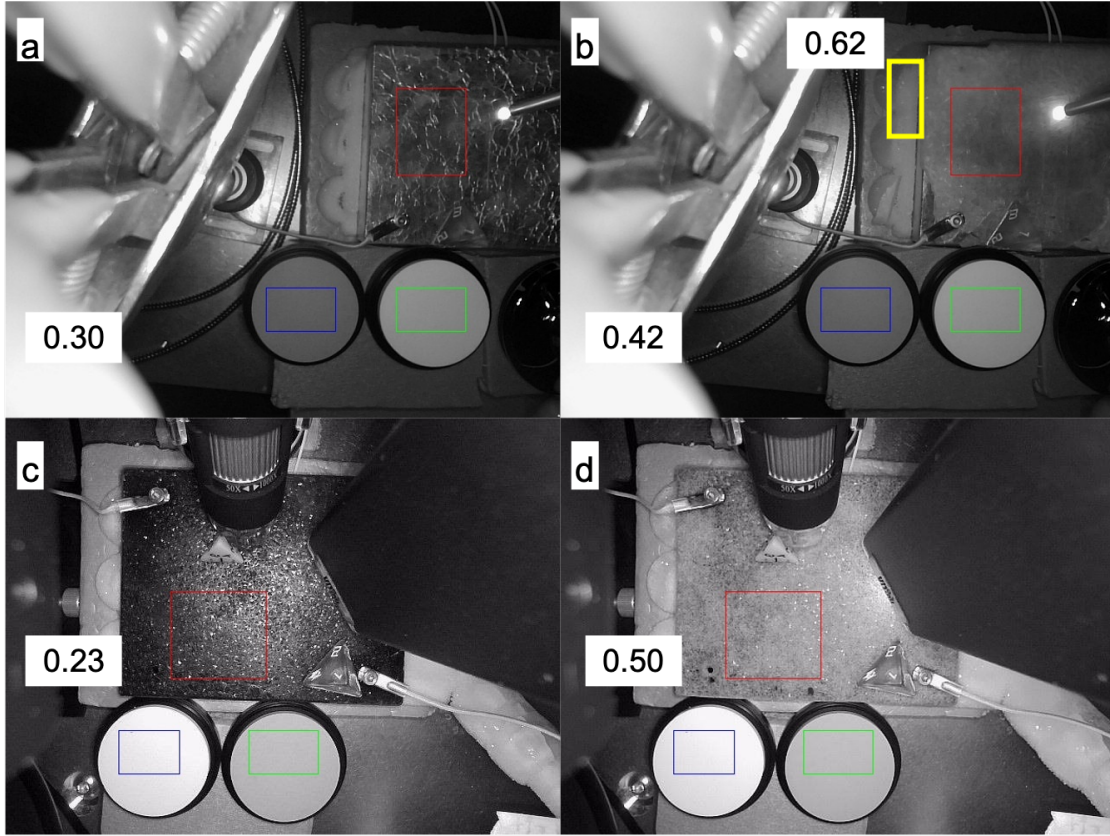


Figure 3.16: IR images of: a) CO_2 slab and b) fractured slab on copper substrate; c) CO_2 slab and d) fractured slab on a substrate painted black. Red rectangles show areas sampled to extract brightness values. Blue and green rectangles are sampled areas on the reference standards (99% and 40%). Albedo calculated for each image is shown on the left. Yellow square in panel b shows an area of fine-grained frost deposition together with its albedo. The same reflectance standards in images a and b appear darker than in c and d, which may be due to lighting conditions between the two experiments and due to difference in image stretch.

darker, less fractured ice and brighter, more fractured surface. Panels c and d show a similar experiment, but the ice was deposited on a copper plate that was painted black. The albedo of the transparent ice grown on the painted plate (0.23) is lower than the albedo of the ice grown on copper (0.30). When this same slab fractured its albedo increased to 0.5. This is higher than the albedo of fractured ice on Figure 3.16 b, however the ice in this case appears more fractured and brighter in the sampled area.

The yellow rectangle on Figure 3.16b outlines the frost deposited onto copper pipes

that circulate LN_2 to the cold plate. This frost is similar to the prismatic frost we see at temperatures below 130 K. It deposits on the copper pipes during every experiment since the pipes always reach below 130 K. The albedo of this frost (0.62) was higher than for any other texture.

All relative albedo values follow the expected trend: albedo is higher for finer-grained and fractured textures, which have more crystal boundaries scattering light compared to polycrystalline ice. Notably, the albedo of the substrate played a role in the measured reflectance of the transparent ice deposited on it. Even without the specular reflection component that skewed our spectral reflectance measurements, the copper sample plate makes the ice grown on it appear slightly brighter than the ice deposited on the painted dark substrate. We are currently working on a method to directly compare the results of reflectance measured by the near-IR spectrometer and relative albedo extraction from the IR camera. The influence of different substrates on both measurements will also be investigated.

Discussion

4.1 Experimental setup: advantages and limitations

The experimental setup, its capabilities, potential use in further experiments, as well as future improvements are described in the methods chapter. Here we highlight some of its advantages and limitations. One of the main advantages is the size of the vacuum chamber: it can fit many different instruments which can access the sample directly, without using any windows. We have used two different microscopes and two cameras simultaneously to record the deposition of CO₂ ice at different scales. This would not be possible with smaller sample plates and vacuum chambers. We plan on placing a small goniometer inside the chamber for measuring the bidirectional reflectance distribution function (BRDF) as one of the future experiments. The length of the platen where we place the instruments (about 90 cm), as well as the space above the sample plate make it possible. Accessing the sample this way provides an opportunity to illuminate the sample and detect the reflectance at very low grazing angles. The area of the sample plate is also advantageous since it provides a large enough surface for a statistically significant number of crystals to grow.

There are cases when the large volume of the chamber might become a disadvantage: long duration experiments require larger amounts of liquid nitrogen for cooling. The large number of ports and components also increases the likelihood of leaks and contamination of the CO₂ atmosphere in the chamber. However, these are closely monitored for and can be promptly fixed. So far we have conducted experiments that lasted several hours (up to 5-6 hours from start of evacuating and cooling), which might have somewhat faster rates of ice

deposition than on the polar caps of Mars. However, these shorter experiments allowed us to vary the conditions and try many configurations in each experimental run.

Another feature that sets apart this setup from other systems in the world is the cooling shroud and platen system. None of the previous experiments had a way to cool the atmosphere in the vacuum chamber and recreate a "cold sky", an essential step in simulating a Martian environment. The shroud and platen are essential in reducing the thermal gradient within the ice, and will become even more important for long duration experiments, lasting longer than two weeks that will require thick layers of ice and a very cold atmosphere. This feature of our setup is unique in the planetary research community and can help advance experimental work in the field of planetary ices.

Future experimenters can certainly use the unique features of our setup to their advantage, and conduct experiments that would not be possible with smaller systems, or systems without the "cold-sky" conditions.

4.2 CO₂ ice textures and their relevance for Mars

The modes of deposition for the seasonal CO₂ ice on Mars are not fully understood. Direct deposition of slab ice and sintering of fine-grained snow into polycrystalline deposits are considered to contribute to the formation of the south seasonal cap (Hayne et al., 2012; Kieffer, 2003). During our experiments, we observed the direct deposition of CO₂ ice, and one instance of fine-grained frost getting incorporated into a pre-existing slab by sintering. Both processes resulted in similar textures, therefore, both direct deposition and sintering can form the polycrystalline ice in the winter on Martian south pole. This agrees with the findings of Grisolle et al. (2014) and Philippe et al. (2015), who also observed both types of slab formation. Microphysical modeling by Eluszkiewicz et al. (2005) mainly considers the sintering mode of slab formation, in order to explain the lower densities of the seasonal deposits than what would be expected if direct deposition occurred throughout the entire ice cap. However, direct deposition still accounts for the formation of the larger share of the seasonal cap, and our simulation of the deposition in the laboratory is well representative of how CO₂ ice is forming at least in some parts of the seasonal cap.

During our direct deposition experiments, transparent slab was the most consistently formed texture of CO₂ ice when the pressure and temperature conditions were close to those found on the south pole of Mars in fall and winter.

We also observed reflective frost texture consisting of prismatic crystals in temperatures below 130 K. The crystalline structure of this frost might be unexpected, since CO₂ ice is known to have a cubic crystal structure (Mangan et al., 2017). We can speculate that the elongated rectangular and triangular prisms are the collections of stacked crystals that are similar to hoar frost formed by water ice. Imaging with a scanning electron microscope would be necessary to confirm this, and would be out of scope of our study. This texture is likely not applicable to present-day Martian poles as it formed when the sample plate was cooled below Martian temperatures and below the CO₂ condensation temperature. However, during the periods of lower obliquity, when the atmospheric pressure is lower, the condensation temperature for CO₂ can approach 130 K. Therefore, this texture might be relevant for other periods of Martian climatic history.

The "blanketing" frost consisting of even smaller crystals was observed during our earlier experiments when the data logging capability was not added, and we have only one case of blanket frost when the data was recorded. It formed at a lower pressure (2.5 mbar) than what we recorded for slab and prism-like frost, and the temperature of the substrate varied between 124 K and 172 K depending on the location on the cold plate. It is hard to infer the exact cause of this type of texture from the available information. We saw this texture when we grew the ice directly on the aluminium cold plate and without shroud cooling, which is why the temperature had such a wide range. Usually, the copper plate on top of the cold plate allows for the temperature to equilibrate and thermal inhomogeneities within the plate do not exceed 2 K. This observation of the different textures might also be dependent on the substrate. The uniformity of temperature, and different microscopic roughness of the two substrates might be affecting the frequency of nucleation sites and the different crystal sizes. The fact that on the microscope images the blanket frost crystals appear aligned with the grooves of the aluminium plate, suggests that the substrate might play a role in the different textures at least in the laboratory setting. This also implies that in order to fully simulate a Martian surface, a Mars regolith analogue needs to be used as a substrate. Our

spectroscopic measurements of ice on such substrate will also be more accurate as shown in the previous chapter, since we can avoid the influence of the high reflectance of the metal substrate on our measurements.

4.3 Reflectance spectra and influence of fractures

We reported reflectance spectra of the CO₂ ice which deposited onto a copper sample plate or onto an aluminium cold plate. Reflectance values are heavily influenced by the very reflective metal substrates and complicate our interpretation and comparison to orbital data. We are measuring reflectance at zero phase angle, where metal's specular reflectance is very strong. Compared to this extremely strong signal, a much more diffuse reflectance of ice and frost is comparatively low. Corrections using spectralon reflectance standards increased the overall reflectance values for all experiments; however, we could not find a meaningful way to fully eliminate the influence of the metal substrate. Even after corrections, increased fracturing still tends to reduce the spectral reflectance we measure during the experiment. It is expected that fractures should increase the overall reflectance or the hemispherical albedo of the surface because they provide more interfaces for the scattering of light. In our setup, the fractures decreased the specular component of the copper plate's reflectance because the surface covered by fractured ice became more diffusely reflecting, meaning the light is scattered in many directions instead of one predominant specular direction. Therefore, the amount of light reaching the detector in our zero phase geometry that picks up mostly specular reflectance, was significantly reduced when we measured more diffusely scattering textures, such as fractured slab and fine-grained frost.

We cannot meaningfully compare the reflectance values of the different textures for the same reason of metallic substrate reflectance. One way to counteract this effect would be to use a substrate with a low albedo, close to the albedo of ice-free surface on Mars which is close to 0.20 (Kieffer et al., 2000). This would not only eliminate the problem of metallic reflectance, but also simulate the Martian environment, where transparent slab ice would appear dark due to the underlying dark material. Our experiments are ongoing and we plan to measure the reflectance on a dark substrate and compare to the remotely sensed

data. These observations also show that in addition to the substrate, the geometry of our measurements plays an important role. Varying the angles of the incoming light and the detector to measure the bidirectional reflectance distribution function (BRDF) would help us better understand the scattering properties of the different textures of CO₂ ice. It would also make it possible to directly compare remotely sensed data to our laboratory observations.

4.4 Relative albedo in near-infrared

The relative albedos we calculated from the images taken with the IR camera varied depending on the substrate albedo, the intensity of fracturing and the type of texture. In this case, the albedo we calculated from the brightness values of the images followed the opposite pattern to the reflectance measured by the spectrometer probe. We record higher albedos for fractured ice compared to the slab ice. The ice that was visibly more intensely fractured also had a higher albedo. The fine-grained frost had an even higher albedo than fractured ice.

The specular reflectance of the substrate that had a strong impact on the reflectance measured by the spectrometer as discussed above, did not significantly influence the brightness recorded by the IR camera. Although the IR camera collects data from a nadir direction as well, the larger lens (compared to the reflectance probe detector) and light source are not perfectly aligned at zero phase angle, but are only close to it. The camera also images at about 10 times larger distance away from the surface than the reflectance probe. Since the intensity of light falls off with the square of the distance from the source, this large distance of the camera from the ice surface together with the nonzero phase angle greatly reduced the effects of specular reflectance on the IR camera images. At the same time, sampling a much larger area of the ice (about 4x4 cm vs. < 1x1 cm) when we extract brightness values increased the contribution of the diffusely scattered light that we measure.

Albedos that we measured for slab ice (0.30 and 0.23) were in the similar range to the albedo of the clean CO₂ slab ice on the south seasonal cap in winter (0.23) as reported by Langevin et al. (2007). Their reported albedo values are measured at 1.08 μ m, which is within our IR camera's wavelength range, however, the phase angle during their data

acquisition was 50 to 70°, which is not comparable to our measurement geometry. We cannot directly compare our calculated values to the reported albedos measured by the OMEGA spectrometer (and other remote sensing instruments). However, we can consider whether our laboratory measured values are roughly in the similar range. All compared values from OMEGA measurements and our calculations are shown in Table 4.1.

Region	Ls	1.08 μm albedo	Texture type	Albedo (copper)	Albedo (paint)
Clear slab	142	0.23	Clear slab	0.3	0.23
Cryptic terrain	223	0.25	-	-	-
Bright seasonal cap	261	0.68	Fractured slab	0.42	0.5
Residual cap	273	0.79	Fine grained frost	0.63	-

Table 4.1: 1.08 μm albedo of different regions on the south pole from OMEGA imaging spectrometer (Langevin et al., 2007) and relative albedos for different textures (on two different substrates: copper and painted copper) created in our laboratory experiments.

The albedo of the Cryptic region is at 0.25, also close to the albedo of our lab-grown slab ice. Our measured albedos for fractured ice (0.42 and 0.50) are however significantly lower than the albedos of the bright regions of the seasonal cap (0.7) and the residual cap (0.8). The values we have calculated might appear lower due to the insufficient thickness of the ice layer. The seasonal ice on Mars is about 1 m thick. It will have more internal reflections that increase the overall reflectance when the ice fractures compared to the <5 mm-thick ice that we grow in the laboratory. Further experiments with thicker samples would be useful in identifying a minimum thickness for a laboratory sample that will have reflectance properties comparable to the Martian CO₂ ice.

The relative albedos we have calculated from IR images have also not been sufficiently tested for consistent performance. Small variations in the lighting conditions might be affecting our measurements non-linearly, and further corrections might be necessary. In future experiments, we will compare the relative albedo values obtained with this method with the reflectance spectra (free of specular effects) measured with the reflectance probe. This will help us test the reliability of our albedo calculations from IR images.

4.5 Rupturing of slab ice during thermal cycling

We have used our capability to rapidly increase the temperature of the substrate using the heater in order to simulate rapid warming that occurs on the substrate from insolation through the translucent ice during early spring. The experiment was repeated twice and in both cases, temperature increase by 1-2 K caused intense fractures, which visibly brighten the slab ice. In one case, large fractures that form due to the thermal gradient were present before the heating started. They were "weak spots" within the ice layer, and when rapid warming started the ice broke apart or "ruptured" along one of the weak spots, exposing the underlying substrate. We believe this is a process analogous to the geyser-like eruptions of CO₂ gas that form spots, fans, and spiders on the Cryptic terrain. We explain the Kieffer model for the formation of these features in Chapter 1. In the model, the ground below the ice is heated by solar radiation that penetrates through the CO₂ ice layer that is transparent in the visible range. The heat is trapped, and the ice sublimates from the bottom where it is in contact with the warm ground. The impermeable slab traps all the sublimed CO₂ gas, the gas travels along channels and builds up to the point when it breaks through the overlying ice bringing along some sediments to the surface.

In our experiment, we simulate the basal sublimation step of this process, when the ice sublimates from below due to the direct heating of the substrate, and breaks the ice along a weak point. Modeling efforts have shown that this process is possible when considering the heat available on the south pole late in the winter and early spring, as well as the strength of the CO₂ ice (Portyankina et al., 2010). Simulating this part of the Kieffer model in the lab is another important step in proving this models feasibility. In our experiment the rupturing occurred within about three minutes when the substrate warmed by 4-7 K. The thickness of the ice was less than 5 mm. In other experiments when pre-existing fractures were absent, breaking of the ice did not occur. The weak spot factor is likely more important in the laboratory, where the ice is grown on a flat smooth surface. In the natural terrain with its small-scale topography, weak points are likely to be very common. We plan on reproducing this experiment and constraining some more of the quantitative values regarding the required temperature increase and ice thickness.

Conclusions

The polar ice deposits of Mars are valuable resources for studying the past and present climate of the planet. While deposits of water ice are important in the context of future human exploration, the seasonal deposits of CO₂ ice play an important role in understanding the current climate of Mars. The weather of the planet is driven by the CO₂ cycle, which also influences the water and dust cycles. Therefore, it is important to understand the processes involved in the formation and retreat of the seasonal CO₂ ice caps. The properties of the CO₂ ice are responsible for many of these processes, Studying the CO₂ ice under Martian polar conditions in the laboratory in addition to remote sensing data is necessary to better understand the seasonal ice caps.

We created an experimental setup to expand and continue previous laboratory experiments on CO₂ ice under simulated Martian environment. The setup has some advantages over the earlier systems created by Brissaud et al. (2004) and Pommerol et al. (2015). We can recreate Martian temperatures, grow larger samples of ice, and have direct access to the sample from inside the chamber. Extensive testing of the system was conducted, and many of the previously reported observations were reproduced. We deposited transparent slab ice at Martian polar conditions similarly to Grisolle et al. (2014) and Portyankina et al. (2019), as well as fine grained frosts at colder temperatures. We noted one case of sintering of fine-grained frost that fell onto a sample of CO₂ slab and got incorporated into the transparent ice. Both of these modes of slab formation are expected to occur on the southern seasonal cap of Mars.

Fracturing due to a thermal gradient in the sample was recorded, as well as "healing" of these fractures within seconds. We additionally created fine fractures by rapid heating of the

substrate by 1 to 7 K, and observed the ice recovering full transparency when the cooling resumed. We note one instance of rupturing of the ice at a pre-existing large fracture due to rapid substrate warming. We consider this as an analogue process to the formation of jets on the Cryptic Terrain of the south seasonal cap.

We measured the reflectance of the ice in the near-IR at nadir with zero phase angle throughout its evolution. Our measurements show decreasing reflectance values over time as the ice grows thicker and more fractures appear. More fractures on the slab usually increase the amount of light scattered from the sample, thereby reducing the specular component. Our observations were affected by the high specular reflectance of the copper substrate. Experiments where ice is grown on a darker substrate with a more diffuse reflectance, potentially a Mars soil analogue, will be conducted to correct for this effect and better approximate the surface of Mars.

In order to acquire additional quantitative data on the albedo of the different CO₂ ice textures and the effects of fracturing on albedo, we acquired images of the ice using an infrared camera. We calculated the albedos from the brightness values of an HSV image relative to diffuse reflectance standards. Our values for slab ice, especially when it was grown on a dark substrate, were similar to the albedo of pure CO₂ slab on the south seasonal cap in winter. The albedos we measured for the fractured slab were significantly lower than the bright parts of the seasonal cap and the residual cap. Future experiments will be conducted in order to investigate the effects of sample thickness on the brightening due to fractures.

Future work will include measuring reflectance values of the different CO₂ ice textures grown on a low-albedo substrate, as well as measuring the reflectance at different illumination and detector geometries. We note some nucleation site differences when different substrates are used. Therefore, experiments with depositing CO₂ ice on Martian regolith analogue would be desirable. We would also like to constrain the conditions for creating ruptures of slab ice by the CO₂ gas pressure from the bottom of the sample. This process has not been reliably recreated in the laboratory, and learning more about it could help validate and constrain models that explain the formation of jets and fans on the Cryptic Terrain.

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Appendix

Date	Shroud On	Texture (starting)	Texture (final)	Fractures	Comments	Freeze start temperature (K)	Freeze start pressure (mbar)	Lowest Temperature (K)	Pressure range
2020-09-28	no	small (~1mm) transparent crystals form at frequent nucleation sites	turns cloudy before slab can form, prismatic crystals develop	no	thick frost layer, grown on Al cold plate	203	2.6	137*	10 to 13*
2020-10-19	yes	identical to 2020-09-28	identical to 2020-09-28	no	thick frost layer, grown on Al cold plate	173	29	140*	2.6-29*
2020-10-21	yes	identical to 2020-09-28	identical to 2020-09-28	no	thick frost layer, grown on Al cold plate	195	6	126*	4.6-6.6*
2020-10-22	yes	blanket frost	prismatic frost	no	some sintering by or sublimation recorded; crystals turning more spherical	176	8	122*	5.3-9.3*
2020-10-26		blanket frost	more rounded crystals to prismatic frost	no	sintering at the end similar to 2020-10-22	143	1.3	111*	1.3-8*
2020-11-03	no	medium sized (3-4mm), "dendritic" crystals, not as transparent, not polycrystalline slab	smaller than original, columnar, prismatic crystals	no	after crystals cover the plate small crystal-size fractures gradually cover the plate, all sample turns white; later grainy "rice-like" crystals are visible	163	7	125*	8-9.3*
2020-11-05	yes	small (~1mm) transparent crystals form at frequent nucleation sites, no polycrystalline layer	small crystals continue to grow vertically, forming columnar crystals	no	thinner layer of frost, can see copper plate between prismatic crystals	140.5	4.14	108.85	4.5-10
2020-12-17	yes	n/a	n/a	n/a	leak in the chamber, no ice deposited	n/a	n/a	159	6.6
2020-12-18	yes	Large (~5mm) flat crystals, widely interspersed nucleation, more square in shape	crystals fully merge, borders disappear, transparent polycrystalline layer	fine fractures caused by heat	very transparent layer. Very fine fracturing of all surface after switching on heater (abrupt temperature rise)	155	10	155	3.3-30
2020-12-22	yes	Very large crystals (5-7mm) at few nucleation sites. Less angular than dec 18	fully transparent polycrystalline layer	fine fractures caused by heat	transparent layer. Very fine fractures when T went from 164K to 167K. Fully healed after plate cooled to 164K again	167	18.6	158	20-27
2020-12-23	no	small (~1mm) transparent crystals form at frequent nucleation sites, no polycrystalline layer	vertical growth, beginning stage of prismatic crystal columns	within individual crystals	individual crystals are turning reflective, either fracture within each crystal or a new crystal with different orientation grows on top. Similar to Nov 3	163	6.6	144	4.5-10
2021-02-03	yes	"flake" crystals, partly transparent	very smooth slab	no	images are too dark; stopped experiment early	148	11.6	148	8-100
2021-02-09	yes	very large crystals (5-7mm) at few nucleation sites. Size limited by space available	slab	larger fractures that heal	transparent polycrystalline layer, many larger fractures, heal at first then healing stops	157	5.6	143	4.5-8
2021-02-18	yes	very large crystals (5-7mm) at few nucleation sites.	slab	larger fractures	recorded sublimation after cooling stopped; transparency returned, fractures heal during sublimation	159	6	145	6.5-9
2021-02-24	yes	very large crystals (5-7mm) at few nucleation sites.	slab	large fractures, before and after sublimation	Frost from shroud fell onto plate, sintered into fractured slab. Slab regained partial transparency after frost and fractures sintered	163	8	148	3.5-42
2021-02-26	no	a mix of smaller (<1mm) and medium crystals (>5mm)	Slab layer, gets more "cloudy" with time	large fractures	many small reflective crystals, does not stay fully transparent, many fine fractures grown directly on Al plate	166	7.5	143	5.5-9.5
2021-05-31	no	blanket frost	prismatic frost	no	heater induced fractures, rupture at weak spot due to basal sublimation	176 and 124	2.5	124	2.5-6
2021-06-02	yes	large transparent crystals	slab	yes	growing ice on dark painted copper plate; heating induced fine fractures; no rupture (lack of weak spots)	154	6.7	152	6-120
2021-06-29	yes	nucleation starts at the microcracks within the paint layer, forming elongated crystals	slab	yes	unpainted copper plate; heater induced fracturing, no ruptures (gas escaping elsewhere)	157 and 163	5.3	144	4.5-6
2021-07-02	yes	large flat transparent crystals	slab	yes		157	3	154	1.5-100

Figure 5.1: An overview table of all conducted experiments. Pressure and temperature values marked with a star denote approximate values from lab notes, data logging capability was not yet available for these experiments.