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High Porosity Chained Aggregates from the Topsoil
of the Lunar Regolith Dust

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Summary: The unusual behaviour of fine lunar regolith like stickiness and low heat conductivity is dominated by the structural arrangement of its finest fraction in the outer-most topsoil layer. Here, we show the previously unknown phenomenon of building a globular 3-D superstructure within the dust fraction of the regolith. New technology, Transmission X-ray Microscopy (TXM) with tomographic reconstruction, reveals a highly porous network of cellular void system in the lunar finest dust fraction aggregates. Such porous chained aggregates are composed of sub-micron in size particles that build cellular void networks. Voids are a few micrometers in diameter. Discovery of such a superstructure within the finest fraction of the lunar topsoil allow building a model of heat transfer which is discussed.

Keywords: Lunar soil**,** soil aggregates, porosity, Transmission X-ray microscopy, regolith

Introduction

Suggestions that the lunar surface is covered by a layer of loose regolith were based on the impact model of its genesis. The lunar surface is covered by numerous impact craters whose diameters vary from tens of kilometres to microscopic in size. Continuous pulverisation of lunar surface rocks and constant mixing develops this type of soil which is unknown on Earth. First accurate estimation of lunar soil grain size was published by Wesselink [1] as a quantitative explanation for the variations in the lunar surface temperature that have been observed during an eclipse of the moon by means of the theory of heat conduction. It is shown that the grains of the lunar powder must be smaller than 0.3 mm. Subsequent measurements on the lunar regolith samples delivered from the Moon during Apollo and Luna missions confirmed Wesselink's estimations and add accurate date from direct measurements [2, 3, 4, 5, 6]. Only 10 wt% of the lunar regolith grains was found to be larger than 0.25 mm and most grains were below 10 μm in size.
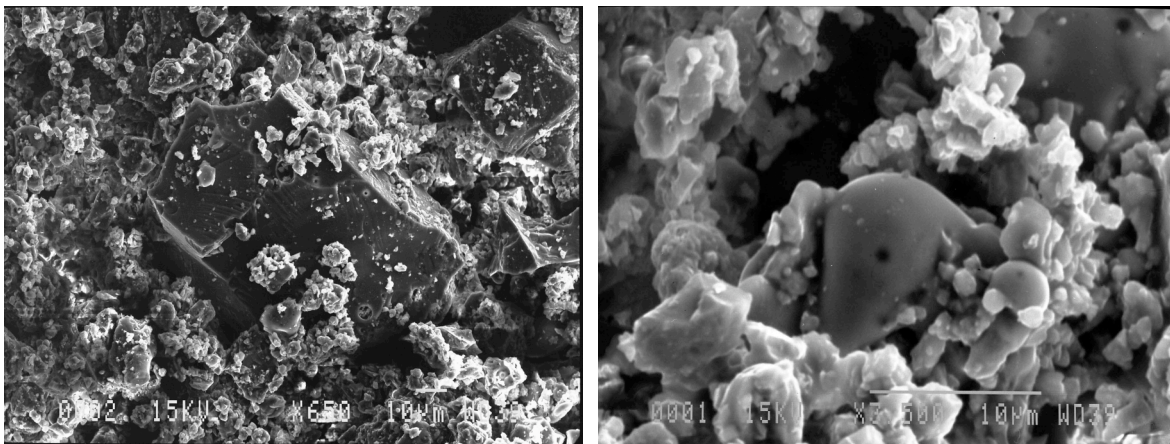

Fig. 1: SEM micrographs of studied lunar soil morphology show (A)- larger grain 100 μm in diameter decorated by fine dust below 1 μm, (B)- fine dust particles show spherical and broken glassy splash and condensation features. (scale bars are 10 μm)

The lunar regolith sample studied in this work came from a depth of 15-18 cm below the lunar surface and drilled and delivered to Earth by first ever automatic lunar mission "Luna 16" and has been obtained from the USSR Academy of Sciences [2, 7, 8]. The features have been described by Żbik, [8]. As shown in SEM micrograph Fig.1A, the lunar regolith consists of larger mineral and rock grains, vesicular glass welds soil particles together called agglutinate. The finest lunar dust fragments, smaller than 10 μm as shown in Fig.1B, consist of glass micro-droplets and their broken irregular fragments. In these SEM micrographs

larger grains (100 μm) are embedded within a cushion of tiny (<1μm) particles. The SEM 2-D micrographs show that particles are arranged in a very porous network, but details of particle 3-D arrangement and contacts between particles are not clearly observed.

Recently, a revolutionary new technique has been applied to the study of nano-material science, called nano-tomography [9]. This method is based on the Transmission X-ray Microscope (TXM) with 60 nm tomographic resolution installed at beamline BL01B of NSRRC in Taiwan. This microscope works with a synchrotron photon source [10]. This new technique [11] has recently been developed to investigate soil even in an aqueous suspension without sample pre-preparation. The big advantage of TXM tomographic study is the possibility of conducting direct observations of aggregates microstructure without sample pre-treatment.

Experimental

A transmission X-ray microscope with 60 nm tomographic resolution has been installed at beamline BL01B of NSRRC in Taiwan shown in Fig. 2. This device has a superconducting wavelength shifter source, which provides a photon flux of 5 x 1012 photons/s/0.1 % bw in the energy range 5-20 keV. X-rays generated by a wavelength shifter are primarily focused at the charge coupled detector by a toroidal focusing mirror with focal ratio nearly 1:1. A double crystal monochromator exploiting a pair of Ge (111) crystals selects X-rays of energy 8-11 keV. After passing through the focusing mirror and double crystal monochromator, the X rays are further shaped by a capillary condenser. Its entrance aperture is about 300 μm, with an end opening about 200 μm and is 15 cm long. This capillary condenser gives a reflection angle of 0.5 mrad with respect to the propagation direction. The condenser intercepts the impinging X-rays and further focuses them onto the sample with a focusing efficiency as high as 90% due to the totally reflecting nature inside the capillary. The zone-plate is a circular diffraction grating consisting of alternating opaque and transparent concentric zones. In the microscope, the zone-plate is being used as an objective lens magnifying the images 44× and 132× for the first order and third order diffraction mode, respectively. Conjugated with a 20× downstream optical magnification, the microscope provides total magnification of 880× and 2640× for first order and third diffraction order mode, respectively. The phase term can be retrieved using the Zernike's phase contrast method that was introduced in light microscopy since the 1930s. The gold-made phase ring

positioned at the back focal plane of the objective zone-plate retards or advances the phase of the zeroth- order diffraction by $\pi/2$, resulting in a recording of the phase contrast images at the detector. The scanning electron microscope (SEM) JSM-840 in a Geology Department of the Tokyo University has been used in secondary electron mode with cathode voltage 15 kV.
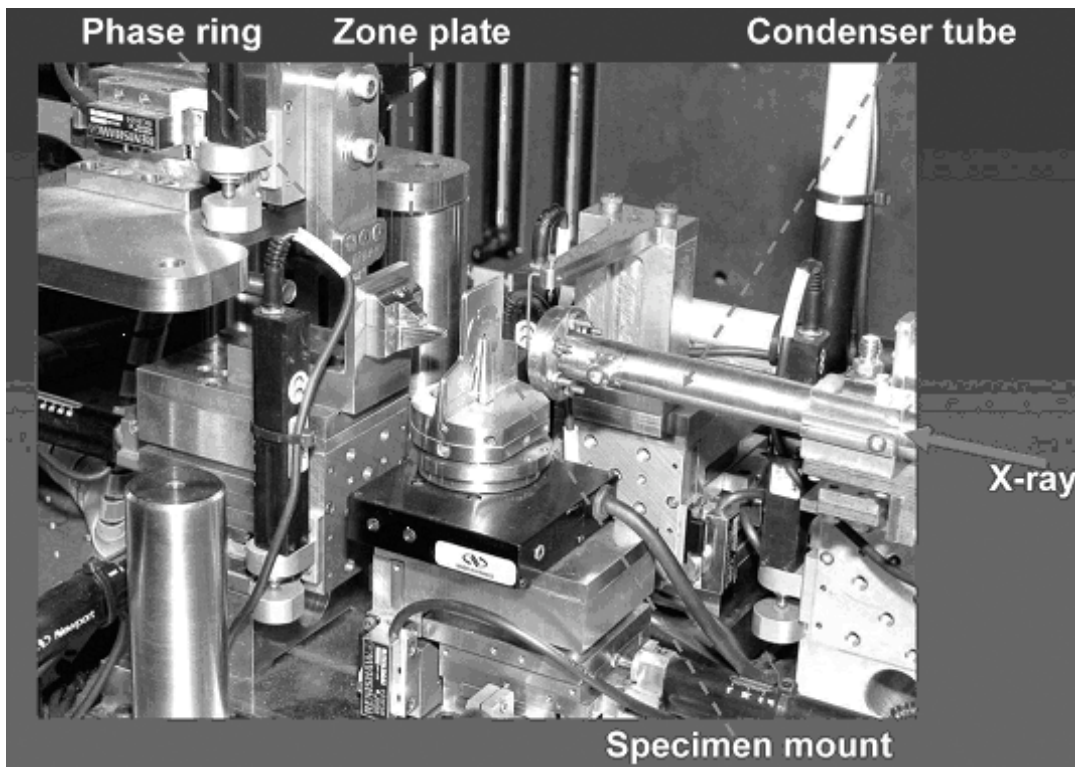

Fig. 2: Schematic arrangement of the beamline 01B1 SWLS-X-ray Microscopy.

The sample used was from Luna 16 mission previously described in [7]. Luna 16 was the first robotic probe to land on the Moon and return a sample to Earth and represented the first lunar sample return mission by the Soviet Union and the third overall, following the Apollo 11 and 12 missions. The drill was deployed and penetrated to a depth of 35 cm before encountering hard rock or large fragments of rock. The column of regolith in the drill tube was then transferred to the soil sample container. After 26 hours and 25 minutes on the lunar surface, the ascent stage, with the hermetically sealed soil sample container, lifted off from the Moon carrying 101 grams of collected material at 07:43 UT on 21 September. Unfortunately the sample was not brought to Earth in a method to prevent its entire shaking and disaggregation.

For the TXM investigations of topsoil outer-most layer, only the smallest aggregates which have been sticking to a Teflon empty sample container wall were collected. This also has a positive outcome because it focuses the TXM investigations to the stickiest aggregates presumably covering topsoil layer of the lunar regolith. In the present article we were limited only to these few dust aggregates remaining within the empty container which limited the observations and restricted the conclusions.

Results and discussion

From the aerogel, the "Stardust" mission which was launched in 2004 to capture particles from comet Wild 2, to interstellar and interplanetary dust, highly porous aggregates are widely used. If the sample is highly porous, 3d images can be taken from a number of 2d slices of material e.g. embedded in epoxy which would give a much better statistics and number of contact points for heat transfer and other considerations. However any sample pre-treatment may introduce artefacts and the chosen TXM method does not need any sample preparation and in consequence show no artefacts.
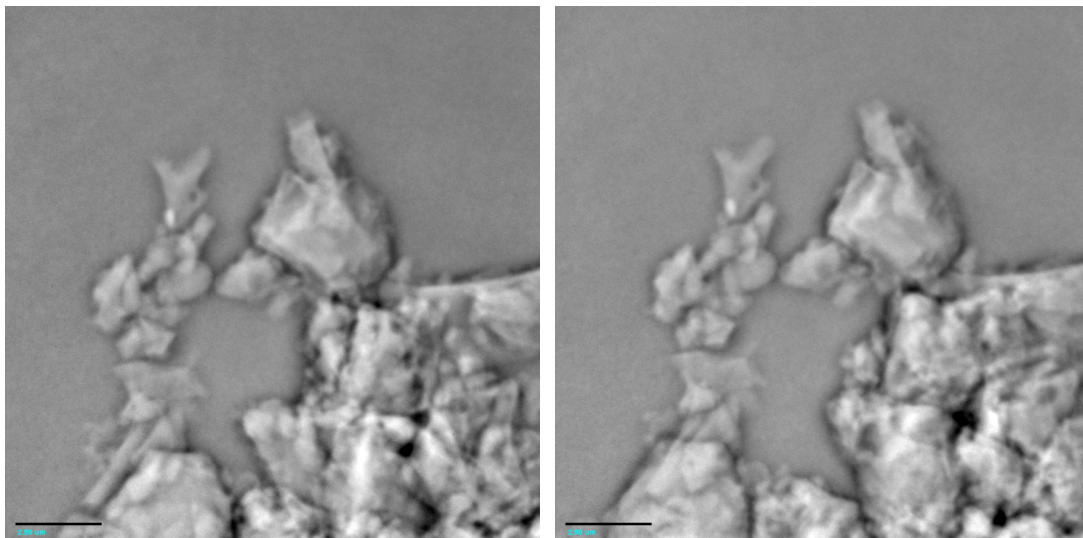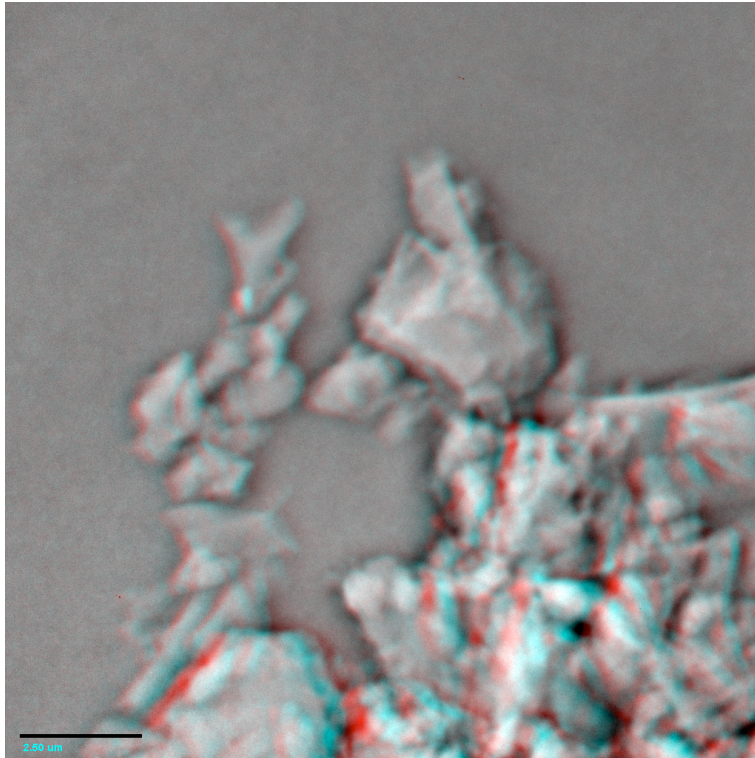
Fig. 3: Stereo- pair and 3-D anaglyph images taken from 181 2-D TXM micrograph (angle difference 1 degrees) showing long chained fine dust particles with large void separating this aggregate from another aggregate cluster (scale bar 2.5 μm).

The stereo pair shown in (Fig 3) has been taken from the two TXM 2-D images in 15 degree angle difference shows the fine lunar dust aggregate superstructure. Particles in this aggregate are loosely connected in a type of arrangement where an angular mineral grain is in contact with irregular sharp edges and spherical dust fine particles. The structure of lunar dust aggregates similar to that studied is abundant in the topsoil layer and looks extremely porous. Aggregates of fine lunar dust, shown on left side of Fig. 3, are built like strings of chained dust particles. In the discussed example, this aggregate consists of 23 particles from 1.8 μm to 120 nm in diameter with a median diameter 480 nm. Morphologically it is mixture with sharp edges and spherical, probably impact generated glassy particles. Some glassy particles show internal nano-bubbles up to 170 nm in diameter. Some particles were decorated with spherical iron rich particles 260 nm in diameter. One aggregate is about 8 μm long and 2-3 μm thick. It is porous and inter-particle voids are elongated and 100-250 nm wide. Particles contact each other in very few points with the contact area smaller than resolution of used TXM (60 nm) which give an appearance that the particles are separated. In such small contact areas the van der Waals attractive forces keeping aggregates together are rather week. They may compete with electrostatic repulsive forces which by trying to break aggregate apart

causing to stretch aggregates in the form of long strings. System is probable in balance depending of the sun position which changing electrostatic charges in dust particles as found in O'Briens work [6]. Between the described aggregate and another similar group of aggregates is about 4 µm which is the half of the aggregate longest dimension. The large void separating aggregates looks regular and cellular in shape. A large probably mineral particle 3.8 µm in diameter and 1.5 µm in thickness lay on the top of the bridge between aggregates. This is the largest grain observed in studied sample.
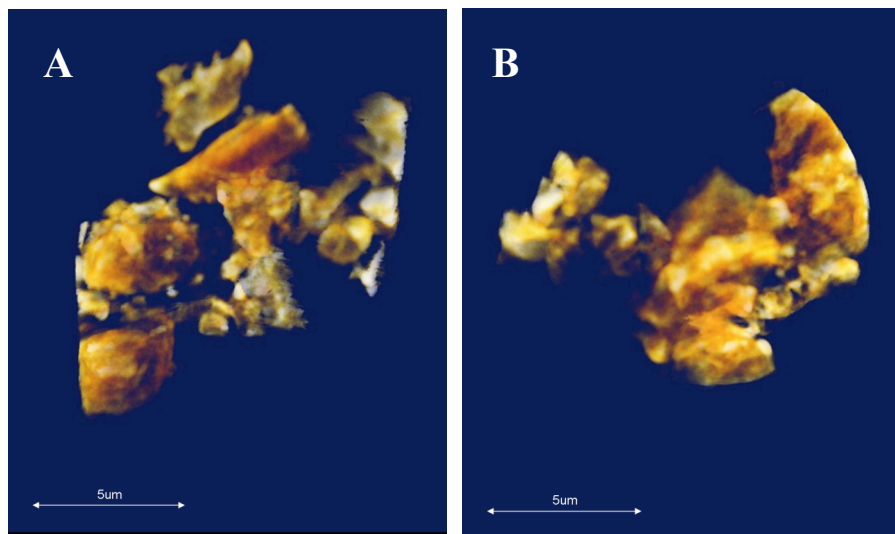

Fig. 4: Space, 3-D reconstruction of the lunar dust particle space arrangement. (A)Cylinder facial cut. (B)- Cylinder vertical cut. Cylinder is 12.1 µm long and 10.2 µm in diameter, scale bar is 5 µm).

In Fig. 4 the 3-D reconstruction from 181 TXM micrographs as shown in (Fig. 3) displays space arrangement between aggregates of the lunar dust particles. In this space reconstruction it is clearly shown that aggregates composed of submicron in size particles are separated by large voids a few µm in size. The void sizes are larger than aggregate dimensions and as shown in the vertical cut (Fig. 4B) vary between 2 and 7 µm in diameter. Because the contact area between particles that bridge aggregates are scarce below resolution of the TXM 3-D reconstruction (60 nm), the particle and aggregates appear not to contact each other. These features are easier to see when rotating the image.

From our observations, we suggest that models for porosity and heat transfer calculations in the lunar topsoil have to consider the presence of a superstructure of highly porous chained aggregates composed of sub-micron size particles that build larger cellular void networks of a

few micrometers in diameter. Such a model may be shown as in Fig. 5 the sandwich sequence of stratification of horizontal parallel and solid slabs of equal thickness ~500 nm. They represent individual dust particles arranged in string-like aggregates. For our proposed model let such slabs be separated by empty spaces, bounded by the parallel plate surfaces of adjacent slabs, so that their distances are all equal and ~250 nm in value. Denote the sum of the thickness of one slab and one space by d. In most of our observations, 3 of these silicate slabs are separated by 2 empty slices that will model the observed aggregates. These string-like aggregates are separated from each other by cellular voids a few microns across (~4 μm in our example). Based upon our observations, the basic module δ of the lunar topsoil will be an assemblage of 3 d plus 4 μm of inter-aggregate space equal to a total of ~6.25 μm. Endless sequences of such modules (δ) will create a simplified lunar dust topsoil model. The Porosity calculated from such a topsoil model is 76 % which is larger than was estimated for loose, bulk, Luna 16 soil sample from its density measurements. The structure of this outer-most, topsoil layer is unique and different from the bulk regolith which is more densely packed. This reflects the different porosity values between the studied outer-most topsoil aggregates and bulk regolith.
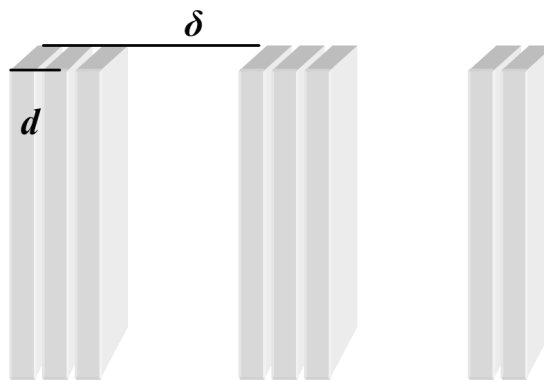

Fig. 5: Simplified model of the lunar aggregated topsoil layers.; d is the thickness of one slab plus one space and δ is the basic module between aggregates of lunar topsoil 6.25 μm in size.

Our simple model, shown in Fig. 5, can be compare with Wesselink's model, the sum of average dimension of grain and interstice is denoted as s, s equals to 750 nm. When the grain size is small enough, the conduction through the contact places is negligible and the radiative transfer prominent. An estimate of an effective thermal conduction (k), can be made by combining the Stefan-Boltzmann law and the heat conduction equation (the per unit area version) and solving the differential equation for k.

$$dQ/dt = \sigma T^4 = k(dT/ds) \Rightarrow k = \sigma \int (T^4/dT)ds = 4\sigma s T^3$$

The heat conductivity k can be considered as irradiative heat conductivity entirely and calculated from $k = 4\sigma s T^3$, where σ is Steven-Bolzmann constant (8.2×10^{-11} cal cm^{-2} min^{-1} °C^{-4}) and T is the absolute temperature in degrees Kelvin. This infers that k equals 1.25×10^{-6} cal cm^{-1} min^{-1} °C^{-1} with T = 370.79°K. It is 373 times less than the value of Wesselink's calculation with s = 0.028 cm. In this case, the porosity is 33% instead of 76% which is estimated in the model described above. That means the practical heat conductivity should be smaller than 1.25×10^{-6} cal cm^{-1} min^{-1} °C^{-1} (8.75×10^{-5} W m^{-1} °K^{-1}) from our observation. It would be of great interest to future insulation in lunar based stations. This low heat conductivity makes the finest fraction of lunar soil heat rapidly from the sun and also loses heat rapidly, because of low heat capacity.

Conclusions

Dust aggregates as shown in Figs 3 and 4 caused by electrostatic attraction between (sub)-micron particles are not stable because they are not really glued together. Even if they are, they easily break contacts with an applied force. Our investigation leaves the nature of the contacts open though saying they are below the resolution of the system. While it is shown in the SEM micrographs, that some particles would be welded but their dimensions are much larger than the observed nano-sized aggregates that may be in fact impacted pulverized rocks and agglutinates. Handling, i.e. extracting aggregates or even handling a larger piece of matter with the aggregates will change the morphology completely as aggregates get restructured. In fact on the lunar surface such restructuring take place even during the lunar day/night cycle. Obviously aggregates have been taken out of the context of other aggregates; at least the 3d image of an aggregate does not show where the aggregate connects to the other aggregates in the real topsoil of the lunar regolith. There is open space around the aggregate in the images so it may refer to aggregates icing the outermost layer where uncompacted lunar soil is exposed to interplanetary space.

Because of the small size of the studied sample (only 101 g was delivered to the Earth), our observations were limited to a few studied aggregates. However, based on our real 3-D observations on this limited sample we may define a void size for an aggregate which only consists of about 20+ particles in each observed aggregate. It is true that in order to apply certain concepts to quantify the aggregate structure requires larger volume scans and this may be done on other lunar samples where larger volume of material can be studied.

As presented, our model of the topsoil has 76% porosity and refers only to the lose aggregates on the lunar outermost surface exposed to interplanetary space. This aggregate can be easily compressed depending on the building blocks as one move down the ground profile. There are numerous references [2, 3, 4] to much more compressed lunar soils of only 40 to 50% porosity in the literature. A thermal shield built from the high porosity cellular outermost topsoil layer may be very important to future planned Moon exploration as well as the cosmic radiation shield suggested in [12].

The results presented in this study suggest that the effective thermal conductivity of lunar regolith is much lower than previously thought due to voids within the substructure. This characteristic of lunar soil increases its attractiveness as thermal insulation material for any future lunar bases.

Acknowledgment.

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