# Habit and texture studies of lunar and meteoritic materials with a 1 MeV electron microscope

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# HABIT AND TEXTURE STUDIES OF LUNAR AND METEORITIC MATERIALS WITH A 1 MeV ELECTRON MICROSCOPE

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Some habit and texture features of the finest and uncrushed grains in the lunar dust samples 10084 and 12070, were studied with the 1 MeV electron microscope of the Institut d'Optique Electronique du C.N.R S., Toulouse, France These features were then compared with those observed in broken fragments extracted either from lunar rocks 10047 and 12063 or from different types of meteorites (Orgueil, Pesyanoe, Shergotty) The finest fraction of the Apollo 11 lunar soil samples is constituted of about 80% of well ordered but rounded crystals which are frequently overlaid by a thin skin of amorphous material. In the Apollo 12 soil the grains are coarser, the fraction of crystalline material is higher ($\sim$90%) and the proportion of crystals showing an amorphous skin is smaller, but both lunar soil samples are very different from meteoritic or lunar rock matter. Some implications of the present results concerning the fabric of the finest fraction of the lunar regolith, the ancient solar wind and meteoritic research are briefly outlined.

## 1. Introduction

It is known [1,2] that habit and texture studies of well dispersed materials can provide informations concerning their fabric. In particular texture features such as the distortion present in the electron or X-ray diffraction patterns of natural samples have already been used to obtain information about the disordering processes acting during their formation and subsequent history [3–5].

Samples from the surface of atmosphereless and magnetic field free planets like the Moon – and possibly the parent body of gas-rich meteorites – have been irradiated by solar and galactic cosmic rays, and by solar wind particles, and also subjected to various types of dynamic processes, such as meteoritic impacts. As a result various forms of lattice disorders should be stored in such samples and their habits and textures should reflect their complex history in the planet regolith.

800, 200 and 100 keV transmission electron microscope studies have been reported [6–11] for grains extracted from the Apollo 11 lunar soil and from lunar rocks. These studies have been generally limited to the analysis of exsolution lamellae present in lunar pyroxenes and have given evidence for a fast cooling history of these crystals and for disordering processes probably not related to the irradiation history of the lunar soil.

In the present work we studied some habit and texture features of the Apollo 11 and 12 uncrushed dust grains by high voltage electron microscopy, with the hope of finding some characteristic signature of the dynamic processes acting in the lunar regolith and evidence for the exposure of the grains in different types of solar radiations.

We saved a tremendous amount of machine time by not studying in detail exsolution lamellae and not indexing our great number (∿300) of electron diffraction patterns. This decision was made because we were using the diffraction patterns almost exclusively as "scaling" devices to estimate the relative abundances of amorphous material and well crystallized and highly disordered crystalline grains in the finest fraction of both lunar soil samples, and because the study of exsolution lamellae only gives a crude classification of pyroxene grains in groups of related cooling histories.

We then compared the lunar soil results to those obtained from the study of small crushed crystals extracted from various types of meteorites and from the surfaces of lunar rocks 10047, 12063. Such comparisons were made in order to get both a better understanding of the fabric of the finest lunar soil grains and some clues concerning the history of meteoritic matter considered either as primitive (Orgueil) or extremely enriched in solar type rare gas (Pesyanoe).

## 2 Experimental Methods and Results

### 2.1. *Experimental methods*

Lunar fines from the Apollo 11 and 12 missions – samples 10084 and 12070 – and small surface chips from lunar rocks 10047 and 12063 were provided by NASA. Small fragments from the type I carbonaceous chondrite Orgueil, from the solar type gas-rich Pesyanoe enstatite achondrite and from the Shergotty eucrite were obtained from B. Mason (U.S. National Museum, Washington D C.).

The preparation techniques applied to the samples differ from those described in a previous paper [12] concerning the nuclear particle track content of the same lunar soil grains The finest fractions of the lunar soil samples were still separated by using a 400 Mesh sieve but they were directly "dry" deposited with a platinum loop on a new parloidion-carbon substrate containing microscopic holes (Fukami-Adachi type substrate). As they were coarser and opaque to the beam the crystals extracted from lunar or meteoritic rocks were crushed before being also "dry" deposited.

The samples were examined with the 1 MeV electron microscope of the Institut d'Optique Electronique du C.N.R.S., Toulouse. Generally dark field and bright field observations and electron diffraction patterns were made as soon as possible after the introduction of the grain in the microscope, with a beam intensity smaller than 0.2 $\mu$A. But we examined occasionally the influences of both the beam intensity (up to 2 $\mu$A) and the duration of the observation (up to 2 hr) on the appearance and stability of some of the most striking habit and texture features of the grains

### 2 2. *Experimental results*

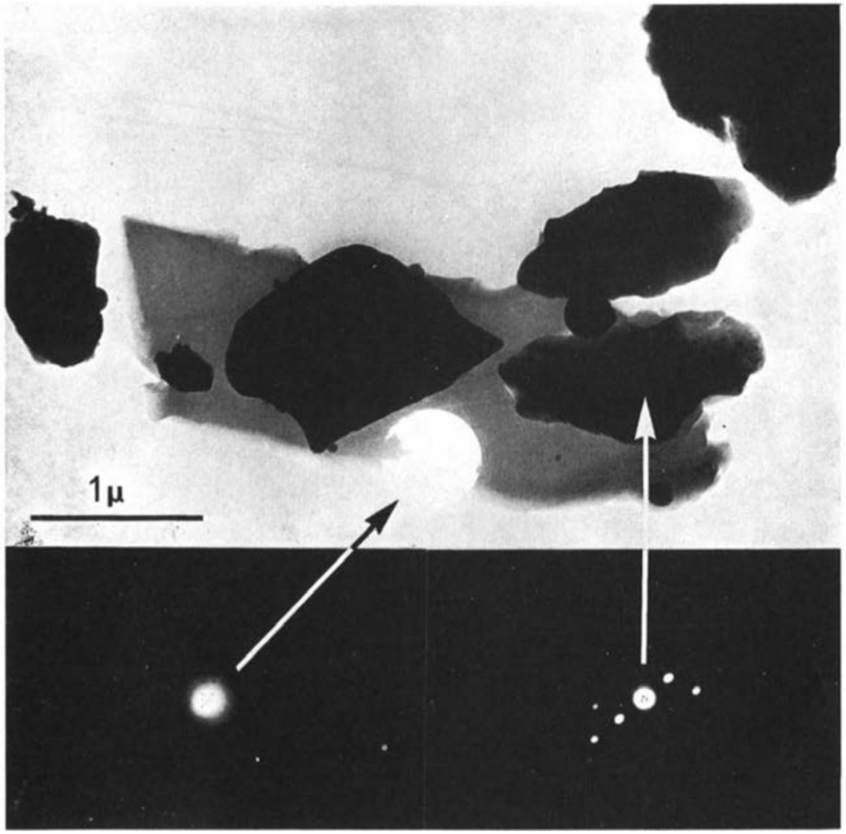
Our habit and texture studies are summarized in figs 1–6 of this paper where we show the high voltage electron micrographs and electron diffraction patterns of the Apollo 11 and 12 uncrushed lunar soil grains (figs 1–3) and those of broken fragments extracted from the following meteorites Pesyanoe (fig. 4), Shergotty (fig. 5), and Orgueil (fig. 6). In addition, several high voltage electron micrographs of individual grains from the Apollo 11 and 12 soils can be examined in figs. 1–5 of another paper [12] for a more detailed comparison of lunar and meteoritic matter, in that paper the Apollo 12 grains were crushed. We analyzed about 100 individual grains for each lunar soil sample, ∿50 grains for each lunar rock but only ∿10 grains for each meteoritic sample Therefore the conclusions concerning meteoritic matter have to be considered as very preliminary.

#### 2.2.1. Lunar fine samples 10084 and 12070

In the Apollo 11 dust, the crystalline component represents approximatively 80% of the material and has the following striking characteristics.

(1) The grain habits are generally very homogeneously rounded (dark grains in fig. 1),

(2) In dark field observation an important fraction of the grains (∿50%) appear to be surrounded with a superficial layer of about 1000 Å in thickness. We verified that these layers, also observed in the Apollo 12 grains, were made of amorphous material by moving the selection diaphragm over the grains and



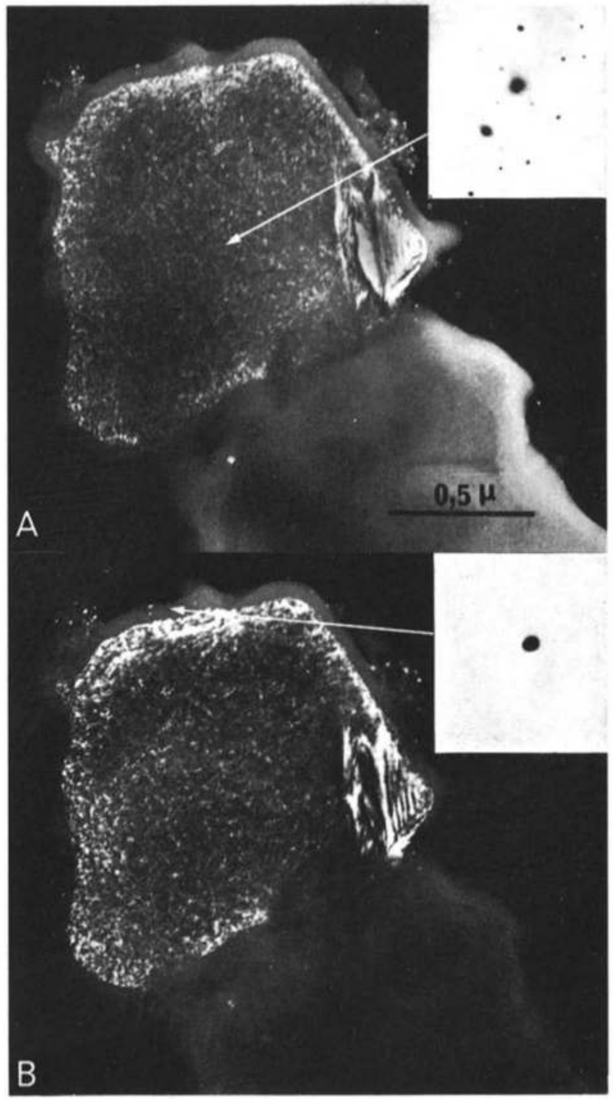
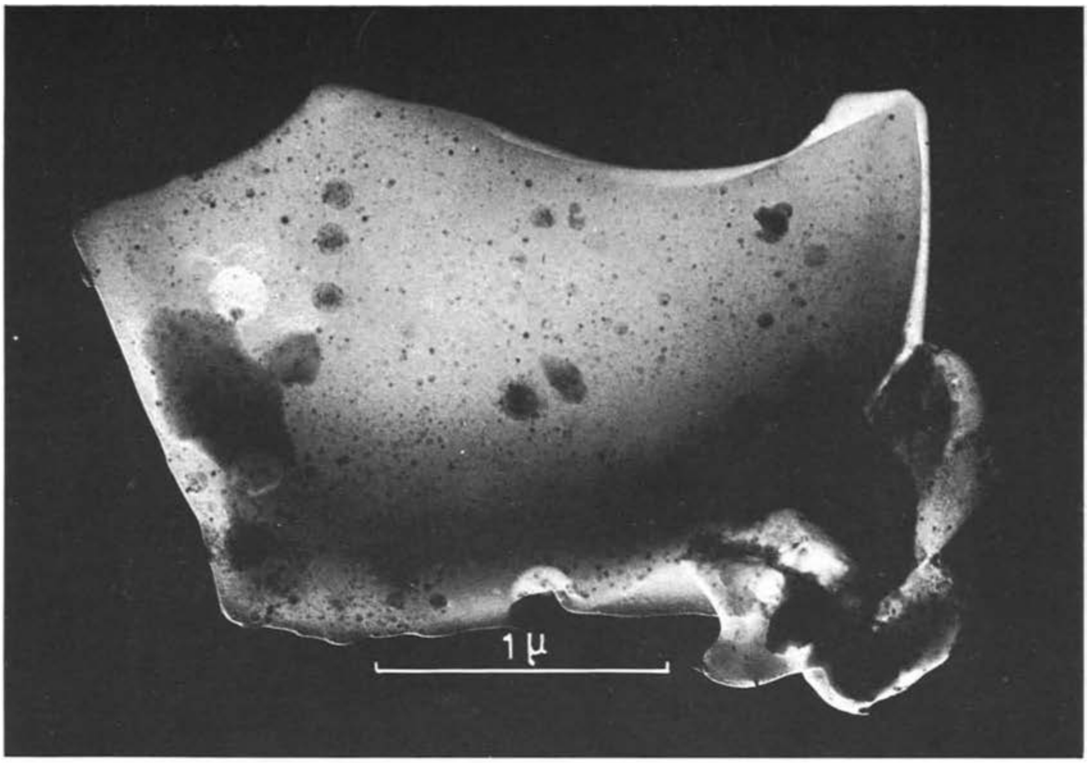




Fig 1. Bright field micrograph of the finest fraction from the Apollo 11 soil (sample 10084). The crystalline grains appearing as the dark phases in this picture are homogeneously rounded.

Fig. 2. Dark field micrographs of a crystalline grain from the Apollo 12 soil (sample 12070). This grain contains a very high density of nuclear particle tracks and is surrounded by a superficial skin of amorphous material, as indicated by the corresponding electron diffraction patterns. Micrographs A and B correspond to 2 very different sets of observation conditions. in A the picture was taken with a beam intensity smaller than 0.1 $\mu$A, 20 seconds after the introduction of the grain in the beam, B represents the same grain after a continuous exposure of 30 minutes in a beam intensity of 0.5 $\mu$A.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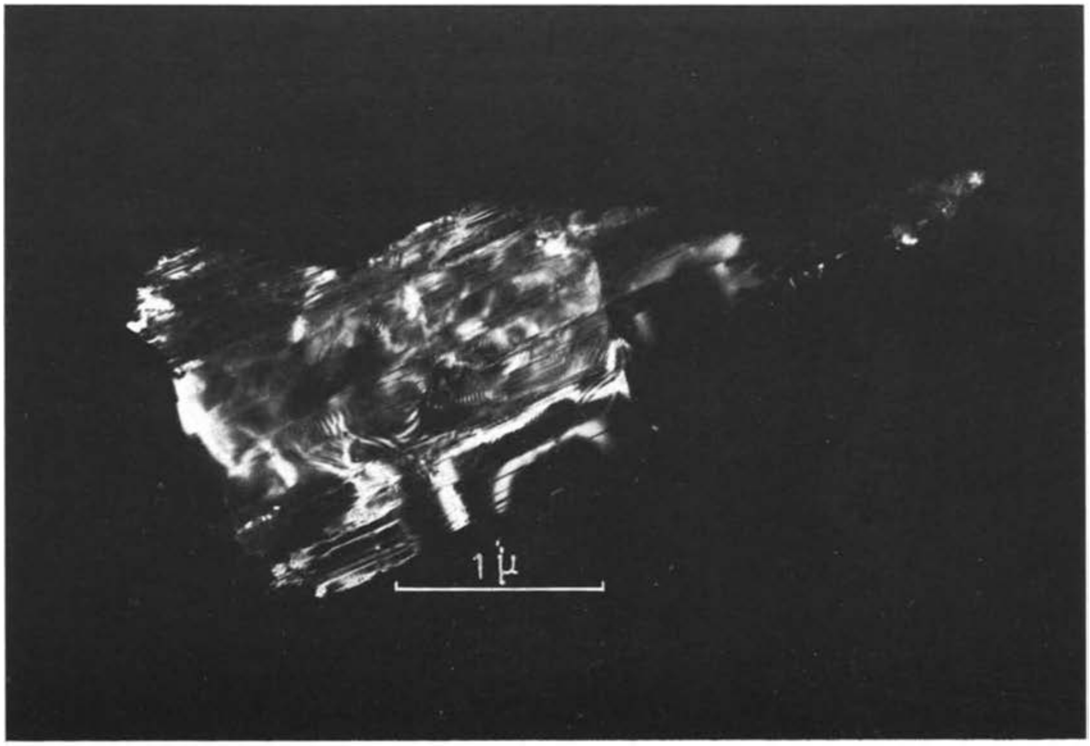
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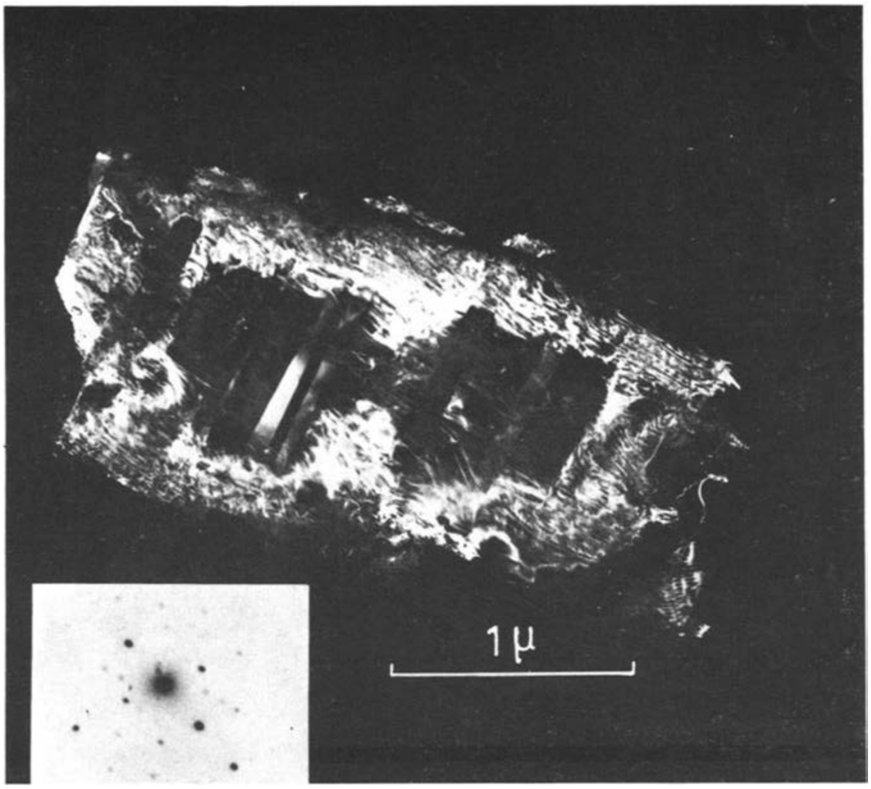


Fig. 3. Dark field micrograph of an amorphous grain of the Apollo 11 lunar soil. This grain was examined with a contrast screen and is beautifully rounded.

Fig. 4. Bright field micrograph of a crystalline grain extracted from a dark part of the gas-rich Pesyanoe meteorite.

Fig 5 Dark field micrograph of a crystalline grain from the shocked Shergotty meteorite. Abundant texture features are visible in almost each individual grain

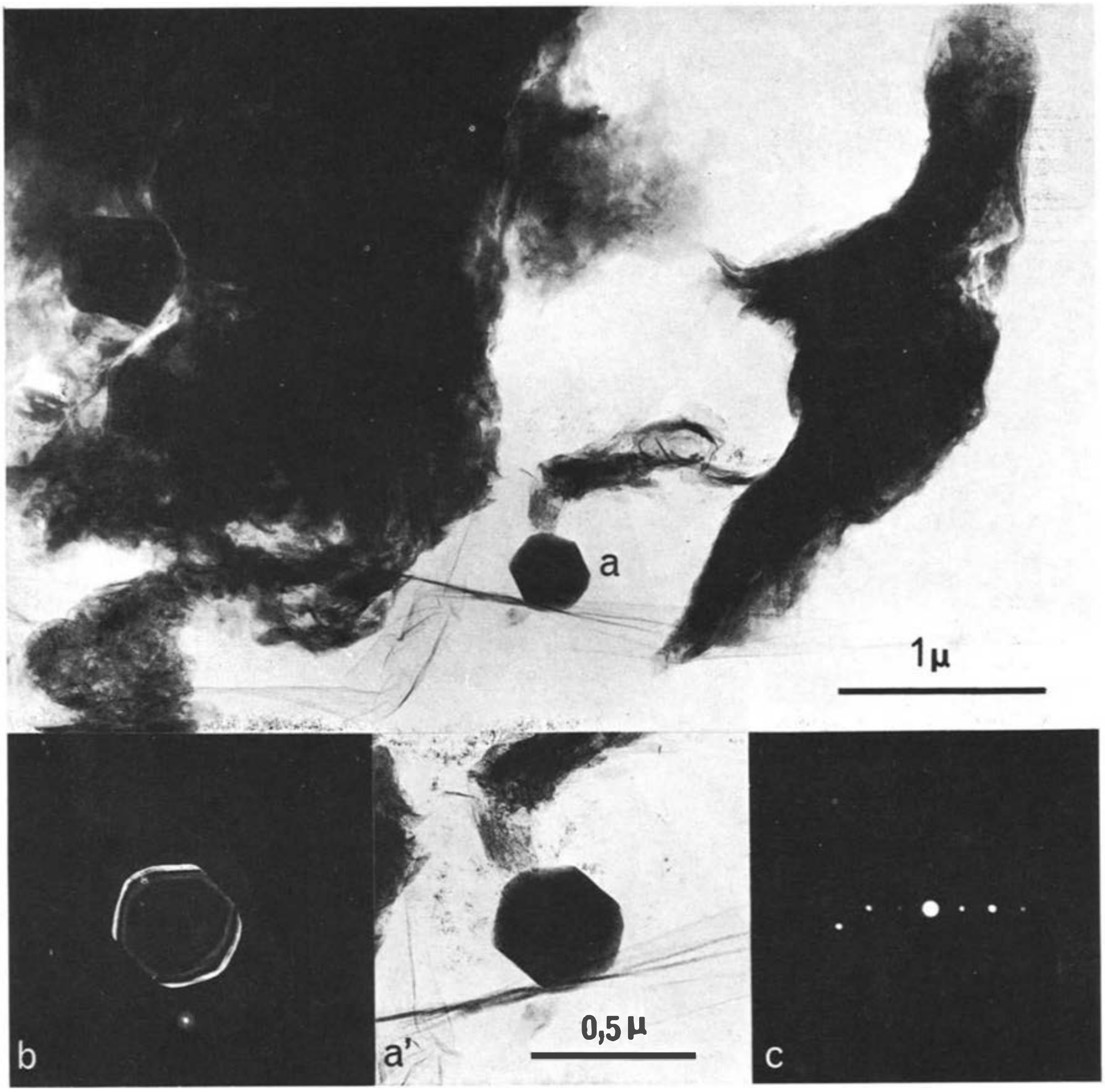


Fig. 6 Bright field micrograph of the fine grained matter extracted from Orgueil by a gentle crushing In the lower part of this picture magnified views of the dark and bright field observations of spherule *a* have been reported in *b* and *a'* with the corresponding electron diffraction pattern in *c*. This pattern indicates that the spherule is probably a circular magnetite platelet. No latent track can be observed in the good dark field micrograph *c* which shows a complicated growth pattern for the platelet

observing that the electrons were no more diffracted when the selected diffracting area (of about 1000 Å in diameter) was chosen in the dark superficial lining. Such an amorphous skin and the corresponding electron diffraction patterns are shown in fig. 2, for an Apollo 12 grain, which was simultaneously studied for other purposes described in paragraph (3),

(3) The diffraction patterns when taken below the amorphous skin, show a good ordering of the grain lattices; thus the proportion of highly disordered crystalline material is remarkably small in thus sample,

(4) A very high proportion of the crystalline grains (∿90%) contain very high densities of nuclear particle tracks perhaps produced – as suggested in another paper [12] – by a flux of cosmic nuclear particles with energies intermediate between those of the solar wind nuclei and those of the solar flare cosmic rays It should be pointed out that the highest track densities ($\gtrsim 10^{11}$ tracks/cm$^2$) were always observed in grains showing an amorphous skin

The non crystalline component representing the remaining 20% fraction in sample 10084 is totally amorphous and consists of very thin lamellae which are either ver[illegible]ngular (light grain in fig. 1) or very homoge[illegible]usly rounded like the crystalline grains (f[illegible]).

Some of the characteristics of the finest fraction of the Apollo 12 soil were different from those of the Apollo 11 soil In the Apollo 12 dust sample the proportion of crystalline material was higher (∿90%) and that of crystalline grains showing an amorphous skin was smaller (∿20%). Furthermore the grains were coarser and for unknown reasons, the nuclear particle track distributions were more contrasted and easier to observe in the Apollo 12 material

In the discussion we shall explain how the amorphous skin could be used as a tracer to study the past activity of the Sun. But for such a purpose it is necessary for this skin to be formed on the Moon and not in the microscope as a result of the interaction between the electron beam and the grains A beam origin for the skin is ruled out by the following evidences, (1) the skin was never observed on crushed fragments extracted from lunar or meteoritic rocks, (2) its thickness and appearance did not vary when the beam intensity, $I_b$, was increased by a factor of 10 or when the duration of observation, $T$, was extended up to 2 hours, therefore the skin cannot be a contamination or a radiation damage layer* (the lack of any combined effect of $I_b$ and $T$ is illustrated in fig. 2 where we show two micrographs A and B of the same grain taken with the following values for $I_b$ and $T$. fig. 2A $I_b \lesssim 0$ 1 $\mu$A, $T \sim 20$ seconds, fig. 2B. $I_b = 0.5\ \mu$A, $T = 30$ minutes, (3) its thickness or appearance showed no discontinuity when the observed grains suddenly stopped being supported by the Fukami-Adachi substrate, this fact excludes any complicated shadowing effect for the skin origin, due for example to the deposition of grain matter on the substrate

Some additional observations relevant to the origin of the skin were made

(1) The skin was still observed unchanged after an annealing of 1 hour, at 800°C in air followed by a very rapid transfer of the grains in the microscope column, this observation seems to rule out the skin as a reaction layer produced by an atmospheric component with the grains, and indicates that the skin matter is not made of pure glass because after this annealing experiment we have not found any spherule or glass lamella in the 400 Mesh residue,

(2) It disappeared completely after 1 minute of etching in a mixture of acids (2 parts HF 40%, 1 part $SO_4H_2$ 80%, 4 parts water) at 25°C, such etching conditions generally remove layers of about several 1000 Å in thickness in silicate material and this experiment shows that the skin is superficially located in the grains

2 2 2 Lunar and meteoritic rocks

Texture features such as exsolution lamellae, striations, microcrackings were much more frequent in crystals extracted from the surface of lunar rocks 10047 and 12063, but no amorphous edge with high track densities below was observed

The grains constituting the dark parts of solar type gas-rich meteorites are considered by several investiga-

* The degradation of insulator grains [13] and the very quick fading of latent nuclear particle tracks [14] in silicate minerals occur frequently at 100 keV and these effects are generally attributed to the ionization produced by the electrons in the samples That they are not observed at 1 MeV is not surprising in view of the marked decrease in the ionization energy losses of the electrons and the much lower beam intensity required to make similar observations with the 1 MeV microscope

tors to have been individually irradiated in the ancient solar wind [15] and solar flare cosmic rays [16,17]. In grains extracted from a dark part of a typical member of this class of meteorites (Pesyanoe) we observed no amorphous fraction and no gradual disordering of the crystals. The angular habits (fig. 4) were similar to those of fragments obtained from the crushing of large single crystals No amorphous edge with nuclear particle tracks below was identified in the crystalline grains.

Shergotty is generally considered as a good example of a shocked meteorite [18] We observed that the material extracted from this meteorite was extremely different from the lunar soil grains and the Pesyanoe material. An important fraction (∿50%) of the grains was amorphous and a gradual disordering of the crystalline component was observed, however no amorphous edge or track was present in the grains. Furthermore abundant texture features, rarely found in the lunar fine and Pesyanoe materials were observed in almost each individual grain chosen at random (fig. 5)

The Orgueil meteorite is often described as a very primitive object [19] The material extracted by gentle crushing from this meteorite (fig 6) was markedly different from the Apollo 11 and 12 dust grains as well as from the Pesyanoe and Shergotty matter (a) the proportion of amorphous material was even more important (∿90%) than in Shergotty. However the abundant texture features observed in the Shergotty grains were almost absent in the Orgueil material, (b) the habits of the amorphous fraction were frequently similar to those of "fibrous" material, (c) no crystalline grain with an amorphous edge and latent tracks below was found in Orgueil, (d) small crystalline spherules were frequently observed The indexation of the electron diffraction pattern of the spherule shown in fig. 6 indicated a cubic lattice similar to that of magnetite Therefore these spherules are probably circular magnetite platelets of a type already discovered by Jedwab with an optical microscope [20]. No spherule with diameter smaller than 3000 Å was observed.

## 3 Discussion of the results

### 3 1 *Fabric of the lunar soil grains*

The fabric of the finest fraction of the lunar regolith is very different from those forming the grains extracted from a primitive meteorite (Orgueil), a solar type gas-rich meteorite (Pesyanoe) or a shocked meteorite (Shergotty) because this fraction, contrary to meteoritic matter, seems to consist of 2 distinct components which are a totally amorphous fraction and a component of rounded crystalline grains showing no tendency for a gradual amorphization These grains are frequently homogeneously rounded and covered by a thin skin of amorphous matter and loaded with very high densities of nuclear particle tracks

Furthermore in the smallest grains of the lunar soil the exsolution lamellae and other striation and microcracking features were apparently less frequent than those observed by us and other investigators [6–11] in coarser lunar rock crystals, in particular in those examined with a 800 keV electron microscope, after an ion bombardment thinning of the crystals [6] This observation could be added to other evidence – such as the 3- to 100-fold enrichment of the lunar soil in Ir, Au, Zn, Cd, Ag, Br, Bi and Tl relative to type A, B rocks reported by Keays et al [21] – to argue that an important fraction of the lunar soil did not originate from the rocks imbedded in it presently

The finest grains of the lunar regolith were probably produced by an erosion mechanism releasing matter from rocks by a flaking off process perhaps triggered by micrometeorite impacts as suggested by Gault [22]. Such a process can in principle produce an amorphous fraction but also well crystallized and highly disordered crystalline materials Then the astonishingly small proportion of highly disordered crystals observed in the present work should help to fix some of the input parameters (such as the characteristics of the ancient micrometeorite fluxes) in Gault's theory.

Immediately after their release in the lunar soil the grains probably had angular habits limited by cleavage or fracture surfaces. Then they should have been processed by a second erosion mechanism having the striking capabilities of homogeneously rounding the grain habits and covering their external surface with a skin of amorphous material without introducing any measurable amount of lattice disorder below the skin.

It is difficult to admit that these erosion features observed in grains of about 1 micron in size are due to micrometeorite impacts, or to microcracking induced by thermal shocks produced during the lunar thermal cycle A more likely process is perhaps the implantation of solar wind ions in small grains which are rolling over the Moon surface during their bombardment. That such an implantation is really occurring is clearly shown by the rare gas data of Eberhardt et al [23]. Then the amorphous skin observed in the grains could be due to the radiation damage accompanying the ancient solar wind implantation. This conclusion is also compatible with the skin thickness (∿1000 Å) which corresponds to the expected range of solar wind ions in silicate material

3.2. *Ancient solar wind*

If further studies* confirm the solar wind origin of the amorphous skin then some interesting results concerning the characteristics of the solar wind in the past could be obtained for the following reasons.

(1) In a given mineral the thickness of the skin will represent a dynamic balance between the loss and the formation of amorphous material but will be a function of the range and therefore of the energy of the solar wind particles Thus it should be possible to look for eventual marked variations in the average energy of the solar wind nuclei in the past by measuring in a given mineral species (such as cristobalite which can be easily identified by its electron diffraction pattern) the thickness of the amorphous skin as a function of depth in a core tube,

(2) for a given set of exposure conditions of the dust grains on the Moon surface, any change in the solar wind intensity in the past would result in a variation with depth inside a core tube of the proportion of grains showing a skin effect,

(3) for a given mineral grain the critical value of the solar wind flux above which the amorphous skin is produced can be determined by artificially irradiating lunar material with solar wind type ions. This value will give an interesting lower limit of the integrated flux of solar wind nuclei which bombarded the lunar soil grains.

* Crushed grains from the central part of several lunar rocks are presently artificially irradiated with solar wind type ions and the eventual growth of the amorphous skin will be studied as a function of the energy, atomic number, dose rate and integrated flux of the ions

3.3. *Meteoritic research*

The first solar type gas-rich meteorite grains we examined looked very different from the lunar soil grains both in the size distribution of the grains, which were much coarser, and by the absence of an amorphous fraction Also there was no rounded edge showing a superficial amorphous skin with high densities of tracks below. These differences have already been emphasized by our high voltage electron microscope nuclear particle track studies [12] and by other evidence summarized elsewhere [24]. They show that the individual mineral grains in the dark part of a typical solar type gas-rich meteorite have not been processed by mechanisms similar in nature or in intensity to those responsible for the irradiation and formation of the finest grains of the lunar regolith, except if the compaction process anneals the tracks and the amorphous skin without introducing any measurable amount of disorder in the lattice of the grains

These conclusions are also valid for Orgueil However it could be argued that this meteorite contains crystals which have been highly disordered by some kind of radiation damage. Therefore before excluding an ancient irradiation for the constituent grains of Orgueil, a thermal annealing experiment of a type proposed by Seitz et al. [25] should be conducted to verify if the proportion of highly disordered crystals has clearly decreased after the annealing, thus indicating that radiation damage remnants are still trapped in Orgueil, even if no amorphous edge with tracks below has been preserved in the mineral grains of this meteorite

As already pointed out the results and conclusions concerning meteoritic matter are preliminary because we examined only a small number of grains (∿10) in very few meteorites. A more extended version of our work, including results obtained from a 2 MeV electron microscope survey of different types of primitive meteorites (Orgueil, Murray, Renazzo, Bjurbole), other gas-rich meteorites (Pesyanoe, Khor-Temiki, Jodzie, Kapoyeta) and the fission xenon rich Angra dos Reis eucrite, is in preparation

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