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Some applications of transmission electron microscopy to space physics

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We have used the techniques of 1 MeV and 100 keV electron microscopy to compare various radiation-damage features in lunar dust grains and in mineral grains that have been exposed to artificial fluxes of low-energy nuclear particles; in particular, amorphous rims to the grains and etched ion tracks were examined. The results of these investigations clearly show the effects of solar wind sputtering and reveal the existence of high fluxes of low-energy particles in interplanetary space.

Some implications of the present work are presented concerning the past and present activity of the Sun, the sticking process taking place in the primitive solar nebula and the variation in the mineralogical composition of the cosmic dust clouds as a function of the age of the galaxy.

1. Introduction

This paper is essentially a summary of the most important results obtained by a collaboration between the Orsay group and the Institut d'Optique Electronique du C.N.R.S. at Toulouse. This work [1-8] concerns radiation-damage features induced by solar particles in small silicate grains extracted from the following materials. 1. Lunar dust samples brought back to the Earth by the various Apollo and Luna missions. 2. Lunar breccias and solar type gas-rich meteorites. 3. The protective mineral paint from the Surveyor III spacecraft that was exposed during three years on the surface of the Moon and subsequently returned to the Earth by the Apollo 12 astronauts. 4. A piece of mica which was exposed during about five days on the surface of the Moon during the Apollo 16 mission. 5. Grains artificially irradiated in our Laboratory in various fluxes of nuclear particles. Some of the results reported here have not previously been published.

We use such silicate grains as radiation-damage detectors. Our main objectives are 1. To detect the effects of an ancient solar wind implantation in lunar dust grains. 2. To search for "low-energy" nuclear particles possibly existing in interplanetary space, with energies intermediate between those of the solar wind nuclei and those of the solar flare cosmic rays. 3. To study the alterations of the irradiation record in the grains that result either from their thermal annealing or from their sintering into extraterrestrial breccias. 4. To determine the exposure conditions of the grains to "ancient" radiations with a view to understanding the very complex transport mechanism responsible for the redistribution of matter in the lunar regolith.

2. Experimental methods

The smallest lunar dust grains (< 1 μm in diameter), as well as ultrathin sections of the Surveyor III paint, were directly examined in dark-field conditions with a 1 MeV electron microscope at the Institut d'Optique Electronique du C.N.R.S. at Toulouse. These samples can also be observed in bright-field conditions with a 100 keV transmission electron microscope, after a slight chemical etching first developed by Barber *et al* [9]. These observations were complemented by a study of the distortion appearing in the electron diffraction patterns of the grains, such as a sharp decrease in the total number of diffraction spots and an increase of the continuous background due to inelastic scattering of the electron beam.

The largest lunar dust grains, as well as thick sections of Surveyor III paint, were mounted in

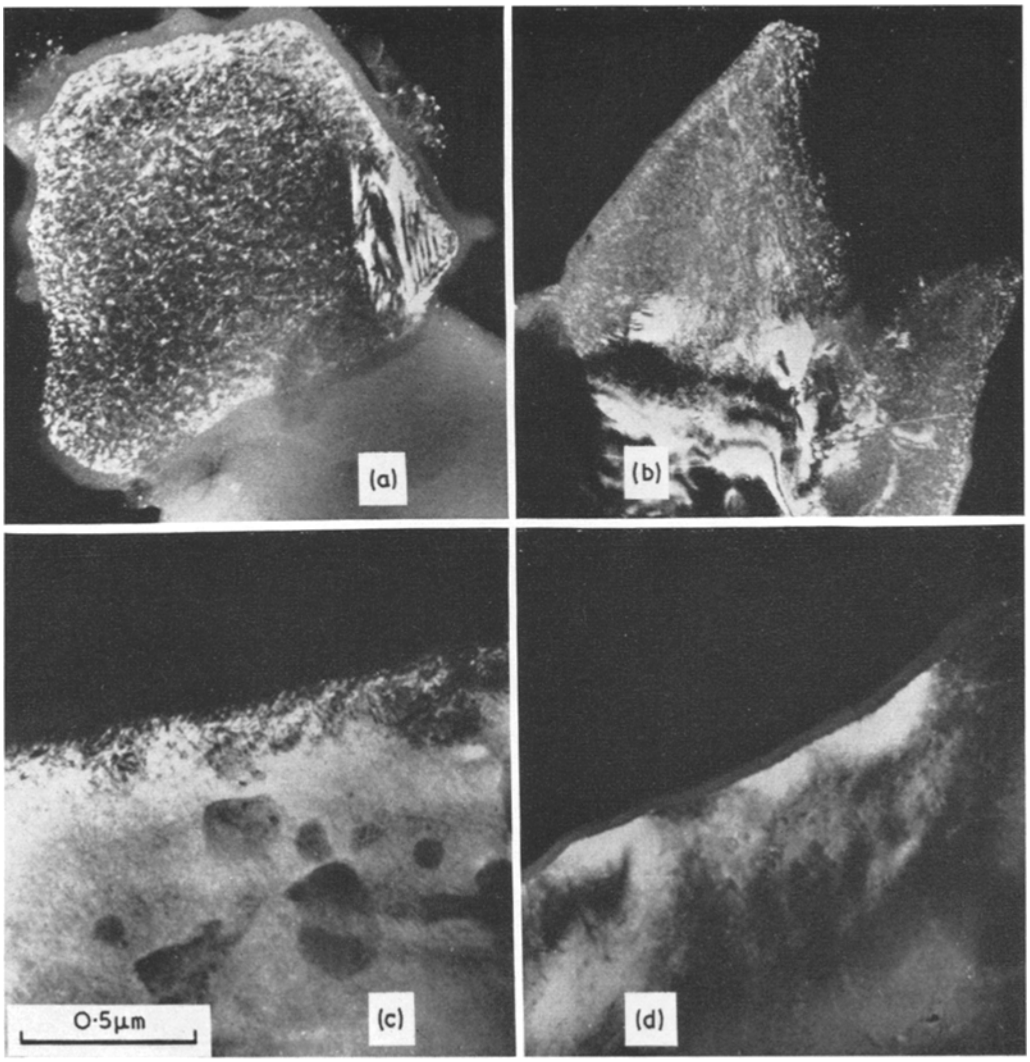

Figure 1 1 MeV dark field micrograph of typical lunar grains from the Apollo 11, 12, 14 and Luna 16 missions, showing rounded habits and amorphous coatings. The coatings of the two grains on the first diagonal have the average thickness of 350Å whereas the ones on the other diagonal show the most extreme variations in coating thicknesses we found (100 to 1200Å).

epoxy discs, polished, then chemically etched and finally the replicas of the surfaces were observed in a transmission electron microscope.

The micas, either naturally or artificially irradiated, were etched for 2 h at 30°C in 40% HF. Their surface was then silvered by vacuum evaporation and examined either with a phase-contrast optical microscope or with a scanning electron microscope.

The artificial irradiations were performed with the isotope separators at our Institute, which produce various solar wind type ions (He to Xe), with energies between 0.2 and 5 keV amu^{-1} Elastically scattered Ni ions of higher energies (up to 200 keV amu^{-1}) were obtained with the reaction chamber constructed by J. Peter at Orsay.

3. Solar wind implantation effects

In all the lunar dust samples (with the exception of Cone Crater sample 14141) so far studied, the finest and uncrushed crystalline grains are frequently rounded and surrounded with an ultrathin superficial layer of amorphous mater-

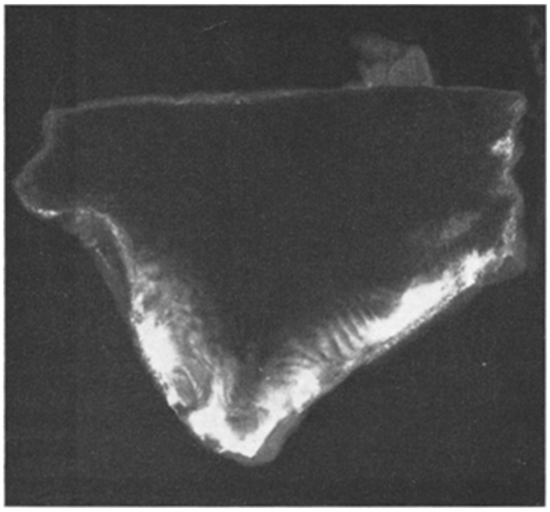

Figure 2 1 MeV dark field micrograph of a micrometer internal fragment of lunar rock 12021 irradiated with a high dose of ^{129}Xe ions of 1 keV uma^{-1}: it is rounded and has an amorphous coating.

ial, about 500 Å thick [1, 2]. These coatings appear as the lines of dark contrast around the grains shown in Fig. 1.

In order to identify the origin of these coatings, we took internal grains in lunar igneous rock 12021, never exposed to solar wind nuclei, and crushed them into micron-sized fragments. These angular fragments were then exposed to high fluxes (between 10^{15} and 10^{17} ions cm^{-2}) of solar wind type ions. After the irradiation, the initially angular grains showed both a marked rounding and an amorphous coating similar to those observed on the lunar dust grains (Fig. 2). By measuring the thicknesses, Δ, of the coatings, we observed that the Δ values vary with ion energy roughly as $E^{0.5}$ and are practically independent of the atomic number of the incident particles, for particles having the same speed.

This dependence of the coating thicknesses on the energy of the ions should be very helpful for determining the thermal properties of the ancient solar wind [2, 3, 6]. (By "thermal properties" we mean the mean flow speed of the solar wind. Suprathermal ions are particles emitted in correlation with the solar wind and having speeds higher than this mean value.)

The rounding of the natural- and artificially-irradiated grains is probably due to ion-sputtering, which erodes away the grains. By observing the "rounding and coating" effects produced by 40 keV argon ions as a function of the type of crystal, we noticed that pyroxenes are eroded away at a faster rate than the feldspars which in turn are eroded away before ilmenite, magnetite and iron. Therefore the relative abundance of silicates in cosmic dust clouds exposed "somewhere" in space to stellar wind sputtering could possibly gradually decrease as a function of the age of the clouds. This effect could have important implications concerning, for example, the formation of planetary systems [8].

When a lunar dust sample has been heavily irradiated in the solar wind, small particles are always firmly stuck to the surface of the coated grains, thus forming micron-sized "fluffy aggregates" of dust (Fig. 3). These aggregates seem to play a major role in determining lunar albedo (light-reflecting capacity) as well as the mechanical properties of the lunar soil [3].

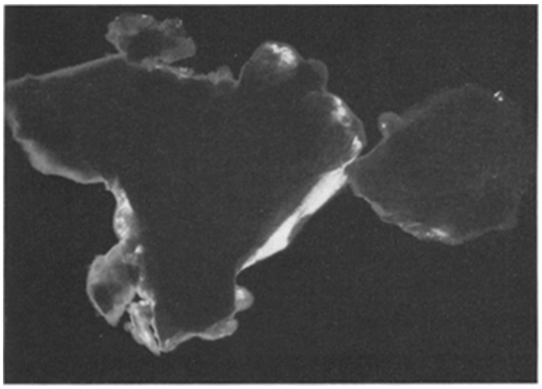

Figure 3 1 MeV dark field micrograph of a "fluffy aggregate" of dust grains. This picture was taken after an attempt of grain dispersion in alcohol, so the strength of cementing seems quite high.

4. Detection of "low energy" nuclear particles in interplanetary space

In the following, we present several lines of evidence that support the existence of low-energy nuclear particles (< 100 keV amu^{-1}) in space and we discuss the various experimental detecting schemes that we have used and that are based on the short penetration ($\simeq$ 2 µm) of such ions in silicate material.

In the micron-sized lunar dust grains, we observed a high density ($\simeq 10^{11}$ tracks cm^{-2}) of "latent" tracks appearing either as the lines of contrast crossing the grains shown in Fig. 4 or as shallow etched canals (Fig. 5) after a slight chemical etching. This strong irradiation of the grains in high fluxes of ions with a short penetra-

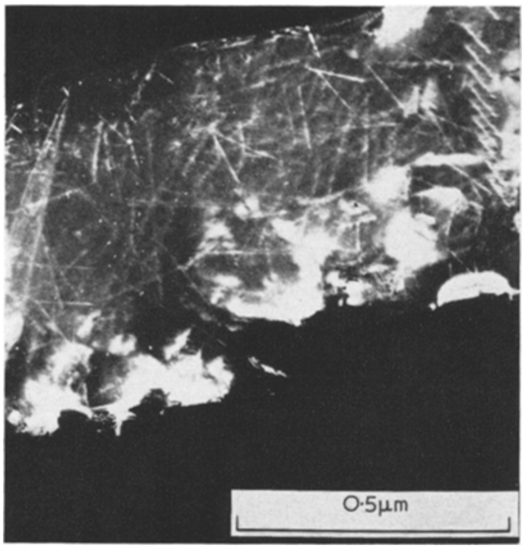

Figure 4 1 MeV dark field micrograph of an Apollo 11 dust grain containing a high density ($\simeq 10^{11}$ tracks cm^{-2}) of latent nuclear particle tracks appearing as the lines of contrast in the grain.

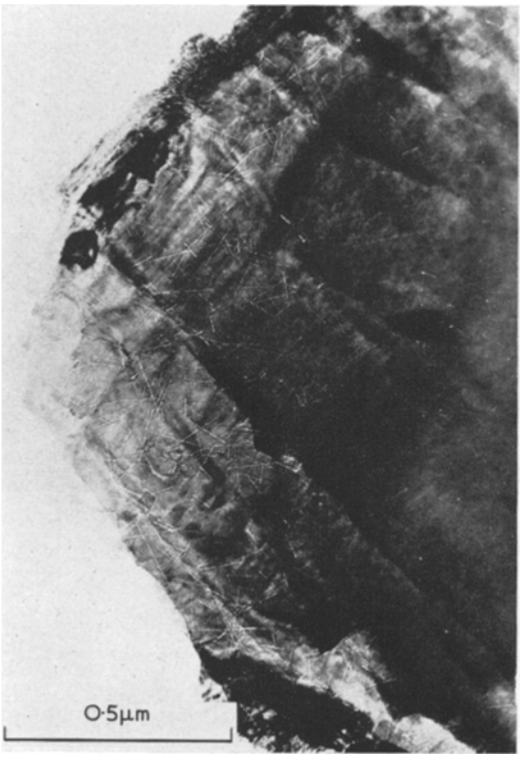

Figure 5 100 keV bright field micrograph of an etched Apollo 11 dust grain showing etched tracks appearing as the lines of white contrast.

tion is confirmed by the electron diffraction studies: the Luna 16 grains we examined had a well-ordered core surrounded with a superficial layer of highly-damaged material extending down to a depth of 0.5 to 1 μm.

We also searched for the tracks of such ions in the superficial layer of much larger grains (100 μm in diameter) by using replicas of slightly-etched polished sections. Such replicas show a very steep variation in the track density with depth inside the grains (Fig. 6). This drop occurs at a depth of about 2 to 3 μm and seems compatible with a "pulse"-shaped differential energy spectrum for the incident particles, roughly similar to that reported by Frank [10] for suprathermal protons. However, other data are necessary before deciding whether these low-energy ions are either suprathermal ions emitted in correlation with the solar wind or low-energy solar flare particles.

We finally attempted to detect the tracks of such low-energy nuclei in the contemporary radiations by using two detectors: the protective mineral paint of the Surveyor III spacecraft and a piece of mica exposed on the lunar surface during the Apollo 16 mission experiment. No latent tracks have so far been detected in the surface layer of ultrathin sections of the mineral paint but it is not excluded that the tracks could have suffered some kind of ionization-annealing under the electron beam of the microscope, similar to that reported for fission-fragment tracks in mica by Price and Walker [11]. On the other hand, replicas of sections of the paint, as well as the direct observation of external etched flakes with a scanning electron microscope, did show evidence for a high density ($\simeq 3.10^9$ tracks cm^{-2}) of short tracks. The integral track length distribution was measured in the piece of mica by Burnett *et al* [12] by means of scanning electron microscope stereoviews. This distribution (Fig. 7) clearly shows a great number of tracks shorter than 2 μm. Calibration irradiations of mica samples with low-energy nickel ions ($E \leqslant 100$ keV amu^{-1}) have been made and they show that the short tracks observed in the mica are most likely due to low-energy iron group nuclei present in the contemporary solar radiations.

5. Thermal annealing of lunar dust grains

Price and Walker [11] showed that fission-fragment tracks in mica can be transformed into small crystallites by heating the sample at

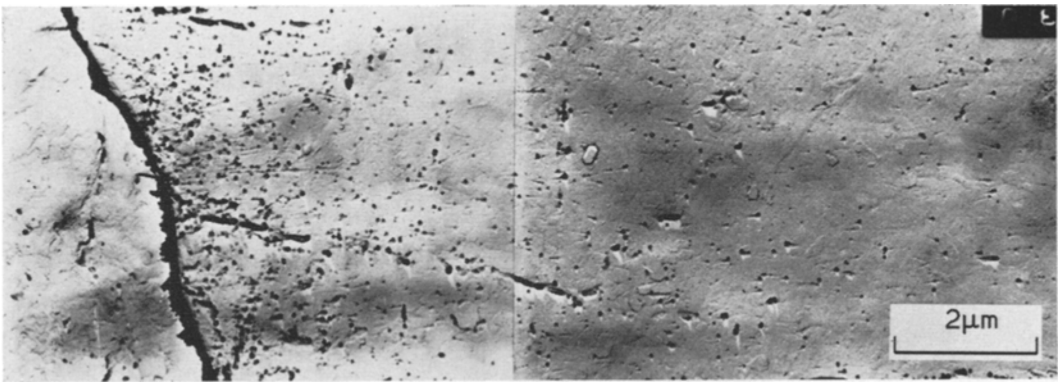

Figure 6 Replica of a slightly-etched polished section of a Luna 16 pyroxene grain, examined with a 100 keV Jeol microscope. A sudden decrease in the track density on a depth of about 2 µm can be observed.

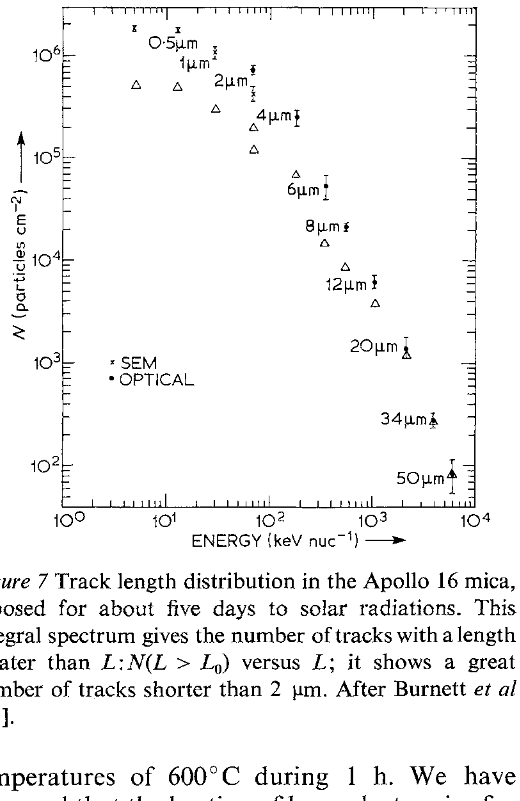

Figure 7 Track length distribution in the Apollo 16 mica, exposed for about five days to solar radiations. This integral spectrum gives the number of tracks with a length greater than $L:N(L > L_0)$ versus L; it shows a great number of tracks shorter than 2 µm. After Burnett *et al* [12].

temperatures of 600°C during 1 h. We have observed that the heating of lunar dust grains for 2 h at 800°C in vacuum, has also the property of transforming the radiation damage features (latent tracks and amorphous coatings) into a high density of small crystallites [13]. This effect was used by our group in the study of lunar and meteoritic breccias (Fig. 8): the spatial extension of such crystallites depends on the annealing temperature and therefore it can be used to fix the temperature reached during brecciation.

Furthermore such studies help in understanding the origin of the irradiation record probably still preserved in meteoritic breccias, such as the gas-rich meteorites that possibly result from the sintering of dust grains individually irradiated in the regolith of the meteorite parent bodies.

6. Conclusions

We have shown that the implantation of solar wind ions in lunar dust grains produces a "rounding and coating" effect, which is a function of both the speed of the incident particle and the mineralogical composition of the target. A new method for studying the thermal properties of the ancient solar wind has been proposed and we have called attention to the possible decrease in the concentration of silicates in cosmic dust clouds as a function of the age of the galaxy.

Evidence has been accumulated concerning the existence of low-energy nuclei in the ancient and contemporary solar radiations. The measurement of their energy spectrum will help in determining the processes responsible for their emission and propagation.

The radiation-damage "thermometry" that we have established, can help in understanding important problems related to the formation of extraterrestrial breccias.

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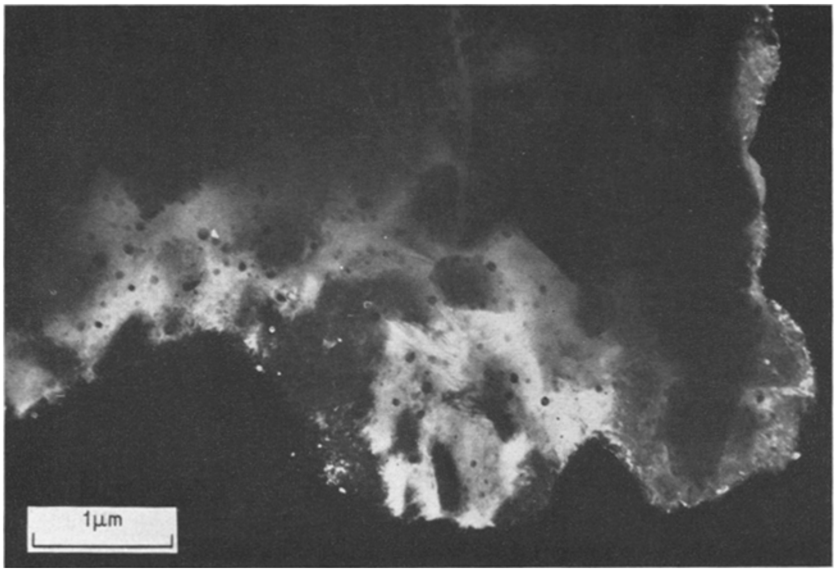

Figure 8 1 MeV dark field micrograph of a "metamorphized" lunar dust grain loaded with inclusions, and extracted from lunar breccia 10046. The existence of such inclusions supports the hypothesis that lunar brecciation results from the heat sintering of lunar dust grains.

criticisms and Professor R. Walker for providing us with a piece of mica exposed on the surface of the Moon during the Apollo 16 mission.

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