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ELECTRON MICROSCOPIC OBSERVATIONS OF HYDROGEN IMPLANTATION IN ILMENITES

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ABSTRACT

Hydrogen ion beams were found to form submicrometer, bumpy textures on the surface of ilmenite grains. From this effect we believe that similar bumpy textures seen on lunar ilmenite, pyroxene, and olivine grains are likely to be caused by solar wind irradiation. As a consequence, the concentration of bumpy textured grains may be a useful index of surface maturity for lunar soils. We believe it is worthwhile to search for grains with these bumpy textures in interplanetary dust and lunar and meteoritic regolith breccias in order to obtain information about the duration of their exposure to the solar wind

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The interaction of solar wind and solar flare atomic particles with lunar minerals is known to cause several types of damage to the minerals including the formation of particle tracks (Crozaz, et al., 1970; Fleischer, et al., 1970), sputter rounding, and the formation of amorphous rims on grain surfaces (Borg, et al., 1970; Dran, et al., 1970). Here I describe another effect which may involve not only structural damage but also chemical alteration.

Blanford, et al. (1981) reported observing bumpy surface texture on 0.5-1.0 μm grains from the lunar regolith from sample 15010,1130 (Fig. 1). Because these soil grains are very small, they most probably were exposed to the solar wind. Consequently Blanford, et al. (1981) inferred that the bumpy texture is some form of solar wind damage. This texture is confined to ironbearing minerals: ilmenite, olivine, and pyroxene. Of these, ilmenite grains have the best developed bumpy texture. Transmission electron micrographs (TEM) of the textured grains show some relatively opaque spots indicating a relatively high density. Precise correlation of these spots with the surface bumps was not always possible, although some of the bumps can be identified in TEM but are not always completely opaque. Energy dispersive X-ray (EDX) spot analyses indicate an enrichment in iron relative to that of the background mineral. For these reasons Blanford, et al. (1981) hypothesized that iron in these minerals was reduced to metal by the solar-wind implanted hydrogen ions.

In order to test this hypothesis I have simulated solar wind irradiation on natural, terrestrial ilmenite. Hydrogen ion beams were directed at small grains and polished sections which were then examined by electron microscopy.

METHOD

Samples were prepared from ilmenite ground with an agate mortar and pestle, ultrasonically stirred in a fluorocarbon solvent, and precipitated onto a copper TEM grid covered with a holey, carbon substrate. Ilmenite grains were identified by EDX analysis. The sample was irradiated with a Denton model DB-1 ion-gun using hydrogen as the operating gas. This gun is a high voltage gas discharge ion source which has an inherently large energy spread, perhaps duplicating solar-wind velocity distributions better than a sharply defined ion source. Irradiations were made with the ion gun at an angle of 45° and the sample was continuously rotated. The sample was 2 cm from the cathode of the gun and the beam spread to an 1 cm^2 area. Ambient pressure was 10 - 30 mTorr. Our average dose rate was $4.3 \pm 0.3 \times 10^{14}$ $cm^{-2}.s^{-1}$ or 10^6 times that of the solar wind on the Moon (3.8×10^8 $cm^{-2}.s^{-1}$; Feldman, et al., 1977). The maximum accelerating potential for hydrogen was 4 kV resulting in a maximum ion velocity of 550 km/s which is typical of the solar wind (Feldman, et al., 1977).

RESULTS

Irradiation with 2 - 5 x 10^{17} cm^{-2} hydrogen ions forms texturing on ilmenite grains (Fig. 2). Sputtering also occurs and, judging from rounding and erosion of small surface features, is estimated to be as much as 50% of the texturing effect (Carter and Colligon, 1968). Some sputtered droplets can be seen in our micrographs, but sputtered droplets are rare on lun· samples. Our analogue grains remain sharp with only some thin features sputtered away; lunar ilmenite grains are sharp and do not form amorphous coatings as do plagioclase grains (Borg, et al., 1980). The texturing effect is definitely not the deposition of sputtered droplets because irradiated grains of iron oxide do not have this texture (Fig. 2).

Identification of the bumpy features continues to be an analytical problem. Microelectron diffraction and EDX analysis have not been useful. Selected area (SA) electron diffraction has given inconsistent results. On lunar ilmenites one can find reflections from iron, but they seem to come from several, unidentified locations on the sample. Freshly irradiated terrestrial samples show ringed SA diffraction patterns typical of finely divided substances. These patterns appear to be those of FeTi which has a CsCl cubic structure with lattice spacing a_0 = 2.976 Å; the pattern is the same as for - Fe which has a lattice spacing a_0 = 2.8664 Å. Although the difference of 0.1 Å is twice the estimated resolving power, I hestitate to identify the bumps by this result. Samples are being prepared for Mössbauer spectroscopy. Another NASA/ASEE summer faculty fellow, Paul Schulze, is using X-ray photoelectron spectroscopy to study ilmenites before and after hydrogen implantation. Hopefully, one of these techniques will produce a definitive identification of the irradiation effects.

In comparing irradiated samples to lunar samples we have found that the simulated samples have finer texturing (Figs. 1 and 2). There is a factor of 2 to 3 difference in the size of the bumps. This effect may relate either to differences in dose rate or to differences in total dose. However, experiments done at a 6 times lower dose rate showed the same effect, but seemed to require a greater total dose by about a factor of 2.

Even though there may be a textural size difference between the simulated samples and lunar samples, we propose that this texturing on lunar samples is caused by the solar wind. This effect has implications which concern lunar reworking rates and maturity indices and it may be useful in studying the space exposure of interplanetary dust grains and grains in meteoritic breccias. We do not have space in this report to discuss these implications.

In conclusion, we have found by simulating solar wind irradiations that bumpy-textures can be formed on ilmenite grains.

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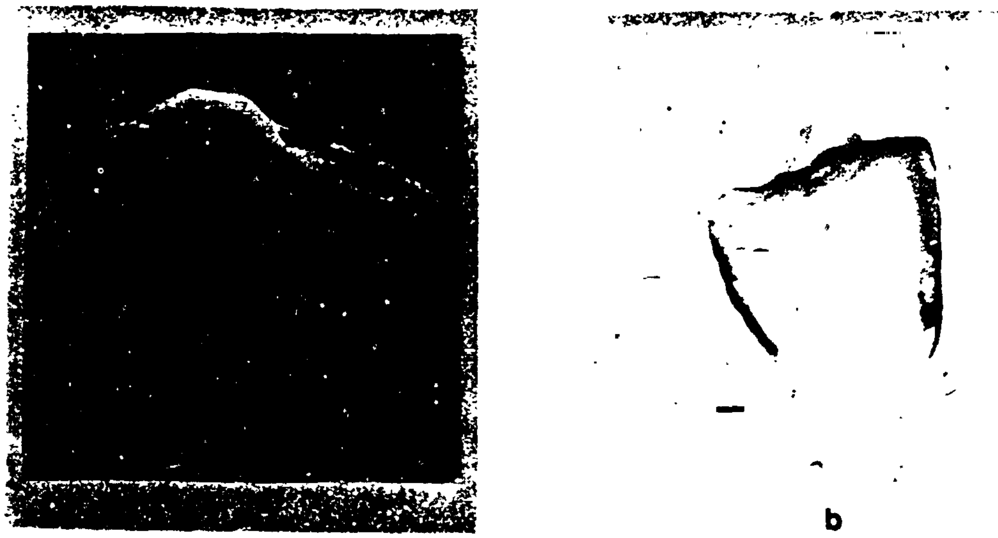

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Figure 1: Scanning electron micrographs taken with a JEOL 100CX microscope of a) an ilmenite, and b) an olivine grain showing bumpy texture from lunar soil (core) sample 15010,1130. The marker represents a length of 0.1 μm on all micrographs.

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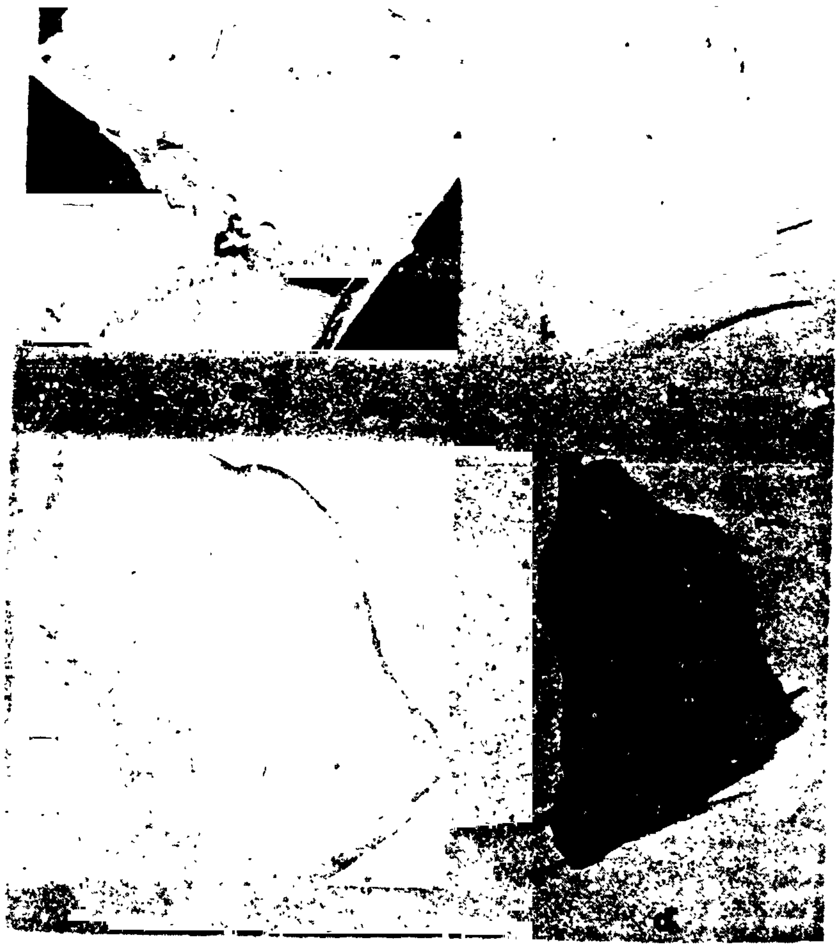

Figure 2: Scanning and transmission electron micrographs of an ilmenite-iron oxide grain before (a and b) and after (c and d) irradiation with 2.5 x 10^{17} cm^{-2} hydrogen ions. The bumpy-texturing uniquely forms on the ilmenite crystal.