MICROSTRUCTURE, MELTING AND CRYSTALLIZATION CHARACTERISTICS OF LUNAR, VITREOUS FINES (14163,14162,14258).

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Melting and crystallization studies were carried out on lunar soil samples (<1 mm and 1-2 mm fines) using thermoanalytical methods, x-ray and electron microscopy. Small, homogeneous fractions were obtained by sieving, by magnetic separation and by picking of individual grains. Approx. 50% of the fines were magnetic especially the black, slag-like particles but also many greyish plagioclase rock fragments. This is mainly caused by metallic, iron-rich inclusions and droplets on the surface. Most of the crystalline grains consisted of plagioclase disregarding their appearance - clear crystals, grey rock fragments, black slags. Olivine and pyroxene were very rare. The majority of the transparent vitreous material were brown glass fragments and also spheres, less glasses were green or colorless. One large, green glass sphere (diameter 1.48 mm) with black, wrinkled surface was investigated separately: scanning electron micrographs proved that the interior was homogeneous, vitreous whereas lath-shaped crystals (plagioclase?) were abundant in one part of the surface (Fig.1). The Ti-concentration was definitely lower in the surface the overall Ca-, Mg-, Fe-, and Ti-concentration of the glass sphere however was very close to that of terrestrial basalts and to that of the black, vesicular spherules (Fig.2), which were observed frequently. Microscopic investigation showed that practically all
glass particles contained gas bubbles of very different sizes. The greatest concentration of bubbles is found close to the surface.

DTA runs on non-separated lunar fines (smaller 100 μm) in vacuum proved that the vitreous portion of the sample recrystallizes at 780°C during heating up (4°C/min), that melting occurs in the range 1140-1170°C, that the sample still contains a small portion of crystals at 1250°C and recrystallized during cooling (6°C/min) at 1045°C to plagioclase. Recrystallization could be avoided if the samples were heated higher (1400°C) and cooled with 15°C/min. The glass obtained was colored brown. When the fines were heated in graphite crucibles in vacuum (10⁻⁵ torr) up to 1300°C a green-brown vesicular glass sphere was obtained which contained many metallic, magnetic spherules and white, lath-shaped crystals on the lower surface, similar as those shown in Fig.1. This reduction is obviously due to formation of CO during heating up. Analogous experiments in which the sample was heated in alumina crucibles in N₂-H₂ up to 1400°C showed a strong loss in weight (12%) above 1050°C. The glass obtained was colorless and contained white, lath-shaped crystals and tiny metallic spheres.

Combined TGA or DTA, heating x-ray and mass spectrometry proved to be very useful for following the melting and crystallization of the small samples available in relation to the evolution of gases. Neon and argon loss was observed first between room temperature and 200°C with a maximum at around 100°C. The largest amount of Ne and Ar is given off above the recrystallization of the vitreous portion of the soil sample, above 900°C. Experiments in which a molecular beam of the evolved gases was directed on a balance pan showed clearly the bursting of individual bubbles in
the course of melting. The heating x-ray pattern (1°C/min) proved in accordance with DTA that the crystalline reflections (plagioclase) disappear not completely in the melting region 1140 to 1170°C. During cooling (1°C/min) recrystallization of plagioclase occurred in the same temperature region, with identical but much sharper x-ray reflections than in the original soil sample. The gas evolution (Ne, Ar) decreases strongly in the region 850 to 650°C but increases again below 600°C. Similar experiments are carried out with homogeneous, separated soil fractions.

Additional experiments in vacuum, N₂–H₂ and O₂ were carried out with terrestrial basalts. The results obtained lead to the conclusion that vacuum and low alkali concentration both favor the formation of divalent iron above the liquidus temperature. This leads to formation of glasses in contrast to oxidized melts (Fe³⁺) which could not be quenched to glasses. Reducing conditions caused the formation of metallic spheres embedded in a colorless glass matrix.

Fig.1. Surface crystallization on green glass sphere 14258 (4750x)  
Fig.2. Black, vesicular spherule 14258 (95x)