Apollo 11 Anniversary: A step back in time where Kemet International Ltd helped prepare Moon rock samples in 1969

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The problems of slicing and polishing hard, high cost materials are well known to electronics manufacturers, metallurgists and others, and it is not uncommon for the price of one boule or crystal to exceed that of the sawing equipment and/or lap. The lunar rock brought back by the three Apollo missions probably constitutes the most 'expensive' material on earth and it is therefore not surprising to find that similar techniques and equipment, involving diamond tools and compounds, are being employed in sample preparation

Nearly 220 lb (100 kg) of moon rock and dust has now been brought back to earth by the three Apollo missions. The crew of Apollo 11, the first men on the moon, collected only 48 lb of lunar material, but even that small sampling from the Sea of Tranquillity-less a proportion kept untouched as a permanent record-had to be distributed amongst the 1000 plus scientists in the USA. Britain. Canada, Japan. Australia and Western Europe who were co-operating with NASA (the US National Aeronautics & Space Administration) in the initial study. Understandably, in view of the financial and other pressures at that time, some quick results were needed and NASA announced that a lunar science conference would be held in Houston, Texas. On 5th January 1970, a bare five months after astronauts Neil Armstrong, Michael Collins and Edwin 'Buzz' Aldrin had returned from their historic mission. In actual fact some groups of investigators only received their allocated samples towards the end of September 1969, so that time became the all important factor in preparing the lunar material for scientific examination.

As this article shows, diamond tools and compounds played a vital part in this race against the clock, and again helped reduce material wastage in the Apollo 12 sample examination programme. The latest versions of almost all the equipment described is available commercially.

Lunar boulder to give indication of size, taken from the Apollo 14 astronauts

Diamond sawing and slicing

Lunar material is classified by NASA into three basic categories: (a) rock, or any material larger than 1 cm, which is catalogued and given its own reference number: the largest single rock to date was brought back by the Apollo 14 mission and weighed some 22 lb: (b) coarse fines, between 1 cm and 1 mm in size: and (c) fine fines, below 1 mm in size, also known as sub-millimetre dust. In view of the time factor, some of the larger Apollo 11 material was sent out uncut and the principal investigators (heads of the examination teams) were therefore obliged to arrange for the sawing and thin sectioning of their samples using the available laboratory equipment.

At the Institute of Geological Sciences, Gray's Inn Road London, large Apollo 11 material was first sawn on an IDP model GF3 machine equipped with a conventional 6 inch diameter by 0.020 inch wide diamond impregnated saw blade.

A different approach was adopted by the University of Tübingen’s Institute of Mineralogy & Petrology, West Germany. Nine 'pebbles’, approximately 1 ½ cm long by½ cm thick were taken by the investigators to the AEG-Telefunken works in Ulm to be sliced into thin sections. Externally, the precious lunar samples resembled terrestrial anthracite. Prior to slicing, the samples were encapsulated in resin and then waxed to a ceramic base plate. Thus mounted, the samples were cut with an ultra-thin annular diamond blade, of the type employed for semiconducting slicing. The internal cutting blade, suitably stressed, was rotated at between 3000 and 5000 rev/min and was fed into the moon rock by about 5 microns per revolution.

The resultant slices, between 0.5 mm and 0.3 mm thick. were taken back to Tübingen for polishing with diamond compound. Finally, the polished thin sections were ready for microscopic Investigation.

The diamond knife, invented by Dr. Fernandez-Moran and described in detail in the January 1962 and September 1963 IDR's, has also proved extremely useful in the analysis of lunar rock. With this ultra-precision tool and its associated ultramicrotome, it is possible to produce sections only 500 Angstroms (0.00002 in.) thick, which are then suitable for examination by very high voltage electron microscopy and electron d1ffract1on techniques.

Apollo 12 rocks

The distribution of Apollo 12 rocks, from the Lunar Receiving Laboratory in Houston, proceeded at a much slower rate than with the Apollo 11 samples. This was because it was decided to cut up all the rocks before they left the Laboratory and in a much more controlled manner. Each step of the sawing being thoroughly photographed as well as many parts of the chipping procedure.

Cut-off machine

Most Apollo 12 rocks were sawn first with a large radial arm saw using standard diamond impregnated cut-off blades. Smaller pieces were sliced with diamond impregnated wire.

Development of the cut off machine carried out in collaboration with Dr. Gerald J. Wasserburg, Professor of Geology and Geophysics at the California Institute of Technology. Involved the provision of several unique laboratory requirements. Special precautions had to be taken to minimise the possibility of contaminating the specimens, particularly with lead, during the cutting-off operations. The California Institute of Technology machined a stainless steel spindle and produced various other stainless steel hardware for the saw. All sawing was done without liquid coolant only dry nitrogen was used to cool the diamond blades, with the rocks enclosed in an air-tight plexiglass cabinet.

Wire saw

At the Lunar Receiving Laboratory in Houston, smaller rock chips are sawn with a diamond impregnated wire which has a steel core covered with a copper sheathing.

Commercially available diamond wire machines can be equipped with either 0.003. 0.008 or 0.015 inch diameter wire: the 0.003 inch wire leaves a kerf of about 0.00325 inch and It is therefore easy to calculate the number of extra slices which can be obtained from a small piece of moon rock, compared with conventional diamond blade slicing. With high-priced exotic crystals as used in the electronics industry, the value of these extra slices can exceed the price of the saw for moon rock. The material saving benefits are of course incalculable A similar type of machine, known as the Sea-Saw employs multiple tungsten wires and loose diamond abrasive compound. The ultra-thin wires up to 60 can be used at a time-pass through a sump containing an oil-based slurry of micron diamond particles and activated by an oscillating spindle system, ride back and forth in see-saw fashion in tension across the workpiece at up to 21 in./mm. Machines of this type are also widely used in the electronics industry, for the slicing and dicing of solid-state materials.

Preparing samples for analysis

The microscopic analysis techniques employed by geologists require that the specimens be polished to the ultimate degree of finish and flatness to ensure that high magnification examination and photographic identification is not distorted or impaired by the reliefs of uneven surfaces. Conventional specimen polishing techniques employ diamond compounds almost exclusively and those used in the preparation of lunar samples were no exception. As it turned out, none of the lunar material was any harder or more abrasive than any of the terrestrial rocks, but the rapid material removal capability of diamond was greatly appreciated in the race to prepare the Apollo 11 samples, so that preliminary results could be obtained in the short time available before the first lunar science conference in Houston.

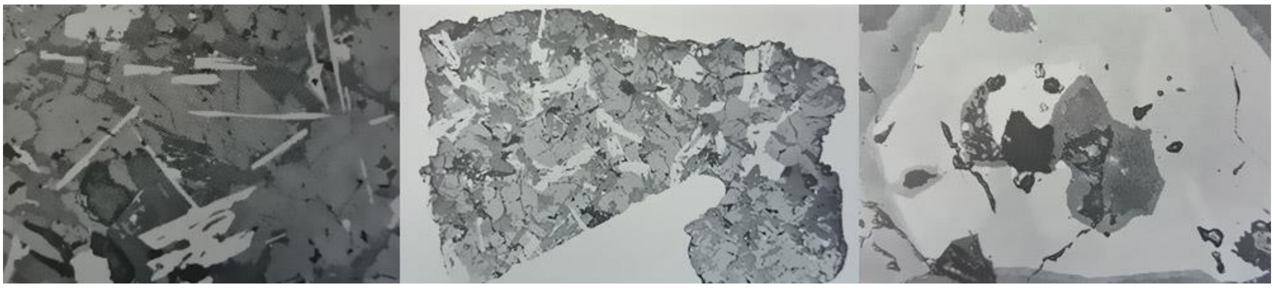
The diamond compounds employed, which are also readily available commercially, contain diamond particles specially crushed and graded to extremely fine limits so that by selecting the appropriate micron size, a predetermined surface finish can be readily achieved. Conventional diamond polishing techniques for thin sections are well known and the following paragraphs will therefore deal only with one specific procedure employed by the Institute of Geological Sciences. London, for polishing samples of sub-millimetre lunar dust.

Polishing dust samples

As Mr. Peter Simpson. co-investigator with Dr S. H U. Bowie at the IGS explained the aim of their technique is to produce specimens both for examination by reflected and transmitted light and for subsequent electron probe micro-analysis. In the latter method of examination, an electron beam is focused by electromagnetic lenses in a vacuum chamber into a fine point of 1 micron diameter on the surface of the specimen, X-rays emitted from the specimen, as a result of excitation by the electrons are analysed by means of x-ray spectrometers and exact chemical compositions can be determined by comparison with known standards. Microscopic examination on the other hand, is used as a means of optical identification of the constituent minerals. Both methods require a highly polished surface.

The first step in specimen preparation is to melt Lakeside 70 cement onto a 1 in. wide biological glass slide heated to 120°c on a hot plate. The glass slide is then slid onto the cold edge of the hot plate and particles or lunar dust sprinkled onto the now solid layer of Lakeside. The fine dust is distributed over the surface with a glass rod, the slide being replaced onto the hotplate for a few seconds so that the grains sink into the Lakeside when it re-melts. The sample is then allowed to cool. When the cement has set (which only takes about ½ minute), the mounted lunar dust is given a pre grind, first with 600 grade abrasive powder and then with Aloxite optical smoothing powder, the slide being held face down on a glass plate and circulated by hand in a figure-of-eight motion. Periodic checks are made on a stereo microscope to ensure that sufficient grain exposure has been achieved.

After diamond lapping of the first side the slide is taken and inverted over a second (blank) slide smeared with Araldite AY 111 resin mixed with HY 111 hardener; this resin has good elasticity and gives excellent adhesion for small particles. The resin is then left to cure overnight. When cured, the slides are heated to the melting point of the Lakeside cement and the upper slide, on which the lunar grains were originally mounted, is removed. The slide is cleaned with industrial alcohol, to remove traces of Lakeside, and is then recharged with a little more Araldite so that the grains are just covered. After curing, the grains are ground down, diamond lapped and polished as before. In this way, grains of exactly the required thickness (30 microns measured optically) are achieved.



Moon rock specimens prepared for high magnification examination. From left to right; ***1.*** *Doleritic rock, polished section, plane polarised reflected light at 20x magnification.* ***2.****Basaltic rock, polished thin section, plane polarised reflected light at 90x magnification.* ***3.*** *Complex ilmenite, intergrowths with some troilite polished section, plane polarised reflected light at 800x magnification.*

Diamond lapping

Diamond lapping is carried out on four Kent Mk 11 machines fitted with Hyprocel-Pellon PAN-K foam fabric polishing discs. Two of these machines are used with 6 micron Kemet diamond compound, the third with 3 micron diamond compound and the fourth with 1 micron compound. If desired, the self-adhesive discs can of course be transferred to another of the machines between preparation sequences. A lap rotation speed of about 180 rev/min is used for the preliminary lapping with 6 and 3 micron compounds, and a speed of about 120 rev/min for the final diamond lapping with 1 micron compound. Each lapping stage takes about 5-10 minutes and the compound is applied to the PAN-K discs in the normal way with a syringe applicator. Before applying the compound. a 'shot' of Kemet OS Fluid is given to the PAN-K discs, one 'shot' of this aerosol fluid providing enough lubrication for a polishing sequence.

A final polish is given to the specimens using 1 micron finely divided alumina on a fifth machine: this much softer abrasive breaks down into even smaller particles during polishing and for this ultimate stage the high cutting ability and dimensional stability of diamond would actually prove a disadvantage. This final polishing, which normally only takes a few seconds, is continued until no scratches or surface deformations are visible at 3000X magnification under an electron or optical microscope.

What the scientists have learnt

Analysis of the lunar rocks and dust brought back by the Apollo 11 and 12 astronauts, based on a study of diamond sawn and polished specimens, has shown that their chemical composition is somewhat different to that of any known earth rock. This finding has led several scientists to suggest that at least one of the three leading theories on the origin of the moon now appears no longer supportable. It may still be that the moon came from somewhere else in space and was captured by the earth's gravity, or that it and the earth were formed at about the same time and out of the same matter. But the notable difference in moon and earth chemistry seem to rule out the third theory that the moon is a fragment that ripped off the earth when the earth was young. Further more, there has been no evidence in the lunar material of life - past or present - or of water.

Physicists have studied tracks made by atomic particles in the minerals of the lunar samples and conclude that the lunar soil lies undisturbed for millions of years. Indeed, the moon's surface provides a record of the early history of the solar system, hitherto unavailable for the scientists to study.

The Apollo 12 samples are better documented than those from the previous historic mission but despite the valuable data already gained it is generally felt that there is no real substitute for a trained geologist in the field. A geologist astronaut will therefore probably accompany one of the future Apollo missions and for post Apollo missions.