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TECHNIQUES FOR THE OBSERVATION OF MICROMETEORITE CRATERS IN METAL SUBSTRATES UTILIZING ELECTRON MICROGRAPHIC REPLICA METHODS

by J. C. Slattery and R. Sloan

Prepared by
TRW SYSTEMS
Redondo Beach, Calif.
for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • APRIL 1967

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INTRODUCTION

On 11 January 1965, the equipment, facilities and personnel of Sloan Research Industries, Inc., were contracted for by TRW Systems to develop, refine and reduce to routine practice the techniques required for obtaining high quality electron micrographs of craters in metallic targets produced by the impact of microscopic high speed particles. It was desired that the electron micrographs be of such quality as to render the measurement of crater diameter to 2% and the depth of penetration below the original surface to 5%. Additionally, the crater profile was to be determined with sufficient accuracy to allow the measurement of crater volume to a maximum error of 10%. The impacted substrates were to be supplied by TRW and would be composed of aluminum and steel targets.

Because the program developed into one which was more complex than initially anticipated, completion date extensions were issued on 25 March 1965 and 9 August 1965.

This report represents the results of the developmental processes which have been completed to this date and is hereby respectfully submitted.

PROGRAM IN GENERAL

The program requires that craters which have been formed in metal substrates by hypervelocity particles, ranging in size from 0.1 to 10μ, be reproduced so that the various crater dimensions can be accurately measured. Because the substrates into which the craters have been formed are far too massive to be inserted directly into the electron microscope, a suitable thin film replica technique must be developed which will accurately reproduce the crater characteristics. When the substrate is composed of aluminum and the projectile composed of iron, the crater depths are in all cases equal to or greater than the crater diameters. This has presented a serious problem in the reproduction

of duplicate replicas of the same crater because of the difficulty encountered in removing the replicas from the specimens without tearing or other alterations occurring. In most cases with aluminum substrates, a degree of roughening has been encountered on the floors of the craters which has not reproducibly replicated.

When the substrates were prepared from stainless steel and impacted with iron particles, the craters formed were in no case deeper than the diameters of the craters and good replication was accomplished by thin film methods.

BASIC REPLICATION PROCESSES

The basic replication process utilized during this study requires the duplication of the target surface in a dependable thin film form, which is of sufficient electron transparency to be viewed in the electron microscope. Fundamentally the process is carried out in six steps.

1. A dilute solution of some plastic material is applied on the specimen and allowed to dry.
2. The plastic replica is removed from the specimen.
3. A coating of a metal is vacuum evaporated on the plastic replica at a predetermined angle (this is the shadowing step).
4. The preshadowed plastic replica is re-replicated by evaporating a material which will be electron transparent and will form a continuous film over the plastic replica.
5. The plastic replica is dissolved.
6. The remaining preshadowed - atomic replica is mounted on an electron microscope viewing grid in such a manner that the craters of interest

will be positioned in the holes and not behind the bars of the grids.

The above outline is basic as there are several possibilities for the selection of materials for use in each step. During this study, most of these possibilities were attempted and specific details will be later discussed.

At this stage it is probably well to define an atomic replica as the expression will be used throughout this discussion. Plastic replicas, as formed from fax film, polystyrene, formvar or parlodian, posses a molecular structure of their own and hence are termed Molecular Replicas. They can be used to reveal all but the finest of structures. Finer detail can be recorded with a second type of replica, conveniently termed an Atomic Replica, and which is prepared by the vacuum deposition of metals or simple compounds of low molecular weight. Our experience has been with that of the molecular replicas; parlodian has less of a gross structure than the other mentioned plastics and can be used to illustrate details at least as small as 50 Å. Naturally if an atomic replica is evaporated upon a molecular replica, the resolution will be no better than that recorded by the molecular replica. This whole discussion of the resolving power of the various replicas is rather academic with reference to the crater study however, because resolutions on the order of 200 Å are quite satisfactory for the study.

SPECIFIC REPLICATION PROCESSES

There are two basic types of replication processes. Both terminate with the microscopist having a preshadowed atomic replica available for viewing. Both types will be discussed.

Direct Replication

Direct replication involved the evaporation of the replicating material directly on the specimen surface, removing this replica from the specimen, shadowing the replica and mounting

for viewing.

In this study, direct replicas prepared from evaporated carbon and evaporated silicon monoxide were attempted. These are the two most commonly used materials for direct replication and, if success were to be had by this process, one of these materials should have produced it. No problems were encountered during the evaporations which were all performed at vacuums of 0.8 microns or less and at rates which were as high as possible, commensurate with the maintaining of the vacuum in the 0.8 micron range. No accurate replica thickness measurements were made, but by varying the evaporation time, replicas were separately formed from very thin to very thick. It is important to realize that the maintaining of extremely low vacuums during the replication process is not a desirable condition. When forming evaporated replicas, one does not wish to establish a situation where the mean free path of the evaporated material is greater than the distance between the evaporation source and the specimen upon which the replica is being deposited. It is not only desirable but quite necessary that the evaporant be able to bend around corners and continuously coat all surfaces of the specimen.

The next step is to remove the replica from the specimen without rupturing it or otherwise disturbing its detail. The most satisfactory method for performing this operation is to dissolve the specimen away from the replica. Unfortunately, this is not a practice which can be used during this study because one would not be able to produce duplicate replicas if the specimens were destroyed and no judgment of replica integrity could be made. The remaining method of replica removal involves the placement of the specimen containing the replica in a dish containing a liquid of sufficiently low surface tension to permit its getting between the replica and the specimen and thereby floating the replica free. The usual liquid for this type of separation is water or water to which has been added small amounts of wetting agents.

These processes were attempted with the carbon and SiO

replicas using water and water plus 0.1% of Eastman Chemical Co. "Photo-Flow" as a wetting agent. In both cases the replicas floated free from the aluminum surfaces, but on close optical microscopic examination of the replicas, it was learned that the replicas had been removed in all areas of the specimens except where the craters existed. In these areas the replicas contained holes. Undoubtedly, the craters possessed a surface of sufficient undercuts to hold the replica while the coating on the smooth surfaces was breaking free.

A final possibility for replica removal which has been claimed by some authors to produce reasonably good results is one which involves the evaporation of a water soluble, surface active agent onto the surface of the specimen before the replica is formed and then after replication, this parting agent is dissolved from the replica-parting agent-specimen sandwich and the replica floats free. A product of the Victor Chemical Company named Victawet is a parting agent of the type previously described. It is water soluble, can be vacuum deposited and can be deposited in essentially monomolecular films.

The use of Victawet as a replica parting agent is discussed in detail by W. H. Bridges and E. L. Long, Jr.* and it is mentioned that two rather small but essential points are required for the successful use of the material as a replica parting agent. The amount of material evaporated by these authors was between 0.5 and 1.0 mg. per evaporation. The evaporated film required baking by raising the evaporation filament to incandescence for a minute or so following the evaporation of the Victawet. Such treatment was thought to align the molecules. The second important step involved a change in the filament design. It was mentioned that Victawet sputtered badly from basket filaments and a flat pancake filament made from five or six turns of 26

*Symposium on Advances in Electron Metallography, A.S.T.M. Special Technical Publication No. 245, A Technique for Easy Removal of Direct Replicas For Electron Microscopy, W. H. Bridges and E. L. Long, Jr., June, 1958.

gage tungsten wire solved this problem. The authors indicated that no artifacts were encountered through the use of Victawet, but other microscopists have indicated that artifacts can and are routinely encountered when this material is used as a parting agent. The particular Victawet used by Bridges and Long was Victawet 35B.

Replicas were prepared using Victawet as a parting agent prior to the atomic replication process but no greater degree of successful removal was achieved when replica-sample separation was attempted than when the separations were attempted without the Victawet. This must indicate that deep structure and undercuts are locking the extremely thin film replicas into the craters.

As a final attempt at direct replication, it was theorized that, if the evaporated atomic replica could be backed up with a sufficiently rigid material, which could later be removed, a greater degree of success might be encountered during the removal process. The atomic replicas of carbon and SiO were separately packed up with heavy films of parlodian (cellulose acetate) and a benzene soluble wax prior to their removal from the specimens. Removal was accomplished by floating free in water containing 0.1% Photoflow, but again holes were present in the replicas where craters should have been.

At this point, direct replication was discontinued and judged not to be applicable to the specimen conditions. A more productive, intermediate replica process was initiated.

Summary of Direct Replication Methods

If the method were applicable to the specimen conditions, the most accurate replicas would be produced in these manners than by other processes. When dealing with specimens containing an infinite number of craters, an occasional crater can be found to be intact as produced by the direct method. Mostof the crater replicas remain in the specimen and the master replica contains holes where the craters once were. This process is first of all

too unreliable from the standpoint of replica production to be used on single crater targets and secondly, even if the process would produce usable replicas, 50% of the time, it is doubtful whether or not it would be satisfactory for single crater targets because of the problems associated with the location of the replica of the crater in an open area on the grid as related to the specimen-replica parting operation.

Indirect Replication

The expression indirect replication implies that an intermediate molecular replica is cast on the specimen and that the final viewing replica is cast from this intermediate replica. The advantage of this method is that the intermediate replica can be made from a castable and subsequently readily soluble plastic material and that stripping or liquid separation of the atomic replica is not required. The plastic intermediate is merely dissolved from the atomic replica after shadowing. The success of this process depends entirely upon the degree of perfection with which the plastic intermediate replica is removed from the specimen without the formation of artifacts or distortions. This condition can relatively easily be judged, since the master specimen is not destroyed and successive replicas can be cast for comparison purposes.

The plastic materials which are most commonly used for intermediate replicas are as follows:

1. Parlodian - cellulose nitrate, soluble in amyl acetate, has good dimensional stability during removal and is quite thermally stable during shadowing and atomic replication. For thin replicas use from a 3% solution in amyl acetate; for thick replicas use from a 10% solution. Mallincrodt stock is the best for use for this purpose. Non-flexible collodian, available in most drug stores, appears to be

identical with parlodian. However, it does not form satisfactory replicas and should not be used.

2. Faxfilm - a replicating tape which is first softened by a acetone and then pressed into the surface to be replicated. Dries quickly and is acceptably stable from a thermal standpoint providing gross mistreatment is not present. The dissolving nature of the tape is first one of swelling and then one of dissolving. As the intermediate replica is quite thick (0.010 - 0.020") it is quite necessary to get acetone to the atomic replica-faxfilm replica interface as quickly as possible during the film dissolving process to break this bond before the swelling of the film replica ruptures the atomic replica. A satisfactory brand of this material is available from Ladd Research Industries, Burlington, Vermont, and is sold as Replicating Tape No. 300.

3. Polystyrene - good quality plastic replicas can be formed from polystyrene which has either been painted onto the specimen from a solution, or thermally pressed into the sample. In any event, the final intermediate replica is a hardened film of polystyrene on the desired surface of the sample. This replica is dry stripped from the specimen and is considerably less thermally stable in the atmosphere of the shadowing and atomic replication than either parlodian or faxfilm. Care must be taken not to overheat it and alter its shape.

Polystyrene is best dissolved in benzene, as the dissolving process in this solvent appears to be completely devoid of swelling, at least as far as this process is concerned. The final replica sandwich is made up of a thick (0.05") styrene replica and shadowing plus an atomic replica. The styrne replica dissolves slowly and any attempt to hasten the process will most usually be met with total rupturing of the desired atomic replica. If styrene is to be used, one must be patient. A satisfactory source of clean polystyrene in solution form is Walsco Electronics, Polystyrene and Coil Dope No. 57-02. This is available from any electronic parts supply store and has in the past been referred to as Q Dope. The styrene dope is of the proper consistency to be used as it comes in the bottle and, if it is desired to use it in the hardened form, some very clean blanks can be cast by allowing small portions of the solution to dry overnight on clean microscope slides. To form replicas with the hardened blanks, it is only necessary to cover the desired area of replication with one of the blanks, place the specimen and blank between two supporting smooth metal plates, and clamp the entire array with a spring "glue clamp." This clamp looks very much like a large metal clothes pin and can be found in most hardware stores. The clamped array is placed approximately 6 inches away from an infrared heat lamp for around 8 minutes and then allowed to cool to room temperature or below, before the clamp is removed. From this point on the replica is treated as described above.

4. Formvar - Formvar is the trade name of a polyvinyl formal resin produced by Shawinigan Resins Corporation of Springfield, Massachusetts. Formvar is soluble in ethylene dichloride and is used much the same as is parlodian. The solutions must be kept fresh. This laboratory does not recommend the use of formvar replicas as we do not believe that they are dimensionally stable during removal from specimens and consequently, dimensionally accurate replicas are not formed.

Of the four plastics available for intermediate replica production, this laboratory has chosen parlodian as the most suitable for the job. The reasons for this choice will become obvious later.

Intermediate Replica Removal Process

One of the most important, if not the most important, steps in the production of replicas which accurately reproduce the specimen surface is the removal of the replica from the specimen surface. This is the step where undercuts will tear out, distortion can take place and artifacts can be introduced. Faxfilm and polystyrene can only be removed from the samples by carefully lifting an edge and then gently stripping the replica away from the specimen in a dry condition. Parlodian is easily separated from the specimen targets with the aid of cellulose tape, if moisture from the breath is first condensed upon the replica surface. A parlodian replica can also be floated free from the specimen surface on a water layer, but this technique is not desirable because of the possibility of contamination of the replica from the water. Formvar replicas, as far as we have experienced, can only be removed from specimens in thin film form by floating off on water surfaces.

Shadowing Metals

The microscopist is not severely limited in the choice

of a shadowing metal for this study. Naturally the low melting and light metals, such as silver, gold and aluminum, should be avoided. The most precise work probably should be shadowed with platinum, but because the magnifications and resolution required in the crater analysis are not the most demanding of the capabilities of the microscopist of his equipment, the use of platinum for shadowing appears to be a waste of both time and funds. Quite satisfactory shadowing can be achieved with chromium, but probably the best all around metal for this task is germanium, as it evaporates at a relatively low temperature, and does not possess any evidence of granulation at the magnifications required for this work. The shadowing angle is best judged depending upon the depth of the crater encountered. An angle of 45^{o} is usable for most work on the aluminum targets, but a somewhat shallower angle is required for the stainless steel targets because the depth of penetration of the projectiles is much less in this material than in aluminum. A $22\ 1/2^{o}$ angle, or half of the customary 45^{o} angle should be quite satisfactory on these samples.

The thickness of the shadowing layer is best judged by the individual microscopist and the contrast producing ability of his electron microscope. Care must be exercised to deposit enough material to produce the desired contrast in the specimen, but the evaporation should be terminated before so much material has been deposited that the fine detail will be filled over. If the specimens are placed on microscope slides and the slides are placed across the table in the vacuum evaporator so that the edge of the table can be seen through the slide containing the specimens, then the shadowing evaporation should be continued to the point where the operator, through a clean point in the evaporator bell jar, just loses sight of the table edge because of the build up of the thickness of the evaporated shadowing film on the slide containing the grids. The evaporated metal is held in a 20 gauge tungsten wire basket and evaporated downward onto the samples.

Atomic Replicas

The microscopist has three choices for the use of materials for the formation of the final viewing or atomic replicas. These materials are carbon, silicon monoxide and lithium fluoride. Either carbon or SiO will give quite satisfactory results when properly evaporated and the choice of which to use is left up to the individual microscopist. Lithium Fluoride should be avoided because even though it evaporates more readily than either carbon or SiO and does form a continuous film, it forms quite a granular film which recrystallizes easily under the influence of the electron beam, producing quite coarse replicas which are not satisfactory for the crater task.

Carbon is available in electrode rod form and can be evaporated easily either up or down. SiO is usually available only as powder or fine pellets and is rarely evaporated except from a boat electrode with the main vapor species projecting upward from the boat to the specimen grids. This laboratory prefers to use carbon for the atomic replica, because of the ease of forming the electrodes for evaporation, the availability of material of reproducible purity, and the convenience of being able to perform the evaporation with the specimen grids sitting on a table rather than being mounted upside down in a fixture held inverted in the bell jar.

Much discussion about special cutters for forming carbon electrodes can be found in the literature. Also much discussion about the importance of precisely the correct amount of evaporation can be found. We find that what may be quite usable for one type of sample, may be totally useless for other types of samples. This laboratory forms its carbon rods for evaporation by pointing them on a grinding wheel approximately one inch of the tip of a 1/8" National Spectrographic carbon electrode. We use the electrodes which are 12 inches in length and break our pieces from these longer rods. These are not the super pure electrodes and are quite suitable for this job. The pointed

electrode is held in contact by spring tension to a blunt end of a 1/4" carbon electrode. This arrangement is located 6" directly above the sample holding table. There is no definite manner for describing how long to evaporate to obtain a satisfactory film which will hold together when the plastic intermediate replica is dissolved. These points can, however, be used as guides. The heat is brought up slowly and continuously on the electrodes by variac control to out-gas the electrodes. Once the evaporation begins (as noticed by visable sparks emanating from the carbon electrode junction) the power should be turned on full and the evaporation continued for approximately two seconds. You will not know whether or not you have produced a satisfactory replica film until you begin to dissolve the plastic replica. At this stage one of three things will occur. If in dissolving the plastic film the carbon replica breaks up into many small pieces, it is too thin. If in dissolving the plastic film the carbon replica rolls up as quickly as it separates from the plastic, it is too thick. Naturally, if the plastic replica dissolves and the carbon replica floats free as a sheet--you are an extremely fortunate person as you have hit upon the proper conditions for making a carbon replica of the sample upon which you are working.

Dissolving the Intermediate Replica

There are two basic methods for dissolving the plastic replicas. The method used is dictated by the plastic used to form the intermediate and the thickness of the intermediate. The following are fairly hard and fast rules and should not be deviated from unless the microscopist has good reason to believe that they are unsatisfactory for the particular application.

For Polystyrene and Faxfilm Replicas: Cut a small piece (about 1/8 x 1/8" or smaller) which contains the area of interest from the master replica and place this piece in a depression in a multicavity Kline Test plate (Van Waters and Rogers catalog

no. 48348), add the appropriate solvent for the plastic and be ready quickly to pick off the carbon replica with fine forceps as soon as it is freed from the plastic intermediate. When you have floated the replica free, grasp a viewing grid by its edge with forceps and pick up the floating replica on the grid. Place the grid containing the replica on a small filter paper and wash the replica free of remaining traces of plastic by saturating the filter paper with the appropriate solvent (benzene for styrene and acetone for faxfilm), letting this solvent leach the remaining plastic off of the carbon replica. Now you can either take a chance that your crater is positioned over a grid opening, or you can attempt to locate it over a grid opening if, through distinguishing features on the sample, you have been able to reference its location. Good optical microscopy is invaluable here, as it is much easier to spend an additional 30 minutes in properly locating the crater in a grid opening than to take your chances in the microscope and, upon finding that the crater is located behind a grid wire, begin the replication process over again. Once a grid has been placed in the microscope and viewed, the chances of either removing any additional plastic by repeated solvent washing, or relocating the replica on the grid, are extremely slim if not zero.

For Parlodian Replicas: As will be later described in the precise process which is used, the parlodian replica is mounted on the grid in proper position before the intermediate replica is dissolved. To dissolve the replica, place the grid (one at a time, please) on a dry filter paper and add amyl acetate to the filter paper edges. As the paper is saturated with the solvent, the solvent will be drawn to the specimen and will dissolve the plastic intermediate from below, without ever wetting the carbon surface of the replica. After several careful washings of this type, the replica should be dried and examined under a low power (25X) microscope for evidence of interference colors on the replica. If interference colors can be seen, it

is a sure sign that residual parlodian is still on the replica and washing should be continued. Care should be taken not to let the replica float free from the grids. It has been prepositioned on the grid and above all you should desire to protect this positioning because it is this feature which has probably influenced you strongly in using the parlodian intermediate replica method.

Viewing Grids

There are several important criteria involved in making the proper selection of the viewing grid for this task. First of all and most important, the grid used must have a central reference opening from which any other opening present on the grid may be located. Secondly, the grid should have as great an open area as possible to permit ease of locating the desired areas in open areas of the grids and not behind support wires. Next the grid should have a large enough area of support wire to keep the extremely thin shadowed carbon replica from sagging and rupturing during the solvation processes. Lastly, in the case of parlodian replicas, where the replica is prepositioned on a grid and stripped against a grid, the grid should be made of a sturdy metal which will not bend as it is being removed from the tape.

After working with this crater system for several years now, we have limited the microscopist's choice of grids to four. Which one he uses again depends upon the type of intermediate replica formed and his own dexterity. Three of these grids are supplied by the E. F. Fullam Company in Schenectady, New York, and are as follows; the fourth mentioned grid must be self-manufactured, or purchased from a model airplane hobby store.

E. F. Fullam No. 2200 - 200 mesh copper, 65% open area.

E. F. Fullam No. 2202 - 75 x 300 mesh titanium, 56% open area.

E. F. Fullam No. 2105 - 100 mesh hexagonal copper, 73% open.

Sequins - Sequins may be made from 0.005" brass sheet having a O.D. of 1/8" and an I.D. no greater than 0.030". This type of washer is available from most hobby shops which feature model airplane supplies and is the propellar thrust washer used on indoor, rubber powered models.

The relative merits of the various grids are as follows. The No. 2202 grid is a must for use whenever the replica is to be taken off of the sample against cellophane tape. It is the only grid which has enough dimensional stability to resist bending when it is removed from the tape. The grid openings are oblong, but a large central opening is available and is ideal for use on single crater targets. No. 2200 is the best all purpose grid in the group. It can be used whenever the intermedite replica is to be dissolved in a dish or on filter paper. Dimensional stability is not especially important here and the microscopist should find the 65% open area quite attractive. The 35% closed area is perfectly sufficient for the support of the replica. The No. 2105 hexagonal grid is new and has not been used extensively. It has a large open area, too large to permit the placement of a grid containing a replica on a smooth filter paper for drying. When this is done, the replica will sag through the grid openings and touch the filter and consequently rupture upon next pick up. If replicas are to be dried on the No. 2105 grid, they should be done on one of the indentations in a rough surface as is found on a No. 150 UHA Scott Towel. The sequin is again a device left up to the ability of the microscopist. Undoubtedly, it is the most satisfactory support which can be used, because it possesses 100% open area with no grid wires to hide the sample. Because there are no grid wires there are also no "tear stoppers" and a tear started in the replica will progress completely across the replica before it stops. A tear similarly started on a support containing grid bars will usually only progress to the next grid bar where it will stop. One should carefully assess the advantages and disadvantages of the sequin before beginning the program, because they are wonderful to work with as long as the

replicas hold together, but they are equally miserable to use when the carbon films are not of the precise correct thickness and tearing takes place.

Oxide Replicas

It would appear that one of the most satisfactory methods of replicating the craters formed in the aluminum targets would be by the anodizing process. Unless the formation of this type of replica is very carefully controlled, the final result will show massive granulation and be useless for this purpose. The process has the same disadvantage that the dissolving of the sample has in the direct replica process in that the sample is destroyed and repetitive results cannot be compared. This laboratory was not successful in preparing usable oxide replicas but feels that the method described by M. S. Hunter and F. Keller* should be a satisfactory process for forming these replicas.

Replica Positioning Methods

As mentioned several times previously in this report, the replica must be able to be accurately positioned on the viewing grid before the final judgment of the success or failure of the replication can be made. This positioning is the most singularly important step in the entire process, because if the microscopist is unable to locate the crater upon which he has been working, he will not be able to evaluate his work.

Before the microscopist can accurately position the crater over a grid opening, he must definitely know where the crater is on the target. This can sometimes be found by examination of the target by metallurgical microscopic methods. A far less painful way of locating the craters is to make a parlodian replica of the

*Symposium on Techniques for Electron Metallography, A.S.T.M. Special Technical Publication, No. 155, Techniques Used in Electron Microscopy of Aluminum Alloys by M. S. Hunter and F. Keller, June 1953.

target, strip it from the target against cellophane tape and shadow it at 45° or 22°, depending on the target material. This is a study replica only and will never find its way into the electron microscope. The replica is examined with transmitted light at 100 to 150 power in an optical microscope and the shadow detail of the crater, which through one stage replication has become a mountain, should point out where the crater exists on the target. After the crater location is mapped (remember that you are looking at a negative replica and that everything is reversed) examination of the target under 25 power stereo microscopy in the area known to contain the crater will usually pinpoint the crater on the target. The location of the crater, with reference to other more obvious marks (usually scratch patterns) on the target can eliminate much eye strain. When it comes to positioning a floating replica on a grid, the microscopist is limited only by his eyesight, patience and luck in being first able to optically find the crater and then locate it over a grid opening. The process for doing this is to optically search the grid at a 150X magnification and, if the crater cannot be found to return the grid to a filter paper. Saturate the filter with the solvent appropriate to the technique which is being used and while holding the grid firmly to the filter with a probe in one hand, move or cause the replica to be moved by either flowing enough solvent on it to float it or by physically moving it with another probe. Perform the optical observation again--and keep repeating the process until you find the crater; sometimes if you are particularly unlucky it takes quite a while.

If the parlodian intermediate replica method is used, and if the crater can be referenced to optically identifiable surface characteristics on the target, the area of the replica containing the crater can be reproducibly positioned on the grid almost every time. This method will now be described in detail, as we feel it to be the most satisfactory and usable method of the plastic intermediate replica techniques.

MICROMETEORITE CRATER REPLICATION PROCESS

It is assumed that the specimen to be replicated is a target containing a single crater and that this crater has been located by the marksman as being somewhere within a 0.050" circle which has been scribed on the target. The size of the crater is not of particular importance, but we will assume that it is at least one micron in diameter. This process from start to finish takes place in several steps. These steps, described in detail, are outlined below:

1. The target is cleaned.
2. The preliminary crater locating replica is taken and processed.
3. The crater is accurately located on the target.
4. The intermediate replica is cast in parlodian.
5. The intermediate replica is removed from the target and simultaneously located on a viewing grid.
6. The intermediate replica is shadowed and re-replicated.
7. The intermediate replica is dissolved.
8. The preshadowed viewing replica is examined optically and finally in the electron microscope.

The details of the process are as follows:

Step 1: The target is cleaned.

By far the easiest and most thorough method for cleaning the target is to cast several parlodian replicas of the target and discard them after they have been stripped from the sample. These replicas will completely remove all surface dirt and the amyl acetate solvent present in the parlodian solution will remove all fingerprints and other oily residues.

Step 2: The preliminary crater locating replica is taken and processed.

Drip several drops of the 3% parlodian-amyl acetate

solution onto the target and incline the target approximately 45° to permit the excess replicating solution to run-off. Have the base of the target resting on a filter paper or other absorbent paper so that the run-off will not puddle about the base of the target. The replica may be judged to be dry when interference colors can be seen on the replica. The microscopist now breathes on the replica on the target and immediately presses a piece of cellophane tape (for this particular replica 3 M Scotch Brand "Crystal Clear" tape works quite well) to the replica. The microscopist now gently lifts an end of the tape and the replica will easily free itself from the target and be attached to the tape. The portion of the tape containing the replica is cut from the bulk of the tape piece and taped down to a microscope slide--replica side up! This slide is now inserted into the vacuum chamber and shadowed. A shadowing angle of 45° will be quite satisfactory for all craters formed by iron pellets in aluminum targets; when iron pellets are used on stainless steel targets the craters are less deep than in aluminum and a 22° shadowing angle should be used.

Step 3: The crater is accurately located on the target

The replica process in Step 2 is observed at approximately 150 power and the crater is located. Switching the microscope up to approximately 450 power should show an annular ring surrounding the crater which represents the lip splash detail and it is this detail which will identify the crater from other structures observable on the replica. The microscopist should now reference the crater location to the 0.050" circle scribed on the target and, if at all possible, to the particular scratch patterns present on the target. It is quite important that the microscopist takes enough time at this stage to become familiar enough with each target to be able to absolutely, positively located the craters repetitively. By taking extra time here, many frustrating hours of trial and error will be saved later.

Step 4: The intermediate replica is cast in parlodian.

The intermediate replica is cast in parlodian in precisely the same way as is the crater locating replica described in Step 2.

Step 5: The intermediate replica is removed from the target and simultaneously located on the viewing grid.

An EFFA No. 2202 titanium grid is dipped into a solution of "benzene glue." Benzene glue can be prepared by dissolving the adhesive from 40 inches of 1 inch cellophane tape in 100 ml. of benzene and then withdrawing the cellophane backing from the container. "Old style" Scotch tape, which had an amber cast to it, worked quite well for this purpose. The new Scotch Clear Cast tape is not usable because the backing is no longer cellophane and dissolves as well as does the adhesive in the benzene. If the glue is to be prepared--obtain older amber tape. The grid is placed on a piece of hard filter paper and allowed to dry. This grid should be used within one minute or re-coated in the benzene glue. The grid is stuck to a piece of crystal clear tape so that the flat edge of the grid runs parallel to the edge of the tape. The crater is located under a stereo microscope on the target and, before the replica is moistened by breathing on it, the tape containing the grid is lowered over the crater in such a manner that the crater is located in the large central opening of the grid. The top edge of the tape is now stuck to the target and the remainder of the tape is folded back on the portion that is stuck. The replica is now breathed on and the tape containing the grid is pressed onto the moistened replica. Again by gradually and easily lifting an edge of the tape, the replica will easily release itself from the target.

Now take a minute to consider what you have. - First of all you have a piece of cellophane tape, stuck to the sticky side of this tape you have a specimen grid and stuck onto this

specimen grid by the benzene glue which dried on the bars of the grid is a parlodian replica of the crater on the target. To remove the grid containing the replica from the tape, place the tape containing the grid under the stereo microscope and carefully cut around the grid that portion of the replica which is bridging the levels from the top of the grid to the tape surface. When this bridge has been cut free, the tape can be bent back across the microscopist's finger and the grid containing the replica can be picked off with a pair of forceps. This grid is placed on a microscope slide for shadowing.

Step 6: The intermediate replica is shadowed and re-replicated.

The intermediate replica is placed in the vacuum chamber and arranged so that it is directly below and between 6 and 7 inches away from the carbon electrodes and that the shadowing basket is related to the grids at an angle of 45^{o}. If you wish to use another angle--go ahead--but be sure you know what it is. The shadowing basket is formed by winding at least four turns of 20 gauge tungsten wire about the tip of a No. 12 wood screw. Place a piece of germanium metal in the basket which is about the size of a B-B shot pellet--or larger. The main idea is to have more material in the basket than you intend to evaporate for in this manner you can cut off the evaporation as you wish, rather than having it cut itself off because the evaporant was used up.

AT THIS STAGE THE PROCESS SPLITS AND IS DEPENDENT UPON THE DEPTH OF THE CRATERS THAT ARE BEING WORKED WITH

If the craters are formed in stainless steel with iron projectiles, they will be shallow and shadowing and atomic replication may be carried out in a single step as follows. When the vacuum in the chamber has reached 0.5 microns or lower, begin the germanium evaporation and continue it until the specimen table edge is no longer visable through the microscope

slide upon which the grids are located. Immediately following the shadowing proceed with the deposition of the carbon replica. Preheat the carbon electrodes and, once the evaporation has begun, turn up to full current and continue for approximately two seconds. Cut the evaporation off sharply. When you vent the vacuum chamber, do it slowly so that the grids containing the replicas are not blown off of the specimen table.

Step 7: The intermediate replica is dissolved.

The grid containing the replica is placed on a piece of filter paper and benzene is flowed onto the filter paper and allowed to leach the adhesive from the benzene glue off of the sample. Do this carefully and the replica will not alter its position with respect to the grid openings. Place the benzene leached replica on a dry piece of filter paper until the replica is dry and then repeat the leaching operation with amyl acetate until the parlodian replica has been removed. Again--do not be in a hurry--you have spent several hours arriving at this stage and you can blow the business in five seconds if you are careless.

Step 8: The viewing replica is examined.

Observe the preshadowed, negative carbon replica under a 25 power stereo microscope and, if any traces of interference colors are visible, continue with the amyl acetate leach. If none are visible, you have probably completely removed the parlodian replica, and can examine the grid under a 150 power microscope. This examination will assure you that the crater is still located in a grid opening. If it is not--proceed with the solvent soaking and replica moving as previously described, until the crater is located above a grid opening. Finally, examine the preparation in your electron microscope.

FOR DEEP CRATERS

If the craters are formed in aluminum with iron projectiles, they will be deep and the atomic replication and the shadowing must take place in 2 separate steps.

Before the replica is shadowed, evaporate the carbon replica directly onto the parlodian replica surface. Slowly vent the vacuum chamber and separately proceed with the benzene and amyl acetate leaching steps. When these operations have been completed and the replica has been judged to be free from all traces of adhesives and plastics, the replica is re-inserted into the vacuum chamber and shadowed. Following shadowing, the replica may be viewed without further treatments.

This two step processing is necessary on the deep craters because when deep craters, or high mountains as the negative replicas represent, are shadowed, the structure is distorted in the direction of the evaporation and misleading results are produced. Examples of this will be shown in the representative electron micrographs presented at the end of the text.

CRATER CROSS SECTIONS

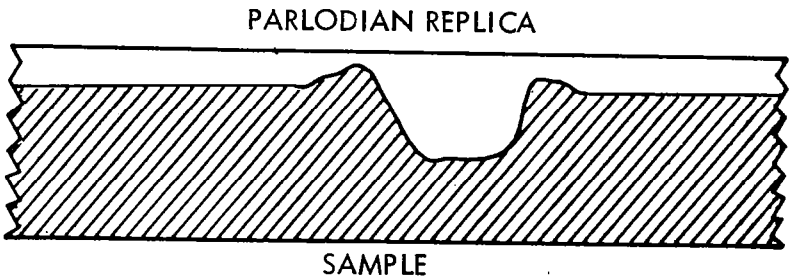
It was noticed in many of the micrographs taken from replicas of the craters, that the geometric bottom of the crater was not usually located in the geometric center of the crater. This condition caused considerable alarm and question as to the accuracy of the replicas. Because the process for duplicate replication had not been worked out at this point, it was decided to metallurgically mount several of the targets containing an infinite number of craters and see if cross sections of the craters could be observed. The targets were mounted in a transparent Epoxy and were cut and polished. The final polishing step should be performed with magnesium oxide on a wet billard cloth at the slow speed of the lap wheel. The polishing action should proceed toward the bottom of the crater. A very quick HCL etch should be applied to the surface of the specimen before replication. The parlodian-carbon replication process described in detail elsewhere in this report was used for replication of these specimens.

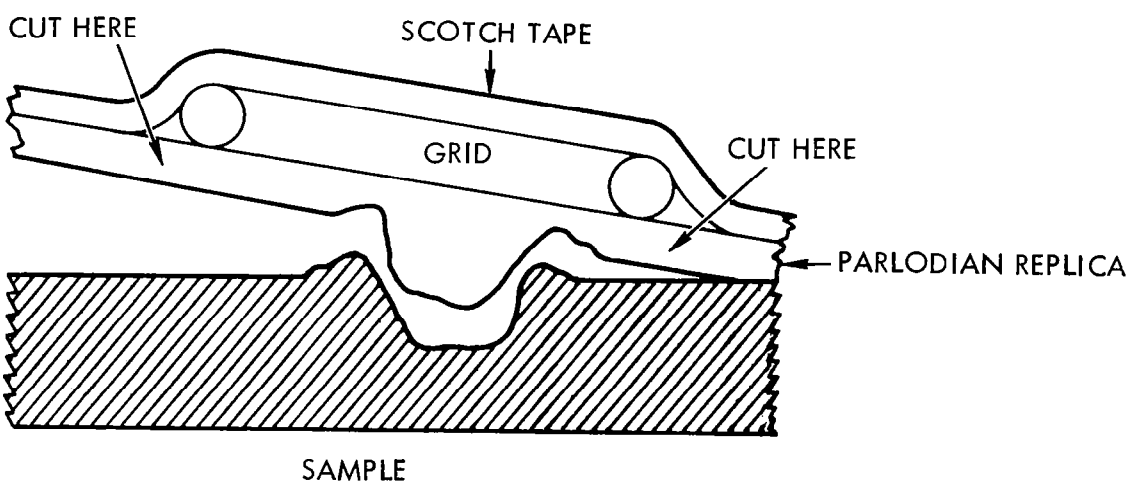
Certain evidence was present in the micrographs of the

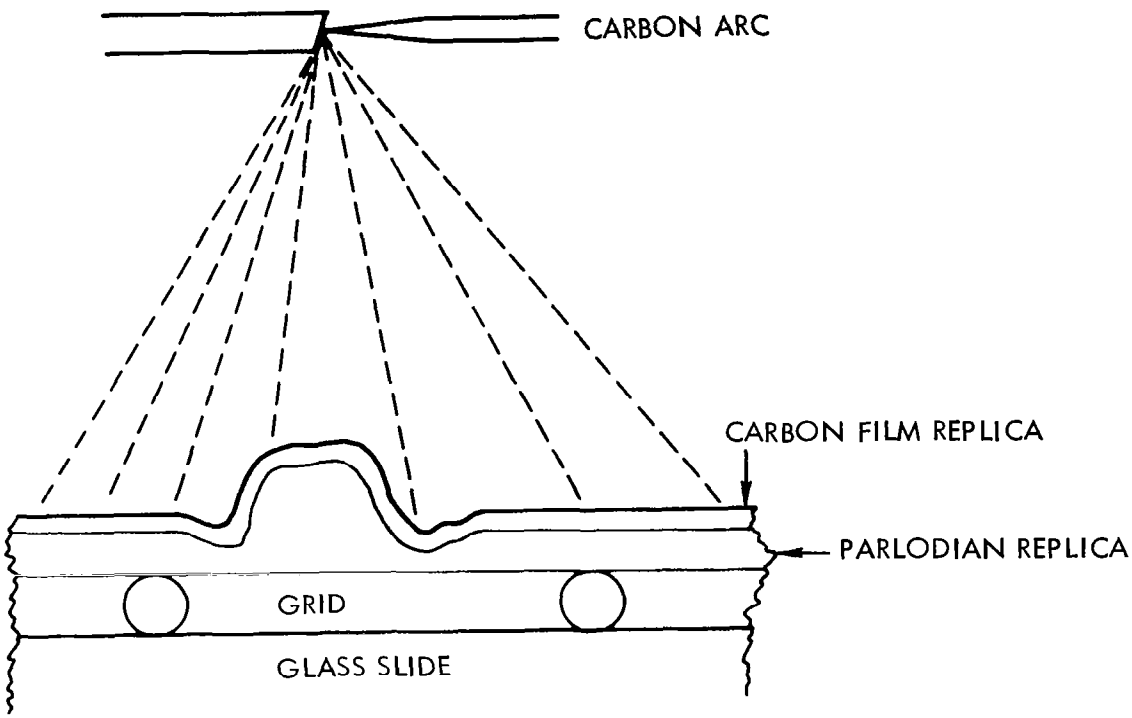
crater cross sections to make one feel that the craters were not being formed normal to the target surface, but a great deal of questions were developed as to the plane upon which the sections were taken and the extreme importance of this knowledge to the interpretation of the micrographs. It was readily realized that control over the cutting and polishing plane was only possible when the utmost of care and, consequently time, was consumed in the process. Even under the best conditions, the determination of this parameter would be an estimate rather than a certainty. It was also readily realized that the cross sectioning studies were not readily applicable to samples containing less than an infinite number of craters, and therefore further development work on the technique was not undertaken.

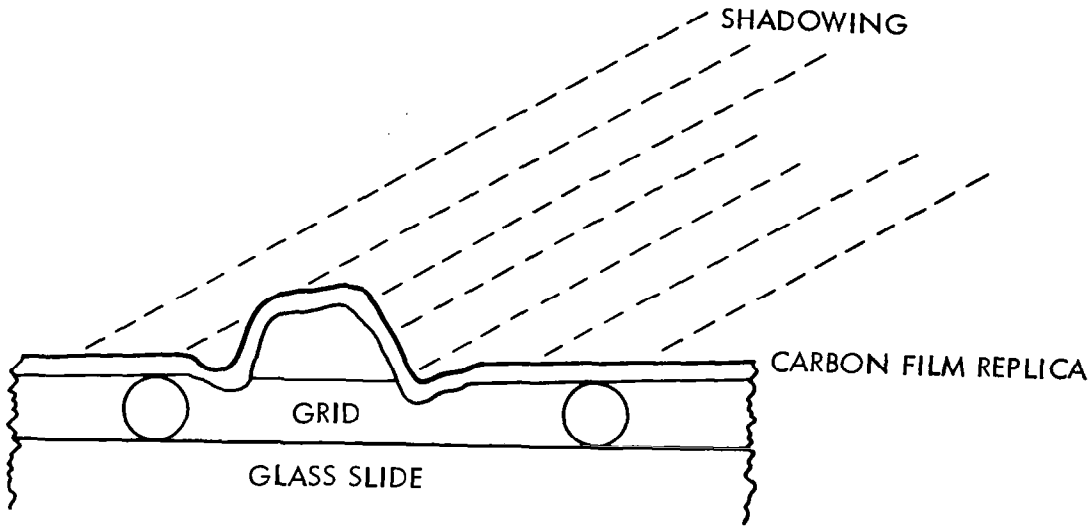
The pictures are of value, however, since they do show crater inner surface characteristics which were heretofore unobserved. These characteristics need not be related to any particular location in the crater.

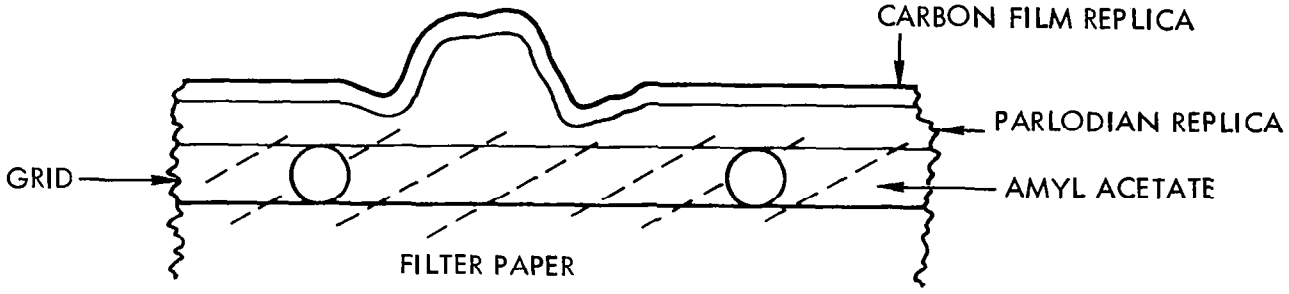
STEPS OF REPLICATION PROCESS



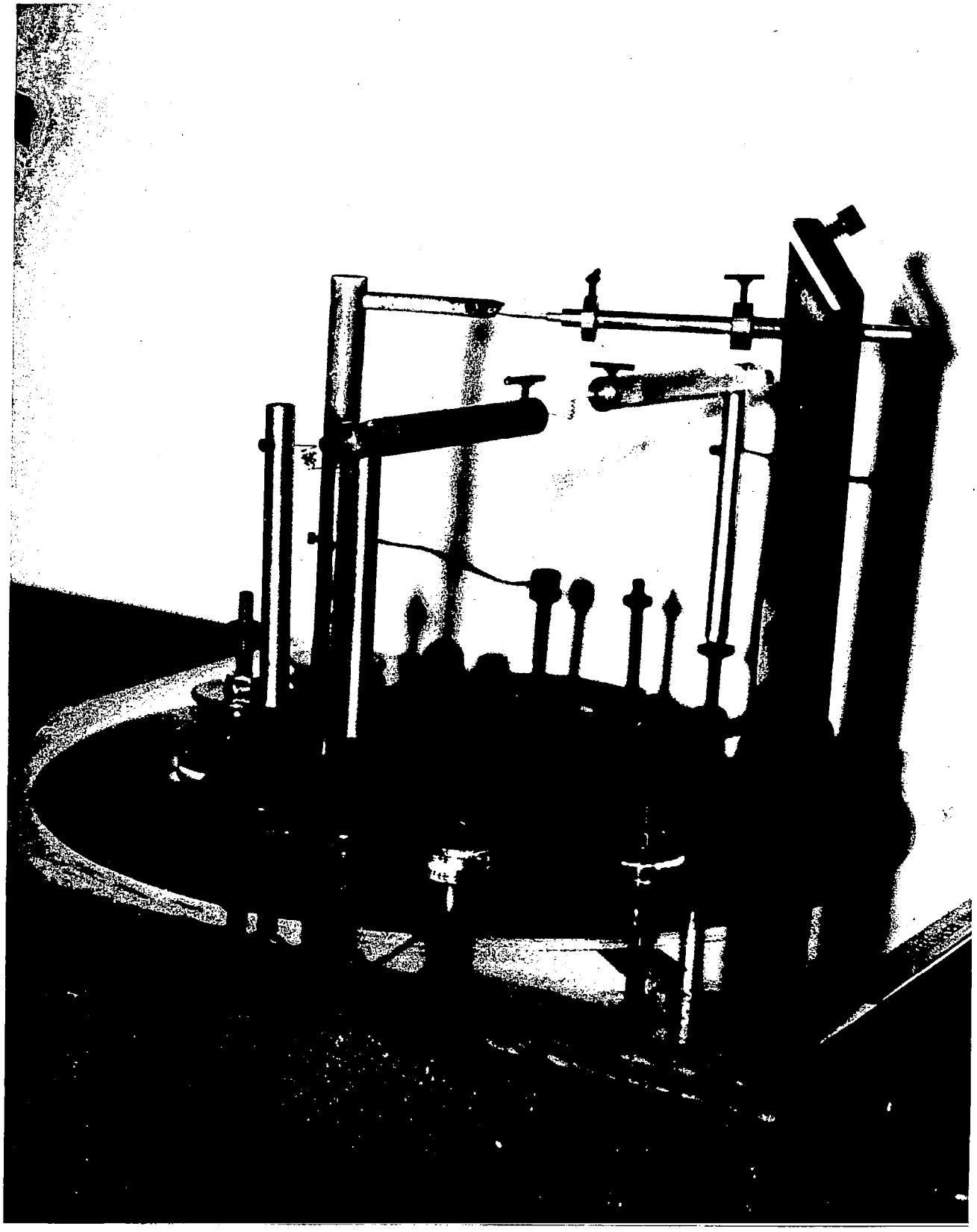


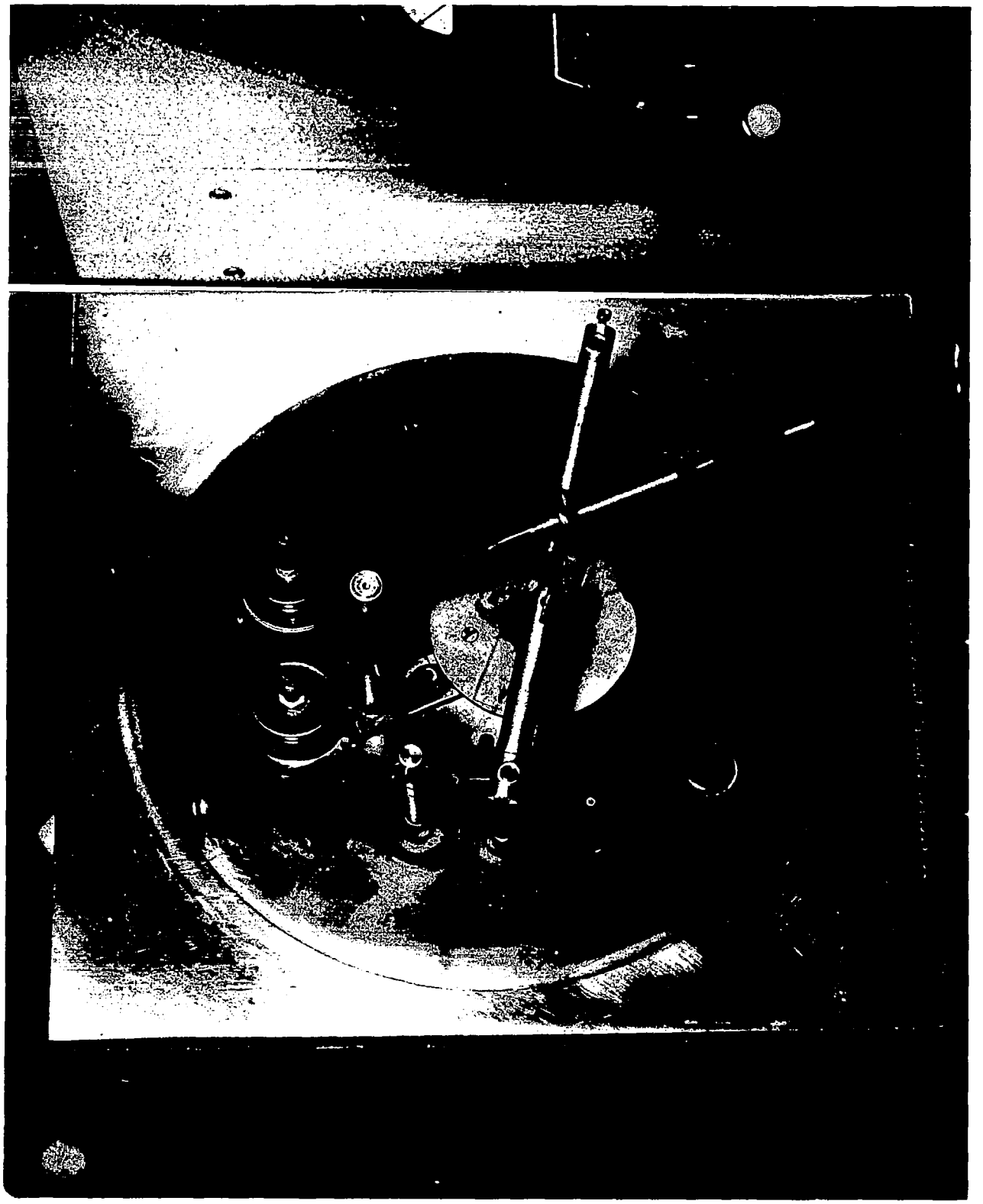

CARBON ARC
CARBON FILM REPLICA
PARLODIAN REPLICA
GRID
GLASS SLIDE


SHADOWING
CARBON FILM REPLICA
GRID
GLASS SLIDE


CARBON FILM REPLICA
PARLODIAN REPLICA
GRID
AMYL ACETATE
FILTER PAPER

On the following two pages, photographs of the arrangement of the vacuum system for victawet evaporation, shadowing and carbon evaporation are presented. The tungsten basket used for shadowing is the filament attached to the periphery electrodes and the filament basket used for the victawet evaporation is the one roughly centered over the evaporation table. The pointed carbon electrode is illustrated quite well. The large 1/2" x 1" brass bar, which serves as the holder for the sliding carbon electrode, has a notch cut in its top front edge. A watch glass slides in this notch and is held with the screw so that a clean port can be maintained in the bell jar by merely changing watch glasses rather than constant cleaning of the bell jar. Actually, it is wise to keep the bell jar well coated to act as a filter against evaporations.

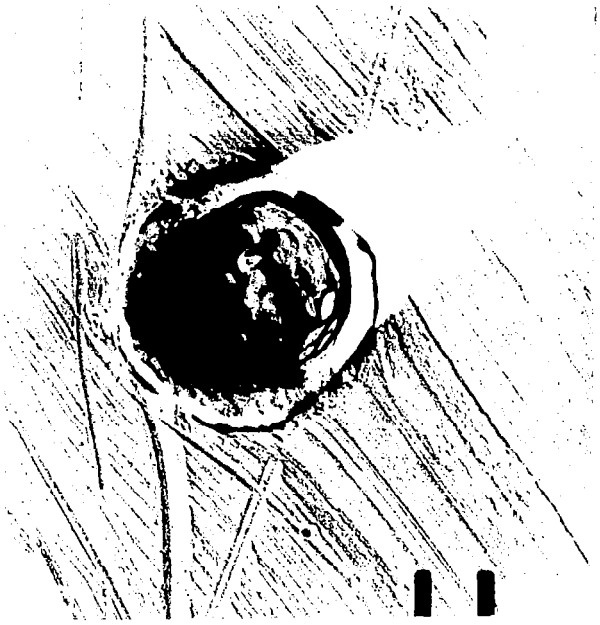




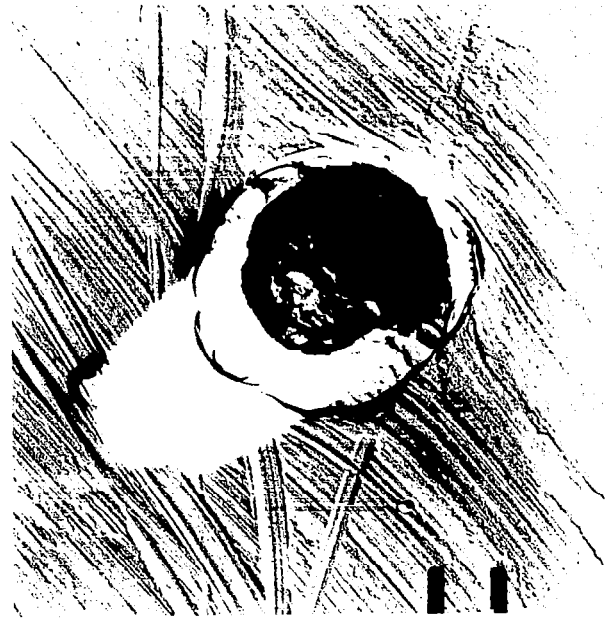
ELECTRON MICROGRAPHS

On the following pages several electron micrographs are presented which illustrate many of the more pertinent details and conditions encountered during the examination of the meteorite craters. Unless otherwise specifically indicated, the micrographs have been prepared from parlodian replicas which were shadowed at an angle of 45°. The magnification of each micrograph may be determined by measuring the distance between the inner edges of the two black marks located about the edge of each micrograph in millimeters and multiplying this value by 1000. This distance between the inner edges of the black marks represents a distance of one micron on the surface of the micrograph. The replicas were all examined and the electron micrographs prepared in a Norelco EM-100 B electron microscope, which is capable of resolving 14 $\mathring{A}$.

Electron Micrographs 1 through 4 represent attempts at the duplicate replication of a single crater impacted in an aluminum sheet from an iron projectile. The duplicates have established the diameter and depth of the crater within the desired limits of accuracy, but have been total failures with respect to reproducing the inner surface character of the crater. Image magnification = 7000.



1



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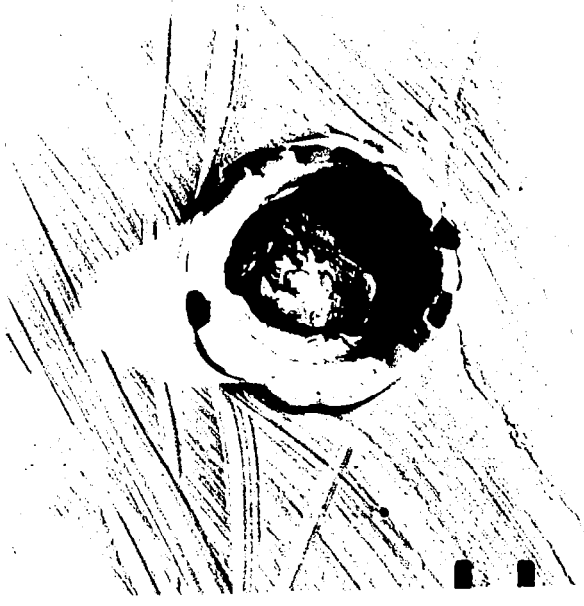

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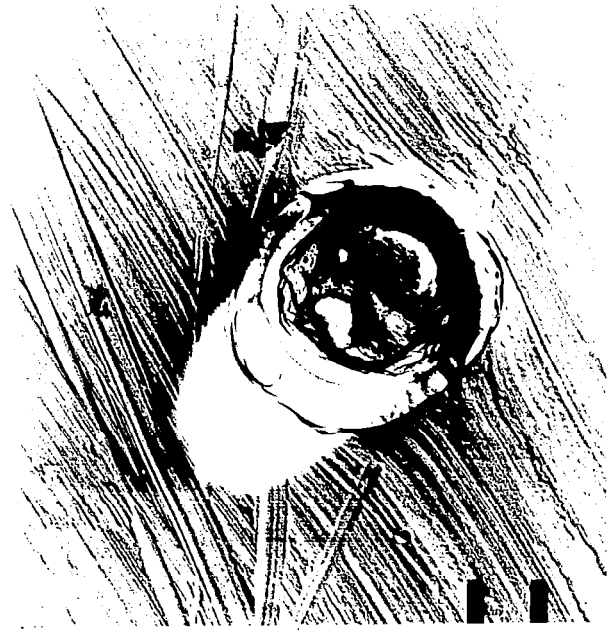
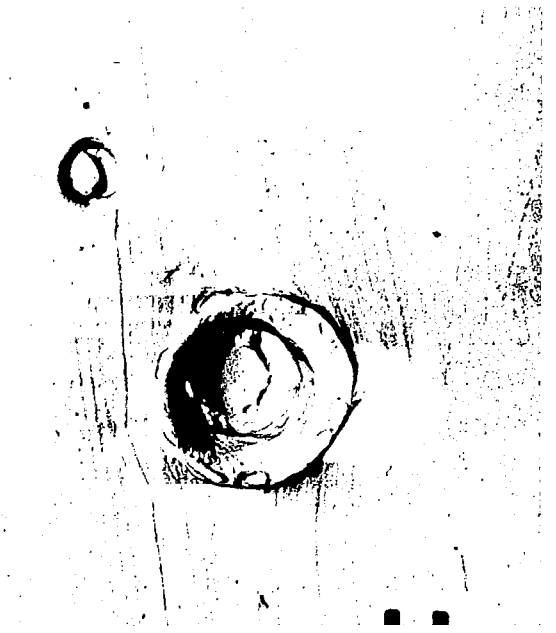


4

Electron Micrographs 5 through 8 represent the somewhat more successful attempts at preparing duplicate replicas of a single crater formed in an aluminum sheet from an iron projectile. Not only do the diameters and depths compare reasonably, but also certain surface characteristics from the inside of the crater can be followed from one micrograph to the next. Image magnification = 5000.

There have been no changes in the replication process between the duplicate replicas represented by Micrographs 1 through 4 and those represented by Micrographs 5 through 8. Only the crater area on the target was varied. In both instances, the large craters are of approximately the same depth.

5



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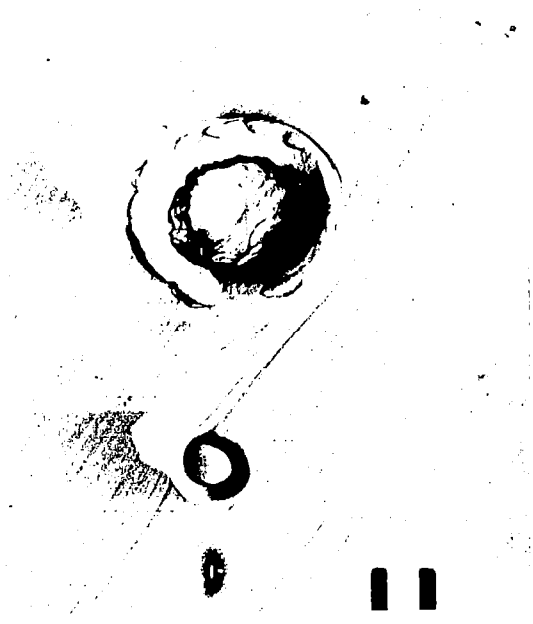

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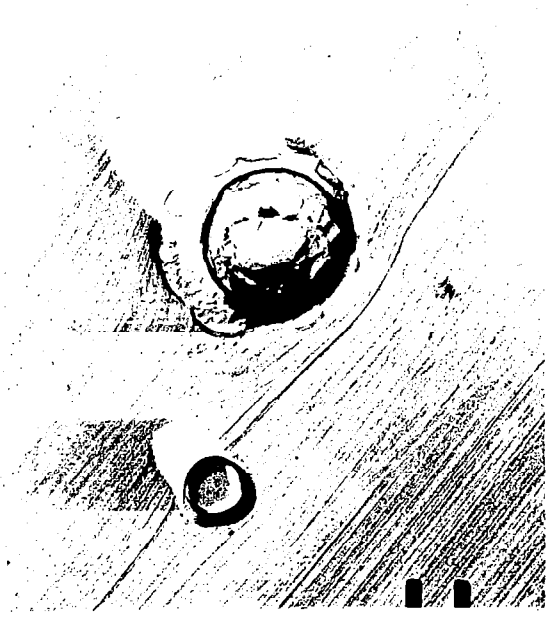
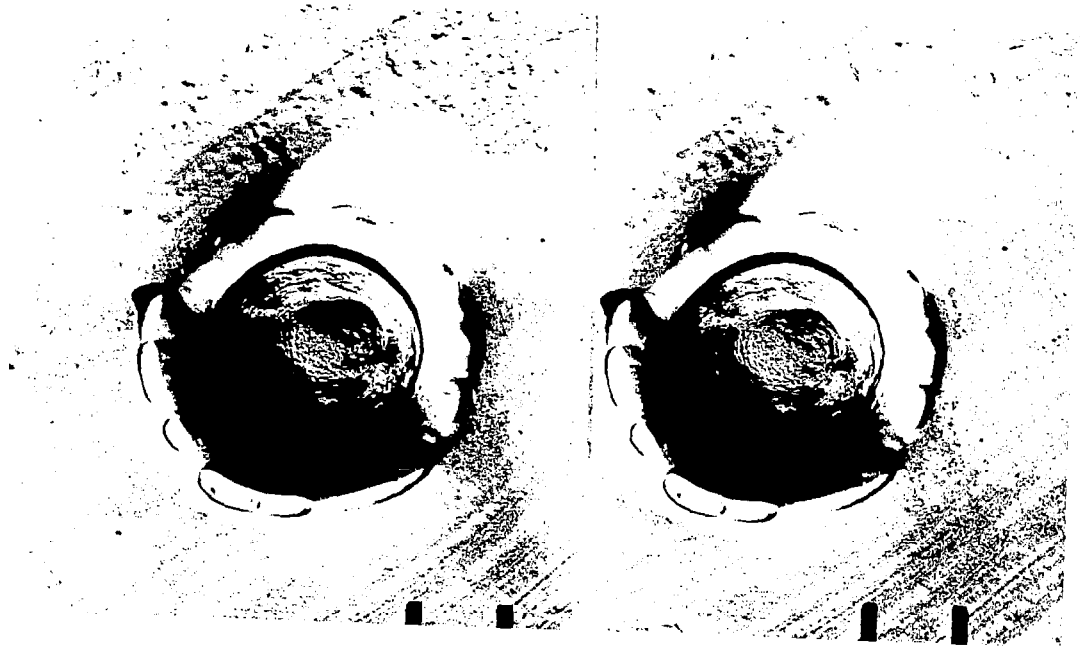


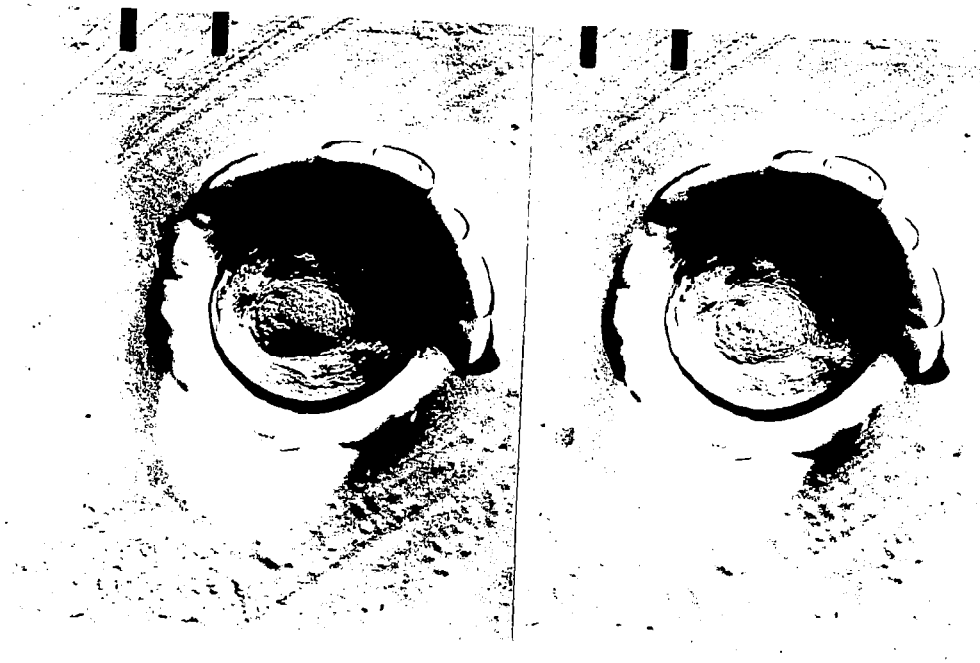
8

Electron Micrographs 9 and 10 are two stereographic sets of the same crater. They are actually duplicate prints of the same two negatives.

Micrograph 9 is mounted in correct register to illustrate the crater as it actually exists on the negative carbon replica. The crater should appear as a mountain and project out of the plane of the picture. Micrograph 10 represents the reverse mounting of the two pictures used in Micrograph 9 and represents the crater as it existed in the target. The crater should appear as a pit and project into the plane of the picture. This mounting was included merely to illustrate that topography reversal could be accomplished without going to the bother of preparing positive replicas. The crater is quite symetrical, does not have the bottom torn out of it and is believed to be one of the more representative replicas viewed. The crater was formed in aluminum foil with an iron projectile. Magnification = 12,000.

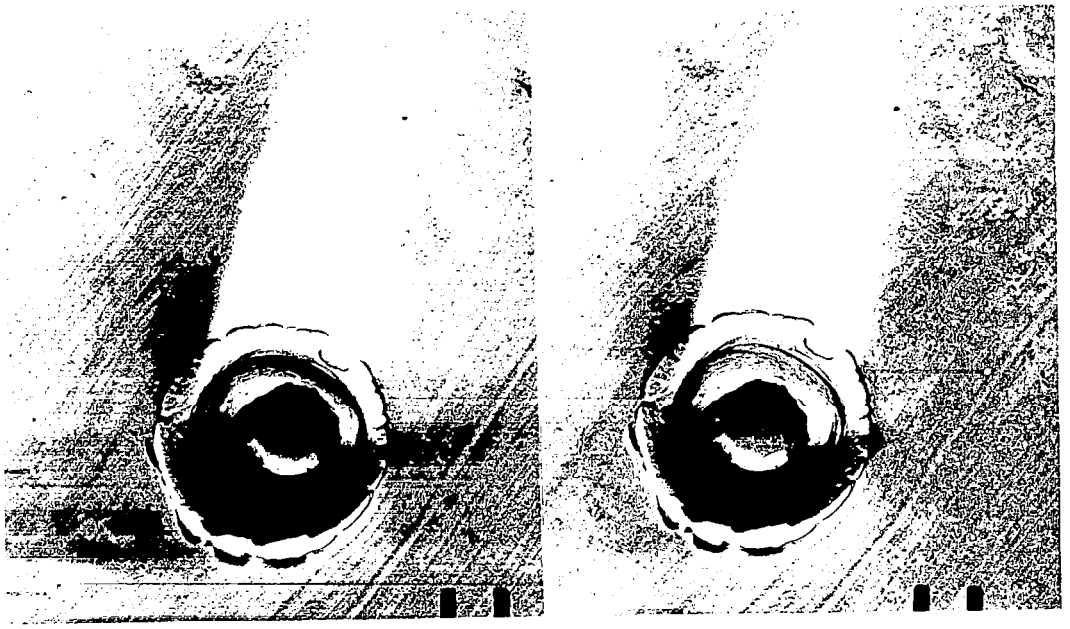


9

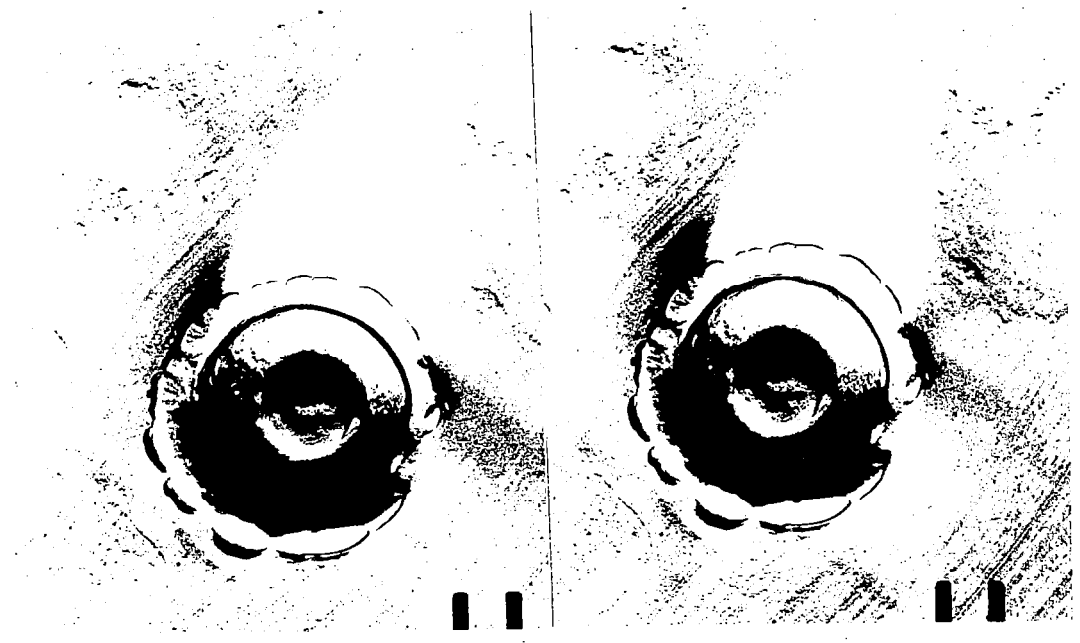


10

Electron Micrographs 11 and 12 again are stereographic pairs of craters in aluminum. It can be noticed that in the bottom of each crater is a smooth structure which has the opposite relief of the crater. Micrograph 11 illustrates the torn remains of portions of the carbon replica existing annularly around the basal smooth area. This occurrence indicates that a micro-crack was present between the smooth structure and the impacted aluminum and that some of the replicating plastic was withdrawn from the crack. Micrograph 12 does not show the crack, but does show a lip separating the smooth structure from the crater wall. A considerable amount of discussion has been centered about what this smooth structure represents and as yet no concrete evaluation has been made. Some believed that the structure represented an air bubble trapped during the replication, while others believed the structure to represent the remains of the iron projectile. The reader, at least at this stage, must be left to his own conclusions.

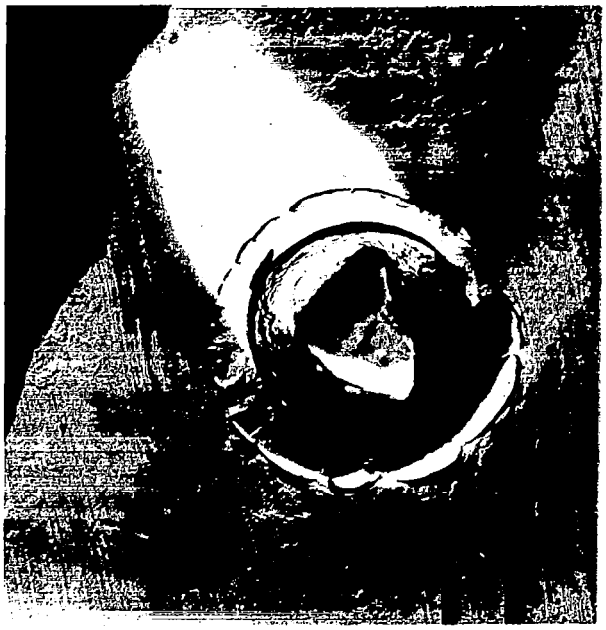


11



12

Electron Micrographs 13 through 16 represent the replicas of four craters impacted in aluminum using iron projectiles. These four craters are of particular interest in that they each have, to some degree, the smooth structure illustrated and discussed in Micrographs 11 and 12. The variation of the structure with size and shape makes the reason for its occurrence even less understandable.



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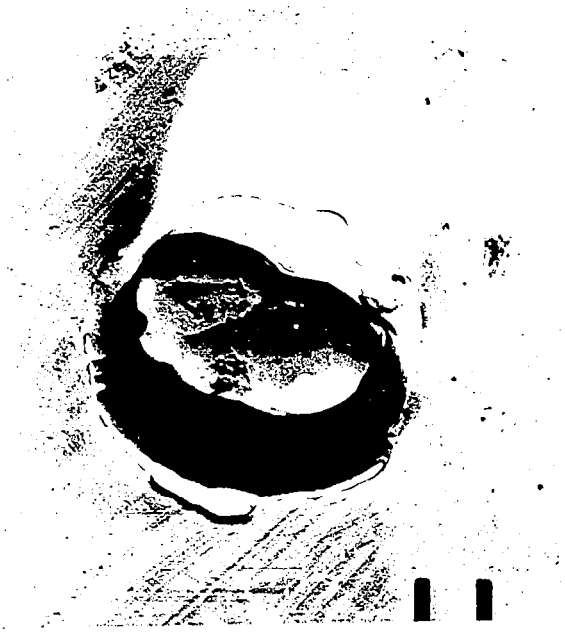

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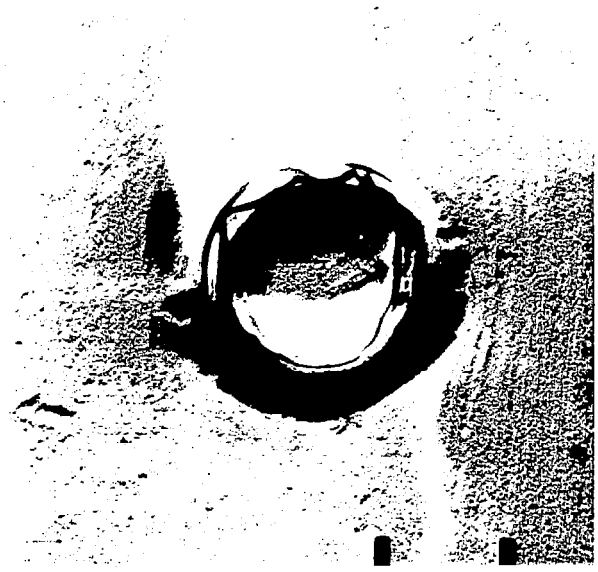
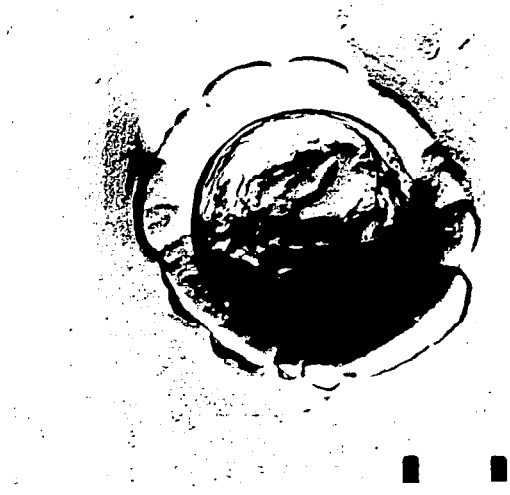


16

Electron Micrographs 17 through 20 again represent craters formed in aluminum with iron projectiles. These craters are relatively small when compared to many of the previous studies. The craters are symetrical and have unusually straight, plug-like sides. The bottom of the craters are pseudo hemispherical and are believed to be quite accurate representations of this type of cratering.

17

18



19

20

Electron Micrographs 21 through 24 are craters from iron projectiles in aluminum foil targets. Micrographs 21 and 22 have been prepared and taken from polystyrene replicas and are included to illustrate the ease with which this process picks up contamination from the various solvent transfer steps. This process is approximately three times as lengthy as the parlodian process and only about half as productive. Micrograph 23 is a comparative parlodian processed replica presented on this page for comparison with the polystyrene replicas. Finally, Micrograph 24 represents a parlodian replica which has been rotary shadowed. Specifically, the grid holding the replica was placed on the center of a table spinning at 60 rpm. while the shadowing process was taking place. As is obvious, it would be impossible to make penetration depth measurements from this micrograph as the heighth shadow is missing.

21

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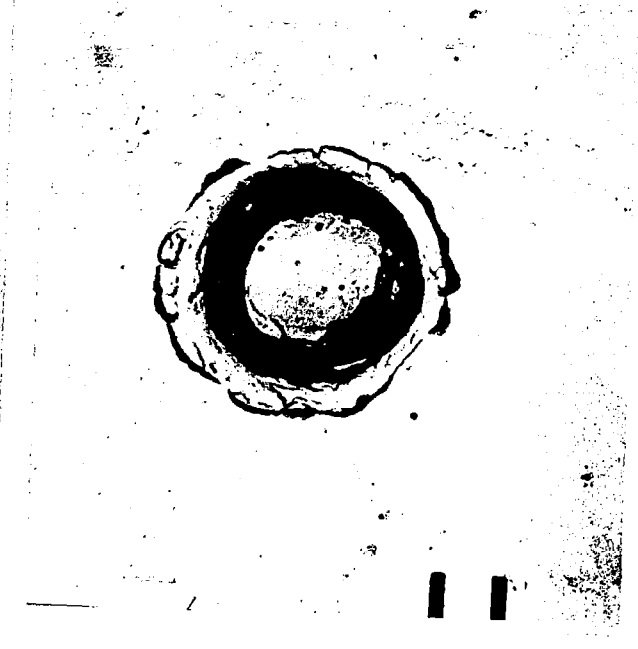
23

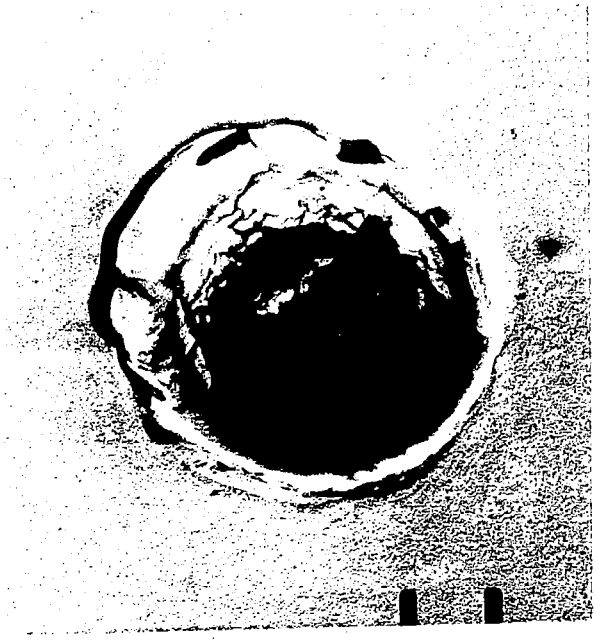


24

Electron Micrographs 25 through 28 illustrate several of the obvious artifacts which can be encountered when attempting to replicate craters made from iron projectiles in aluminum. Micrograph 25 was taken from a crater in aluminum foil and 26-27 and 28 were taken from craters impacted in electropolished aluminum sheet. Micrograph 25 illustrates the manner in which the crater can be pushed over in the direction of the shadowing if the replica is shadowed before the carbon replica is prepared and the plastic replica dissolved. Micrographs 26 through 28 illustrate several manners in which the replica of the crater body can collapse during processing of the replica.

25

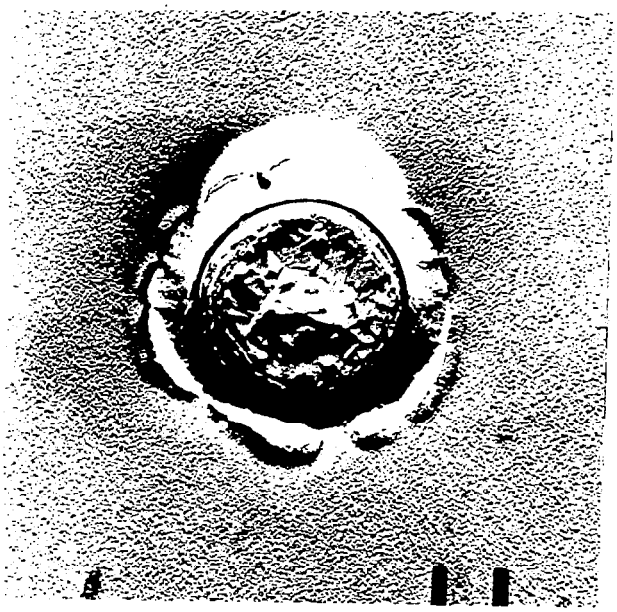
26



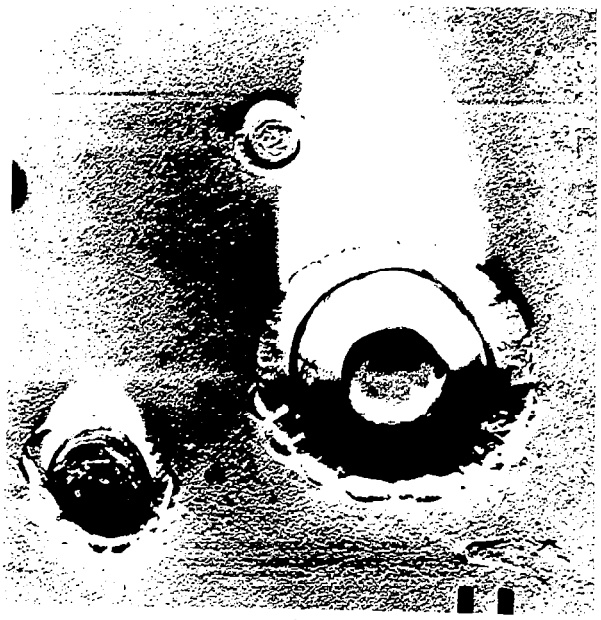
27

28

Electron Micrographs 29 through 32 were prepared from craters which had been impacted in electropolished aluminum by iron projectiles. They are included to illustrate the texture of the electropolished aluminum.



29



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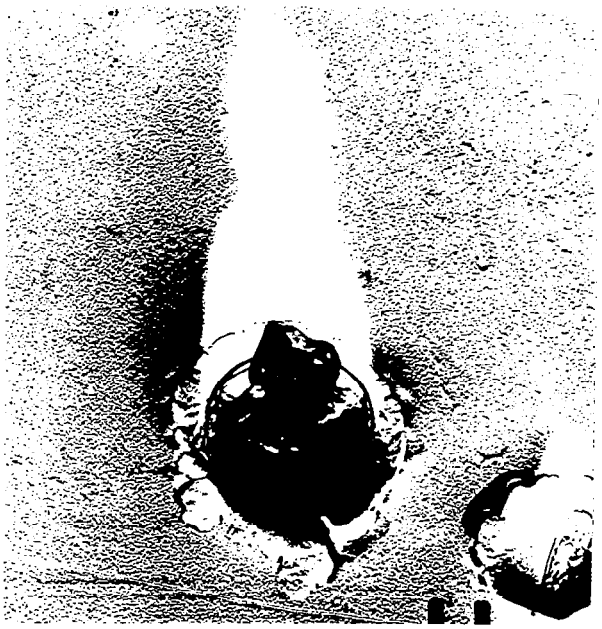

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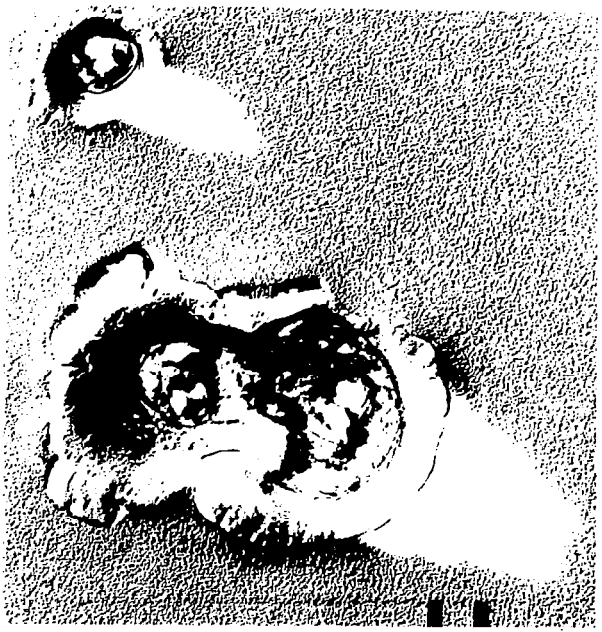
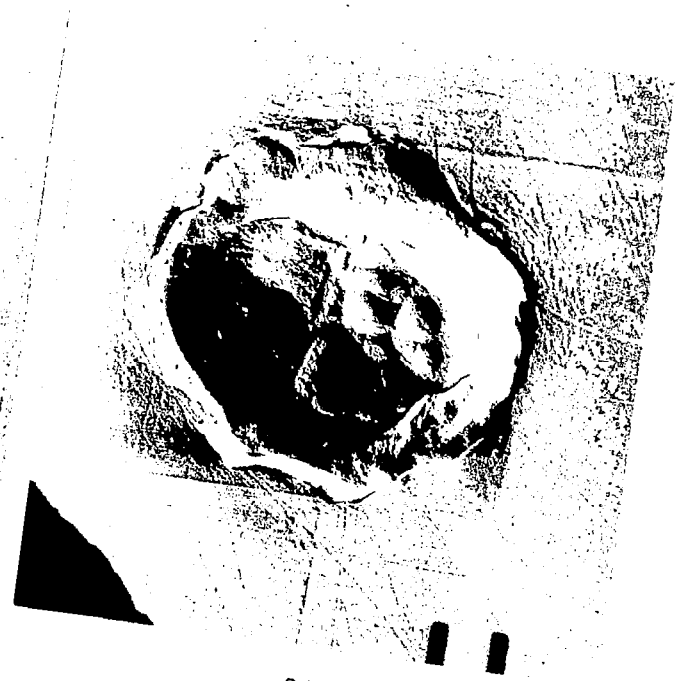


32

Electron Micrographs 33 through 36 illustrate the cratering effect that can be expected when iron particles are impacted upon stainless steel targets. As can be noticed, the cratering is much shallower than in aluminum and that the floor of the craters takes on quite strange contours.

33



34

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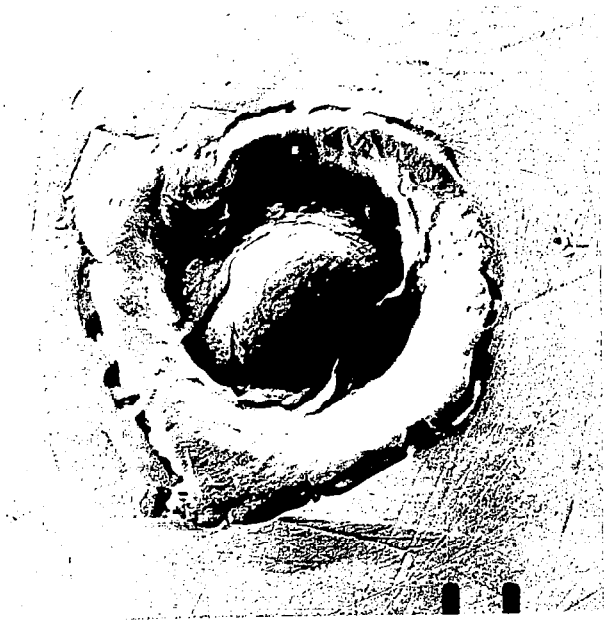
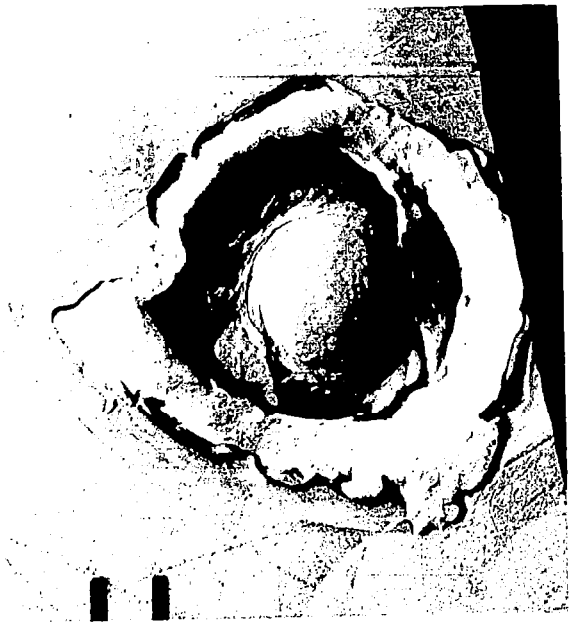
36

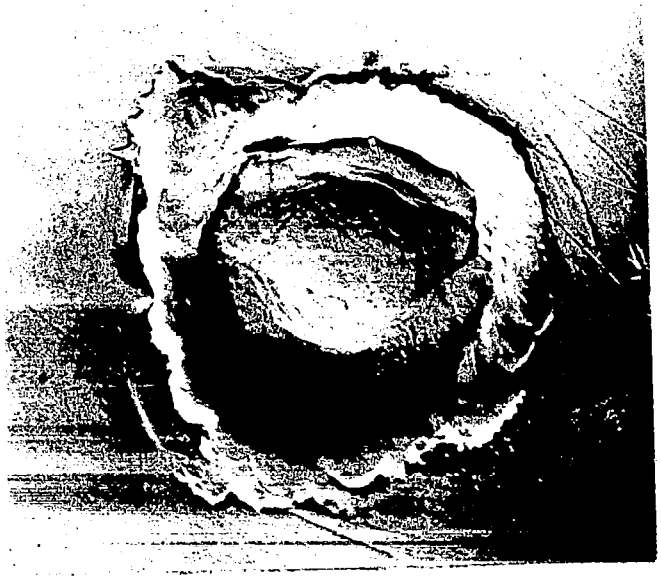
Electron Micrographs 37 through 39 represent the duplicate replication of a crater in a stainless steel target. As can be noticed during the comparison of the three micrographs, the details duplicated quite well.



37

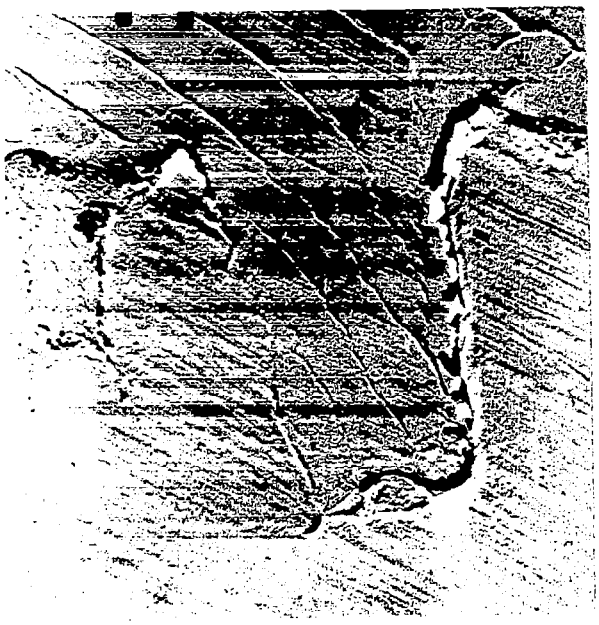


38

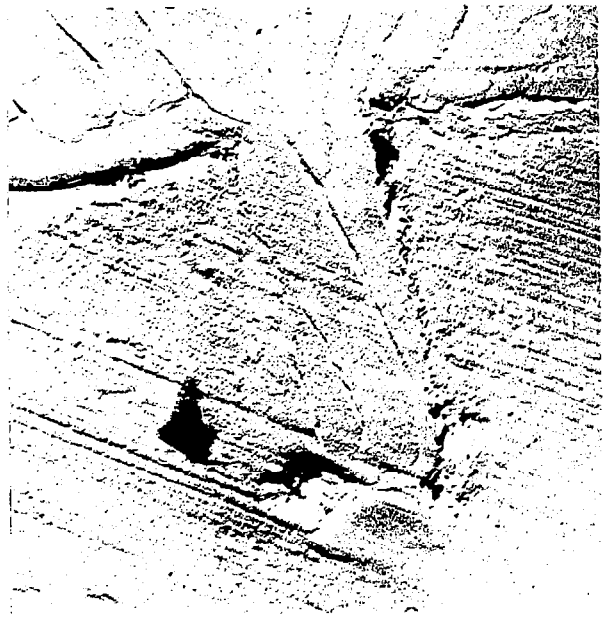


39

Electron Micrographs 40 through 43 illustrate the cross section of several craters which have been prepared by metallurgical mounting as indicated in the text of this report. In three cases the craters have been straight-walled with relatively flat or reverse curved floors. Micrograph 42 illustrates a conical crater. We tend to believe that the actual craters may be more straight-walled than conical in shape. If one considers the possibility of cutting a cone to produce a parallel walled section, one realizes that there is only one angular relationship which can produce this result. On the other hand, there are essentially an infinite number of ways in which a cylinder can be cut to appear like a cone. We feel that the illustrated results prepared from craters which were intentionally sectioned in a near to a longitudinal manner as possible, favor the cylindrical type of crater.



40



41

42

43

Electron Micrographs 44 through 47 again represent the results of the longitudinal cross sectioning of craters formed in aluminum from iron projectiles. The crater splash lip is much more apparent in these micrographs than in micrographs 40 through 43 as is the compressed zone in the bottom of the craters.