Battelle

Pacific Northwest Laboratories
Richland, Washington 99352

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Research Report

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FINAL REPORT

PART I

EXPLORATORY DEVELOPMENT AND SERVICES FOR PREPARING AND EXAMINING ULTRATHIN POLISHED SECTIONS OF LUNAR ROCKS AND PARTICULATES

NAS 9-11993

211B00862, Revision 1

to

NASA MANNED SPACECRAFT CENTER
HOUSTON, TEXAS 77068

March 23, 1972

R. H. Beauchamp
J. F. Williford
E. L. Gafford

PACIFIC NORTHWEST LABORATORIES
a division of
BATTELLE MEMORIAL INSTITUTE
P. O. Box 999
Richland, Washington 99352

TABLE OF CONTENTS

PART I

PROGRAM NARRATIVE

TABLE OF CONTENTS (CONT'D)

PART II

PROCEDURAL GUIDE

LIST OF TABLES

PART I

PROGRAM NARRATIVE

PART II

PROCEDURAL GUIDE

SUMMARY

This Final Report covers developmental activities, services and technology transfer efforts under Contract NAS-9-11993 for the period June 23, 1971, to March 23, 1972.

Successful development of improved procedures is reported for three classes of lunar materials; dense rocks, breccias and particulates. High quality ultrathin sections of these materials of unprecedented thinness provided to the Technical Monitor, Dr. Michael B. Duke, and to a co-investigator in the Geochemistry Branch, Dr. David S. McKay, are summarized. Lists of equipment and supplies, procedures, photomicrographic documentation and training have been provided.

Advantages of ultrathin polished sections for conventional and unconventional optical microscopy methods are described.

Recommendations are provided for use of ultrathin sections in lunar rock studies, for further refinement of ultrathinning procedures and for additional training efforts to establish a capability at the Manned Spacecraft Center.

A two-part report has been prepared, separating procedural details from the program narrative and technical discussion for NASA's convenience.

PART I

PROGRAM NARRATIVE

INTRODUCTION

During the past few years, Battelle has developed methods for thinning brittle materials to thicknesses on the order of one to five microns, polishing both surfaces. This work is an outgrowth of our research and development of mixed oxide nuclear fuels and advanced materials such as plasma spheroidized oxides and carbide fiber reinforced metals. In recent years, the methods have been applied to a variety of rocks and minerals, including local Columbia Basin basalts.

As a result of this prior work and some sections we provided to NASA of the Allende meteorite and Lunar breccia 12073 without cost, the value of ultrathin sections for investigations of lunar rocks became clear to investigators at the Manned Spacecraft Center.

We were requested to provide a proposal to further refine procedures and to transfer our technology to the Lunar Receiving Lab petrography facility. In addition, we were requested to provide sections of lunar breccias, dense rocks and particulates.

This report describes advances made in ultrathinning methods during the nine-month period of the resultant program and summarizes the technical significance of the new type section for optical microscopy. It is divided into two major, separately bound parts. Part I is a narrative describing the chronology, accomplishments and significance of the program efforts, and is addressed to the interests of the Contracting Officer and the Technical Monitor. Procedural details have been pulled together into a concise guide for use by the preparator in Part II.

ORGANIZATION

Part I of this final report deals with matters of contractual compliance, technical accomplishments and remaining problems. It is divided into the following major sections:

- Compliance
- Technical Discussion
- Conclusions and Recommendations

COMPLIANCE

The following section deals with our compliance to the terms of Contract NAS 9-11993. In summary, we have provided developmental and service tasks as described in the original proposal, 211B00862, Revision 1, made part of the subject contract by reference. The narrative below is organized to follow the original Task description sequence:

Task 1 - Providing a list of needed equipment and supplies to NASA.

Task 2 - Providing written procedures for ultrathinning of lunar dense rocks, breccias and particulates.

Task 3 - Providing instruction for NASA-MSC personnel in Battelle ultrathinning methods.

Task 4 - Providing services in ultrathinning to support Dr. D. S. McKay's investigations of breccias and particulates.

Task 5 - Devising ways to work on glass slides or to transfer ultrathinned sections to grids for transmission electron microscopy.

Total sectioning tasks were listed in Revision 1 to our proposal, and relate to our performance under Tasks 2 and 4. Total sectioning activities are summarized in Table I. A more detailed listing by rock identity is provided in Table II.

TABLE I

SECTIONS PREPARED UNDER CONTRACT NAS 9-11993

Type of Section	Contracted for	Made
Dense Lunar Rock	6	7
Lunar Breccias	4	6
Lunar Particulates	4	5
McKay	4*	6**
Allende Meteorite	0	1
	18	19

* Dr. McKay's samples were concurrently used in Tasks 2 and 4.

** Due to concurrent use of Dr. McKay's samples in Tasks 2 and 4, these sections were already counted above, and are not added to the total. The number is provided to indicate sections returned to Dr. McKay for his use.

TABLE II

LUNAR ROCK AND PARTICULATE SAMPLES SECTIONED

Identification	Description	Number
14303,33	Recrystalized microbreccia	1
14321,110,2	Breccia with large basalt fragments	1
14261,2,4	Chip from veined breccia	1
14162,40 GA-A 14162,40 GA-B	2 bowl-glazed aggregate fragments	1
14163.76,09,a	Speck of ropy glass	1
14066,44	Loosely bonded breccia	1
14003,28	-20 micron particulates	1
10048,82	Well-indurated chip	1
15102,3,T	1 to 2 mm fines	1
15103,7,T	Four fragments about 3-4 mm	1
14063,11	Resin impregnated butt	1
14311,62	Resin impregnated butt	1
12045,2	Dense lunar rock	1
12047,4	Dense lunar rock	1

TABLE II (CONT'D)

Identification	Description	Number
12053,3	Dense lunar rock	1
12051,71	Dense lunar rock	1
10003,17	Dense lunar rock	1
10050,12	Dense lunar rock	1

TASK 1

Our original intent for this task was to supplement existing equipment at the Lunar Receiving Laboratory to the extent possible, thereby minimizing costs to NASA for procurement of new equipment and supplies. Due both to delays in receiving a listing of equipment at the LRL and to differences in procedures between our two laboratories, it was not possible to apply discrimination of this sort. Accordingly, we submitted a preliminary listing of all equipment and supplies used in our laboratory to Mr. James Townsend on August 4, 1971. By October, we had identified two additional supply items of definite usefulness and one of potential value. A revised final listing was accordingly supplied as Enclosure A to the first Quarterly Report, dated October 22, 1971. That listing is reproduced here as Table III.

TASK 2

Task 2 principally consisted of developing procedures for ultrathinning of dense lunar rocks, breccias and particulates. Some delays were experienced, since dense lunar rocks were not made available to us at the beginning of the program (as planned), but arrived October 1, 1971. We accordingly delayed the first Quarterly Report so that we could include a procedure for the initial side preparation of these rocks. Two breccias were made available during the first quarter by Dr. McKay, so that we were able to provide a complete (although preliminary) procedure for breccias in the first Quarterly Report, dated October 22, 1971, rather than in the second Quarterly, as planned.

TABLE III

LIST OF EQUIPMENT AND SUPPLIES FOR ULTRATHINNING

Quantity	Description	Supplier	Price
	Equipment		
1	AB Duomet Belt Surfacer	Buehler, Ltd. 2120 Greenwood St. Evanston, Ill. 60204	$1,420
1	Buehler 3-unit Polishing Apparatus (two standard and one low-speed polishers, two-speed controllers, 2 wash basins)	Same	$1,515
(4)	Metal-bonded diamond lapping wheels to fit Buehler machines	Same	
	1-45 micron No. 60-4458		$ 400
	1-30 micron No. 60-4468		$ 400
	1-15 micron No. 60-4478		$ 400
	1-9 micron No. 60-4488		$ 350
1	Electric Drying Oven (NAPCO) ±1°C controller, range to 200°C. VW&R Cat. No. 52345-107.	Van Waters & Rogers	$ 95
1	Vacuum Oven (NAPCO) ±0.5°C, range to 200°C, vacuum to 1 micron. VW&R Cat. No. 52344-002	Same	$ 300
1	Vacuum pump, Welch "Duo-Seal" Model 1400BG-075. VW&R Cat. No. 54973-075.	Same	$ 210
4	Syntron Vibratory Polishing Machines Model No. LP-01, with controller, bowl and clamping ring	Syntron	$2,552 $638 each)
8	1-1/4 in. specimen holders for each vibratory polishing machine		

TABLE III (CONT'D)

Quantity	Description	Supplier	Price
	Supplies		
2 bags	SCAN-DIA cloth 12 in. diam. self-adhesive Finale 300 MM	T. C. Jarrett Co. Denver, Colo.	$ 69
8 bags	Pellon Type PaN-W 300 MM 12 in. diam.	Same	$ 80
2 bags	Pellon Type PaN-K 300 MM 12 in. diam.	Same	$ 20
2 units (9 lb)	Maraglas Type A casting epoxy #655 and hardener #555	The Marbelette Co. Long Island City, N.Y.	$ 34
4 units	Spectrum diamond lapping compound, #1/2, Spectrum color grey, Formula DW-57, diamond concentration 100, 25 gram units	Penn Scientific Abington, Pennsylvania	$ 270
4 units	Spectrum diamond lapping compound, grade #1, Spectrum color white, formula DW-57, diamond concentration 100, 25 gram units	Same	$ 180
4 units	Spectrum diamond lapping compound, grade #3, Spectrum color violet, formula DW-57, diamond concentration 100, 25 gram units	Penn Scientific	$ 400
6 doz.	SiC cloth abrasive belts for belt surfacer, 4 x 36 in., waterproof, VW&R No. 40204-083.	Van Waters & Rogers (VW&R)	$ 150
100 sheets	600 grit SiC wet or dry papers, 9 x 11 in., VW&R No. 40265-248.	Same	$ 15
400 sheets	2/0 Emery polishing paper, VW&R No. 40262-160.	Same	$ 64
400 sheets	3/0 Emery polishing paper, VW&R No. 40262-206.	Same	$ 64

TABLE III (CONT'D)

Quantity	Description	Supplier	Price
5 lb.	Linde A alumina polishing powder, VW&R No. 40404-000.	Same	$ 85
1 lb.	MgO polishing powder (e.g., VW&R No. 40393-000).	Same	$ 4
75	Buehler petrographic well slides- glass, Cat. No. 40-8001.	Buehler, Ltd. 2120 Greenwood St. Evanston, IL 60204	$ 10
1 lb.	Silastic E RTV for making casting molds	Dow Corning	$ 5

We reported a complete procedure for dense rocks in the second Quarterly Report, dated December 27, 1971.

Particulates were thinned during the third quarter, and included some fragments and chips supplied by Dr. McKay as well as a -20 micron size fraction of lunar soil. Procedures for these materials are given in Part II.

In essence, we have been able to apply a uniform procedure to the diverse materials with good results. The principal variation in preparation of differing materials consists of omitting one polishing step, and is spelled out in Part II.

As planned under Task 2, we provided ample photomicrographic documentation of the appearance of the three types of lunar material in varying kinds of illumination. Approximately forty high quality photomicrographs, many in color, were provided in the two quarterly reports and in a letter to Dr. McKay. Optical methods shown have included brightfield transmission, orthoscopic transmitted light, brightfield reflection, darkfield illumination and Nomarski interference contrast (in both reflected and transmitted light). Petrographic descriptions have been provided to define the rock types which have proven to be responsive to established procedures.

We have shown diligence beyond contractual requirements by supplying 14 mounted display boards documenting the sections and optical methods. A summary of these is provided in Table IV. This extra effort was commended by the Technical Monitor, Dr. Duke.

TASK 3

Task three consisted of three training sessions; two in Houston and one in our laboratory. Mr. R. H. Beauchamp visited the LRL in Houston in December 1971, late in the second quarter rather than early in that quarter as planned. Equipment and supply procurement by NASA had been delayed. Progress was reviewed during this visit, and lunar materials were selected for the remaining sectioning work under Tasks 2 and 4.

During January 1972, Mr. Travis Allen, then a preparator at the LRL, spent a week in our laboratory receiving training from Mr. Beauchamp, the Battelle Principal Investigator. Mr. Allen got some hands-on experience

TABLE IV

DISPLAY BOARDS PREPARED

Identification	Description	For
14321,110,2	160X Photomosaics (2) reflected light and orthoscopic transmission with 1st order red plate.	Dr. D. S. McKay
14303,33	160X Reflection photomosiac plus selected areas in transmitted light.	Dr. D. S. McKay
14261.2,4 14162,40 GA-A 14162,40 GA-B 14163.76,09,a 14003,28	One board of photomicrographs and mosaics of particulate materials.	Dr. D. S. McKay
14063,11	Selected areas of the microstructure.	Dr. M. B. Duke
14311,62	(2) Selected microstructural areas in varying kinds of illumination, including Nomarski interference contrast.	Drs. Duke & McKay
12045,2 12047,4 12053,3 12051,71 10003,17 10050,12	(6) Low Magnification Mosaics, plus high magnification color photomicrographs of selected areas.	Dr. M. B. Duke

with basalt stand-in materials, and observed the preparation of lunar breccias and particulates.

Mr. Beauchamp again visited the Manned Spacecraft Center during February 1972. On his arrival, he found that equipment for ultrathinning had not been installed, and that two needed size fractions of diamond polishing material specified in October 1971 had not been procured. Accordingly, only limited training was possible. Mr. Beauchamp was able to observe existing procedures and equipment problems in the thinning laboratory at Houston, and made his conclusions and recommendations known to the NASA technical monitor during his visit. These are listed in a later section (pp. 22-23).

Task 4

Task 4 consisted of supplying sections of lunar breccias and particulates for Dr. McKay to support his own investigations. To some extent, sections under this task and those spelled out under Task 2 were the same. This was as expected. During Mr. Beauchamp's December visit, particulates and breccias were selected by Drs. Duke and McKay for the balance of the program. In the main part, these consisted of materials of interest to Dr. McKay.

Two sections were prepared for Dr. McKay during the first quarter, and four during the third quarter.

TASK 5

Task 5 was originally proposed as an either/or case. We were to develop ways to get sections onto glass slides or to transfer ultrathin sections to grids for transmission electron microscopy.

We tried working directly on glass slides early in the program, using the Allende meteorite as a stand-in material, and were immediately successful. The Allende section was sent to Dr. McKay. All sections of lunar materials have been prepared on glass slides. Procedural details are provided in Part II.

We have prepared a number of ultrathin sections of the Allende meteorite, but have not yet evolved a successful procedure for transferring these to grids for the electron microscope. Additional time and funding would be needed to achieve this objective.

TECHNICAL DISCUSSION

The following discussion summarizes what we have learned during the contract period, and is divided into three principal sections:

- Progress in Ultrathin Section Preparation
- Examination Methods
- Remaining Problems

PROGRESS IN ULTRATHIN SECTION PREPARATION

Significant progress has been made during the program period in improving the ultrathinning procedures. Accomplishments include working directly on glass slides, improved thickness uniformity and enhanced surface polish quality. These are discussed separately below.

Glass Slide Substrates

When this work was originally propsoed, we were preparing thin sections in polymer mounts. Essentially, the first slide was prepared, a new mount was cast against the polished face, and the section was thinned and polished. Subsequently, the polymer mount was sawn to reduce its thickness to the order of 1/8-inch and was polished on the back side. Although such polymer-mounted specimens are breakage resistant, they have two large drawbacks, both related to the polymer thickness. The first problem is the excessive path length between the substage condensor system and the section plane. This prevents critical focussing of the condensor for high magnification work, so that the intrinsic advantages of ultrathin sections as phase objects for enhanced resolution cannot be exploited. The second drawback is the strain birefringence of the polymer, which causes superimposition of higher order colors and irrelevant extinction behavior on the object plane.

We anticipated extreme difficulty in providing an adequate glue line so that the sections could be directly prepared on glass slides. Although there are some remaining difficulties, it has proven to be readily feasible to prepare ultrathin sections on glass slides, using the same casting epoxy (Maraglas A) that we have used all along. The problems of optical path length and strain birefringence were thus overcome early in the program. The preparation of glass-mounted ultrathin sections is treated in the Procedural Guide, Part II.

There are still significant remaining stresses in the assembly following gluing of the first surface to the glass slip. We have experimented with various cooling rates for the sections in the curing oven, but so far have been unable to completely alleviate the problem. It is essential to use an oven for curing rather than a hot-plate (as used at the LRL) to provide a sufficiently slow and uniform cooling of the assembly. Gluing difficulties are discussed later under Remaining Problems.

Thickness Uniformity

Another advancement during the program period has been to improve the uniformity of section thickness. Early in the program we considered the slightly lenticular shape of the ultrathin sections and did some elementary computations of surface flatness requirements to minimize the difference between center and edge thickness. A discussion of the importance of flatness was provided in the first Quarterly Report. We systematically checked the flatness of laps and polishing tables, and determined that the principal cause of surface curvature was bumping and rocking of the polishing weights on the vibratory polishing table. An acrylic template was made to space the polishing weights apart on the polishing table, and the flatness was immediately improved beyond the sensitivity of the ring-base spherometer we were using to check specimen surfaces. A striking improvement in the uniformity of section thickness and obtainable section size was accordingly obtained, compared to the state-of-the-art nine months ago.

Polishing Quality for Planar Interphase Relationships

Significant advances were made in refining polishing procedures for lunar rocks. Prior to the contract period, we used half-micron diamond alone for polishing. Significant improvements in rate and interphase flatness were obtained with basaltic rocks by adding a rough polishing step using 3-micron diamond, followed by 1-micron diamond. Improvements were made in preparation of breccias and particulates by using 1-micron diamond slurries for vibratory polishing.

EXAMINATION METHODS

During this program we have provided extensive photomicrographic documentation of the appearance of ultrathin sections in various modes of illumination. The following discussion reviews this work briefly in two categories:

- Conventional petrographic methods
- New optical methods

Conventional Petrographic Methods

Conventional methods used by petrographers and ore microscopists include both transmitted and reflected illumination modes. These are:

- Transmitted light modes
- Brightfield reflection
- Darkfield methods in reflected or transmitted light

These are discussed separately below.

Transmitted Light Examinations. The principal reason for making and using ultrathin sections is the improvement in resolution and optical transparency. The sections are extremely useful for delineating microstructural details in portions of rocks previously dismissed with the catch-all phrase "groundmass." The textures of fine-grained "groundmass" portions of the microstructure can be resolved in ultrathin sections using conventional transmitted light microscopy. This advantage has been shown in a striking manner with the lunar breccias, where the shape, size and optical activity of grains in the matrix can be both illuminated and resolved. A great deal of optical information is accordingly made available about important details such as recrystallization and sintering phenomena.

A great many of the details we have shown with polished, ultrathin sections of lunar breccias lie in areas of the microstructure which are opaque in conventional sections. Further, the superposition of overlapping diffraction patterns in conventional sections (due to a plurality of grains through the depth of the thirty-micron thickness) compromises resolution even when light can be gotten through the structure.

The textures of dense lunar rocks do not present the same problems of fine grain size and opacity as do the lunar breccias. The advantages of ultrathin sections are thus not immediately apparent at low magnification with dense rocks. However, gains in resolution and field flatness become readily apparent at higher magnifications (i.e, 160X and greater). Excellent resolution of detail is retained up to the limits of optical microscopy, revealing a new level of ultrastructure in late-crystallizing interstitial material, immiscible phases and reaction rims--notably around ilmenite grains.

<u>Reflected Light</u>. Reflected light microscopy does not benefit from reduced section thickness, but the quality of reflected light information is definitely enhanced by our preparation methods compared to sections we have seen from the Lunar Receiving Laboratory. Compared to the LRL methods, our preparation techniques show an elimination of artifacts and smearing, as well as much better interphase flatness. Our first side polish has consistently been better in these respects than the second side polish. It is well to do some characterization of material in reflected light prior to thinning and polishing the second side, not only because of the slightly higher quality of the first side polish, but also to assure that microcracking observed in the final section is related to the true microstructure rather than to preparation.

Examining ultrathinned sections in reflected light is troublesome due to internal reflections within the specimen from the back face. The net effect is a reduction in contrast. This problem can largely be overcome by placing a matt black piece of paper behind the specimen or temporarily blacking the back side of the slide with a marking pen.

Darkfield Methods. Darkfield methods are not extensively used by petrographers, but bear some comment. Reflection darkfield methods and use of reflected polarized light have similar uses (i.e., to look within the section without the bright, masking effect of normally reflected light from the section surface). Darkfield reflection is rather more powerful than polarized reflection in accentuating grain boundaries and microcracking details. Darkfield transmission has similar uses in providing bright delineation of grain boundaries and microcracks against a black background.

The advantages of ultrathin sections in darkfield illumination are principally evident with fine-grained materials or extensively cracked microstructures. The problem with darkfield illumination and standard thickness sections is that overlapping structures obscure detail. Due to the shallow depth of field with optical microscopy, the dark background is substantially destroyed by light scattered from grain boundaries and cracks below the plane of focus. Substantial reductions in thickness below thirty microns (e.g., to the three-to-five-micron thickness of many of our breccia sections) allows darkfield illumination to be used for unequivocal delineation of grain boundaries and cracks.

The method of using darkfield transmission appears to be particularly powerful in bringing out the extent of reaction rims and textural differences in recrystalized breccias, notably around phenocrysts. Inclusions within phenocrysts (e.g., Schiller structure in pyroxene, gas inclusions and small crystalites) can be observed with darkfield methods. Some additional attention to these methods with the lunar rocks might be appropriate.

Unconventional Methods

The term "unconventional" is used in the sense of optical methods familiar to the petrographer. Contrast enhancement methods we have used are quite familiar to the biological microscopist, but have not been widely used by petrographers. Nomarski interference contrast in transmitted light is a particularly powerful method for examining ultrathin sections, but is useless with conventional sections. This method vastly exaggerates apparent relief due to very small differences in refractive index or thickness. Nomarski transmission methods are most powerful in showing inhomogeneities in glasses and the relationships in zoned or intergrown crystalline mineral phases.

Nomarski reflection methods do not depend on thickness of the section, but are based on an enhancement of surface topographical details using the optical path differences resulting from surface relief between phases. The method is most powerful where polishing relief has been minimized and an artifact-free, highly polished surface has been provided. Where relief is excessive, the method simply does not work. Nomarski reflection is a powerful tool for examining the textures of rocks in reflected light, and for distinguishing between phases which are similar in plain reflected light.

Although Zernicke's phase contrast methods can be applied to ultrathin polished sections, we currently believe that the Nomarski methods are more powerful and easier to interpret with geological materials.

FUNDAMENTAL REMAINING PROBLEMS

Basically, there are two types of problems remaining with the new technology of making and using ultrathin polished sections. One problem is technical and centers around preparation difficulties. The other problem is the degree of existing conservatism in petrography, which we had underestimated. These problems are discussed separately below.

Preparation Problems

The most important remaining problem is that of residual stresses in the gluing operation, wherein the polished first side is joined to the well side of a petrographic slide. There is a large difference in the coefficient of thermal expansion for polymeric materials and ceramics. Since the epoxy we used is cured at 80°C, considerable stresses accumulate on cooling to room temperature. In addition, there is some shrinkage of polymeric glues during curing and after some bonds have been developed at the interfaces within the system. The net result of these factors is a significant amount of residual stress, with very high shear stresses in the glue line, peaking near the section perimeter. The upper glass slide surface is loaded in compression, and the back surface in tension. If the stress is sufficiently high, the glass will simply break. Alternately, with many of the polymers we have tested, the bond line (generally at the glass/polymer interface) will fail.

There are a number of polymers having relatively low shear strength that exhibit plastic deformation in the form of creep (stress relaxation) at room or moderately elevated temperatures. With such systems, residual stresses may be substantially reduced. Unfortunately, we have been unable to find a polymer that will exhibit the desired stress relaxation behavior without also exhibiting excessive compliance and inadequate bond rigidity.

We have gotten around most of the problem of residual stress by attention to slow and uniform cooling (so that thermal stresses are minimized) and by taking care not to over-cure the epoxy (which produces excessive shrinkage and hence greater stress). Further, we perform rough grinding to relieve stress as soon as possible after oven cooling, since the tensile failure of glass is known to be time-dependent with constant stress. Added to these precautions is the long experience and highly developed skill of Mr. Beauchamp, our chief preparator, which has given him a "feel" for the amount and kind of pressure that can safely be applied in lapping.

Although promise has been shown by some room-temperature-curing epoxies, more work is needed with these to improve bonding strength to glass. Progress in reducing residual stresses is probably the greatest single procedural advancement needed to reduce the skill requirement for successful ultrathinning.

Conservatism

The science of preparing and examining rock sections is an old and well-established way of identifying minerals and describing textures. Methods of preparation and examination have not changed greatly in the past 100 years, and a vast amount of information is available in the literature for making and using thirty-micron-thick sections. Until recent years, these methods have provided as much information as anyone could want or need.

More recently, we have become interested in better preparation methods for use with relatively new techniques such as high resolution (i.e., small beam diameter) electron microprobe analysis. For such methods, coplanar relationships between phases are desirable, along with an artifact-free surface. Smearing of one phase over another during the vigorous polishing methods traditionally applied can unnecessarily limit the microprobe in discriminating between adjacent phases and in resolving reaction details.

In addition to the new non-optical methods, increased attention is needed for finer microstructural details in the microscopic examinations of newer ceramics as well as in extraterrestrial rocks such as stony meteorites and lunar rocks. The gain attainable through use of ultrathin sections in making fine textural detail visible is comparable to the first application of high magnification to carefully prepared metallurgical specimens--a development which took place in the 1800's.

The interrelationship between metallography and the development of the entire science of metallurgy are well-known. However, the correlation of visible "ultrastructure" in rocks and minerals with their genetic history and geochemical characteristics has hardly begun. Accordingly, most petrographers do not recognize what they are now able to see, and the new information is thus of little value to them. Further, access to a new level of microstructural detail is purchased at the price of the familiar color information seen in thirty-micron thicknesses. Relief, using traditional methods, is also absent. In summary, the petrographer is unable to see many familiar optical phenomena in ultrathin sections, and is unable to understand much of what he can see for the first time.

We had underestimated the degree of conservatism in the field of petrography. Also, we had not been aware of the separation between preparators and investigators in Houston. We are accustomed to working in much closer collaboration here, principally because we are almost always interested in very fine microstructural detail and collaboration is necessary so that both the preparator and the investigator realize what is important in the material and how access to particular detail relates to the preparation methods.

CONCLUSIONS AND RECOMMENDATIONS

THE PLACE OF ULTRATHIN SECTIONS

The advantages of ultrathin sections are not always important. Their inherent superiority as phase objects for enhanced resolution and transparency is irrefutable in terms of optical theory and is clearly evident in practice. However, where all microstructural detail of interest is on the order of tens of microns and optical density is not a problem, there is little need for making an ultrathin polished section. The only real advantage in such instances is the photographic field flatness due to elimination of significant optical path differences.

The real strength of ultrathinning is in resolving the textures of rocks or portions of rocks normally not accessible to transmitted light methods. Particularly in examination of breccias for sintering, devitrification and recrystallization phenomena, remarkable gains have been made with ultrathin sections.

The whole area of Nomarski interference transmitted light microscopy of rocks and minerals remains virtually untouched.

We recommend that new optical methods such as Nomarski and Zernicke contrast enhancement, sensitive tint methods and darkfield techniques be systematically applied to breccias and lunar glasses particularly, and that efforts be made to correlate visible microstructural detail with other data gathered by electron microprobe, X-ray diffraction and other analytical methods. Other materials of interest include chondritic meteorites and various fine-textured terrestrial materials.

We recommend that the uses of ultrathin sections be explored with PI's involved in lunar rock studies, that new techniques be described in the literature, and that efforts be made to increase the number of high quality sections available for use by Principal Investigators.

PROCEDURAL DEVELOPMENT

We recommend that further work be done on improving gluing materials and procedures. In particular, effort should be directed toward surface treatments (i.e., cleaning and use of coupling agents) to improve interfacial bond-shear strength. In addition (or alternatively) prestressing methods should be investigated to cancel or at least reduce residual stresses.

Further work on mounting and sectioning very fine particulates (-20 micron fraction) is needed to improve section flatness generally and edge retention around the particles.

Some additional study to compare our procedure with the traditional methods of lapping and polishing could serve to identify where difficulties lie with conventional methods. Identification and clarification of some key factors (e.g., depth of sub-surface fracturing during lapping operations, responsiveness of various prepared surfaces to vibratory polishing, etc.) might be persuasive in overcoming more preparator conservatism or in identifying some traditional methods which may be retained. Better understanding of alternative ways to prepare specimens might lead to further improvements or simplifications of procedures or equipment requirements.

In addition to its usefulness in ultrathinning, vibratory polishing appears to have some advantages for microprobing. The relative virtues of conventionally and vibratorily polished specimens ought to be compared to clarify which is best in a persuasive way.

The ultrathin section should be examined as a jump-off point for the preparation of specimens for transmission electron microscopy. We have not been able to progress as far in this direction as we had hoped. Essentially, we would suggest that state-of-the-art ion milling methods be applied to ultrathin sections, keeping the total amount of material removal needed to the order of one to three microns. We would expect that problems due to differing etch rates could be significantly reduced by starting with such thin samples. We would also like to examine the applicability of directly thinning specimens by our methods for the megavolt electron microscope. We have access to such an instrument on site.

RE-DOING NASA SECTIONS

During the course of our investigations we were asked to prepare three sections previously impregnated with polymer in Houston. We found that the heat distortion temperature of this polymer was substantially less than the cure temperature of our epoxy system. We were able to prepare one very good section of breccia (14311,62) and one usable section of basalt (14063,11). These were both relatively thick potted rock butts. A thinly sliced NASA-impregnated breccia (14307,25) curled up in the curing oven so much that it could not be prepared.

During our visit to Houston last month, Dr. Brett asked us if we thought we could re-do some particularly interesting NASA sections. We would like to do some further work on this problem, either through exploratory studies in our laboratory with the NASA polymer or through use of a room temperature curing epoxy to remount the NASA sections.

FACILITIES AND STAFF TRAINING IN HOUSTON

Mr. Beauchamp's impression following his last trip to the LRL may be summarized as follows:

1) Preparators training to make ultrathin sections need to be relieved from production quota pressures.

2) Better optical instrumentation is needed by the preparators in the lab to assure the quality of their efforts.

3) Closer collaboration between preparators and investigators is needed so that preparators can better appreciate the needs of the investigators and the investigators can better appreciate what can and should be done in the thinning lab.

4) More space is needed for clean, efficient working and improved section quality. This is particularly a problem with the high workload.

5) Preparators should be trained to some degree in a number of optical microscopy techniques and in photomicrography so that

they can develop a direct appreciation for the problems involved as they relate to section quality and preparation.

6) Preparators who are sufficiently conscientious and skillful to prepare ultrathin sections need to receive some recognition and assurance that they are more than piece workers.

We are very much interested in helping you set up an entirely new petrographic facility in Houston, and particularly encourage you to consider vibratory polishing as a standard process for reflection, standard thickness and ultrathin specimens.

DIFFICULTY OF ULTRATHINNING

It is much harder to learn to make an ultrathin polished section than it is to make one of conventional thickness. Once the skill is developed, and given the desire in all cases to produce high quality surface polishes, sections of any thickness from thirty microns down to about five microns all require about the same amount of time to make.

During the past nine months, one of the authors (JFW) and one Battelle technician (Mr. D. H. Parks) have learned to make polished thin sections substantially below standard thickness (i.e., about 5 to 8 microns thick). Neither has worked to the one-micron thicknesses produced by Ray Beauchamp, and neither can consistently turn out sections of equal quality. Both of these trainees have had previous experience with critical surface preparation by hand methods and are well acquainted with the basic principles and problems. Both are competent microscopists.

Based on our own experience, we can safely say that the art of ultrathinning can be learned, but with great difficulty and with a large investment of time. On the other hand, application of vibratory polishing methods to standard thickness specimens or to reflection mounts can be readily learned and yield immediate benefits in section quality.

We recommend that additional training of NASA staff members take place, and that continuing provision of thinning services by Battelle be provided for as a source of some comparison standards to gauge progress in Houston. We would further recommend that further process refinements be studied to reduce the skill requirement insofar as possible. Some needed refinements in gluing have already been indicated. Systematic examination of more automated surfacing methods in our laboratory might also be of great benefit, but we would need some additional equipment to do this.