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Boron contamination in polished thin sections of meteorites: Implications for other trace-element studies by alpha-track image or ion microprobe*

D. M. Shaw, M. D. Higgins,** M. G. Truscott, T. A. Middleton
Department of Geology, McMaster University, Hamilton, Ontario L8S 4M1, Canada

Abstract

Alpha-track images (ATI) of B and Li in ordinary and carbonaceous chondrites were prepared from polished thin sections. The first sections examined gave ATIs showing much petrographic detail, but terrestrial contamination was suspected.

The contamination was confirmed and comes from B, chiefly but not only in diamond polishing paste used in section preparation. In addition to enhancing textural detail, the contamination is also revealed by spots of high track density unrelated to petrography.

Specially prepared (uncontaminated) sections of Murchison and Antarctic meteorites gave ATIs of weaker intensity but which still distinguish chondrules, rims, clasts, and matrix. The Antarctic samples also show B and/or Li to be localized in red-brown alteration.

ATIs can give valuable information on B and Li distribution within meteorites, but contamination must be excluded during section preparation. Polished sections for ion-microprobe study require similar care.

Introduction

Grain-scale variation in chemical composition within rocks and meteorites can be studied using various kinds of microprobe or autoradiography, such as induced alpha-track imaging (ATI), after preparation of a very flat surface, usually a polished thin section (PTS). The ATI has been used to examine the distribution of B and Li in meteorites and rocks by many, including Fleischer and Lovett (1968), Carpenter (1972), Kleeman (1973), Seitz (1973), Seitz and Hart (1973), Furst et al. (1976), Weller et al. (1978), Truscott and Shaw, (1984), and Truscott et al. (1986).

Our studies of trace levels of B and Li in meteorites, using this technique, encountered problems that were not initially recognized but proved ultimately to have arisen from contamination, principally during PTS preparation, but possibly elsewhere also. The subject of contamination during specimen preparation is not new; for example, Steele et al. (1981) have described contaminants in materials for preparation of ion-microprobe sections, as have Phinney et al. (1979) for B, Li, and Be. The present results will be of particular interest to others working on B or Li, but also have relevance to studies of other elements, using the ion microprobe.

Other sources of contamination in B analysis have already been reported by Weller et al. (1978), who documented how their samples acquired B from the atmosphere, and more recently by Curtis et al. (1980) and Curtis and Gladney (1985).

The samples used for this study are listed in Table 1 in the sequence in which they are discussed.

Analysis methods

The ATI utilizes the nuclear reaction $^{10}B + n = {}^{7}Li + {}^{4}He$ + 478-keV gamma-ray. The surface of a polished thin section or slab is placed in contact with a sheet of cellulose nitrate film (CN-85, Kodak-Pathe Ltd.) and irradiated with a thermalized fluence of 10^{12-13} neutrons/cm^2 in the McMaster Nuclear Reactor. During the irradiation, some of the alpha particles generated at the surface of the section leave trails of damage, i.e., tracks, in the cellulose nitrate. Following radiation cooling, the film is removed and etched in 2.5*N* NaOH solution at 50 °C for 10–20 min. The alpha tracks are preferentially etched and can be observed by using a microscope. At high magnification, individual tracks may be counted; but at low magnification, the aggregate track density is seen.

Li generates tracks that are indistinguishable from those produced by B, by the reaction $^{6}Li + n = {}^{3}H + {}^{4}He$, but with terrestrial isotopic proportions, B is 10–15 times more effective in generating tracks per unit element mass. In addition to these two elements, the only others to be considered are ^{17}O and ^{14}N, but their effects are almost negligible in meteorites and rocks. In the photographs used in this paper, high track density is represented by a dark image, which is proportional to the B plus Li concentration below the film.

Some authors have attempted to use this method to analyze for B quantitatively, using powdered and ho-

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** Present address: Sciences de la Terre, Université du Québec à Chicoutimi, Chicoutimi, Quebec L8S 4M1, Canada.

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TABLE 1. Meteorite specimens used

Name	Type	WG*	Source**	Comments
Murchison	C2M		FM	Polished section first prepared and used for other purposes
RKPA78004, 23	H4	A	JSC	Polished section prepared by standard NASA/JSC methods
Murchison	C2M		SI	Five sections cut from sample no. 5470, using various methods; one prepared dry
Allende	C3V		FM	Slab, no. Me2629, prepared with no epoxy, but using diamond paste

* WG indicates weathering grade for Antarctic meteorites, as given in the "Antarctic Meteorite Newsletter," i.e., A = minor; B = moderate; C = severe.
** FM = Field Museum, Chicago; JSC = Planetary Materials Branch, NASA/JSC, Houston, Texas; SI = Smithsonian Institution, Washington, D.C.

mogenized samples (e.g., Weller et al., 1978); it is difficult, however, to correct for Li. In the present work we have used the method in a qualitative way.

Preliminary studies

It is necessary to say a few words about the abundance levels of B and Li in meteorites. Curtis et al. (1980), Curtis and Gladney (1985), and our own analyses (Shaw et al., 1986, 1987) indicate that few chondrites contain >1 ppm B, and most fall in the range 0.1–1 ppm. Li is somewhat higher, although not well known, in the range 1–4 ppm (Nichiporuk, 1971; Nichiporuk and Moore, 1974; Murty et al., 1983). At these low concentration levels, contamination will clearly be of greater importance than for more abundant constituents. No B-rich or Li-rich phases have been reported in meteorites.

The initial suite of samples was obtained from the Field Museum, Chicago, through the courtesy of E. J. Olsen, in the form of nine polished thin sections prepared and previously used for other purposes. The principal feature of the ATIs obtained from these samples was the vivid delineation of petrographic relationships within each meteorite and, in particular, the clarity of the rims around chondrules, clasts, and inclusions. These features appear in all the sections, but are represented here by the C2M carbonaceous chondrite Murchison in Figure 1.

This meteorite shows a low level of track-forming elements in many chondrules (the significance of which will be discussed elsewhere), but tracks do occur in cracks, minute inclusion mineral grains, or glass. The low-level track background comes in part from the low ^{17}O concentrations in silicates, in contrast with metal and sulfide grains that contain virtually no track-producing elements, but also comes from ^{17}O and ^{14}N in the cellulose nitrate.

In contrast with the rather featureless ATI of chondrules, many clasts and inclusions show detailed internal structures. However, the most striking features are high track density rims to chondrules and clasts. In Figure 1, almost every such object has either a single or a multiple rim, usually with the densest part adjacent to the chondrule or clast. In some of these, the rim is uneven in thickness or may not surround the whole inclusion. In one case, a portion of matrix or a small clast adheres to the chondrule, and both are surrounded by the rim.

The rims just described are also visible optically in the PTS, but they do not always overlap completely the track-defined rims.

Presentation of the foregoing results verbally (Higgins and Shaw, 1985) and in an earlier version of this article elicited comments to the effect that the ATI features just described might be artifacts, resulting from contamination of the meteorites during residence in a museum or departmental collection, during the preparation of the PTSs, or subsequently. A given PTS might have acquired contamination from an already-dirty hand-specimen or after preparation of the PTS. Since many chondrules are not held tightly within a meteorite, their rim regions could be particularly liable to contamination from soluble B compounds that are widely used in laboratories (detergents, X-ray fluorescence pellets, rock-analysis fluxes, etc.). Curtis and Gladney (1985) have already shown that the exterior portions of many meteorite specimens are contaminated with B.

Further studies

Antarctic meteorites

In the light of the foregoing it was decided next to study meteorites unlikely to have been exposed to terrestrial contamination, either natural (weathering) or artificial (laboratory). The Antarctic meteorites appeared at first sight to meet these requirements. Those available comprised four Yamato meteorites (kindly supplied by the Japanese Committee on Antarctic Meteorites), nine from the Allan Hills, and two from Reckling Peak (courtesy of the Meteorite Working Group, NASA/JSC). The polished sections had been impregnated with epoxy resin, then prepared with standard methods, ending with diamond polishing followed by alumina polishing, taking care to clean scrupulously between steps with ethanol.

Bulk B analyses of the Antarctic meteorites had been made by prompt gamma neutron-activation analysis (Higgins et al., 1984). None of the results was anomalously high, so it is unlikely that the meteorites were contaminated before PTS preparation; weathering effects are present, however, and will be discussed elsewhere.

The ATI textures of all these Antarctic meteorites show the same features as those described for the Field Museum specimens, but the overall track densities are lower and are lacking in good resolution of petrographic detail. One example will serve to illustrate the observations and is shown in Figure 2. The bulk B content of the H4 chondrite RKPA78004 is 0.44 ppm B according to Curtis and Gladney (1985). This stone also contains much metal,

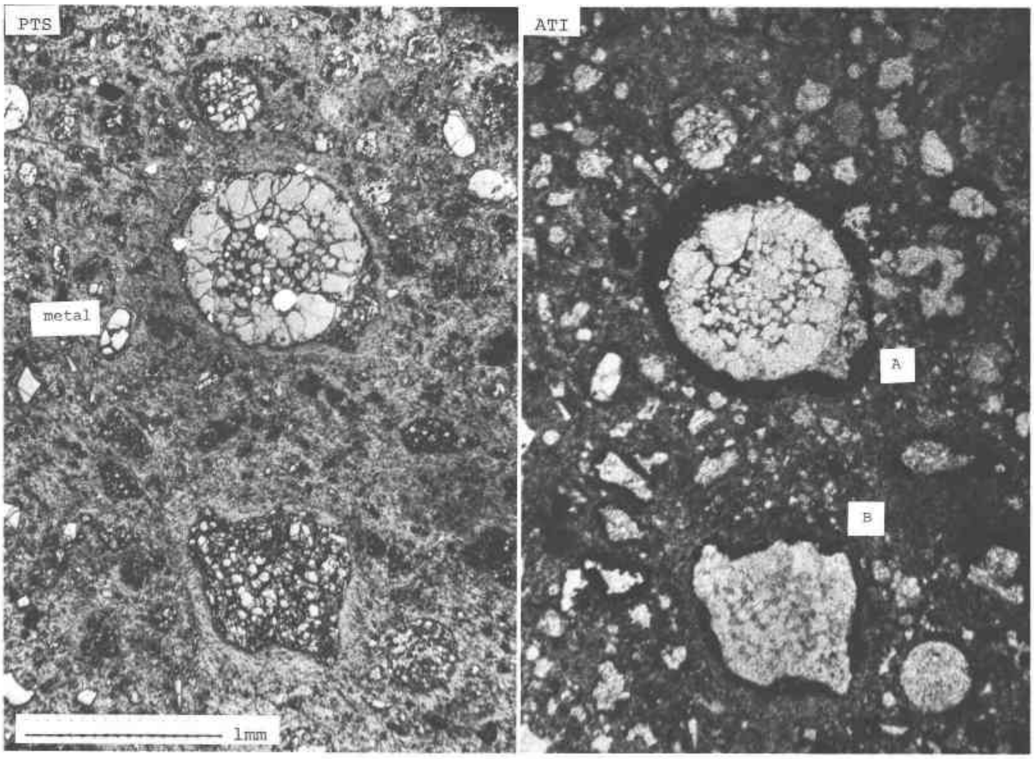

Fig. 1. Murchison, C2M carbonaceous chondrite. The PTS is illuminated with reflected light, revealing matrix, chondrules, inclusions, and white spheroids of metal, both in the matrix and within the chondrules. In the ATI, the chondrules, aggregates, and inclusions show low track density, in contrast with the matrix. There are irregular rims of varying thickness around several of the inclusions: these rims are also visible, but not so clearly, in the PTS. Note that the rim around the large chondrule encloses a portion of matrix or clast (A), adhering to the chondrule. The lower inclusion has a multiple rim (B): the inner portion is irregular, with high track density, but the outer segment is more circular and shows lower density. The matrix appears to consist of fragments or aggregates, down to the limits of resolution. The rims, and perhaps other features in the ATI, are believed to be enhanced by contamination from diamond polishing paste.

which can be clearly distinguished from silicate minerals in both the PTS and the ATI. A fusion crust (now consisting of a fine-grained aggregate of orthopyroxene crystallites) forms the top edge of this specimen. Although this crust has a lower track density than the anterior matrix, it and the adjacent epoxy contain numerous point sources or spots of intense track abundance; these have the form of spherical bubbles and some are visible in the PTS. It became evident that they were artifacts (see next section).

Section preparation materials

The presence of prominent chondrule rims and track-producing bubbles in some ATIs suggested contamination from the materials and techniques used to prepare PTSs; Weller et al. (1978) and Phinney et al. (1979) had already signaled this possibility. Materials used in the preparation of cut and polished sections of meteorites and lunar samples were provided by the Planetary Materials Branch, NASA/JSC. They were analyzed for B using prompt gamma neutron-activation analysis, and in some cases for Li also, using atomic adsorption analysis (see Table 2). Analytical details are given in Shaw et al. (1988); precision for each element is 5–10% of the amount present, and the sensitivity is 0.3–0.5 ppm.

It is known that epoxy resins and microscope slides would contain some B, and the low levels measured (Table 2) confirm what was already suspected, namely that neither could generate artifacts that would obscure indigenous meteorite B; i.e., one might confuse epoxy and meteorite, but epoxy would not be the source of unusually high B signals.

Most of the other analyses in Table 2 indicate that preparation materials are not potential sources of B contamination, with the striking exception of diamond-grit polishing paste, the first analyzed sample of which contained 150 ppm. Additional samples of this were obtained, and also of polishing grade alumina. The new diamond paste samples showed significantly less B, although the 3-μm paste is a source of concern; it is possible that the B level varies with the manufacturer, or even with the batch. Li is not present, as shown in Table 2, at significant levels. Our B analysis of alumina is similar to the value of 1 ppm obtained by Weller et al. (1978).

It was desirable to find out whether the B occurs in the abrasive itself, or in the carrier liquid. A small amount

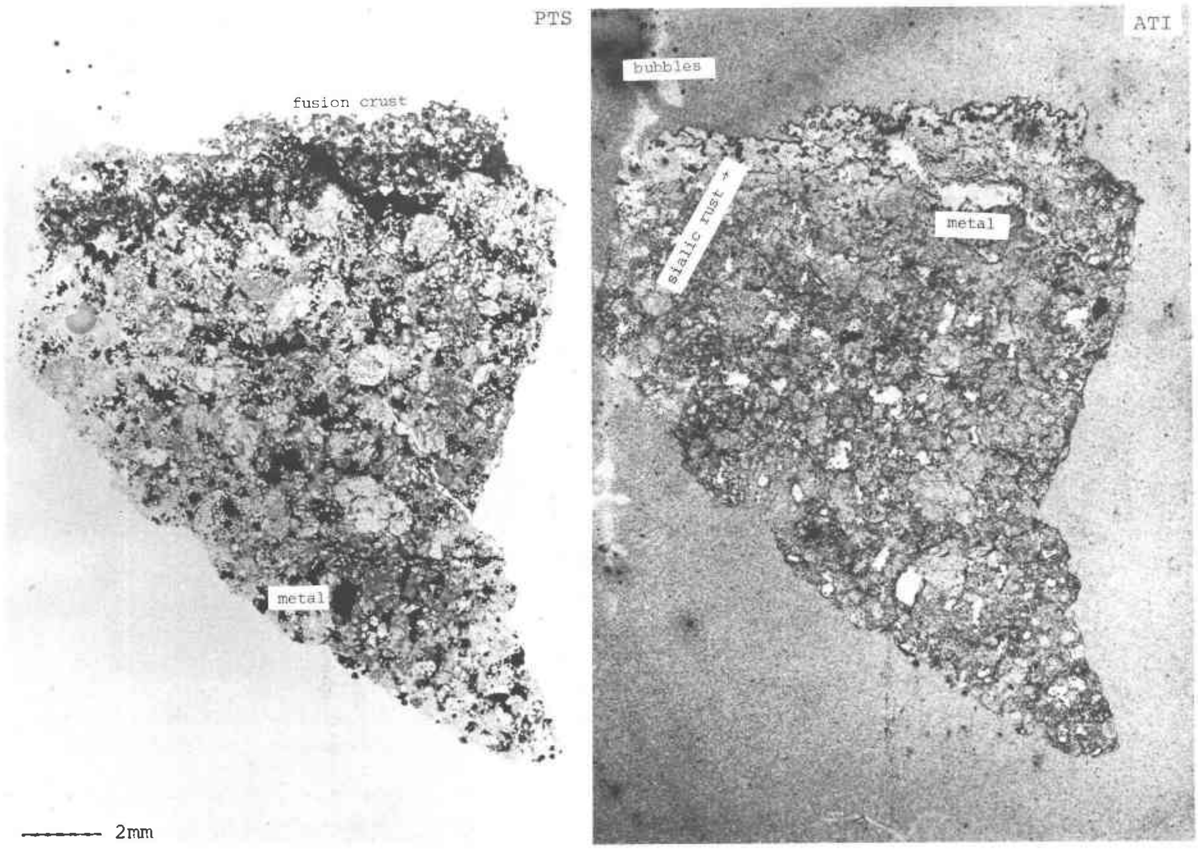

Fig. 2. RKPA78004,23. The top part of this H4 chondrite is fusion crust, much of which now consists of a microcrystalline aggregate of orthopyroxene. Within the meteorite there is abundant metal, which is black (opaque) in the PTS, but shows up white (low track density) in the ATI. Much of the fusion crust produces a homogeneous low-density track image, but areas of red-brown alteration ("sialic rust" of Gooding, 1986) give a darker image. Within the crust and also in the epoxy occur small point sources of intense track-density that look like bubbles and are visible in the PTS also; these tracks indicate contamination by B (see text).

of the 3-μm diamond grit was extracted with distilled water, then washed, dried, and mounted on epoxy for preparation of an ATI. The grit was partially dispersed into loose individual grains, but much remained in dense aggregates. The ATI showed that the aggregates are the source of the alpha-tracks and individual grains give no record. This indicates that B is in the carrier liquid, which had not been completely removed, but not in the grit itself.

It is concluded that any PTS prepared using diamond-grit polishing paste will acquire B contamination, by adsorption onto receptive surfaces. In addition, spots of paste adhering to the underside of a section, or to the adjacent epoxy, might embed themselves to form subspherical point sources of the kind shown in Figure 2; spots in the epoxy beneath a section would presumably not generate tracks, except in cases where the grinding has abraded through the section.

In one of the first studies of ATIs of rocks and meteorites, M. G. Seitz described (1973, p. 592), in a ground and polished specimen of the chondrite Saint-Severin, the inhomogeneous distribution of B that "occurs mostly

TABLE 2. B content (ppm) of materials used in the preparation of meteorites and lunar rocks

Material	B ppm	Li ppm
First set (materials used at JSC)		
Mineral spirits	0.1	
Almag grinding oil	<0.1	
Mineral polishing oil	<0.1	
Cotton rag paper	0.7	
Epoxy cement no. 208	0.2	
Epoxy cement Araldite no. 506		
low track density sample	0.55	
high track density sample	0.42	
Glass microscope slide	1.96	
Silica microscope slide	5.3	
Diamond compound, M 1	150	
Second set (various materials)		
1-μm diamond compound, M 1*	0.96	
3-μm diamond compound, M 1*	8.6	
3-μm diamond compound, M 2*	21.0	<1.6
3-μm diamond compound, M 3*	1.4	<1.2
Aluminum oxide (alumina) powder	1.82	

* M 1 = Metallurgical Systems Inc.; M 2 = Metadi-II; M 3 = Micro-Metallurgical M-3.

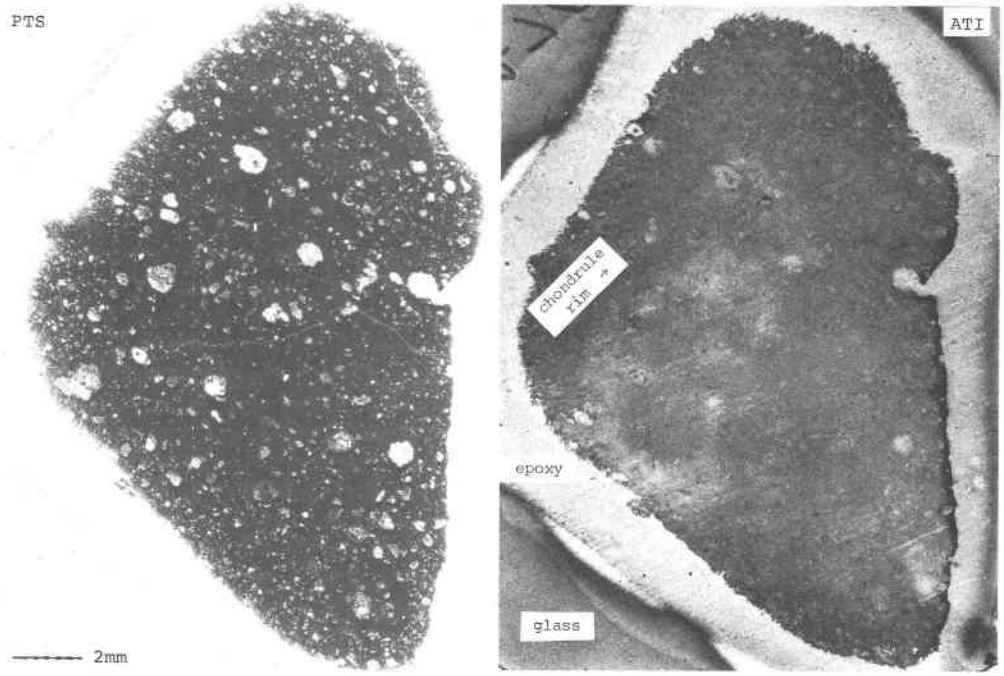

Fig. 3. Photograph and ATI of a thin section of Murchison prepared dry, i.e., without any lubricating liquids or diamond polishing paste during the sawing, grinding, and polishing operations. The section has a normal thickness overall, but some parts are thinner and show scratches from the grinding grit, and the matrix is nearly opaque; there are some holes that resulted from plucking, but chondrules and inclusions are also present. All three give rise to track densities lower than the matrix; a few of the chondrule images show dark rims, although very weakly. The dark line on the RH side of the ATI is an artifact that results from over-etching. The meteorite is embedded in an epoxy resin (light ATI) whose image is surrounded by a darker glass image.

in grain boundaries or patches of fine-grained material." He attributed the B-rich fine-grained phase to chlorite or serpentine, but some of this may have been contamination, of the kind just described.

Specially prepared sections

The possibilities of contamination by extraneous B suggested the desirability of studying specially prepared clean sections. Three experiments were made.

First, through the good offices of Roy S. Clarke, Jr. of the Smithsonian Institution, Washington, D.C., a series of five special sections were prepared by Richard Johnson, Tim Rose, and Frank Walkup, of the same institution. The five sections were prepared from a sample of Murchison, using progressively fewer preparation materials, culminating in no. 5, which was prepared with no lubricants in either sawing, grinding or in the final polishing with tin oxide.

In spite of the extraordinary difficulty of preparing dry a section of a friable material such as this, PTS no. 5 was superficially little different from the others; the size was no less than usual, the thickness quite normal, and the only signs of different treatment were the presence of striations from the coarse silicon carbide grinding, and a few holes that presumably followed plucking of chondrules (see Fig. 3).

After the care that was put into the preparation of these sections, it would be pleasant if it could be demonstrated that they succeeded in resolving the contamination problems. But to our surprise, their ATIs were all strikingly similar to the one in Figure 3, uniformly weak and lacking in detail, with much less structure visible than in the specimen of Murchison shown in Figure 1. Because of this the sections were irradiated again, to make a second set of ATIs; these were etched more thoroughly, but yielded little improvement.

The track images nevertheless resembled other unequilibrated chondrite images, with a concentration of track-forming elements in the matrix and weaker images in chondrules and clasts. Dark rims are weakly visible in each ATI (the opacity of the matrix conceals almost every one in the PTS photographs), even in no. 5, which was not exposed to diamond paste (see Fig. 3).

Allende slab

A second experiment in preparing a "clean" specimen was kindly arranged by E. J. Olsen at the Field Museum, with the particular aim of excluding epoxy cement; however, the final polish used diamond paste, which was not at that time known to be a source of grief. In order to use a rather friable carbonaceous chondrite, this plan necessitated the substitution of a slab for a thin section.

The PTS photograph in Figure 4 was taken in reflected light and in fact reveals texture better than expected (the

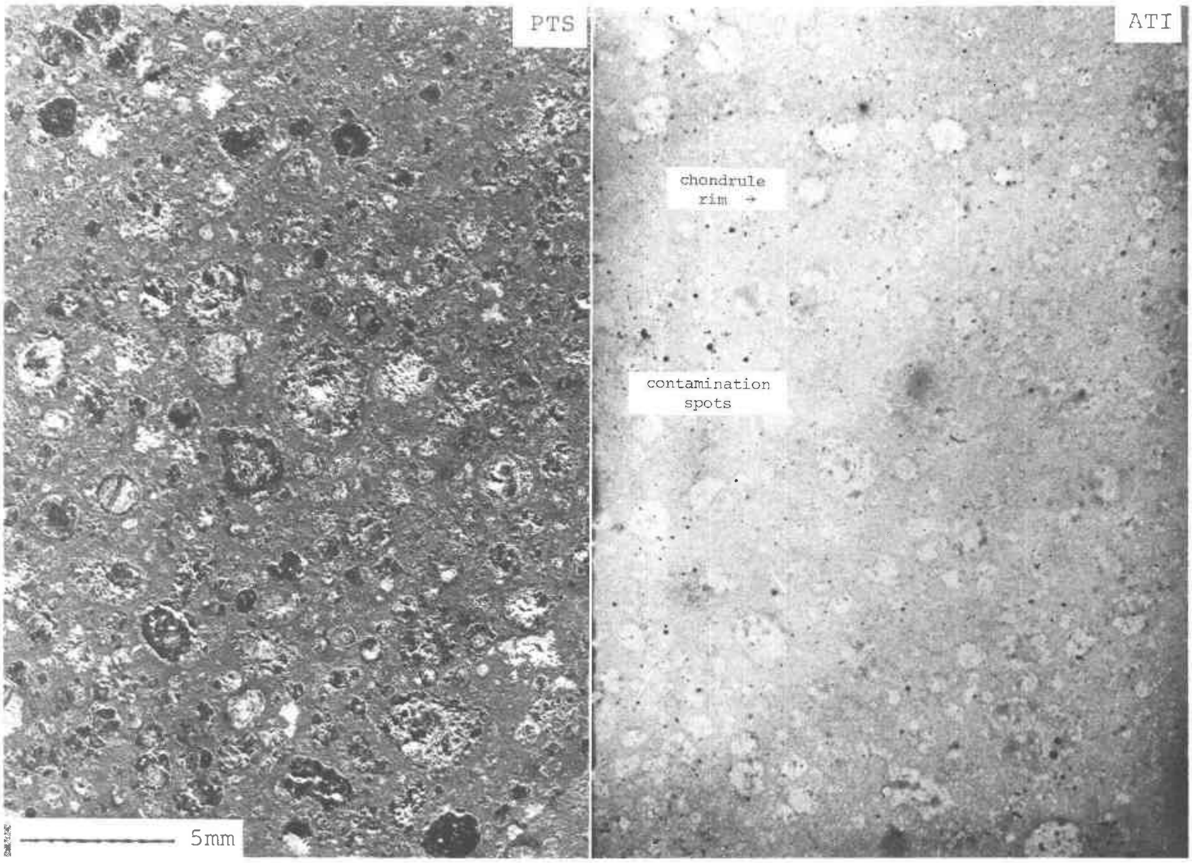

Fig. 4. Reflected light photograph and ATI of a slab of Allende (3 cm long), cut, ground, and polished without using any epoxy or detergent. The contrast is low overall in the ATI, probably because of the imperfect polish, but chondrules and inclusions have weaker track images than the matrix; one or two chondrules show rims. In addition there are many track-intense point sources, which may mark residues of diamond polishing paste.

surface had not as high a polish as it would have had with epoxy impregnation). Among the wide variety of chondrules and inclusions, a few have unequivocal rims, although others only appear to do so because the softer matrix has been ground down below their level.

The first ATI prepared was weak and showed poor contrast and resolution, so was repeated to give the example in Figure 4, which still needed strong photographic enhancement to bring out the textural detail. The record is similar to what was seen in the Murchison sections, except that the chondrules are clearer. In addition there are numerous track-dense point sources, which may be attributed to residual spots of diamond paste. The evidence of this specimen confirms that epoxy cement does not present problems in the interpretation of ATIs.

Other clean sections

Finally, a series of specially prepared sections was obtained from the curatorial facility at the Planetary Materials Branch, NASA/JSC. These were skillfully prepared by J. Holder and R. Marlow using only silicon carbide sandpaper (3M plastic sheets), followed by polishing with alumina powder in ethanol. The sections were cut from the "potted butts" (sawed meteorite fragments impregnated with epoxy) remaining from the previously examined Antarctic sections (e.g., Fig. 2).

The ATIs obtained from these sections show all the petrographic features described earlier, including weathering effects, but lacking the high-track-density bubbles. Examples will be shown in a forthcoming paper.

It was concluded that diamond paste is a source of serious B contamination, but that the other petrographic features in "clean" ATIs indicate indigenous B or Li.

Summary

Meteorite ATIs may include the effects of both indigenous and introduced B; the problem in each case is to distinguish the two and to estimate their relative importance.

Alpha-track images reveal useful petrographic detail in meteorites and may supplement optical petrography; they give qualitative indications of B and Li distribution.

Chondrule rims are visible in the ATIs of some meteorites of low grade. In many cases, these rim enhancements are artifacts produced by B contamination during section preparation, resulting from the use of commercial diamond polishing paste; where such contaminants have not been used, chondrule rims may have cosmochemical significance.

Point sources of intense track abundance occur in many ATIs; they are particularly noticeable within fusion crusts but also occur outside the meteorite slice, within the epoxy cement, showing spherical outlines; they appear to be bubbles lined with track-producing elements and result from the use of diamond polishing paste.

Indigenous B and Li may be mapped using alpha tracks. But for meaningful results, it is essential that B-bearing materials should be stringently avoided in preparing meteorites (and many rocks) for alpha track study.

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References cited

Carpenter, B.S. (1972) Quantitative applications of the nuclear track technique. Microscope, 20, 175–182.

Curtis, D.B., and Gladney, E.S. (1985) Boron cosmochemistry. Earth and Planetary Science Letters, 75, 311–320.

Curtis, D.B., Gladney, E.S., and Jurney, E. (1980) A revision of the meteorite based cosmic abundance of boron. Geochimica et Cosmochimica Acta, 44, 1945–1954.

Fleischer, R.L., and Lovett, D.B. (1968) Uranium and boron content of water by particle track etching. Geochimica et Cosmochimica Acta, 32, 1126–1128.

Furst, M., Lowenstam, H., and Burnett, D.S. (1976) Radiographic study of the distribution of boron in recent mollusc shells. Geochimica et Cosmochimica Acta, 40, 1381–1386.

Gooding, J. (1986) Clay mineraloid weathering products in Antarctic meteorites. Geochimica et Cosmochimica Acta, 50, 2215–2224.

Higgins, M.D., and Shaw, D.M. (1985) Induced alpha-track mapping of chondrites: A petrographic tool (abs.). Proceedings, 48th Annual Meeting, Meteoritical Society, July 1985, Bordeaux, p. 67.

Higgins, M.D., Truscott, M.G., Shaw, D.M., Bergeron, M., Buffet, G.H., Copley, J.R.D., and Prestwich, W.V. (1984) Prompt gamma neutron activation analysis at McMaster nuclear reactor. In O.K. Harling, L. Clark, and P. von der Hardt, Eds. Use and development of low and medium flux research reactors, Supplement to vol. 44, p. 690–697. Atomkern-Energie Kerntechnik.

Kleeman, J.D. (1973) The role of lithium and boron in forming Lexan plastic prints. Geochimica et Cosmochimica Acta, 37, 1321–1326.

Murty, S.V.S., Shukla, P.N., and Goel, P.S. (1983) Lithium in stone meteorites and stony irons. Meteoritics, 18, 123–136.

Nichiporuk, W. (1971) Lithium. In B. Mason, Ed., Handbook of elemental abundances in meteorites, p. 67–72. Gordon and Breach, New York.

Nichiporuk, W., and Moore, C.B. (1974) Lithium, sodium and potassium abundances in carbonaceous chondrites. Geochimica et Cosmochimica Acta, 38, 1691–1703.

Phinney, D., Whitehead, B., and Anderson, D. (1979) Li, Be and B in minerals of a refractory-rich Allende inclusion. Proceedings of the 10th Lunar and Planetary Science Conference, 885–905.

Seitz, M.G. (1973) Boron mapping and partitioning in synthetic and natural systems: Crystal-melt assemblages, garnet lherzolite, chondrites. Carnegie Institution of Washington Year Book 72, 588–592.

Seitz, M.G., and Hart, S.R. (1973) Uranium and boron distributions in some oceanic ultramafic rocks. Earth and Planetary Science Letters, 21, 97–107.

Shaw, D.M., Higgins, M.D., and Truscott, M.G. (1986) Status of alpha track imaging of chondrites for B/Li distributions (abs.). Meteoritics, 21, 510.

Shaw, D.M., Higgins, M.D., Truscott, M.G., and Middleton, T.A. (1987) Alpha-track imaging of chondrites and boron cosmochemistry (abs.). Terra Cognita, 7, 378–379.

Shaw, D.M., Truscott, M.G., Gray, E.A., and Middleton, T.A. (1988) Boron and lithium in rocks and minerals from the Wawa-Kapuskasing region, Ontario. Canadian Journal of Earth Sciences, in press.

Steele, K.M., Hervig, R.L., Hutcheon, I.D., and Smith, J.V. (1981) Ion microprobe techniques and analysis of olivine and low-Ca pyroxene. American Mineralogist, 66, 526–546.

Truscott, M.G., and Shaw, D.M. (1984) Boron in chert and Precambrian siliceous iron formations. Geochimica et Cosmochimica Acta, 48, 2213–2320.

Truscott, M.G., and Shaw, M.D., and Cramer, J.J. (1986) Boron abundance and localization in granulites and the lower continental crust. Bulletin of the Geological Society of Finland, 58, part 1, 169–177.

Weller, M.R., Furst, M., Tombrello, T.A., and Burnett, D.S. (1978) Boron concentrations in carbonaceous chondrites. Geochimica et Cosmochimica Acta, 42, 999–1010.

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