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# Distinguishing shocked from tectonically deformed quartz by the use of the SEM and chemical etching

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#### Abstract

Multiple sets of crystallographically-oriented planar deformation features (PDFs) are generated by high-strain-rate shock waves at pressures of > 12 GPa in naturally shocked quartz samples. On surfaces, PDFs appear as narrow (50–500 nm) lamellae filled with amorphosed quartz (diaplectic glass) which can be etched with hydrofluoric acid or with hydrothermal alkaline solutions. In contrast, slow-strain-rate tectonic deformation pressure produces wider, semi-linear and widely spaced arrays of dislocation loops that are not glass filled.

Etching samples with HF before examination in a scanning electron microscope (SEM) allows for unambiguous visual distinction between glass-filled PDFs and glass-free tectonic deformation arrays in quartz. This etching also reveals the internal 'pillaring' often characteristic of shock-induced PDFs. This technique is useful for easily distinguishing between shock and tectonic deformation in quartz, but does not replace optical techniques for characterizing the shock features.

Keywords: scanning electron microscopy; etching; quartz; deformation; shock metamorphism

#### 1. Introduction

After three decades of study, the importance of correctly identifying shocked minerals has become obvious (e.g. [1] and references therein). When evidence of shock metamorphism is found in grains of quartz or feldspar, it has the implication that a shock pressure wave greater than 10-12 GPa has passed through the sample, requiring exposure to a hypervelocity impact by a meteorite or cometary body. In a

number of cases, such as the Cretaceous/Tertiary (K/T) boundary layer, this information has proven pivotal for interpreting major geological events [2,3]. However, visual identification of shock metamorphic features such as PDFs in the optical microscope can be somewhat ambiguous and controversial, as witnessed by the considerable debate over the origin of microstructures in volcanic materials from the Toba Caldera and, by extension, their implications for the K/T boundary event [4–7]. Similar features in quartz grains from a sandstone near the K/T boundary within the Deccan Traps of India were likewise optically misidentified as shock lamellae [8,9].

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Shock metamorphism in quartz produces planar deformation features (PDFs), submicron Brazil and Dauphiné twins, fractures (sometimes accompanied by crystal rotations producing optical mosaicism), diaplectic glass, high pressure phases (notable coesite and stishovite), and melting (Table 1). For the purposes of this paper, which focuses on etching, PDFs refer only to glass-filled shock features. The development of each feature type varies significantly as a function of shock pressure and duration, target temperature and, possibly, other parameters [10]. Transmission electron microscope (TEM) studies have produced detailed descriptions of the planar feature defects induced by shock [11-17]. Microfault zones are shear fractures on which melting has occurred. Although often grouped with other planar structures, they form by a different mechanism and can occur at any pressure above  $\sim 7$  GPa. Narrow (< 10 nm) Brazil twins and transformation lamellae (glass layers) are the earliest features to appear, at pressures of 10-15 GPa, and are not visible optically. The optically visible 'planar deformation features' (PDFs) most commonly studied are not, in

Table 1

Features seen in quartz shocked naturally and in the laboratory

fact, due to deformation but, rather, to transformation (i.e. planar fractures formed in tension during initial release from shock loading [15]). They consist of 50-500 nm wide crystallographic or subcrystallographic transformation lamellae comprised of layers of glass, highly fractured quartz, and possibly trace amounts of high-pressure phases. In some cases a **sublamellar** structure is present within individual PDF lamellae [18]. Such PDFs form at 12-40 GPa and are the loci of collapse of the quartz lattice, largely by solid-state amorphization, when it is compressed above its stability field. Many of these shock-derived microstructures produce trains of inclusions when annealed. These data are summarized in Table 1.

Because all of these features are narrower than 1  $\mu$ m, the optical microscope is not ideally suited to their identification and it is possible for even fairly experienced workers to confuse visually tectonic deformation (Boehm) lamellae with PDFs. The former, the product of geologic strain rates, are dislocation features with no glass production and are subject to annealing by thermal recovery into bubble trails (e.g.

Feature	Description	Occurrence	Minimum pressure to form
Fractures *	Open	All shocked quartz	Any pressure
Microfault zones *	Crystallographic shear cleavages, esp. on {1011}, containing quenched melt	Most shocked quartz, especially in large grains	7 GPa
Narrow transformation lamellae	Fractures ± glass, esp. on {0001}, < 10 nm wide	Only in laboratory shock experiments	10 GPa
Brazil microtwins	Narrow basał twin lamellae with dissociated partial dislocations	Natural, some laboratory samples, in areas lacking PDFs	10 GPa
Wide transformation lamellae *	50-500 nm layers of diaplectic glass ± fractures ± high-pressure phases, esp. on {10Tn}	Natural and laboratory samples	12-15 GPa
Dislocation bands *	Dense, planar tangles of glass-free dislocations	Only seen in natural samples; may be inherited	Unknown (tectonic)
High-P phases (coesite, stishovite)	Growing from intergranular melts; trace amounts on lamellae?	Most impact sites, rare or absent in shock experiments	20 GPa
Diaplectic glass	Completely amorphized areas without signs of flowage	All quartz shocked to above $\sim$ 30 GPa.	30 GPa
Lechatelierite	Bubbly, amorphous material (glass) as inclusions from melting of quartz grains	Naturally and laboratory-shocked rocks	35 GPa
Melting	Flowed, amorphous material	All quartz shocked to above $\sim 50$ GPa	50 GPa

\* Features visible as planar structures (PDFs) in the optical microscope.

[19]). Furthermore, PDFs must be oriented nearvertically to be seen optically, requiring either a universal or spindle stage to observe some of these shock features. For these reasons, either an SEM or TEM is the optimal tool at present for detailed visual characterization of shock features. These tools allow not only the unequivocal identification of shock features, but also considerable analysis of their structure and any modifications they may have experienced after formation.

However, TEM techniques are not available to all researchers, do not allow examination of more than a few grains in a hand specimen, and sample preparation is difficult and time-intensive. Some workers have come to rely on etching techniques and the use of a scanning electron microscope (SEM) for identifying PDFs [2,20,21]. An SEM is easier to use and more generally available than is a TEM. This article describes results of three etching techniques for distinguishing shock features that may appear superficially similar to tectonic lamellae with optical examination alonc.

# 2. Experiments

The use of etching in structural geology and materials science is based on preferential chemical attack on deformed crystals or on different phases [22,23]. Fluorine-based solutions are commonly used to attack silicates, and several recipes exist for mixtures optimized for attack of quartz, feldspar, olivine,



Fig. 1. HF liquid-etched (3 min) shocked quartzite from Haughton impact crater, NW Territories, Canada (SEM photos). (a) Parallel sets of shock lamellae with pillaring, running SW-NE in photo. Note irregular line of curved dislocations trending normal to PDFs (SE-NW); these are inherited dislocations formed tectonically in this sample prior to impact. (b) Close-up of PDFs shown in (a) at intersection with dislocation; note pillaring. (c) Two different sets of intersecting PDFs. These multiple sets commonly occur in shocked quartz, but are rare to nonexistent in tectonically deformed quartz. (d) Extremely thin lamellae (basal?) that would not be visible optically; these probably contain only a thin coating of glass. Note absolutely straight parallel, planar character of all of the glass-filled shock transformation features in this figure.

and other rock-forming minerals ([24] and references therein). Etching may also be induced by sublimation or by ion sputtering. An etchant specific to PDFs provides an interesting variation on more conventional methods: such a procedure should preferentially attack glass, the main component of most transformation lamellae, without significantly affecting the dislocations which make up tectonic lamellae.

To this end, we experimented with several different etchants. Four samples were etched and then examined by optical microscopy and SEM: two shock-metamorphosed samples, one from the 23 Ma Haughton crater [25] and another from a K/Tboundary site in Western North America [26] and two samples containing abundant tectonic deformation lamellae, including a Cambrian novaculite from Arkansas and a Cambrian quartzite from Scotland. In addition, a few laboratory-shocked quartz samples [7] were studied. Unetched samples were also examined optically and by TEM. PDFs seen in the Haughton impact samples consist of a combination of glassy and fractured material, along with bubbles, voids, and dislocations due to post-shock alteration. They generally have a strong sublamellar structure. K/T boundary shocked grains have glass lamellae frequently annealed to similar mixtures of glass and bubbles. Samples from low-pressure (12 GPa) shock experiments contain narrow transformation lamellae, microfault zones, Dauphiné twins, and extensive



Fig. 2. HF fluid-etched (3 min) Arkansas novaculite with tectonic deformation (Boehm) lamellae (SEM photos). (a) Set of partial dislocation loops aligned into NE-SW oriented lamellae. These lamellae look solid and slightly curving in the optical microscope. (b) Another set of these dislocation lamellae; white box on left photo enlarged  $3 \times to$  form right photo. (c) Another lamella with en echelon partial dislocation loops. (d) Curved segments of dislocation loops formed en echelon into lamellae. Note that none of these tectonic deformation lamellae seem to be glass-filled, none are parallel-sided or straight and planar, multiple intersecting sets are lacking, and the lamellae are composed of linear arrays of individual dislocations.

fracturing, whereas higher-pressure samples (27 GPa) contain abundant wide transformation lamellae, often with a sublamellar structure.

#### 2.1. HF liquid etching

Past studies by Bohor and co-workers [2,20] have made considerable use of liquid HF etching combined with SEM examination in characterizing K/T boundary distal ejecta. Applying these techniques here, grains were rinsed for several minutes in a 30% HF solution. Liquid HF deeply etched PDFs in shocked samples, yielding narrow (< 1  $\mu$ m), sharply planar trenches, often consisting of planar, transverse, sublamellar pits (Fig. 1). Etching of the K/T sediments produced similar results. The same etchant



Fig. 3. HF vapor-etched shocked quartzite from Haughton impact crater, NW Territories, Canada (SEM photos), showing two different sets of intersecting PDFs. These multiple sets commonly occur in shocked quartz, but are rare to nonexistent in tectonically deformed quartz. Note absolutely straight parallel, planar character of all of the glass-filled shock transformation features in this figure.



Fig. 4. Hydrothermally etched (with alkaline solutions) lamellar features (SEM photos). (a) Intersecting sets of PDFs, Haughton impact sample. Note etching of quartz boardering planar features. (b) Sinuous, parallel tectonic deformation lamellae in Northwest Highlands quartzite. Note that none of the tectonic deformation lamellae seem to have been glass-filled, none are parallel-sided or straight and planar, multiple intersecting sets are lacking. The etched lamellae are composed of broad, semi-parallel arrays of individual dislocations.

applied to tectonic lamellae produced wide, linear  $(5-10 \ \mu m)$  arrays of curved dislocations in the tectonized Arkansas novaculite containing deformation lamellae (Fig. 2). The method, while requiring considerable caution in handling HF, is rapid and easily used on loose grains. After etching, workers should be careful to neutralize samples thoroughly (e.g. in 5% Na<sub>2</sub>CO<sub>3</sub>) to avoid etching microscope lenses by HF vapor. The low vapor pressure (No. 6)

etchant of Wegner and Christie [27], ammonium bifluoride  $(NH_4HF_2)$  and fluoboric acid  $(HBF_4)$  was not tested, but might provide alternative, somewhat safer procedures.

### 2.2. HF vapor etching

A second approach is to etch with HF vapor for 1-5 min, a method originally used by Christie et al. [28]. Fig. 3 shows that etch structures are comparable to those produced by liquid HF; no attack whatever was noted on tectonic lamellae. The method is very safe: a bottle of liquid HF is taped to the side of a fume hood, and a thin section or grain mount is simply laid on top of the bottle for 1-5 min. A single bottle may be reused almost indefinitely. On the other hand, vapor etching has not proven effective on loose grains, possibly due to precipitation and/or beading on the surface.

### 2.3. Hydrothermal etching

Hot alkaline solutions  $(0.001-0.01 \ M$  KOH at 166-211°C for ~4 h) in a rocked, Teflon-lined vessel [29] aggressively attacked shock lamellae (Fig. 4a), but also readily etched tectonic lamellae (Fig. 4b). There were clear differences between the morphologies of the resultant etch structures: shock lamellae were sharply planar with a distinctive sub-lamellar texture, whereas tectonic lamellae were wide (~10  $\mu$ m) and curved, with a cheesy or spongy texture similar to that of highly deformed synthetic crystals [30]. The more intense etching of both transformation and tectonic lamellae produced hydrothermally might be reduced by lowering the temperature or solution alkalinity.

### 3. Discussion

SEM analysis reveals that for all etchants shockmetamorphic PDFs are sharply planar features < 1 $\mu$ m wide, frequently with a sublamellar structure. In contrast, tectonic lamellae are only weakly and irregularly attacked by etchants; these lamellae are  $\gg 1$  $\mu$ m wide bands of partial dislocation loops that are often sinuous. These differences allow a clear distinction to be made between shock-induced and tectonic features. Etching rates vary somewhat from sample to sample, but show qualitatively similar behavior for a range of shocked materials. Caution dictates that unknown samples be sequentially etched, starting with very short exposures.

Which shock-induced features are revealed by etching? Narrow transformation lamellae were not obvious after chemical etching. We suspect that, due to the very fine scale of the narrow lamellae, any such etching is manifest as faster dissolving regions rather than as individual etch structures. We also saw no evidence for etching of microtwins, again possibly due to their fine scale. Etching also attacks microfault zones, but reveals little detailed information about these features which is not available from optical microscopy.

Instead, etching reveals those features (wide transformation lamellae) that are also optically prominent as PDFs. These lamellae etch distinctly in both natural and synthetic samples, and reveal their sublamellar structure if the sublamellae are widely spaced (e.g. Fig. 1a). Vapor etching (with SEM and TEM study) on the same laboratory-shocked samples yields strikingly similar images (Fig. 3a). Because sublamellae are usually normal to  $\mathbf{c}$  or  $\mathbf{a}$  (directions of rapid dissolution), etching times should be kept to a minimum to prevent removal of the crystalline material separating glass sublamellae.

Chemical etching can be used effectively for mapping PDFs in a thin section, and the reconnaissance can be extended to explore PDF abundances and distributions. For example, vapor-etched shock lamellae from the Haughton crater terminate at the edge of most overgrowths (Fig. 3b), although in rare cases lamellae extend into overgrowths. This suggests that impact occurred in the early stages of the overgrowth process, and that most overgrowths formed after the impact event. Severe authigenic silica overgrowth on quartz grains commonly occurs in a marine environment, making visual identification of PDFs both optically and using the SEM difficult (e.g. [31]). In these cases, breaking open the overgrown shocked grains by crushing and then reetching and remounting for SEM examination will reveal the PDFs on internal fracture surfaces.

One characteristic of etched transformation lamellae is the 'pillaring' that can be observed (Fig. 5). These 'pillars' probably represent intersecting fine



Fig. 5. SEM photo of HF-etched shocked quartz grain from the K/T fireball layer, Berwind Canyon site, Colorado. Note strong 'pillaring' in both intersecting sets of PDFs. 'Pillars' have the same orientation across the two sets, suggesting that they are caused by a separate set of fine crystalline lamellae (probably basal). From [20].

crystalline lamellae having either (0001) or (0111) orientations. These lamellae are either normal to the planar boundaries of the glass-filled PDFs or at high angles to them [14]. Gratz et al. [15] have also reported seeing fine basal (0001) lamellae in laboratory-shocked quartz viewed with a TEM. The scale of these features indicates that they can only be seen at the high magnifications provided by an SEM or TEM, and not optically. Large, open (glass-free) microfractures in quartz typically do not show this pillaring after etching.

Much of the confusion and contention about PDFs in quartz grains at the K/T boundary and their ability to define a shock-metamorphic origin could have been avoided by following a procedure of HF etching and SEM examination. Lyons et al. [32] commented that optical examination alone cannot distinguish fully between shock and tectonic effects, and suggested that TEM examination might be necessary for this characterization. We reiterate that etching and SEM examination is normally adequate for this task, as shown by the examples in this paper.

The basis for distinguishing between the effects of shock-induced transformation and tectonic deformation in quartz is the unique formation of diaplectic silica glass by the former process. This glass is present ubiquitously in quartz shocked above 10 GPa, and increases in amount as some function of increasing shock pressure (e.g. [33]). The presence of

this glass can be revealed by measuring the decrease in refractive index of individual quartz grains, as by [2,3] for shocked grains for K/T boundary clays. These refractive index measurements, the etching effects seen in PDFs and direct observation by TEM [14,15] all confirm the presence of shock metamorphism in quartz. In contrast, glass has never been reported as a normal component of tectonic deformation features in quartz (J. Christie, pers. commun., 1994). We emphasize that optical techniques are still primary in the characterization of shock metamorphism in quartz. Only with optical techniques can the crystallographic orientations and frequency of occurrence of specific orientations of PDFs be ascertained [1]. Likewise, only optical techniques can measure the decline in refractive index of the grain with increasing formation of diaplectic glass within lamellae as a function of increasing shock pressure (e.g. [33]). Examination of etched samples with the SEM, in order to distinguish confidently between shock transformation lamellae (PDFs) and tectonic deformation (Boehm) lamellae, can then be combined with optical measurements for further characterization of these shock effects.

# 4. Conclusions

Chemical etching combined with the SEM is a useful tool for revealing features due to shock metamorphism, and can also provide some information on their substructure. It facilitates a rapid search for shocked quartz, and can likely be adapted for use with other minerals which amorphize under pressure. It is simple to apply and requires no specialized equipment other than an SEM, which is necessary to resolve most of the ambiguity inherent in the optical identification of shock and tectonic microstructures. After screening with chemical etchants, samples can be selected for more detailed study by TEM and other techniques. SEM examination of shocked samples provides information on the same shock features (PDFs) which can be seen optically, but with greater detail and less sensitivity to grain orientation; these features form at above  $\sim 12$  GPa. Current etching techniques are less useful for studying material shocked to < 12 GPa, where narrow transformation lamellae and microtwins predominate. The chemical etching technique takes advantage of the phenomenon of glass formation within transformation lamellae and other features generated during shock metamorphism. Because tectonic deformation does not generate glass along the dislocations it produces, high magnification electron beam analysis of etched samples can easily discriminate between shocked and tectonic modes of deformation.

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