SCANNING ELECTRON MICROSCOPE STUDY OF CRACKS AND PORES IN CRYSTALLINE ROCKS

by

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ABSTRACT

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Direct observation of the microstructures in rocks was made possible through combined use of ion bombardment in sample preparation and the scanning electron microscope for viewing. Samples from six granites, a diabase, and a gabbro were observed. The porosity can be divided into pore-like and crack-like features. Pore concentrations are characteristic of plagioclase in granite. Most of the crack-like features are composed of rows of slots. For granites the aspect ratios are on the order of 10^3 or less; most cracks were 1 µm or less wide; and 2 to 4 cracks frequently met at a common point.

A preliminary study of the effects of mechanical and thermal stress was done, using two granites and a diabase that had been stressed in triaxial compression to over 95% of their fracture strength and a granite that had been heated to 400°F. In the mechanically stressed granites the rows of slots observed in the unstressed material were replaced by single high aspect ratio cracks. The cracks tended to be oriented parallel to the direction of maximum compression. In the thermally stressed granite, existing cracks extended and more grain boundaries were cracked. Quartz grains showed more new cracks than the other materials.

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INTRODUCTION

History of the Problem

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Cracks and pores are thought to have important effects on the physical properties of crystalline rocks [summarized, for example, in <u>Brace</u>, 1972]. Compressibility, electrical resistivity, thermal conductivity, and the velocity of compression and shear waves exhibit large changes, which have been attributed to the closure of cracks, over the first one to two kilobars of effective pressure.

Adams and Williamson [1923] were the first to suggest that cracks and pores were responsible for the non-linearity of elastic properties of rocks. As cracks close in a compressibility test, the bulk modulus increases and the pressure-strain curve steepens. After the first kilobar or two of pressure the cracks are closed and the curve is linear because the behavior of the rock is determined by constituent minerals which are nearly perfectly elastic [Walsh and Brace, 1966; Walsh, 1965a; Brace, 1965]. Walsh derived expressions for elastic constants by considering the effects of cracks and pores [Walsh, 1965a, 1965b, 1965c; Walsh and Brace, 1966]. The cavities were modeled as isolated simple geometric shapes in an infinite linearly elastic material.

In water-saturated crystalline rocks electrical conduction is almost totally through pore fluids. At low pressure a drastic increase in electrical resistivity is observed due to the closing of cracks. Porosity is the sole

property which determines the high pressure resistivity of water-saturated rocks composed of non-conducting minerals [Brace and Orange, 1968; Orange, 1965]. The correlation between electrical resistivity and porosity requires that most of the cracks in rocks be interconnected.

Cracks form effective barriers to the flow of heat when rocks are dry. When the rock is saturated or when cracks are closed by stress, the thermal conductivity increases [<u>Walsh and Decker</u>, 1966]. Since saturation increases the thermal conductivity, most of the cracks are probably interconnected.

In dry rocks with porosity in the form of cracks, the velocities of both compressional and shear waves at atmospheric pressure are often significantly lower than at a few kilobars of pressure. When such rocks are saturated with water the difference between compressional velocity at low pressure and at high pressure is reduced. The difference between dry and saturated compressional velocities increases with increasing crack porosity. There is no correlation with volumetric porosity. The shape of the cavities is important because the compressional velocity is only increased when the saturated cavities are in the form of cracks [<u>Nur and</u> Simmons, 1969].

Porosity of crystalline rocks, including those with low porosity can be measured with an accuracy of .001. This

suggests that most cavities are interconnected [Brace, 1971b]. In a study of permeability of granite, Brace et al [1968] used a model consisting of a single channel of constant crosssectional area. Measured permeability and porosity determined the hydraulic radius and allowed the shape of cavities in granite to be estimated. At low pressure, the cavities would be flat slits, while at higher pressure they would be equidimensional pores.

What is the real form of the cavities? Are the cracks interconnected or isolated? How concentrated are the cracks and pores? Where are they located relative to the grain boundaries? What kinds of aspect ratios do the cracks have? These are just a few of the questions that studies of the physical properties of rocks have raised.

Present Investigation

In this paper we show as reported earlier [Brace et al., 1972] that direct observation of details of porosity and grain structure in crystalline rocks is possible through combined use of ion bombardment for sample preparation and the scanning electron microscope for viewing. Details of the preparation of six granites, a diabase, and a gabbro are given. Cavity shapes, aspect ratios, connectivity and location are reported and compared with predictions from indirect measurements by other investigators. A preliminary study is made of mechanically and thermally stressed rocks and the observed characteristics are compared with those of the unstressed rocks.

Previous Attempts to Map Cracks in Rocks

Many attempts have been made to map cracks in rocks using thin or polished sections. In some instances an effort was made to decorate the cracks before sectioning.

Koide and Hoshino [1967] investigated microfractures in experimentally deformed rocks using thin sections. Their criteria for an open crack was one that appeared dark under crossed nicols and clear under uncrossed nicols. From their study they obtained the preferred orientation of microcracks. Willard and McWilliams [1969] determined the orientation of defects in thin sections. By their definition defects include grain boundaries, cleavage planes, twin planes, slip planes, inclusion planes, deformation bands, and microfractures. Defect orientation analysis records all cracks irrespective of their origin in time. Paulding [1965] observed many transgranular cracks in thin sections of undeformed Westerly granite. From thin and polished sections he reported that at stress differences of about half the fracture strength cracks began to grow from the pre-existing transgranular cracks and grains and were inclined to the direction of maximum compression and generally en echelon. Crack growth continued as stress was raised and was predominantly parallel to the direction of

maximum compression. <u>Wawersik and Brace</u> [1971] used polished sections to study crack densities, crack orientations, crack location relative to grain boundaries and orientation for the longer partial fractures in stressed rocks. One difficulty they noted was in distinguishing cracks from grain boundaries.

Baldridge and Simmons [1971] impregnated rocks with two types of water-thin compounds. One of the compounds used Canada Balsam to mark the cracks with its yellow color. The other compound was a mixture of furfuryl alcohol, which easily penetrated the pores, and hydrochloric acid, which polymerized the alcohol. The impregnated sample was heated to 750°C to complete the polymerization of the alcohol and to convert the polymer to carbon. Sub-micron sized particles of carbon colored the cracks. After the impregnation procedures the cracks were sectioned and polished. Both compounds enabled them to differentiate between original cracks, which were decorated, and cracks introduced in the thin sectioning process. From their studies they were able to get crack densities, crack orientations, average lengths, and crack intersections. Hadley [unpublished] attempted crack mapping using radioactive tritiated furfuryl alcohol. It was hoped that the introduction of a radioactive polymer into the cracks would permit a three-dimensional picture of the fissures to be built up by progressively sectioning the rock and placing it in contact with photographic film. Unfortunately, film with a fine enough grain size was not available.

<u>Timur et al</u> [1971] used the scanning electron microscope to observe samples that had been polished by a technique similar to that used in making thin sections. They were primarily concerned with the structure of pores.

All of the above studies produced unreliable results for one or more of the following reasons. In the projects based upon the observation of ordinary thin or polished sections, it is difficult, if not impossible, to differentiate between the original cracks and those created in the sectioning process. Often in polished sections it is a matter of personal bias whether a feature is a crack or a grain boundary. Crack decoration schemes enabled positive identification of some of the pre-sectioning cracks, but failed to show cavities that had received none of the marking fluid because they were disconnected from the outside.

No attempts were made to examine the fine details of cavity structure. Aspect ratios which depend on measurement of both length and width were not given.

Of all the results reported from these studies the crack orientations are probably the most accurate. If the cracks produced in the sectioning process were randomly oriented, they would appear as background in the measurements of the preferred orientation of the total crack population. The total number of cracks would be increased, but the preferred orientation would remain the same.

METHOD

Cores 1.04 cm in diameter were drilled from large oriented blocks. After the ends were ground perpendicular to their axes, the cores varied from 1.14 to 1.27 cm in length. Along the axis of each core was cut a groove which when viewed from one end served as an origin of coordinates.

The following procedures gradually eliminated the damaged layers from the surface of the sample. About 300 to 400 μ m of rock was removed by hand grinding with increasingly fine silicon carbide abrasives (240, 400, and 600 grit) on a metal wheel, followed by polishing with aluminum oxide abrasives (5, 1, and .3 μ m) on cloth laps. The next step was the crucial one. A layer about 30 to 40 μ m thick was removed by ion bombardment (which is also known as ion thinning) in a Commonwealth Scientific ion milling instrument. An ionized argon beam of about 6 kv bombarded the sample at an angle of 17°. This ion thinning was the vital difference between our specimens and the ordinary thin or polished section.

Ion thinning does not produce a perfectly smooth surface. Olivine and orthopyroxene thin at about 1 μ m/hr. Plagioclase thins at 2 μ m/hr. Quartz thins at 3 to 8 μ m/hr. Mica thins even faster than quartz. Thinning time was kept close to the minimum period necessary to remove the damaged layer (six hours for most rocks) so as to avoid very large topographic steps between different minerals. Bombardment also tends to produce gentle mounds much smaller than the grain size. These mounds appear to be a function of the beam rather than the mineralogy. Areas of weakness such as the regions surrounding cracks and pores do not thin faster than the rest of the mineral. Grain boundaries are not etched out.

Since specimens viewed with a scanning electron microscope (SEM) must have conducting surfaces, approximately 400 Å of a 40% gold 60% palladium alloy was vacuum evaporated onto the surface. There are many good references on the use of the SEM [Kimoto and Russ, 1969; Majumdar, 1970; Oatley et al., 1965].

To get a three-dimensional picture of the porosity, some samples were serially sectioned after photographs had been taken of selected fields under the SEM. First the metal coating was removed with 1 and 0.3 µm aluminum oxide abrasives. Then the surface was ion bombarded for six hours. If polishing did not precede thinning, any small irregularities in the coating seemed to cause very uneven thinning of the rock.

The effects of thermal stress were studied in a sample which, after being prepared as described above and photographed, was placed for $3\frac{1}{2}$ hours in a furnace at 400°F, which was calculated to be more than sufficient to induce many cracks in the rock. The sample was allowed to cool inside the furnace overnight. Recoating was unnecessary and the sample could be viewed immediately after removal from the furnace.

The mechanically stressed samples came from triaxial compression tests of dogbone-shaped specimens. The rocks were recovered intact after being loaded to over 95% of their fracture strength [Brace, Paulding and Scholz, 1966; Paulding, 1965]. The dogbones were cast in a mold, sawed in half lengthwise, and cores were drilled from their centers. The cores surfaces were polished, thinned, and coated, as described above.

ROCKS STUDIED

The rocks arranged in order of increasing total porosity are listed in Table 1, along with their crack and pore porosities, grain size, and modal analysis. The Westerly granite is from a block of so-called Westerly granite referred to in <u>Birch</u> [1960]. Many experiments have been conducted on other blocks of similar granite from Westerly, including permeability [<u>Brace</u>, Walsh and Frangos, 1968], linear compressibility [<u>Brace</u>, 1965], resistivity [<u>Brace and</u> <u>Orange</u>, 1968], shear wave velocity [<u>Simmons</u>, 1964], and compressional wave velocity [<u>Birch</u>, 1960]. Maryland diabase is from Block I [<u>Birch</u>, 1960] and was used in many of the same studies as Westerly granite [<u>Brace</u>, 1965; <u>Brace and</u> Orange, 1968; Simmons, 1964; Birch, 1960]. The Chelmsford granite is similar to that studied by <u>Todd and Simmons</u> [1972] and <u>Birch</u> [1960]. San Marcos gabbro was used by <u>Brace and</u> <u>Orange</u> [1968] and <u>Birch</u> [1960]. Raymond granite from the Raymond Quarry in Madera, California, granodiorite of Red Lake (referred to as Red Lake granite) near Shaver Lake, California, and two samples of Katahdin granite from Baxter State Park, Maine, were also studied.

OBSERVATIONS - UNSTRESSED ROCKS

General

Our samples when viewed with an SEM look radically different from thin sections of the same rocks viewed with an optical transmission microscope. SEM fields resemble fields seen with a reflecting optical microscope. With both the SEM and the reflecting microscope, only surface structures can be seen. An important difference in the capabilities of the two microscopes is the depth of focus; for the SEM it is 30 to 40 times greater than for the light microscope at a given magnification [White and Roy, 1968]. The other difference is in the range of magnifications available; an SEM is capable of magnifying an object from 100 to 30,000 times.

In SEM micrographs, cavity rims and sharp topographic steps frequently appear bright, because in these areas the

secondary electrons have an unusually large surface through which they can escape. Dark regions such as the centers of cracks may result from the shadowing of one region by another from the incident beam, or from the failure of electrons from a depressed region to reach the collector [Majumdar, 1970; Oatley et al., 1965; Kimoto and Russ, 1969].

As mentioned earlier, ion bombardment tends to produce gentle mounds, 40 μ m or less in diameter, which are a function of the beam. In Figure 6 the cracks near "c" are seen to have no effect on the mounds.

Mineralogy was determined from structural details such as cleavage or characteristic crystal form, or from composition determined with the electron microprobe.

When a rock is viewed at low magnification, the cavities seem to fall into two classes, pore-like and crack-like. The pore-like cavities are usually equant, but may have been aspect ratios as high as 10. Frequently they are randomly oriented as in plagioclase (Figure 2), but they also appear in lines (Figure 3). At high magnifications the crack-like features often resolve into rows of slots. A <u>slot</u> is a blunt-ended cavity of nearly rectangular cross-section. Many slots have aspect ratios greater than 10 and may be referred to by the general term, high aspect ratio cavity (HARC). Other cracklike features resolve into true cracks, which are HARCs with sharp ends.

HARCs

Long, narrow, sharp-ended cracks as imagined in t are relatively rare in many rocks. The term HARC is u to avoid some of the ambiguity and implicit shape invothe use of the term crack to refer to individual caviti The use of the word crack is reserved for sharp-ended cavities and high aspect ratio features that appear cracklike at low magnification.

Figure 1 shows a typical crack intersection in Westerly granite. The slots have black interiors and white edges. At "a" and "b" are two of the many delicate bridges dividing the cracks into slots. At lower magnifications these bridges are invisible, so these cracks seem to be uninterrupted HARCs. Individual slots often have blunt ends as seen here.

In most granites cracks have widths of 1 μ m or less. Along their length cavities vary in width, making it difficult to determine their aspect ratio. Measuring HARC length was difficult because the many tiny bridges made it hard to determine if a HARC were continuous and because the curves made it difficult to measure total length. The indicated problem is treated in more detail when the comparison between observed and predicted aspect ratios is discussed. Most HARCs are continuous for only 10 to 100 μ m, which is much less than a grain diameter. However, cracks composed of many slots had lengths on the order of a grain boundary in all of the granites except Westerly. Occasionally a mica grain was cracked for its entire length by a HARC. Extremely narrow HARCs, such as those 0.1 μ m wide, accounted for the highest aspect ratios, which were on the order of 10³. The aspect ratios for rocks with very low crack porosities such as Maryland diabase and San Marcos gabbro were about the same, but the widest cracks were about 0.33 μ m wide.

Pores

Pore-like features appear in several configurations, one of which is as scattered cavities. Pores of this type can be seen below point "b" in the quartz grain in Figure 2. Some of these pores are connected by fine cracks and others appear isolated in this section.

Dense concentrations of pores in feldspar, especially plagioclase, are common in granite. Three granites, Westerly (an_{17}) , Chelmsford (an_{20-25}) , and Red Lake (an_{30}) have concentrations of pores 7 µm or less in diameter, with many pores about .4 µm in size. In Katahdin granite I almost all of the plagioclase pores are less than .7 µm in diameter, while in Raymond granite many are about 10 µm in diameter. In Chelmsford granite (Figure 2) plagioclase grains have pore porosities of 2 to 3%. The microcline in Chelmsford granite has a high pore porosity, but not as high as the plagioclase. In Westerly granite the plagioclase, as mentioned, contains pore concentrations, but the microcline is relatively porefree. The plagioclases in San Marcos gabbro (an_{42}) and Maryland diabase (an_{67}) are not sites of pore concentrations.

Pores also appear in linear arrays. In some instances cracks connect these pores. Other times the pores are isolated inclusions enclosing daughter crystals (Figure 3). These trains of pores may mark the site of a healed crack.

Crack Intersections

Isolated HARCs, often with blunt ends, are not uncommon, but such HARCs may be connected with other cavities beneath the surface. Sometimes a thin band of uncracked rock lies between two HARCs which at low magnification seem to connect as at point "c" in Figure 1.

The number of cracks observed sharing a common point of intersection is generally 2 to 4 in granite. In Red Lake granite, 6 cracks meeting seems to be an upper bound, while in Westerly, 3 cracks meeting is maximum.

Sometimes it is difficult to determine whether a feature consists of one or more cracks, because the feature is very sinuous and/or is composed of slots. Some of the problem can be explained with reference to Figure 2. At "a" the crack strikes to the northeast, but at the plagioclase-quartz boundary, the crack curves until it strikes almost due east. Then it makes several sharp zig-zags until it curves gently near "b" so that it again strikes to the northeast. Since most of the major direction changes occur at places with relatively large radii of curvature, the entire feature was considered a single crack, although aspect ratios were only measured between bridges. When a large change in direction occurs at a sharp corner, the feature is generally considered to be two cracks. Crack counting tends to be subjective because of the continuous spectrum of radii of curvature possible at corners.

Grain Boundaries

The percentage of the grain boundary that is cracked varies significantly from rock to rock. In San Marcos gabbro (Figure 3) and Maryland diabase, the grain boundaries seem to be crack-free. From 20 to 85% of each grain boundary is cracked in Westerly granite. Examination at high magnification of the guartz grain in Westerly granite seen in Figure 4 shows that about 20% of its grain boundary is cracked. The HARCs all have aspect ratios less than 100 and are located in the bright regions designated "a", "b", and "c". In Chelmsford, Raymond and Katahdin I granites about 20 to 90% of each grain boundary is cracked. Almost all of the grain boundaries in Red Lake and Katahdin II granites are cracked. Even when most of a grain boundary is cracked, the cracking usually consists of many HARCs whose lengths are all much shorter than the apparent grain diameter.

OBSERVATIONS - MECHANICALLY STRESSED ROCKS

Specimens

Two samples of Westerly granite and one of Maryland diabase were made from cores that had been stressed in triaxial compression to within a few percent of their fracture strength. The strength of the rocks and the confining pressure are listed in Table 2.

Comparison with Unstressed Rocks

Westerly granite: The sharp contrast between stressed and unstressed Westerly granite can easily be seen by comparing Figure 4 and Figure 5. In the stressed rock the grain boundary is occupied by HARCs of lengths equivalent to a grain diameter. At point "a" in Figure 5, a HARC with an aspect ratio of a little more than 200 is located in the grain boundary. The HARCs are much broader and longer than in unstressed rock. At point "c" in Figure 5 there are cracks running from the grain boundary into the interiors of the grains on both sides. This type of cracking, beginning at the grain boundary, is typical of stressed Westerly granite but is never seen in the unstressed rock. Multiple crack intersections are common as at points "b" and "c" in Figure 5. In some places fine cracks crisscross the rock.

Figure 6 shows some of the details of cracks in stressed Westerly granite to be compared with Figure 1 for unstressed material. The delicate bridges are gone and the cracks are composed of single HARCs and exhibit a high degree of interconnectivity. Many of the cracks tend to be subparallel to the direction of maximum compression, but in feldspar the cracks are often at a large angle to the principle stress direction because they follow cleavage planes.

The orientations of the cracks in Figure 7a were measured and plotted in the histogram in Figure 7c. Although only 217 non-grain boundary cracks were plotted, a definite peak is evident near zero degrees to the direction of maximum compression. In a similar sized field in unstressed Westerly granite, there were only 83 cracks, and their scattered angular distribution is indicated on the histogram for comparison.

Maryland diabase: Although this rock was stressed nearly to fracture, most of the grain boundaries are uncracked. Steep topographic steps exist at the grain boundaries due to differences in thinning rates. In Figure 8 a crack can be seen crossing a steep plagioclase-pyroxene boundary which is intact. Many intra-granular cracks are also visible. Other fields show that the mineral cleavage planes exert a strong influence on intra-granular cracking.

OBSERVATIONS - THERMALLY STRESSED ROCK

A sample of Chelmsford granite was heated to 400°F in a furnace at atmospheric pressure. The sample was heated for 3¹/₂ hours and allowed to cool inside the furnace to room temperature. Before the thermal stressing, selected fields in the surface had been thoroughly examined and photographed with the SEM. These same fields were re-examined and rephotographed after the heat treatment.

The bridges across the cracks were broken. Existing cracks extended and intersected. Some cracks more than doubled in width. Sharp ends were observed on many HARCs. At some sharp corners of cracks which were present before heating, new cracks formed. Many previously uncracked grain boundaries were opened. Within some of the large quartz grains there were many new intersecting cracks. Although the feldspars are extremely porous (2.5%) the pores remained disconnected in the plane of observation.

The new cracks were observed at places which would be expected to be weak-grain boundaries, crack tips and corners. More new cracking marred the quartz than the other minerals. However, it is difficult to determine whether a crack is really new or just an extension of a previously existing crack, because we are limited to two dimensions. The "new" cracks could be extensions of cracks, which did not intersect the plane of observation before thermal stressing.

DISCUSSION

Is the Section Really Undisturbed?

There are several arguments in favor of the hypothesis that we are seeing cracks and poresas they exist in the interior of the rock. One argument is based on the observation that only a few of the cracks appear to be of purely brittle origin. Cracks of brittle origin, such as those seen in stressed rock would have sharp ends and would usually intersect each other. Instead, blunt-ended cracks which stop short of intersection are frequently seen. Delicate bridges, which could be due to healing since the rock was cracked early in its history, span the cracks. Paulding [1965] reported many transgranular cracks in thin sections of unstressed Westerly granite, which we have never seen. More support for the success of our damage removal technique comes from the fact that some rocks such as Maryland diabase are virtually devoid of cracks. Furthermore, the crack porosity of Chelmsford granite determined in our study is in close agreement with that measured by linear compressibility experiments.

Comparison of Observations and Predictions

<u>Brace</u> [1964, 1961] and <u>Brace and Walsh</u> [1966] have predicted that microcracks have lengths on the order of the grain size. This length estimate was based on the Griffith theory for tensile strength. $T_{O} = \left[\frac{2E\gamma}{\pi c}\right]^{\frac{1}{2}}$

 T_{o} = tensile strength

- E = Young's modulus
- γ = specific surface energy
- c = half the length of the Griffith crack
- 2c = the longest straight section of grain boundary
 (maximum grain diameter)

HARCs with lengths equivalent to the grain diameter of one of the moderate to large grains in the granites have not There are features of grain boundary size in been observed. some granites (Red Lake, Katahdin II and Chelmsford), but they are either long series of en echelon slots or cracks which are bridged every 20 to 30 µm. These features could be cracks which formed early in the rock's history and have since undergone varying amounts of recrystallization. In some places the bridges seem to be from recrystallization. With time, such bridged cracks might become a trail of pores as in Figure 3. Another possibility is that the slots were formed by stresses which were not large enough to create continuous cracks, such as those seen in the laboratory stressed samples. It is difficult to determine how extensive the bridging must be before the crack ceases to behave elastically like a single crack. A three-dimensional picture of the bridges is not available, so we do not know if they are pillars or more substantial walls. Do they crumble at relatively low pressures, or do they survive so that the aspect ratios of the slots between them can be used in predicting elastic properties? The longest continuous HARCs in granites are about 200 to $400 \mu m$ long. Long, straight HARCs are often found in mica.

In most granites almost all cracks are less than 1 μ m wide. The exception is Katahdin II, which contains large cracks about 100 μ m wide. Cracks in San Marcos gabbro and Maryland diabase are much less common, and those observed are less than .4 μ m wide. <u>Brace</u> [1971b], because of the failure of attempts to obtain a detailed picture of crack structure, predicted that cracks would be less than 10 μ m wide.

The aspect ratio (or ratio of crack length to crack width) is frequently referred to in crack theory. <u>Walsh</u> [1965b] and <u>Brace, Walsh and Frangos</u> [1968] have predicted from the pressure to close elliptical cracks that aspect ratios should be on the order of 10^3 . Problems arise in getting both the correct length and the correct width of a crack for determining its aspect ratio. The cross-section of a HARC is frequently very irregular, so that, for example, in some places it might be 1 µm wide and in others only .5 µm wide. The average width was used for our ratios. For bridged cracks the lengths of the individual HARCs were used, but conceivably if the bridging were not too substantial the length of the entire row of slots should have been used.

Can the aspect ratios measured be used in the formulas derived in crack theory? Elastic analyses invariably treat the case of an isolated cavity in an infinite medium with separation of one crack length being a good approximation to infinity [Walsh and Brace, 1966]. Cracks are frequently much closer together than one crack length. Our aspect ratios for granites have about the order of magnitude as those predicted, 10³, but a great many cracks have ratios less than 500.

Crack porosity can be determined to ±.05% from the intercept on the strain axis of the linear portion of the pressure-strain curve extrapolated to zero pressure [<u>Walsh</u>, 1965b]. Using this method, <u>Todd</u> [personal communication] found the crack porosity of Chelmsford granite's rift face to be .29%. By mapping cracks in a field of Chelmsford in the same direction, we found the porosity to be .26%.

Lack of a pronounced low pressure toe in the resistivity pressure curve indicates zero crack porosity [Brace and Orange, 1968]. The resistivity curve for Maryland diabase does not have a low pressure toe. In agreement, we found that the crack porosity of Maryland diabase is negligible.

Pore porosity can be determined to ±.1% by immersion. Total porosity is given by

$$\frac{\ell_{s} - \ell_{b}}{\ell_{s}}$$

$$\ell_{s} = \text{mineral specific gravity}$$

$$\ell_{b} = \text{bulk specific gravity}$$

The mineral specific gravity is found by immersion in carbon tatrachloride. Bulk specific gravity was measured in dried, precisely-ground cylindrical samples. Pore porosity is found by subtracting the crack porosity from the total porosity [Brace, 1965]. For Chelmsford granite the total porosity was 1.0%, so the pore porosity is 0.7% [Todd, personal communication].

By determining the typical porosity in photomicrographs of the major mineral phases in Chelmsford granite and multiplying each porosity by the percentage of the rock it comprises, a porosity of 1.2% was determined for that granite. The high estimate could be due to the pores in the plagioclase. Many of these pores are probably isolated fluid inclusions that do not contribute to porosity as measured by immersion.

The range in size of the pores in plagioclase from 10 μ m in diameter in Raymond to less than .7 μ m in diameter in Katahdin I may be related to pressure and hence to the depth at which the granite solidified. On this basis, Katahdin granite would be a shallow granite, whereas Raymond granite would be a deep one. Chelmsford, Westerly, and Red Lake would lie somewhere between those extremes. Another possibility is that the pores are a function of whether or not the rock is water-saturated.

A porosity-resistivity relation known as Archie's law is commonly observed for rocks.

$$\frac{\rho}{\rho_0} = \eta^{-r}$$

 ρ = the resistivity of the brine-saturated rock ρ_{o} = the resistivity of the brine η = pore porosity r = constant (2.0 ± 0.1)

Greenberg and Brace [1969] found that networks of resistors behaved like brine-saturated rocks. Networks with 3 to 6 intersecting elements gave r values in the range 2 to 3. For most granites, 2 to 4 cracks are commonly observed to intersect. The number for Westerly granite is somewhat lower, 2 to 3. For granites, various upper limits have also been placed on the number of cracks sharing an intersection (Table 3). In Red Lake granite as many as six cracks are observed intersecting at a common point, while for Westerly granite the maximum number is three. The other granites fall in between.

Scale of Features

Many important microstructures in low porosity crystalline rocks are a few microns or less in size. The extremely fine scale of the microporosity rules out detailed crack investigations based upon ordinary thin or polished sections. Most of the structures revealed by the scanning electron microscope in ion-thinned surfaces would be hidden by polishing damage in ordinary sections.

Ordinary polishing tends to etch out grain boundaries. Delicate bridges and slots are destroyed so that instead of a row of slots a groove is observed. The abrasives striate the mineral surfaces and fill the pores with debris. In unthinned specimens, only the largest pores are visible. Thin sectioning creates many transgranular cracks such as those seen by <u>Paulding</u> [1965] in Westerly granite. All of the features we have observed in Westerly granite have lengths less than a grain boundary.

Radioactive decoration of cracks in retrospect is unfeasible because the grain size of the film is larger than the features in the rocks. Other decoration schemes would fail to reveal the finer structures, because not enough coloring matter can be concentrated in the microstructures.

The Concept of Cracks and Pores

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Pores are approximately equidimensional cavities which are believed to provide networks of conduction paths to very high pressure [Brace, 1971a; Walsh, 1965a]. Cracks are extremely narrow openings such as cleavages and grain boundary separations which can be closed by application of a few kilobars of pressure. These are the definitions of cracks and pores used in the theories explaining the various physical properties.

In many micrographs such as Figure 2 a natural division between cracks and pores seems to be present. The feldspar grain is filled with more or less equant cavities. The quartz

grain contains a few equant cavities. Also, the crack in regions such as "a" intersects pores. However, are these really pores in the sense of the definition above? To satisfy the definition the equant cavities must be connected. Are these equant cavities tubes of nearly circular cross-section? Are they connected by fine cracks in another plane?

When the same field was rephotographed after about 30 \pm 10_µm had been removed, there was still a crack in the same general area with approximately the same curve. In the quartz, there was still a cluster of pores at "b". Thus these pores could be tubes. Unfortunately it was impossible to unequivocably follow any individual pores.

The cracking observed in various stress experiments seems to indicate that most of the pores in the feldspar are not connected, although some of those in quartz are. When the same area was viewed after thermal stressing, only one new crack appeared in the feldspar. Presumably cracks connecting the pores just below the surface prior to heating would have grown when the rock was thermally stressed and would now intersect the plane of view. After thermal stressing, more, but not all, of the pores in the quartz were connected. When rocks are mechanically stressed, the feldspars generally crack along cleavage planes rather than preferentially through the pores.

Further evidence arguing against the interconnectivity of most of the pores in feldspar is that if they were connected

the porosity as measured by immersion should be higher. As mentioned earlier, the porosity measured from the micrographs is significantly higher than that measured by immersion. Many of the pores in the feldspar could be fluid inclusions.

What then provides the conduction paths at high pressure? Some of the pores in quartz show evidence of being interconnected by cracks in another plane. Also, the crack in Figure 2 does intersect some pores. Another consideration is that many cracks are very irregular in cross-section and would probably not close completely with pressure.

The concept of cracks and pores may need some redefinition because many of the pores are not pores in the sense of the models. Some of the behavior attributed to pores may come from cracks which cannot close completely.

SUGGESTIONS FOR FUTURE RESEARCH

The method used here for viewing microstructure has enormous potential. Other researchers have reported crack orientations, crack lengths, crack densities, and crack intersections. In addition to measuring these parameters I have recorded crack aspect ratios, crack widths, and details of crack and pore shape for six granites, a diabase, and a gabbro, but many rock types have yet to be examined.

A preliminary investigation of the effects of mechanical stress on rocks showed that many cracks develop parallel to the direction of maximum compression. Determination of the onset of brittle cracking and a thorough SEM study of the stages into which the typical stress-strain curve has been divided [<u>Brace</u>, 1971b; <u>Brace</u> <u>et al.</u>, 1966] would be very valuable. Before cracks preferentially grow parallel to the direction of loading, do pre-existing cracks close, do randomly-oriented cracks form, and does sliding occur on pre-existing surfaces as hypothesized? In later stages the specimen spalls and buckles. Steeply inclined shear fractures appear, and finally an open fault develops. What microscopic changes accompany these large-scale fractures?

When Chelmsford granite was heated to 400°F, many new cracks formed. The minimum temperature change that causes crack growth is still unknown. Hydrostatic pressure is widely believed to prevent thermal cracking. How much pressure is necessary to balance a given temperature change? Does hydrostatic pressure by itself cause cracking? SEM studies could resolve these questions.

The porosity of the feldspars as seen with the SEM is a new puzzle. Why does the size of the pores in the feldspars vary from granite to granite? Why don't the plagioclases in Maryland diabase and San Marcos gabbro contain pore concentrations? Possible suggestions are that the porosity

depends on the plagioclase composition, or the water saturation of the melt, or the pressure at which the mineral solidified.

The use of solid cores has proved to be a very good way to study structure, because the core can be further sectioned or stressed. However, the determination of the mineralogy of a given field is difficult, because the electron microprobe should be used to find the composition. In many cases it might be very advantageous to work with ion-bombarded thin sections. Such sections could be examined on an ordinary microscope to determine the mineralogy, coated, and then viewed with the SEM.

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Thanks are also due to Madge Slavin who typed the final copy. In general I would like to say how very much I enjoyed working with everyone on the seventh floor of the Green Building.

APPENDIX I

POLISHING HOLDER

Since the samples were very small, a polishing holder was designed to grip the samples while they were ground on the metal wheel and polished on the cloth laps. Not only did the holder provide an easier size object to grasp, but it also insured that the sample surface stayed perpendicular to its axis during the polishing and grinding. A diagram of the holder is shown in Fig. 9.

The rock was inserted into the center hole. Shims made of pieces of paper of known thickness with holes of the same diameter as the sample were placed on top of a glass plate. The rock and the holder were then placed on top of the shims and the rock was tapped gently until it touched the glass plate so that a measured amount of rock protruded from the holder. Then the holder's screw was tightened to grip the rock firmly and the protruding rock was polished away. Assuming that the abrasive produced a damaged layer directly proportional to its size, the process was repeated six times with progressively finer abrasives and thinner shims. The polished rock surface was then ready for ion bombardment.

APPENDIX II

CHOICE OF COATING

Specimens viewed with the SEM must have conducting surfaces. In electron microscopy many substances including gold-palladium, gold, platinum, silver, graphite, and aluminum are vacuum evaporated onto sample surfaces to provide a conducting layer. I chose a 40% gold 60% palladium alloy after experimenting with several metals.

At first gold was used, but it had two disadvantages. The gold coating tended to crack. Thus the approximately .5 µm or smaller cracks might have obscured the fine scale structures. Also at high magnification the surface charged and darkened so that when the magnification was reduced a dark square was visible.

In an attempt to avoid cracking, silver was tried. Unfortunately the silver reacted with the air to form an oxidized layer.

Platinum produced very good results, because it did not crack or charge. However, its high melting temperature made vacuum evaporation more difficult.

The gold-palladium alloy did not crack and had a lower melting point than platinum. At high magnification it did charge, but not nearly as fast as gold did.
APPENDIX III

HOLDER FOR THE ROCK

Standard specimen mounts for the Jeolco scanning electron microscope are designed to hold .375" or 1.00" diameter specimens. Fig. 10 is a diagram of the holder for the rock, which is meant to be used in the standard 1.00" diameter mount. Holders with different inside diameters can be used to adapt other size cores to the standard microscope mounts.

APPENDIX IV

PRECISE RELOCATION OF A PARTICULAR SEM FIELD

Several aspects of this study were based upon observing changes in a given SEM field and thus upon the ability to precisely relocate it. For serial sectioning it was necessary to return to the same field after approximately 30 μ m of material was removed. To study thermal stress selected fields were examined both before and after heating.

The problem was to retain the orientation of the cylindrical sample so that the coordinate system used in the first viewing would not be rotated. To accomplish this, the rock core was placed inside a holder (Appendix III) whose set screw fitted into the groove along one side of the rock core. The holder was then placed in the microscope mount, and a groove in the side of the holder was aligned with the screw in the mount. The entire configuration can be seen in Fig. 11. The sample was then inserted in the SEM in the same general orientation every time.

The rock's groove when viewed on end looked like a notch, and was used as the origin of coordinates. The precise coordinates of the notch usually shifted slightly from one viewing to the next. The shift was recorded and the new coordinates of the desired field were calculated. The TV mode of observation at lowest magnification (100X)

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was used to relocate the field using the computed coordinates as a guide. Once the field was found, the correct coordinates were recorded. The TV was turned off and the cathode ray tube display was used for viewing because it provided better resolution and because photographs could be taken of the display.

APPENDIX V

MICROPROBE ANALYSIS

Although the SEM was an excellent tool with which to study microstructure, it left many mineralogical questions unanswered. The SEM samples were solid cores, so the standard petrological techniques of the optical transmission microscope could not be used. Instead an electron microprobe was used to determine the composition of the various grains and to verify the location of the grain boundaries.

The electron microprobe had several important limitations. An optical reflecting microscope with a magnification of 400X was used to select the points for analysis. The magnification and the shallow depth of field of the microscope were severe handicaps. The microprobe beam can theoretically be reduced to $1 \mu m$, but generally a wider beam ($6 \mu m$) was used. This beam samples to a depth of $6 \mu m$. The SEM saw only surface structures, while the probe analysis reflected subsurface composition. If for example a thin piece of quartz lay on top of plagioclase, the SEM saw structures in quartz, where the probe gave a high calcium count.

The microprobe study revealed that the plagioclase grains in granite contained pore concentrations. The microcline in Chelmsford Granite was almost as porous as the plagioclase, but the microcline in Westerly Granite had so few pores it was frequently confused with quartz, which typically has only a few scattered pores.

Traverses across several grains (Fig. 12) were made in an attempt to verify whether or not we were correctly identifying grain boundaries with the SEM. The raw data in Fig. 12 implies that the grain was thinner towards its edges. The microprobe data concurred with the SEM photomicrographs. Unfortunately features observed at 1000X to 10,000X with the SEM were invisible to the microprobe microscope (400X), so it was impossible to verify whether or not they lay precisely at the grain boundary.

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LIST OF ILLUSTRATIONS

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 - III. Comparison of Observations to Predictions.

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Fig. 1: Photomicrograph of intersecting cracks within a quartz grain in Westerly Granite. The cracks have black interiors and white edges. The thinning mounds are the irregular gray areas.

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Figure l

Fig. 2: Photomicrograph of Chelmsford Granite. The highly porous grain to the left is plagioclase (pg), while the grain to the right is quartz (qtz). The bright band running down the middle is the topographic step at the grain boundary, caused by the difference in thinning rates between the grains. A crack runs along the grain boundary until halfway up the picture, it turns and crosses the quartz grain above "b".

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Figure 2

Fig. 3: Photomicrograph of a line of pores in a plagioclase (pg) grain in San Marcos Gabbro. The grain in the bottom left corner is an amphibole. The grain boundary is crack free.

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Figure 3

Fig. 4: Photomicrograph of a multigrain field in Westerly Granite. The center quartz (qtz) grain is surrounded by microcline (mi), biotite (bi) and plagioclase (pg) grains. The bright areas at "a", "b", and "c" are cracks which occupy about 20% of the quartz grain boundary. The irregular mounds are from the ion bombardment.



Fig. 5: Photomicrograph of Westerly Granite (#18) which was stressed in triaxial compression to over 95% of its fracture strength. The white arrows show the direction of maximum compression.



Figure 5

Fig. 6: Photomicrograph of Westerly Granite (#18). The white arrows show the direction of maximum compression. The quartz (qtz)-plagioclase (pg) boundary runs from top to bottom to the right of "c".



Figure 6

Fig. 7A: Composite of three photomicrographs of stressed Westerly Granite (#18). The white arrows show the direction of maximum compression.

Fig. 7B: Map of the cracks in the field of 7A. Fig. 7C: Histogram of the crack orientations with respect to the direction of maximum compression. The angular distribution of the cracks in 7A is shown by the solid line while the dashed line shows the distribution for cracks in a similar field of the same size in unstressed Westerly Granite.

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Figure 7B

Crack Orientation with respect to the direction of Maximum Compression



Fig. 8: Photomicrograph of Maryland Diabase stressed in triaxial compression to over 95% of its fracture strength. The pyroxene (pyr)-plagioclase (pg) grain boundary is the site of a large topographic step because of the difference in the minerals' thinning rates, but the grain boundary does not contain cracks. In the center of the micrograph a crack crosses the grain boundary.



Figure 8

Fig. 9: Polishing Holder. For details on its use please see Appendix I.



POLISHING HOLDER

Fig. 10: Holder for Rock. For details on its use please see Appendix III.

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Fig. 11: Sample ready for insertion into the SEM.

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Fig. 12: Electron microprobe study of quartz-plagioclase grain boundaries in Chelmsford Granite.



RAW MICROPROBE DATA:

Point	Potassium	Calcium
l	.00	.02
2	.00	•00
3	.Ol	•00
4 [°]	.02	•29
5	.05	1.13
6	.05	.85
7	.00	•07
8	•00	•00

The points were roughly 18 μm apart.
TA	B	LI	Ξ	Ι
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Rocl	ks	S	tu	d	i	ed	
1.00		~	$\sim \infty$	~	-	$\sim \infty$	

Rock	Porosity (%)		Grain	Modal	_	
	Crack	Pore	Total	Size (10 ⁻³ mm)	Analysis	Reference
Diabase I Frederick, Md.	0	0.1	0.1	180	48 an ₆₇ , 49 pyr, 1 mica, 1 ol	Brace & Orange, 1968 Birch, 1960
Gabbro San Marcos, Cal.	0	0.2	0.2	2000	70 an ₄₂ , l2 mica, 8 pyr, 7 am, 3 ox	Brace & Orange, 1968
Raymond granite Madera, Cal.	.22	0.4	0.6	2000	47 an ₁₆₋₂₀ , 35 qu, 10 mi, 7 mica	Sprunt (present study)
Katahdin granite I Baxter State Park, Maine	.20	0.7	0.9	2000	28 an ₁₈₋₂₄ , 35 qu, 27 mi, 9 mica	Sprunt (present study)
Chelmsford granite Massachusetts	.36	0.7	1.0	1500	31 qu, 31 mi, 31 an ₂₀₋₂₄ , 7 mica	Birch, 1960 Todd & Simmons, 1972
Westerly granite Rhode Island •	.2	1.2	1.1	750	27.5 qu, 35.4 mi, 31.4 an ₁₇ , 4.9 mica	Brace, 1965, 1964a Birch, 1960 Simmons, 1964
Granodiorite of Red Lake Shaver Lake, Cal.	.29	0.8	1.1	2000	49 an ₃₀ , 27 mi, 17 qu, 5 mica, 2 misc	Sprunt (present study)
Katahdin granite II Baxter State Park, Maine	.46	0.7	1.2	3000	29 an ₁₈₋₂₄ , 29 qu, 33 mi, 9 bi	<u>Sprunt</u> (present study)

ox = oxide qu = quartz bi = biotite mi = microcline

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TABLE II

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Rock	Confining Pressure	Strength	Reference
Westerly granite #18	1.5 kb	10.52 kb	Paulding, 1965
Westerly granite #28	0.5 kb	6.30 kb	Paulding, 1965
Maryland diabase	1.6 kb	13.21 kb	Wawersik & Brace, 1971

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TABLE III

Comparison of Observations to Predictions

Property	Rock	Observed (SEM)	Predicted	Reference
Crack length	Granites	Less than a grain diameter, usually 200-400 µm	Order of the grain size	Brace & Walsh, 1966 Brace, 1961 Brace, 1964a
Aspect ratio	Granites	\sim 10 3	~10³	Walsh, 1965a Brace et al., 1968
Crack porosity	Chelmsford granite	.26%	0.29%	Todd, personal communication
	Maryland diabase	.05%	0	Brace & Orange, 1968 Brace, 1965
Pore porosity	Chelmsford granite	1.2%	0.78	Todd, personal communication
Number of cracks intersecting at a point	Granites	2 to 4	2 to 6	<u>Greenberg & Brace</u> , 1969