ROCK FRACTURING UNDER MICROWAVE IRRADIATION: DEVELOPMENT OF ANALYTICAL METHODS AND INVESTIGATION OF THE CONTRIBUTION OF BOUND WATER

by

Marion Nicco
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Golden, Colorado

Date ______________________

Signed: ______________________________

Marion Nicco

Date ______________________

Signed: ______________________________

Dr. Elizabeth A. Holley
Thesis Advisor

Golden, Colorado

Date ______________________

Signed: ______________________________

Dr. Priscilla P. Nelson
Professor and Head
Department of Mining Engineering
ABSTRACT

Microwave irradiation has been considered as a potential method for weakening rock in mining and civil engineering applications. Most rocks in Earth’s crust are predominantly composed of silicate minerals, which are weak microwave absorbers, and the behavior of these rocks under microwave irradiation is not completely understood. This contribution examines the mineralogical and textural controls governing microwave-induced cracking in granites and the influence of the presence of hydrated minerals.

A review of existing methods to characterize cracks indicated that there is no single method that provides comprehensive data on crack location, morphology, mineralogy, textural controls, and reduction of rock strength. Therefore this study developed an integrated suite of methods to assess cracks. Mechanical properties are quantified using relative P-wave velocity measurements and Uniaxial Compressive Strength tests. The morphology of microwave-induced cracks is assessed using new micro-computed tomography and 3D imaging tools. The mineralogical association of cracks is quantitatively described using a combination of optical microscopy, Scanning Electron Microscopy, automated mineralogy, and point counting.

Results show that contrary to hypotheses presented in the literature, the color, water, or iron content of constituent minerals cannot be used to predict the microwave behavior of a rock. The texture of the rock is an important governing factor. Coarse grained rocks develop complex networks of narrow cracks whereas fine grained rocks will develop few wider cracks. Fine pre-existing weaknesses in granite do not appear to be affected by microwave irradiation and are easily discernable from microwave-induced damage. Adjacent mineral grains with contrasting properties (microwaving, thermal, physical or chemical) appear to be critical to the development of cracks.

If microwave technology is applied to rocks made up of minerals that are weak microwave absorbers, the ideal candidates constitute coarse grained (1-5 mm) rocks containing randomly disseminated minerals with contrasting properties. This includes granites, granodiorites, diorites, and gabbros, conglomerates, breccias, coarse sandstones, and metamorphic rocks without small-scale fabrics. Granite hosted ore deposits such as porphyry copper, certain skarn (tin, tungsten, and
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CHAPTER 1
GENERAL INTRODUCTION

1.1. Overview

Understanding the creation and propagation of fractures is important in multiple fields such as mining, geothermal, civil, and petroleum engineering. Microwaves have been proposed as an alternative rock fragmentation method that could reduce the cost of excavation and particle size in civil and mining engineering applications (Didenko et al. 2005, Peinsitt et al. 2010, Hartlieb et al. 2012, Hartlieb et al. 2016, Hartlieb et al. 2017), as well as providing pretreatment for specific ore processing (Walckewick et al. 1991, Salsman et al. 1996, Hua and Liu 1996, Kingman et al. 2004, Scott 2006, Bradshaw et al. 2008, Guo et al. 2011, Rizmanoski 2011, Batchlor et al. 2015, Li et al. 2015, Singh et al. 2016), but the underlying mechanisms that govern microwave-generated fracturing are not well understood.

Fundamentally, rock fractures induced by microwave irradiation rely on the dielectric (Church et al. 1988) and thermal (Skinner 1966, Robertson 1998) properties of the rock. Microwaves affect the polarization of molecules inside the minerals, which creates kinetic energy that translates into heating (Church et al. 1988). However most rocks are polymineralic, and no relation has been established between the dielectric losses of each mineral and the behavior of the whole rock under microwave irradiation.

In the literature on rock fragmentation by microwaves, few studies have addressed the fact that most rocks are polymineralic, and the result of microwaving two minerals at the same time is not just an addition of the dielectric properties of the individual minerals. For example, Jones et al. (2007) and Meisels et al. (2015) assumed that the mineral grains in numerically modeled samples were of a single composition, hosted in a non-absorbing matrix. Those authors modeled these grains based on thermal and mechanical properties intended to represent the average of the dielectric properties of realistic rocks (basalt and granite in Meisels et al. 2015 and a pyrite-calcite ore in Jones et al. 2007). Thus questions such as the following remain unanswered:

- Which mineral fractures first?
- How do microwave-induced fractures propagate?
- Does the presence of water in hydrated minerals contribute to the fracturing?
- How do thermal expansion and thermal conduction interact to generate fracturing?
Understanding these questions would improve our ability to predict how rocks break under microwave treatment, hence improving the potential for applications of the technique in many fields such as mining.

A recent study by Robinson et al. (2014) suggests that the mineral composition could have a considerable impact on the heating ability of a rock. The presence of hydrated clays (kaolinite, illite and others) led to a higher maximum temperature of the microwaved specimen, which in their study led to a better recovery of oil from low grade oil sands. In the present study, initial observations from microwave irradiation of granodiorite showed that chlorite group minerals (biotite and clinochlore) appear to be spatially associated with microwave-generated fractures at a higher proportion than their modal abundance in the sample. Building on those two observations, this study will attempt to test the following hypothesis: **The presence of bound water in the mineral composition facilitates more effective fracturing of the rock.**

In order to test this hypothesis, rocks containing varying amounts of biotite and amphibole in their composition will be compared following microwaving treatment, using a suite of microanalytical techniques. Biotite and amphibole are two minerals that contain water in their mineral structure, and that are found in common Earth’s crust rocks. Biotite minerals are sheet silicates of the mica group, and can contain up to 4% of water in their mineral structure (Miers 1929, Kohl 1960, Deer et al. 1962). Amphibole minerals are hydrous chain silicates and can contain up to 2% of water in their structure as hydroxyl groups (Deer et al 1966).

In order to understand where rock fractures are located, how the microwave treatment influences the fracturing patterns, and how hydrated minerals fracture, a comprehensive method for studying all aspects of the fractures must be established. The first part of this study focuses on evaluating various methods of studying fractures. These methods were classified based on the type of data provided by the method, as well as practical advantages and disadvantages. From this classification, a protocol was developed involving a suite of methods to provide the most comprehensive data possible. In order to validate the methods, they were tested on granodiorite samples subjected to microwave irradiation. Data were obtained on the strength changes caused by microwave irradiation, as well as the characteristics of the fractures including morphology, locations, and mineralogical associations. In the second part of the study, a suite of granite samples containing different proportions of hydrous minerals were subjected to microwave irradiation. Data were obtained
on the characteristics of the fractures including morphology, locations, and mineralogical associations, in order to assess the role of hydrous minerals in microwave-induced fracturing. In the third part of the study, additional methods were developed to assess the role of hydrated minerals, based on microcomputed tomography of samples before and after microwave irradiation.

1.2. Review Of Pretreatment Methods For Rocks Prior To Comminution

1.2.1. Comminution Definition

Comminution refers to the process of reducing the particle size of mined material. “Run of mine” material typically comprises meter- or decimeter-sized particles generated during blasting. During the comminution process the material is reduced from meter size or smaller (depending on the size of run of mine material) to centimeter or millimeter size. Comminution can comprise crushing, or crushing followed by milling. The comminution process generates either a final product or a concentrate, which is further processed to extract the commodity of interest. Energy consumption is a major consideration for crushing and grinding. According to Somani et al. (2017) and the Canadian Industry Program for Energy Conversation (2005), comminution represents on average 36% of the total energy consumed in mines. Comminution represents 1.3% of Australia’s gross electric consumption (Somani et al. 2017) and is assumed to represent 1.5% of the world’s consumption of energy in 2003 (Gwarek and Celuch-Marcysiak 2004). Therefore there is considerable motivation to investigate technologies that would reduce the energy bill of comminution.

The main research focus of such investigations is to develop a pretreatment process to weaken mechanical properties of rocks prior to comminution. Somani et al. (2017) provide a detailed review of the most recent advances in the five methods that were globally the most investigated:

- Thermal breakage (conventional heating)
- Microwave heating
- High voltage pulse breakage
- Ultrasonic breakage
- Thermal shock

The main objective of these methods is to weaken the mechanical strength of the rock, which leads to a decrease in energy consumption (Somani et al. 2017 and references therein, Kingman et al. 1999, 2003). The present study focuses on microwave heating.
1.3. Literature Review: Microwave Irradiation

1.3.1. Molecules and Microwaves

The behavior of molecules under microwave irradiation is well documented by numerous studies in many fields. Here the water molecule is used as an example. Under the electric field of the microwave, molecules that are very polar (i.e. the moment of inertia is unbalanced) such as water will align with the changing orientation of the magnetic field (Figure 1.1). The molecule rapidly aligning back and forth creates kinetic energy, which then translates into a thermal energy. The microwave energy is thus derived from electrical energy and relies on an energy transfer, and not a heat transfer like most conventional heating methods such as furnaces etc. Microwave heating occurs without contact, and it is potentially quick and material dependent.

![Figure 1.1 Creation of kinetic energy by polarization of the highly polar water molecule (Haque 1998).](image)

Depending on their dielectric properties, materials can be transparent, conductors or absorbers of microwave irradiation (Figure 1.2) (Haque 1998, Microwave Power Industry 1994), therefore heterogeneous materials may not heat uniformly. The heating of the sample relies on the differential absorption of microwave energy by minerals in the sample.

In terms of frequency, microwaves are on the high end of the electromagnetic spectrum, ranging from 300MHz to 300 GHz. There are hundreds of ways of microwaving a sample. Ali and Bradshaw (2010) showed that delivery method and power density both play major role in the damage
caused by microwaves. Therefore it is important to keep those components of the testing identical throughout the study.

**Figure 1.2 Interaction of microwave and materials (Haque 1998).**

### 1.3.2 Previous Studies

The first studies performed on microwave irradiation for rocks were aiming at understanding the reaction of different minerals after a microwaving treatment (Church et al. 1988, Hua and Liu 1996). At the time the focus was to document dielectric properties and maximum irradiation temperature of various minerals.

Once these properties were acquired, the focus of studies shifted to using microwaves as a pretreatment or as a treatment for ore processing. Many studies have evaluated the efficacy of microwaves as a pretreatment for copper ore (Scott 2008, Bradshaw et al. 2008, Rizmanoski 2011), manganese ore (Singh et al. 2017), ilmenite ore (Guo et al. 2011), lead-zinc ore (Kingman 2004), iron ore (Walckiewick et al. 1991, Barani et al. 2011, Barani et al. 2012, Singh et al. 2017), disseminated sulfide ore (Salsman et al. 1996) and coal (Li et al. 2016, Singh et al. 2017). These studies examined the efficacy of microwaves as a pretreatment on the milling and liberation characteristics of the mentioned ores.

Almost all of these studies were made on ore bearing minerals with one valuable element, which is usually the case in concentrated ore before processing. However, the ore in-situ can be associated with either a matrix or other minerals. The interaction between two minerals cannot be
interpreted simply as an addition of the changes that occurs in each of them. Depending on the nature of the mineral(s) (transparent, conductor or absorbent to microwaves), the interaction of two or more minerals will change consequently. Recent studies have tried to model those interactions; Jones et al. (2007) simulated the interaction of a pyrite and calcite mixture ore, and Meisels et al. (2015) studied thermal mechanical consequences of a material composed of two materials with different dielectric properties. In addition, Batchelor et al. (2015) studied 14 different nickel, copper and lead-zinc ores and their reaction to microwaving. In this study the mineralogy, grain size, dissemination, textural consistency and mineral associations were taken into consideration. Their study (Batchelor et al. 2015) aimed at filling the gap between studies of single mineral ores, whole rock studies, and two material modeling studies.

Another approach to the problematic nature of polymineralic rocks involves characterizing the effects of microwaving on bulk rock. Previous studies on bulk rock include granite, basalt, sandstone (Hartlieb et al. 2012, Hartlieb et al. 2016, Hartlieb et al. 2017), and kimberlite (Didenko et al. 2005). However, these studies focused on the impacts of microwaves in terms of breakage efficiency and maximum temperature reached for bulk samples. The details of the mineralogy were not discussed.

1.3.3. Microwaving Applied to Comminution

Previous studies specifically focused on microwave-enhanced comminution are reviewed below. These studies have employed various ways to assess the efficacy of the treatment. Some studies have focused on the strength weakening of the material, which can be directly assessed by bond work index and impact breakage indexes. The work index (or bond work index) is the most widely used and defines the equivalent amount of energy to reduce one ton of the ore from a very large size to 100 microns. Point load testing can also be adapted to predict the comminution behavior of a rock (Bearman et al. 1997) indicated by the point load strength index $I_p$. Other studies have used indirect methods such as evaluating the temperature of a sample. This approach assumes that the higher the temperature reached, the more fractures are created and the material weakened.

Ball mill testing is another method that can be used to assess the efficiency of microwaving ore. Zhu et al. (2016) showed that the main driver for microwaving lignite is the moisture content. Vorster et al. (2001) demonstrated that the reduction in work index of microwave treated material leads to a decrease in the circulating load of a ball mill and hydrocyclones (both used in grinding);
therefore enabling the installation of lower capacity, lower energy demanding crushers and screens in the plant without affecting its productivity. Point load testing was used by Kingman et al. (2004b) in order to determine the decrease in strength of a copper bearing carbonatite ore after microwaving. They showed that an exposure of 0.1s at 10kW in a single cavity mode device leads to reduction of the $I_{50}$ of approximately 40%. Kingman et al. (2004b) also make the case that point load index is useful but difficult to interpret in terms of changes in processing efficiency and the resulting energy savings. Finally the impact breakage index $t_{10}$ presents two parameters (referred to as A and b) that are good indicators of the ability of the ore to size reduction by impact (Kingman et al. 2004b) and is measured using the Drop Weight Test. Kingman et al. (2004b) show that microwave treated material has a higher b value, suggesting a softer ore, therefore enhancing its breakage ability. By measuring the maximum temperature reached, Omran et al. (2014) proved that microwave heating is an effective method to weaken iron ore prior to magnetic separation, with recovery of iron ore peaking at 97.95% when pretreated with microwaves (900W power level and 90 s exposure) against 39.54% for untreated ore (both at 1T magnetic intensity). Microwaving has also shown to improve the ease of grinding of coal (Sahoo et al. 2011).

Kingman (1999) and Kingman et al. (2004b) show that there is a threshold effect in the results of changes microwave power level. The reduction in strength of the rock is more significant at 10KW ($I_{50} = 0.9 \times 10^{-6}$MPa for 0.5s) than at 5kW ($I_{50} = 3 \times 10^{-6}$MPa for 0.5s); the gap is less noticeable with 15kW exposure ($I_{50} = 0.6 \times 10^{-6}$MPa for 0.5s). This observation shows that there is an “ideal” microwave power level for a given type of ore. Related work by Whittles et al (2003) shows the same trend for exposure time; after a big drop in strength there is little benefit gained by increasing the exposure time. Kingman et al. (2004b) studied the influence of the type of cavity employed and concluded that for 10kW of power level a strength reduction of 50% of the material is obtained in 5s for a multimode cavity, whereas only 0.1s is needed in a single mode cavity. This is explained by the fact that in a multi-mode cavity a uniform high power density within a sample is very hard to achieve and very energy consuming, whereas a single mode cavity only uses one wavelength, and therefore less energy, but requires a better understanding of the positioning of the sample compared to the power source (Kingman et al. 2003)
1.3.4. Large Scale Implementation

It is important to notice that up to the present date, microwave-enhanced comminution has been proven to be efficient at laboratory scale but has not yet been converted to commercial feasibility for large-scale mining operations (Somani et al. 2017). Large scale implementation is inhibited by the need to address engineering problems like treating large tonnages of material and continuous system development (Gwarek and Celuch-Marcysiak 2004).

Recent work from Buttress et al. (2017) investigates a pilot scale system using a single mode cavity system (reduced energy needed for a large scale operation and better understanding of breaking mechanisms). The experiment has been ongoing since early 2010 at the University of Nottingham and is supervised by microwave research pioneers Andrew Batchelor, Samuel Kingman and David Jones. The basic design is a vertical flow configuration, with the material going through a feed hopper into a processing tube that leads to where the microwaves are applied using a single (100kW, 100 ton per hour) or dual applicator configuration (2x 100kW, 150 ton per hour), as shown in Figure 1.3. The study is the first of its kind and proved the method viable, treating porphyry copper ores at throughputs of up to 150 tons per hour. The study is focused on engineering design and does not address the energy consumption of the system nor the comminution efficiency.

Microwave assisted comminution is a realistic possibility for reducing the global mining energy consumption. Five different variables of microwaving should be investigated in order to make it a viable pretreatment method for comminution: time of exposure, microwave power level, single or multi pulse treatment, the applying device (single or multi cavity mode), and the characteristics of the rock. It is very important to know the mineralogy of the sample, as well as the grain size range, texture, anisotropy etc. in order to predict the efficiency of microwaving a particular type of ore. Just as different types of ore require different milling treatments, the nature of the ore is of vital importance in considering pre-treatment methods for comminution. Since the objective is to reduce the strength of the ore by inducing fractures, it seems vital to understand how the fractures propagate in the rock and the mineralogical controls on fracturing. **This is the focus of the present work.**
1.3.5. The Role of Water

None of the above-mentioned studies included hydrated minerals except for the limited observations by Robinson et al. 2014, so the contribution of hydrated minerals in microwave induced fracturing has not yet been thoroughly investigated.

The water in bulk rock samples can be present in two forms:
- Free water filling pores (can also be referred to as surface water in certain studies)
- Bound to the crystalline structure of certain minerals such as clay minerals, chlorite group etc.

Those minerals are referred to as hydrated minerals.

The water molecule is small and polarized, therefore its sensitivity to microwaves is high, and its presence in a material should have an impact on microwave behavior. Peinsitt et al. (2010) investigated the changes in mechanical properties (through UCS and P-wave velocity testing) on dry and water saturated granite, basalt and sandstone. The results showed that water saturation led to more extensive weakening in sandstone, to a lesser extent in granite, and no observable effect in basalt (Peinsitt et al. 2010). The study also noted that very large cracks occurred in sandstone leading to bursting in some cases. A similar study showed that water saturation between 25% and 50% lead
to optimum crack formation in coal ore (Huang et al. 2019). The presence of free water in the sample has therefore been proven to be a contributing factor to rock weakening during microwaving. The difference in the results between rock types could also be explained by the fact that sedimentary rocks such as sandstones are more likely to be porous compared to igneous rocks (basalt and granite).

Recent studies mention the presence of hydrated minerals (and therefore bound water) as a contributor to the microwave reaction of a material. Robinson et al. (2014) suggests that the mineral composition could have a considerable impact on the heating ability of a rock, and the presence of hydrated clays (kaolinite, illite and others) especially leads to a higher maximum temperature of the sample in the microwave and hence a better recovery of oil from low grade sands. Dos Anjos and Barbin (2015) investigated vermiculate and its changes in AC conductivity, which is strongly influenced by the dielectric properties. Vermiculite is a hydrated phyllosilicate mineral composed of oxides (among N₂O, K₂O, MgO, SiO₂, Fe₂O₃ and Al₂O₃) and bound water in the interlayers. Results showed that the tangent loss increases with the frequency and permittivity decreased when frequency increased.

**Nota Bene:** Dos Anjos and Barbin (2015) write “The water dipoles present a good response to external fields, this characteristic is well known by many researchers” which emphasizes the fact that even though it is “common knowledge” that water is very receptive to microwave irradiation, no study has really proven why. In 1988, Church et al. wrote:

Where it is desirable to heat a mixture of two or more materials, the rate of heating will depend upon the dielectric properties of each. Different heating rates will then occur and in many cases this effect can be used to selectively modify the chemical or physical properties of one of the constituents, thereby enhancing the potential for mineral separation.

Numerous studies have cited that statement to justify that the dielectric properties of a single mineral cannot be extrapolated to the behavior of the same mineral in a polymineralic rock. Together these statements clearly show the gap of knowledge regarding the contribution of water in rock behavior under microwave irradiation.

### 1.4. Methods

The present study focuses on empirical observations of the mineral-scale behavior of rocks subjected to microwave irradiation. Prior to developing the analytical methods, a comprehensive
review was conducted of all the methods that have been used to describe rock fractures, cracks or pores in the last century. Of all of the methods cited in the literature, those that were most promising for the present study were identified and investigated in more detail. The following sections describe that subset of methods, including optical light microscopy, scanning electron microscopy, X-ray computed tomography, nuclear magnetic resonance, Uniaxial Compressive Strength (UCS), Brazilian Tensile Strength (BTS), Drop-Weigh, Differential Strain Analysis (DSA), Point-Load tests, P-wave velocity, Nuclear Magnetic Resonance (NMR) and acoustic emissions. The review also includes an evaluation of the efficacy of each technique for obtaining data on the changes in rock strength and the characteristics of the fractures.

The main takeaway from this review of existing methods is that no single technique is perfectly suited. Thus a combination of methods is needed.

1.4.1. Mineralogical Studies

This section describes the different microscopic methods that were considered in order to build a comprehensive method to identify cracks (presented in Chapter 2), as well as the sample preparation method used prior to the mineralogical studies.

1.4.1.1. Sample Preparation

The samples that have been microwaved usually show large cracks (Figure 1.4) and it is crucial to conserve them for mineralogical studies.

Prior to preparing the thin sections, the core was impregnated with blue epoxy resin, in order to preserve the cracks and prevent the specimen from disintegrating (Figure 1.5). The impregnation process was done in the Thin Section Laboratory at the Colorado School of Mines. The microwave-irradiated sample was wrapped in foil and brought to a temperature of 50°C and the epoxy was slowly poured on the sample using a syringe. The foil was then closed, the heat was turned off, and the sample was left to dry for 24 hours. The method was developed in order to avoid using vacuum that could cause the cracks to expand. The epoxy-impregnated sample was then cut into thin section billets for microscopic investigations.
1.4.1.2. Optical Microscopy

Traditional optical light petrography is an effective way to determine sample mineralogy and fracture geometry. Optical microscopy is the only way to locate grain boundaries and their relationship to fractures, which is a key element of this study. The disadvantages are the time-intensive nature of traditional petrography, the effort required to make quantitative measurements (traditionally conducted by point-counting of a thin).

Figure 1.4 Example of large cracks induced by microwave irradiation on samples of Pikes Peak Biotite (A, B) and Mount Rosa (C).

Figure 1.5 Blue epoxy impregnation sequence showing the epoxy being poured with a syringe (A), the sample enclosed and resting to let the epoxy diffuse (B) and the results on the entire core (C) and a section with arrows pointing at the cracks (D).

section), and the challenges inherent in differentiating some optically similar minerals. In order to help with the visualization of the fractures, samples can be impregnated with colored epoxy as described in the sample preparation section above. It should be noted that the epoxy only infiltrates fractures that
are connected to the surface, but the epoxy will not impregnate any internal porosity that is not interconnected with the surface pores.

1.4.1.3. Scanning Electron Microscopy (SEM) based Automated Mineralogy

Traditional petrography can be supported by more detailed analyses using Scanning Electron Microscopy (SEM) based automated mineralogy. The development of automated SEM systems began in Australia, the United Kingdom and Canada in the late 1970s and early 1980s (Gottlieb 2018) and was focused on plant optimization, recovery and grade optimization (Miller et al. 1982). Automated mineralogy quantifies the modal mineral abundance and textural information, and it can be used to obtain semi-quantitative mineral chemistry data. In this study, carbon-coated thin sections were loaded into a VEGA3 tungsten filament SEM from TESCAN. Scan parameters were input using the TESCAN TIMA (Tescan Integrated Mineral Analyzer) software. The pixel size was set between 5 and 20 µm and depends on sample grain size and textural heterogeneity. Four energy dispersive X-ray (EDX) spectrometers in the TIMA instrument collected 1,000 total X-ray counts for each analysis point in an established grid for each sample measurement. The Back Scattered Electron image (BSE) was analyzed on a point by point basis following the method developed by Reid (Gottlieb 2018). That absorption signature was matched to a library of mineral definitions in the TIMA software. The minerals are assigned, which can be presented as a false colored mineral map as shown in Figure 1.6. However the SEM based automated mineralogy is a chemical analysis; therefore grain boundaries cannot be detected.

1.4.1.4. X-Ray Computed Tomography

X-ray computed tomography (CT) is a non-destructive imaging technique, first introduced in medical sciences in the early 1970s (Hounsfield 1972, 1973) and applied to the geosciences starting in the 1980’s with soil science (Petrovic et al. 1982), petroleum (Vinegar 1986), and geophysics (Fredrich and Wong 1986). The technology is based on the energy loss of X-rays as they pass through material and is governed by the Law of Beer (Equation 1.1).

\[ I = I_0 \exp\left[-\int \mu(s)ds\right] \] (1.1)
Figure 1.6 Example of false colored mineral map of a thin section of Boulder granodiorite obtained by SEM based automated mineralogy.
where $I_0$ is the initial intensity of the X-ray, $I$ is the final density, and $\mu(s)$ is the linear attenuation coefficient along the path. The attenuation coefficient is strongly related to the density and the atomic composition of the material. X-ray CT relies on the acquisition of one- or two-dimensional radiographs for different positions followed by computational reconstruction of a 3D image. The variation in density and atomic composition of the components (minerals, fractures, fluids etc.) allows a 3D reconstruction showing the internal structure of the sample (Mees et al. 2003). There is a tradeoff between sample size and resolution; for a typical sample size of 1-2 mm diameter, maximum resolution with currently available microCT instrumentation is typically 0.5 to 1 μm. Variation in density and atomic composition of the components (minerals, fractures, fluids etc.) allows a 3D reconstruction showing the internal structure of the sample (Mees et al. 2003), with some limitations. For example, attenuation contrasts between minerals in the sample must be sufficiently large in order to resolve individual phases.

X-ray tomography can also be done using synchrotron radiation (i.e. particle accelerator) instead of lab X-ray sources in order to achieve better resolution or detection of smaller features. Synchrotron radiation produces a brilliance that is a billion times higher than typical laboratory X-ray sources, achieving a much better resolution. The high energy beams of the synchrotron radiation also penetrate deeper into the material and combined with small wavelength this enables study of microscopic features. This technique was employed by Renard et al. (2009) to study hydraulic fracture propagation in limestone. However, synchrotron facilities are expensive and have limited access (Van Geet and Swennen, 2001).

1.4.1.5. Analysis Of Microscopy Images

1.4.1.5.1. Cracking Density

Underwood (1970) developed a method for calculating linear cracking density based on drawing horizontal lines across an image and point counting the number of times it encountered a fracture. This linear cracking density has been used in multiple studies over the years to calculate the “cracking efficiency” of a heating method (Fredrich and Wong 1986; Wang et al. 1989; Menendez 1999).

Pereira and Voorwald (2008) used computed image analysis to assess microcracking density. The principle relies on the Underwood equation (Underwood, 1980) but the grid line and points of
intersection (intercepts \( N_i \)) are determined by Image Pro-plus 4.0 and Material Pro Analyzer. Results are expressed in terms of number of intercepts (\( N_i \)) for the test line \( L_t \) and area of cracking \( N_i / L_t \) (cracks/cm), the method is shown in Figure 1.7. Arena et al. (2014) developed a computational technique based on SEM grayscale images that are converted to black and white images using adapted software. These images were then treated with three different scripts (filters) in Matlab to obtain the following fracture parameters: width, length, area, aspect ratio, and orientation as shown in Figure 1.7.

These computational methods are very useful to determine the cracking density, however none of them takes into account the mineralogy of the fractures, which is a main focus of this study.

1.4.1.5.2. Point Counting and Fracture Classification

This technique expands on traditional point counting by recording two types of data:

1. Type of fracture

Figure 1.7 Example of grid line test as presented by Pereira and Voorwald (2008). This method allows for isolation of the cracks using Matlab to obtain width, length, area, aspect ratio, and orientation of the cracks.
a. Intergranular fractures between two grains of the same or different compositions (also called boundary fractures)

b. Intragranular, within one grain

2. Mineralogy of the grains

The process of manual point counting can be eased by using software which automates the data recording and processing, such as the free software Jmicrovision v.1.2.7.

1.4.1.6. Nuclear Magnetic Resonance (NMR)

Two teams from different universities led by Purcell (Harvard) and Bloch (Stanford) respectively, separately discovered the NMR in 1946 (Kleinberg and Jackson 2001). However the technique started to be developed by Schlumberger and Chevron in the 1960’s (Kleinberg and Jackson 2001). NMR allows for the determination of the content and purity of a sample as well as its molecular structure. It can be used to assess fractures and fluid distribution in reservoirs (Chang et al 1997). NMR testing is widely used in petroleum and gas engineering. However NMR requires fractures to be filled with fluid, and in this case study the fractures will be air filled therefore this method will not be used.

1.4.2. Strength Tests

This section describes the different strength measuring methods that were considered in order to build a comprehensive method to identify cracks (presented in Chapter 2).

1.4.2.1. Uniaxial Compressive Strength Test (UCS)

Uniaxial Compressive Strength tests are the most common way to assess the strength of a rock or rockmass. This test is based on the application of an increasing axial force, F, until a maximum load bearing capacity of the sample in this direction is determined. This maximum capacity is reached when the specimen breaks under pressure. The result of that peak stress (F/area) is the UCS of the specimen.

The relationship between UCS and the degree of fracturing of a rock or a rockmass is intuitive. If a rock is more fractured it will have a lower strength thus a lower UCS. By comparing UCS for an intact rock and a thermally treated rock, previous authors have assessed the degree of
fracturing of the rock caused by thermal treatment (Wang et al. 1989, Chaki et al. 2007). Since UCS testing is destructive of the sample it is important that the rockmass from which samples are obtained is homogenous. UCS tests have been used in many studies related to fracturing rocks because of the accuracy and simplicity, although pilot data collected in this project suggest that the results are unreliable.

1.4.2.2. Brazilian Disc Test

The Brazilian disc test (or Brazilian test) was introduced independently by both Carneiro (1943) and Akazawa (1943).

![Figure 1.8 Typical Brazilian tensile test loading configurations: a flat loading platens, b flat loading platens with two small-diameter steel rods, c flat loading platens with cushion, and d curved loading jaws (from Li and Wong 2013)](image)

It is an indirect testing method that gives a measure of the tensile strength of a material. A thin circular disc of the brittle material is diametrically compressed to failure using one of the four methods shown in Figure 1.8, and the tensile strength is calculated by assuming that the failure occurs at the point of maximum tensile stress i.e. the center of the disc; using equation 1.2 (ISRM 1978; ASTM 2008):

$$\sigma_t = \frac{2P}{\pi Dt} = 0.636 \frac{P}{Dt}$$  \hspace{1cm} (1.2)

where P is the load at failure (N), D is the diameter of the test specimen (mm), and t is the thickness of the test specimen measured at the center (mm) (Li and Wong, 2013).
1.4.2.3. Drop Weigh Test (DWT)

This technique measures the strength of a sample by dropping a mass on it and evaluating the sizes of the breakage products (Napier-Munn et al. 1996). Even though the test is very simple the results are supported by detailed analysis: the collected broken sample pieces are sieved in five size fractions. Depending on the ratio of each fraction the operator can evaluate how effective the breakage was. The test is standardized for every sample. Rizmanoski (2011) used the DWT to evaluate the reduction in strength of a low grade copper ore subjected to microwave treatment. The method is destructive and does not give any information on the mineralogy of the fractures and where they are located (boundaries or intragranular). Therefore this test was not considered in this study.

1.4.2.4. Differential Strain Analysis

For this testing method, strain gauges are attached to a sample in three orthogonal directions. Pressure is applied, and the strain is determined in three directions. The intercept of the line tangent to the strain curve at a pressure P represents the cumulative amount of strain in that direction (Wang and Simmons 1978). According to a study by Simmons et al. (1974) the compression (pressure versus strain) curves obtained using the differential strain analysis can give indication of the fracturing of the sample. Depending on the aspect of the compression curves (linear segments or smoothly varying segments), the crack aspect ratio can be deducted as shown in Figure 1.9.

![Figure 1.9](image.png)

Figure 1.9 The DSA curves for Chelmsford, Massachusetts, granite. Additional cracks were produced in the sample by uniaxially loading a cylindrical specimen to 80% of the failure strength. The stress-induced cracks have different characteristics from those of the virgin specimen (from Simmons et al. 1974).
1.4.2.5. Point Load Test (PLT)

This method is widely used as an alternative to UCS in the estimation of rock strength. A typical setup is presented in Figure 1.10. The PLT is an efficient method to assess intact rock strength properties from drill core samples. It has become an accepted test in geotechnical evaluations based on its portability (Rusnak and Mark 2000) and involves compressing a rock sample between conical steel plates until failure occurs (Broch and Franklin 1972).

![Figure 1.10 Typical Point Load test setting (From Broch and Franklin 1972).]

1.4.2.6. P-Wave Velocity

In the acoustic test, ultrasonic waves are sent through the sample and the time it takes to travel the length of the specimen is recorded on an oscilloscope. A wave generator sends a pulse through the sample from one transducer (emitting cell) while simultaneously transmitting that same pulse to the oscilloscope. The time difference that is measured by the oscilloscope between the emitted pulse and the arrival pulse sent by the receiving cell allows for the determination of the velocity, which can be used to calculate the elastic modulus (E) and Poisson’s ratio (ν). The P-wave (or compressive wave) is calculated in meters/second (m/s).

Throughout all studies conducted on heating rocks (Wang et al. 1989, Menendez et al. 1999, Chaki et al. 2007), P-wave velocity were used. P-waves; or compressive waves; are defined by a longitudinal mode of propagation in an isotropic and homogeneous solid. The particles vibrate along the axis of propagation of wave energy. The relationship between the degree of fracturing and the
change in P-wave velocity is such that if the sample is more fractured (less homogeneous), the P-wave velocity should decrease. Changes in P-wave velocity can be indicated as a ratio; the relative velocity as function of an applied fracturing treatment, with 100 % being the initial P-wave velocity of each sample ($v_{p0}$) and $v_{p1}$ the measurement after treatment. If the relative velocity is high, the following equation 1.3 shows that there was insignificant change in the sample structure, whereas a low relative velocity indicates fracturing due to the treatment:

$$\text{Relative Velocity} = \frac{v_{p1}}{v_{p0}}$$

The main advantage of using this method is that it is non-destructive of the sample and thus one sample can be measured for P-wave velocity before and after the fracturing treatment is applied.

1.4.2.7. Bulk Sample Porosity By Mercury Injection

The method of bulk sample porosity determination by mercury injection relies on the calculation of the volume of mercury injected in a sample and gives information on the throat diameter of the pores using the Laplace equation and the total volume of connected pores (comparable to fractures patterns) (Ritter and Drake 1945, Webb 2001). This method is widely used in oil and gas studies and often suggested when it comes to studying cracks in mining applications. However from the mining perspective all fractures are important, not just connected porosity, which is why this method will not be used.

1.4.2.8. Acoustic Emission Testing

Acoustic emission (AE) refers to the elastic wave generated during the release of energy within a material (Lockner 1993). The set up consists of an acoustic sensor placed on a flat surface of the sample (usually a piece of drill core) that records the sound emitted by the release of energy when a fracture occurs. In the mining industry, acoustic emissions have been studied in the context of rock bursts and mine failure (Lockner 1993). Acoustic emission monitoring was successfully used to monitor fracture creation and propagation of rock samples heated in a furnace (Wang et al. 1989), under compression (Rodríguez et al. (2016), and in concrete during cryogenic cooling (Kogbara et al. 2016). This technique has not been considered for this study because a typical AE sensor would be damaged during microwaving, the sensor being composed of metal that would melt in the microwave.
1.5. Materials

This section describes all of the materials that were considered for the present study, including those that were ultimately selected for study (Boulder granodiorite, Mount Rosa complex granite suite), and those that were investigated and not selected for further study (Valmy quartzite, Buffalo Valley skarns, pegmatite, and marble).

1.5.1. Boulder Granodiorite

Specimens of the Boulder granodiorite (Figure 1.11) were collected from a road cut exposure of the Boulder Creek batholith in Boulder Canyon, Colorado. Boulder Creek is one of several Precambrian calc-alkaline batholiths in central Colorado (Gable 1980). The Boulder granodiorite was selected because of its mineralogical and physical homogeneity throughout the batholith and on outcrop scale, the absence of hydrothermal events in the area, and the well-constrained thermal history of erosion and uplift (e.g. Johnson et al. 2014). The site was selected because the outcrop is relatively fresh due to recent road widening and blasting. Alteration of the outcrop is limited to minor 2-5 mm coatings of iron oxides that exist on some of the recently blasted faces. Some joints and faults are present in the road cuts. Specimens comprised homogeneous, unjointed blocks of approximately 400 x 400 x 400 mm size that were separated from the outcrop along outcrop-scale joint surfaces. The specimens that were selected contained no macroscopically visible preexisting weaknesses or Fe-oxides. The mineralogical composition of the specimens is dominated by feldspar (~60%), quartz (~30%) and biotite (~5%).

1.5.2. Mount Rosa Complex Granite Suite

The suite of specimens comprised three types of granite: the Pikes Peak biotite granite (PPB), the Mount Rosa granite (MR), and the Mount Rosa amphibole granite (MRA) presented in Figure 1.12 ABC respectively. These granitic rocks were collected in Mount Rosa area of Teller and El Paso counties, Colorado. The three different rock types were described in detail by Gross and Heinrich (1965) and Persson (2017). The suite of samples was chosen to carry the study on mineralogical and textural controls because, even though all three rock types are geologically related and mineralogically similar, their mineral modal abundances vary (i.e., biotite versus amphibole versus a lack of a hydrous mineral phases), as well as contrasting textures. A detailed description of the mineralogy and texture of each of the three rock types is given in Section 3.3.
Figure 1.11 Specimen of Boulder granodiorite.

Figure 1.12 Specimen of Pikes Peak Biotite (A), Mount Rosa (B), and Mount Rosa Amphibole (C) granites specimens.
1.5.3. Valmy Quartzite

The Valmy Formation is the primary host to ore at Marigold Mine, NV, USA, where it was sampled during this study. The Valmy Formation is composed of an upper sequence of folded and faulted quartzite (80-100%), argillite (0-20%), and sparse metabasalt, and a lower sequence of calcareous mudstone, siltstone, volcaniclastic and siliciclastic debris flows, and metabasalt (Fithian et al. 2018). Specimens of mineralized quartzite at different mineralization stages were sampled in a trench at the Marigold mine, as presented in Figure 1.13. The goal was to study the potential for microwave irradiation treatment of an ore from barren to highly mineralized. Since the mineralization at Marigold is highly associated with faults, the trench would allow sampling across different degrees of mineralization.

Figure 1.13 A shows the fine grained Valmy quartzite unaltered and barren. It is grey color with white quartz veins. Figure 1.13 B shows a less altered less mineralized sample taken a couple of meters from the first one in the trench. It is a fine grained quartzite grey to brown color, with pink-orange surface alteration. Figure 1.13 C shows a sample of highly altered quartzite composed of quartz, argillite and oxides. The red color and friable texture is indicative of high grade ore.

Most of the samples collected from the Marigold mine turned out to be too hard to drill cores with the available equipment (Figure 1.13 A), too fractured (Figure 1.13 B), or too altered and crumbly (Figure 1.13 C) to be usable for the aimed study. Therefore they were not chosen for continued study.

1.5.4. Buffalo Valley Skarns

Skarns from the Buffalo Valley Mine (NV) were sampled. Skarns at this deposit are composed of an assemblage of quartz, pyroxene and garnet with varying degrees of alteration (Reid 2010) (Figure 1.14). Two problems arose with the Buffalo Valley skarns. Skarns are by definition altered material and the alterations visible at the Buffalo Valley mine varied at a meter scale, making samples of skarns too difficult to sample homogenously. Second, skarns in the Buffalo Valley were extremely difficult to retrieve because of the remote location which is currently only accessible on foot. It would have been too difficult to get samples big enough to core (100 mm by 50 mm diameter) for the present study.
Figure 1.13 Specimens of Valmy quartzite from the Marigold mine at different stages of mineralization: barren Valmy quartzite (A), ore grade altered Valmy quartzite (B) and high grade altered Valmy quartzite (C).
1.5.5. Pegmatite

Pegmatite samples were collected from underground exposures at the Colorado School of Mines's experimental mine (Edgar mine), located in the Idaho Springs district (CO, USA). The rock is mainly composed of quartz, microcline, plagioclase and locally abundant biotite and magnetite (Moench and Drake 1966) as shown in Figure 1.15. The pegmatite presents an interesting opportunity to study the effect of hydrous minerals (biotite) under microwave irradiation. However, the samples were too coarse grained (some grains measuring up to 50 mm) to allow the making of mineralogically representative thin sections (50x150 mm). The Edgar pegmatite was the basis of a numerical modeling study on microwave irradiation presented in Li et al (2019).

1.5.6. Marble

Specimens of Yule marble were collected from the Marble mine in Marble (CO) and cored (Figure 1.15). The specimens were collected from fresh excavations and were unaltered. The rock is homogeneous and fine grained (0.5-1. mm, Knopf 1949; McGee 1998) and is practically a pure calcite (99%) marble with very few inclusions of other minerals (Knopf 1949 ; McGee 1998). The Yule marble was not used during this study because it is monomineralic and not suited for a study of the mineral interactions. However marble samples were used in a strength reduction study on microwave irradiation (R. Kaunda, unpublished data) which showed that longer irradiation times up to 300 seconds do not appear to reduce significantly the strength of the material.
1.6. Dissertation organization

Following the general introduction presented in this chapter, the bulk of the dissertation comprises three papers addressing the methods for characterizing microwave-induced rock fracturing and the mineralogical controls on the fracturing.

Chapter 2 is a paper published in the Journal of Rock Mechanics and Rock Engineering. It reviews an exhaustive list of possible methods used to characterize fractures from several fields of
engineering. No single method enabled study of both the location and mineral associations as well as quantity and rock strength reduction of the sample after microwave irradiation. Therefore a combination of methods was established to obtain these data, and then this approach was tested on granodiorite samples.

Two important observations were made during the granodiorite case study:

1. The integrated suite of methods provides a dataset on microwave-induced changes in rock strength, fracture mineralogy and morphology; these data can be used to investigate the mechanisms causing the fracturing, as well as how the fracturing treatment could be optimized.

2. Fractures on one specimen tended to be preferentially associated with biotite/chlorite (15.6% of fracture boundaries in contact whereas biotite/chlorite account for 9.4% of the sample composition normalized to 17.03% porosity), compared to feldspar (46.7 % of fracture boundaries in contact for 53.6% of the sample composition normalized to 17.0% porosity) and quartz (31.8% of fracture boundaries in contact for 33.5% of the sample composition normalized to 17.0% porosity).

Chapter 3 is a paper submitted to the Journal of Rock Mechanics and Rock Engineering. In this paper a suite of three granites was studied, with a focus on the mineralogical and textural controls on microwave-induced cracking, using the techniques presented in Chapter 2, except for microCT. The results showed that the behavior of the minerals was consistent across rock types in the sample suite. Albite and amphibole tended to be disproportionately associated with the cracks, whereas biotite, orthoclase and quartz tend to be disproportionately disassociated with the cracks. However there was no single chemical, physical or microwave property that seemed to consistently explain the behavior of the minerals. The biotite in samples of PPB was disassociated with the cracks, in contrast with results from Chapter 2. This could be explained by either the contrasting biotite texture between the two rocks or the presence of a different amount of bound water; the latter is tested in Chapter 4. Chapter 3 also presents the textural controls that seem to govern a rock’s behavior under microwave irradiation, the main driver being the grain size. Chapter 3 also presents a possible approach to recognize pre-existing weaknesses or fractures by studying their grain boundary association. The preexisting cracks tended to be straight throughout the sample while microwave induced cracks were predominantly around the grains. This study concludes by recommending rock types that are ideal
candidates for applications of microwave technology despite only containing low dielectric loss minerals.

Chapter 4 is a paper in preparation for submission to the Journal of Rock Mechanics and Rock Engineering. The paper presents the first application of microCT to a large microwave-irradiated core specimen in order to determine the contribution of pre-existing weaknesses and the expansion of hydrated mineral biotite submitted to microwave irradiation. The specimen was large enough to be representative of a realistically sized run of mine rock fragment (in contrast with the proof of concept on a 5mm$^3$ sample in Chapter 2. This study was done in parallel and on the same granite used in Chapter 3, Pikes Peak granite. This particular rock type was chosen among the three studied in Chapter 2 because of its textural properties (e.g., grain size) that allowed for better observation using the microCT. The study used deep machine learning to identify cracks generated by microwave irradiation. The study also used the microCT data to conduct an analysis of the volumetric expansion of biotite caused by microwave irradiation. In addition to the conclusions regarding the mechanisms of microwave-induced cracking, the paper provides the methods of the machine learning protocol involved in the reconstruction and segmentation of the data in the software Dragonfly (v4.1). This allows any user of the software who would like to segment cracks in a polymineralic rock to directly use the extraction-reconstruction method that was develop in this study.

Chapter 5 is a global discussion of all three studies and provides recommendations and guidelines for future research on the topic.

1.7. Committee and Co-authors

This section clarifies the roles of the co-authors of the three papers presented in this dissertation. Together the co-authors form the dissertation committee.

Marion Nicco is the Ph.D. candidate, primary researcher, and first author of the papers. She conducted all of the work presented in the papers, analyzed the data, prepared the figures, and wrote the text incorporating edits from the committee.

Dr. Elizabeth Holley is an Assistant Professor in the Mining Engineering Department at the Colorado School of Mines. She is the student's advisor and provided guidance on experimental
design, data interpretation, and presentation of the results in the papers. She is a co-PI on the NSF grant that partially funded this project and provided the remainder of the financial support for the work.

Dr. Katharina Pfaff is a Research Assistant Professor and is the manager of Mineral and Materials Characterization (MMC) facility at the Colorado School of Mines. Dr. Pfaff helped with the acquisition and analysis of the SEM based automated mineralogy data.

Dr. Philipp Hartlieb is a Senior Researcher at the chair of the Mining Engineering and Mineral Economics Department in the University of Leoben, Austria. Dr. Hartlieb has extensive knowledge on microwaving research and technology. He operated some of the microwave tests and was involved in the interpretation of the data.

Dr. Rennie Kaunda is an Assistant Professor in the Mining Engineering Department at the Colorado School of Mines. Dr. Kaunda advised on rock strength tests and was the PI on the NSF grant that partially funded this project.

Dr. Priscilla P. Nelson is a Professor and the Head of the Mining Engineering Department, and was a co-PI on the NSF grant that partially funded this project.

1.8. References


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Rusnak J, Mark C (2000) Using the Point Load Test To Determine the Uniaxial Compressive Strength of Coal Measure Rock. 19th International Conference on Ground Control in Mining, 8-10 August, 362–71.


Vinegar HJ (1986) X-ray CT and NMR imaging of rocks. Int. j. pet. technol. 38 257-259


2.1. Abstract

A detailed understanding of how induced cracks form and propagate in rock is important in many fields, particularly in mining where size reduction of material is a major cost. This contribution provides a comprehensive review of previously published methods for characterizing rock cracks. The existing methods generate one of two types of data: crack-related changes in bulk rock properties, or the nature of the actual cracks, such as aperture, location, morphology, and mineralogical boundaries. However, no single technique provides both types of information. Using a suite of granodiorite specimens which were cracked under microwave irradiation, this contribution illustrates how multiple non-destructive and destructive techniques can be integrated to provide both types of data on cracks induced in rock. Rock property changes were quantified using P-wave velocity measurements and Uniaxial Compressive Strength tests. The cracks were characterized qualitatively and quantitatively using a combined approach of micro-computed tomography, optical microscopy, and automated mineralogical analysis of scanning electron microscopy images. The resulting dataset provides information on the mechanical and mineralogical effects of the cracking. The microwave irradiation led to a significant reduction of rock strength, caused by networks of intergranular and intragranular cracks in granodiorite, disproportionately associated with biotite. Based on the strengths and limitations of each method as illustrated by the granodiorite results, recommended protocols are provided for evaluating cracks in four different types of studies: 1. Quick assessments of damage; 2. Geotechnical investigations; 3. Mineralogical investigations, and 4. Comprehensive evaluations of the effects of cracking, requiring data on mechanical properties and the characteristics of the cracks.
2.2. Introduction

Size reduction of material is a major cost in mining and mineral processing. Mining can be considered the first step in reducing decimeter-scale blocks to smaller fragments which are supplied to subsequent processing steps. Researchers have examined a range of alternatives to the traditional sequence of drilling, blasting and comminution, including breakage mechanisms such as high-pressure water jets, electric pulses, and high-power microwaves (Kingman and Rowson 1998 and references therein, Kingman et al. 2000, Graves et al. 2002, Graves and Bailo 2004, 2005, Jones et al. 2005, Andres 2010, Guo et al. 2011, Rizmanoski 2011, Hartlieb et al. 2012, Ciccu and Grosso 2014, Hartlieb et al. 2016, Singh et al. 2017). These fragmentation methods can either be used independently or in conjunction with traditional mechanical excavation methods. The success of these technologies will depend on introducing a network of artificial cracks in the rock mass, leading to more efficient extraction of the rock.

In order to optimize rock fragmentation, it is critical to understand damage propagation and especially the influence of pre-existing cracks in rock. Numerous studies have investigated the relative merits of breakage mechanisms by artificially inducing damage in rock and monitoring the ensuing changes in various rock properties, with the aim of understanding why the rocks break, and how to optimize breakage mechanisms. However, each study has employed different monitoring techniques for the damage, and a standard practice has yet to be agreed upon even among investigations with similar goals. The present work attempts to rectify this lack of consistency by proposing workflows for four targeted applications: quick assessments of damage, geotechnical investigations, studies which require mineralogical observations (e.g. mechanistic or mineral processing investigations), and comprehensive evaluations of cracking.

2.3. Characterization of Induced Cracking

A number of previous studies have examined rock breakage by high-power microwaves for applications in civil and mining excavations (Peinsitt et al. 2010, Hartlieb et al. 2012, 2016, Toiff et al. 2016) and mineral processing (Salsman et al. 1996; Kingman et al. 2004a, 2004b, Scott 2008; Jones et al. 2007, Guo et al. 2011, Rizmanoski 2011, Li et al. 2016, Singh et al. 2017). Microwave-induced cracking of the rock has most commonly been assessed through strength tests, including point load (Kingman et al. 2004a, 2004b), Uniaxial Compressive Strength (UCS; Whittles et al. 2003; Jones et al. 2007, Peinsitt et al. 2010), Drop Weight Tests (DWT) (Kingman et al. 2004a, 2004b, Rizmanoski,
2011), and ultrasonic velocity testing (Peinsitt et al. 2010, Hartlieb et al. 2012). The strength reduction has been quantified by comparing non-destructive test results before and after the microwave treatment, or by comparing destructive or non-destructive test results in treated and untreated specimens. One study examined the cracks directly (Hartlieb et al. 2012): a penetration spray was applied to the cracked specimen to assist in visual identification of crack locations, and a portion of the specimen was impregnated with resin and examined in thin sections using optical light microscopy. Numerous mineral processing studies have attempted microwave-assisted grinding of strong microwave absorbing minerals in a poorly absorbing matrix (see Pickles, 2009 for a review). In order to examine the effects of microwave treatment in mineral processing, liberation analyses are commonly conducted using scanning electron microscopy (Wang et al 1989, Walkiewicz et al. 1991) and automated mineralogy (Kingman et al. 2004a; Scott 2008; Singh et al. 2017).

Investigations of other types of thermally induced rock damage have used a range of techniques to evaluate cracking. For applications in geothermal energy extraction and nuclear waste storage, rocks subjected to pressure-temperature cycling were examined using acoustic emission measurements, differential strain analysis and Scanning Electron Microscopy (SEM; Wang et al. 1989), and P-wave velocity measurements (Wang et al. 1989, Menéndez et al. 1999, Darot and Reuschlé 2000, Sone and Condon 2017). Several geological studies have induced thermal cracks in rocks by heating specimens in a furnace. The mercury intrusion porosimetry method (Ritter and Drake 1945; Webb 2001) has been used to evaluate the effects of the cracking (Géraud 1994). The thermally-induced cracks have been examined using optical light microscopy of thin sections (Kranz 1983 and references therein), SEM (Fredrich and Wong 1986, Géraud 1994, Homand-Etienne and Houpert 1989, Mahanta et al. 2016, Sone and Condon 2017), confocal scanning laser microscopy (Menéndez et al. 1999), and by UCS testing and evaluation of macroscopic failure patterns (Tian et al. 2017). Griffiths et al. (2017) developed an algorithm to automatically filter optical light microscopy images of thermally-induced microcracks to determine the 2D crack density and length, although mineralogy was not considered.

Studies on rock breakage by laser (Graves and Bailo 2004, 2005) evaluated the efficacy of the technique by thin section petrography, CT, a pressure decay profile permeameter, as well as P-wave and S-wave velocity measurements. The performance of water jets in rock breakage is typically assessed by recording crack depth, radius, volume and angle (e.g. Ciccu and Grosso 2014, Oh and
Cho 2014). In electrical pulse studies, rock damage has been characterized and quantified using optical light microscopy (Andres 2010).

X-ray computed tomography (CT) has been applied to the study of cracks induced by loading in a polyaxial testing cell (Sellers et al. 2003), under uniaxial compression (Jia et al. 2014), in Brazilian disc tests (Weerakone et al. 2017), and by hydrofracturing (Renard et al. 2009, Zhang and Sheng 2017 and references therein). Numerous studies have attempted to measure the apertures of induced or pre-existing rock cracks using CT or microCT (e.g. Johns et al. 1993, Keller 1998, Bertels et al. 2001, Van Geet and Swennen, 2001, Vandersteen et al. 2003, Weerakone et al. 2017).

Porosity and fluid flow in petroleum reservoirs are commonly assessed using nuclear magnetic resonance (NMR), and several studies have used NMR to characterize the apertures of pre-existing or induced fractures in rock (Chang et al. 1997, Kumar et al. 1997; Renshaw et al. 2000, Nakashima and Kikuchi 2007). Another study used NMR to examine the porosity effects of microwave irradiation of coal ores (Li et al. 2016).

Each of the above techniques can be broadly classified as an assessment of bulk rock properties, or a means of characterizing crack features such as aperture, morphology, and mineralogy (Table 2.1). No single method provides comprehensive data on rock properties and crack characteristics, necessitating a combination of methods to obtain both types of data. Non-destructive methods are ideal because they allow one to compare the same rock before and after a cracking treatment is applied. However, limitations on the types of data generated, as well as practical constraints such as specimen size or resolution, motivate the use of both non-destructive and destructive techniques to fully characterize the efficacy of a rock cracking treatment.

Table 2.1 Published methods for assessing cracks induced in rock. Techniques employed in this study are underlined.

<table>
<thead>
<tr>
<th>Technique</th>
<th>Capabilities</th>
<th>Limitations</th>
<th>Applications to induced rock cracking</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Rock Property Tests</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Non-destructive</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>P-wave velocity measurements</td>
<td>Quantitative measurement</td>
<td>Specimen preparation requires perfectly parallel planes</td>
<td>Wang and Simmons (1978); Wang et al. (1989); Menéndez et al. (1999); Darot and Reuschlé (2000); Sone and Condon (2017); Peinsitt et al. (2010);</td>
</tr>
</tbody>
</table>
Table 2.1 Continued

<table>
<thead>
<tr>
<th>Method Type</th>
<th>Method Description</th>
<th>Detection Details</th>
<th>Authors Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-destructive</td>
<td>Acoustic emission</td>
<td>Allows detection of the cracking and location as well as real time propagation of cracks</td>
<td>Hartlieb et al. (2012)</td>
</tr>
<tr>
<td>Non-destructive</td>
<td>Differential strain analysis</td>
<td>Specimen equipped with strain gauges in three orthogonal directions; measures cumulative strain in each direction. Differentiates populations of cracks that created under different conditions.</td>
<td>Simmons et al. (1974); Siegfried and Simmons (1978); Wang and Simmons (1978); Wang et al. (1989)</td>
</tr>
<tr>
<td>Non-destructive</td>
<td>Bulk porosity by mercury injection or comparison of dry, water immersed and water saturated specimen weights</td>
<td>Quantitative measurement of primary and crack-induced porosity: throat diameter of pores, total volume of connected pores</td>
<td>Ritter and Drake (1945); Géraud, (1994); David et al. (1998); Méndez et al. (1999); Webb (2001)</td>
</tr>
<tr>
<td>Destructive</td>
<td>Point-Load</td>
<td>Determines intact rock strength properties from drill core</td>
<td>Kingman et al. (2004a,b); Guo et al. (2011)</td>
</tr>
<tr>
<td>Destructive</td>
<td>Drop-weight</td>
<td>Rock strength is evaluated by dropping a mass on a specimen</td>
<td>Kingman et al. (2004a,b); Rizmanoski (2011)</td>
</tr>
<tr>
<td>Destructive</td>
<td><strong>Uniaxial compressive strength (UCS)</strong></td>
<td>Quantitative measurement of the rock strength</td>
<td>Whittles et al. (2003); Jones et al. (2007); Peinsitt et al. (2010)</td>
</tr>
<tr>
<td>Non-destructive</td>
<td><strong>Computerized tomography (CT)</strong></td>
<td>Spatial distribution and aperture of cracks. Some mineralogical data possible.</td>
<td>Johns (1993); Keller (1998); Bertels et al. (2001); Sellers et al. (2003); Vandersteen et al. (2003); Van Geet and Swennen (2001); Renard et al. (2009); Jia et al. (2014); Hartlieb et al. (2017); Weerakone et al. (2017); Zhang and Sheng (2017)</td>
</tr>
<tr>
<td>Non-destructive</td>
<td>NMR</td>
<td>Spatial distribution and aperture of cracks in fluid-saturated rock</td>
<td>Kumar et al. (1997); Chang et al. (1997); Renshaw et al. (2000)</td>
</tr>
</tbody>
</table>

*Crack Characterization Methods*
| Destructive | Confocal microscopy | A series of 2-D images are used to achieve 3-D analysis. | No mineralogical data. 3-D requires hundreds of images. | Menéndez et al. (1999) |
| Destructive | Thin section optical microscopy | Mineralogy and crack characteristics. | 2-D. Typically qualitative unless point-counting or image processing is used. Some mineral phases may be difficult to identify petrographically. | Kranz (1983) and references therein; Graves and Bailo (2004; 2005) Hartlieb et al. (2012); Griffiths et al. (2017) |
| Destructive | Scanning electron microscopy (SEM) | Qualitative mineralogy and crack characteristics. | 2-D. Typically qualitative unless point-counting is used. Cannot differentiate individual grains in aggregates of the same composition. | Brace et al. (1972); Tapponnier and Brace (1976); Wang and Simmons (1978); Kranz (1979b); Padovani et al (1982); Fredrich and Wong (1986); Homand Etienne and Houpert (1989); Wang et al. (1989); Walkiewicz et al. (1991); Géraud (1994); Mahanta et al. (2016); Sone and Condon (2017) |
| Destructive | Transmission electron microscopy (TEM) | Qualitative mineralogy and crack characteristics. | 2-D. Typically qualitative unless point-counting is used. Cannot differentiate individual grains in aggregates of the same composition. | Boland and Hobbs (1973); Tullis and Yund (1977), Marshall and McLaren (1977); Dunning et al. (1980) |
| Destructive | Automated mineralogy | Quantitative modal abundances of minerals and cracks, liberation analysis. | 2-D. Cannot differentiate individual grains in aggregates of the same composition. | Scott (2008); Kingman et al. (2004a); Singh et al. (2017) |
In the present study, we propose a comprehensive method for evaluating induced cracks, based on rock properties and crack characteristics, using both destructive and non-destructive techniques. The method comprises petrographic and automated mineralogical analysis of mineral phases and cracks in thin section, microCT, UCS tests, and P-wave velocity measurements. We demonstrate the efficacy of the method by characterizing cracks that were induced in granodiorite specimens by treatment with 3.2 kW microwave irradiation.

2.4. Materials and Methods

2.4.1. Rock Specimens

Specimens were collected from a road cut exposure of the Boulder Creek batholith in Boulder Canyon, Colorado. Boulder Creek is one of several Precambrian calc-alkaline batholiths in central Colorado (Gable 1980). The Boulder granodiorite was selected because of its mineralogical and physical homogeneity throughout the batholith and on outcrop scale, the absence of hydrothermal events in the area, and the well-constrained thermal history of erosion and uplift (e.g. Johnson et al. 2014). The site was selected because the outcrop is relatively fresh due to recent road widening and blasting. Alteration of the outcrop is limited to minor 2-5 mm coatings of iron oxides that exist on some of the recently blasted faces. Some joints and faults are present in the road cuts. Specimens comprised homogeneous, unjointed blocks of approximately 400 x 400 x 400 mm size that were already separated from the outcrop along outcrop-scale joint surfaces. The specimens that were selected that contained no macroscopically visible preexisting weaknesses or Fe-oxides. The mineralogical composition of the specimens was dominated by feldspar, quartz and biotite (Table 2.2, column A). Figure 1 shows the sequence of preparation and analytical methods applied to the specimens.

2.4.2. P-wave Velocity Measurements

The intact blocks were cored to NX diameter in the laboratory, resulting in 55 mm diameter by 150 mm length core. Twenty-four of the core specimens were subjected to ultrasonic velocity testing to determine P-wave velocities prior to irradiation. Only twenty of the original twenty-four specimens retained sufficient physical integrity to measure P-wave velocities after irradiation. P-wave velocity is a good indication of a rock’s density; extremely dense rocks can reach P-wave velocities of up to
6,000 m/s. Rocks with more cracks and less dense packing will exhibit lower P-wave velocities in the range of 3000 to 4000 m/s (Gebrande and Angenheister 1982). This principle can be applied to provide an assessment of cracking in a specimen by comparing the reduction in P-wave velocities resulting from specimen cracking can be expressed in a relative manner according to \( v_{p_{rel}} = \frac{v_p}{v_{p0}} \), where \( v_{p_{rel}} \) is the relative P-wave velocity, \( v_p \) is the P-wave velocity after treatment and \( v_{p0} \) is the P-wave velocity before treatment. The axial P-wave velocity was measured with a Geotron Geoelektrik® USG40 ultrasound generator, using a UPG250 transmitter (operating at 250 kHz) and a UPE receiver. A pneumatic device maintaining a constant contact pressure and a special ultrasound contact gel ensured proper coupling of the measurement devices with the rock. The sensors were placed at the end faces of the core specimens.

Table 2.2 Modal abundance (%) of major minerals and porosity in Boulder granodiorite specimens (A) B0 prior to irradiation and (B) B17 after irradiation for 146 seconds. (C) shows the same results as in (B), recalculated to exclude porosity; this allows comparison of mineral modal abundances in B0 and B17, as in the standard deviations shown in (D). (E) shows the results of a liberation analysis, which was obtained by first calculating the total length of crack boundaries in irradiated specimen B17, and then calculating the percentage of those boundaries in contact with each mineral phase.

<table>
<thead>
<tr>
<th>seconds irradiated</th>
<th>A. B0 (after 0 s of irradiation)</th>
<th>B. B17 (after 146 s of irradiation)</th>
<th>C. B17 recalculated to exclude cracks</th>
<th>D. Modal abundance std dev</th>
<th>E. % crack boundaries in B17 in contact with each mineral</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
<td>146</td>
<td>146</td>
<td>N/A</td>
<td>146</td>
</tr>
<tr>
<td>Quartz</td>
<td>31.7</td>
<td>27.8</td>
<td>33.5</td>
<td>1.1</td>
<td>31.8</td>
</tr>
<tr>
<td>Feldspar</td>
<td>59.5</td>
<td>44.4</td>
<td>53.6</td>
<td>4.4</td>
<td>46.7</td>
</tr>
<tr>
<td>Muscovite</td>
<td>0.6</td>
<td>0.3</td>
<td>0.3</td>
<td>0.1</td>
<td>0.4</td>
</tr>
<tr>
<td>Biotite/Chlorite</td>
<td>5.5</td>
<td>7.8</td>
<td>9.4</td>
<td>2.6</td>
<td>15.6</td>
</tr>
<tr>
<td>Hornblende</td>
<td>0.0</td>
<td>0.7</td>
<td>0.9</td>
<td>0.6</td>
<td>1.1</td>
</tr>
<tr>
<td>Carbonates</td>
<td>0.4</td>
<td>0.5</td>
<td>0.6</td>
<td>0.2</td>
<td>0.9</td>
</tr>
<tr>
<td>Titanite</td>
<td>0.1</td>
<td>0.1</td>
<td>0.2</td>
<td>0.0</td>
<td>0.3</td>
</tr>
<tr>
<td>Apatite</td>
<td>0.2</td>
<td>0.2</td>
<td>0.3</td>
<td>0.1</td>
<td>0.5</td>
</tr>
<tr>
<td>Allanite-(Ce)</td>
<td>0.0</td>
<td>0.1</td>
<td>0.1</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Hematite/Magnetite</td>
<td>0.5</td>
<td>0.3</td>
<td>0.3</td>
<td>0.1</td>
<td>0.3</td>
</tr>
<tr>
<td>Porosity</td>
<td>0.5</td>
<td>17.0</td>
<td>-</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>other minerals</td>
<td>0.2</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>unidentified pixels</td>
<td>0.8</td>
<td>0.7</td>
<td>0.8</td>
<td>0.2</td>
<td>2.4</td>
</tr>
</tbody>
</table>

Std dev = standard deviation, calculated excluding porosity.
2.4.3. Microwave Irradiation of Rock Specimens

For induced crack experiments, the 24 core specimens initially subjected to ultrasonic testing were subsequently exposed to microwave irradiation. The irradiation was achieved in a standard household Panasonic NE-3240 microwave operating at 2450 MHz with a power of 3.2 kW in a multi-mode configuration. Following the procedure described by Peinsitt et al. (2010), individual specimens were placed on top of a cylinder made of quartz glass in the center of the microwave cavity. The cylinder allows one to position the specimen in the location with the highest field strength inside the cavity. Quartz glass was chosen because it is penetrated by microwaves without any absorption and thus has no further influence on the experiment. Irradiation times were between 15 and 360 seconds, with duplicate or triplicate specimens for each irradiation time. The surface temperature of irradiated specimens was measured during treatment with an Ahlborn MR721420 infrared thermometer with adjustable focus and emissivity $\varepsilon$, according to the specific emissivity of the rock type calibrated with a standard thermocouple.
2.4.4. UCS Testing

Uniaxial Compressive Strength (UCS) tests were conducted on 18 specimens (16 irradiated specimens in addition to two control specimens that were not irradiated). Prior to testing, the irradiated 55 x 150 mm core specimens were trimmed in length to 110 mm to produce standard sized UCS specimens, and the remaining 40 mm length of trimmed core was retained for mineralogical analyses. The UCS testing was accomplished using an electronic servo-controlled MTS stiff testing machine with a capacity of 220 kips (1MN) using ASTM standard D7012. Experimental data were retrieved using a computer-based acquisition system and analyzed using a spreadsheet program.

2.4.5. X-ray Computed Tomography

X-ray computed tomography (CT) is a non-destructive imaging technique, first introduced in medical sciences in the early 1970s (Hounsfield 1972, 1973) and applied to the geosciences starting in the 1980’s with soil science (Petrovic et al. 1982) and petroleum (Vinegar 1986). The X-ray CT instrument acquires one- or two- dimensional radiographs for different positions, from which a 3D image is computationally reconstructed (Mees et al. 2003). There is a tradeoff between specimen size and resolution; for a typical specimen size of 1-2 mm diameter, maximum resolution with currently available microCT instrumentation is typically 0.5 to 1 µm. Variation in density and atomic composition of the components (minerals, cracks, fluids etc.) allows a 3D reconstruction showing the internal structure of the specimen (Mees et al. 2003), with some limitations. For example, attenuation contrasts between minerals must be sufficiently large in order to resolve individual phases in order to gather mineralogical data. The non-destructive nature of this test enables one to conduct repeat testing of the same specimen before and after application of cracking treatment(s).

For the present study, microCT analyses were conducted on specimens of granodiorite before and after irradiation. The analyses were conducted at the Colorado School of Mines using a ZEISS Xradia 520 Versa 3D X-ray microscope. The analyses were run at 80 kV and 7W, translating to a current of 0.0875 mA, using the 4x objective and LE1 filter provided by Zeiss. Acquired data were then reconstructed using the 3D visualization and analysis software Dragonfly 2.0. A resolution of 2.25 µm was deemed necessary for the observation of cracks in the present study. At this resolution, the maximum analytical volume that can be examined is a 2 mm diameter by 2.3 mm height cylinder inside a slightly larger cubic specimen (5 by 5 by 5 mm).
2.4.6. Thin Section Optical and Electron Microscopy

Mineralogical composition and cracks were compared in thin sections of untreated and irradiated specimens. Polished thin sections (27mm width, 46mm length, 30 µm thickness) were prepared for standard transmitted light petrography, scanning electron microscopy and automated mineralogy. Prior to preparing the thin sections, the core was impregnated with blue epoxy resin in a vacuum, in order to preserve the cracks and prevent the specimen from disintegrating.

Traditional transmitted light petrography was conducted on the thin sections in plane and cross-polarized light in order to assess the mineralogy and the geometry of the cracks. These data were supported by quantitative mineralogical analyses by automated scanning electron microscopy, which was used to generate false-color compositional maps of the thin sections. The automated mineralogy analyses were conducted at Colorado School of Mines on a TESCAN-VEGA-3 Model LMU VP-SEM platform equipped with the control program TiMA3. Four energy dispersive X-ray (EDX) spectrometers acquired spectra using a beam stepping interval (i.e. spacing between acquisition points) of 30 µm, an accelerating voltage of 25 kV, and a beam intensity of 14. Interactions between the beam and the specimen were modeled through Monte Carlo simulation. The EDX spectra were compared with spectra held in a look-up table allowing one to determine the composition of each acquisition point. The assignment makes no distinction between mineral species and amorphous grains of similar composition. Results were output by the TiMA3 software as a spreadsheet giving the area percent of each composition in the look-up table, with compositional assignments grouped appropriately. The following sections describe how the transmitted light and electron microscopy were used to examine the cracks, their spatial associations with minerals, and whether they occurred within or between grains.

2.4.6.1. Crack Detection

Under optical microscopy, cracks were detected in the thin sections based on the presence of the blue epoxy that was used to impregnate the specimens (see previous section). On the SEM, cracks could be detected in backscatter electron mode based on the compositional difference between the epoxy and the mineral grains. In automated mineralogy, pore space is not ordinarily analyzed or factored into the total volume of a thin section. In order to avoid detection and acquisition of data from epoxy or voids in the specimens, the brightness is typically set to a minimum detection
limit of 15 to 20 percent. However, in the present study the void spaces are the targets of interest, and cracks need to be accounted for in the automated mineral mapping and subsequent statistical analyses. In order to achieve this, a separate definition was created for the pore space in the instrument's Species Identification Protocol. Because the crystalline granodiorite examined in this study contains only 0.5% modal porosity prior to heat treatment (Table 2.2), nearly all of the pore space in the treated specimens can be attributed to induced cracks. This technique makes the cracks easy to detect and study. Assessment of the exact quantity of cracks created by heating would require a complex statistical analysis as mentioned in Sone and Condon (2017).

2.4.6.2. Mineral-Crack Associations

The spatial relationships between cracks and grains of a particular composition can be determined using liberation analysis of the automated mineralogy data. The liberation analysis mode of the TiMA3 software provides statistical data on the spatial association of minerals in the specimen. This is typically used to determine the degree to which ore mineral grains are liberated from the matrix material; for example, the percentage of sulfide grain boundaries in contact with void space. One of the features in this software package calculates the number of pixels of one mineral phase in contact with each of the other mineral phases, enabling calculation of the percentage of contact between two phases. In a traditional ore study, this feature might be used to calculate the percentage of sulfide grain boundaries in contact with quartz.

The methods employed in the present study build on traditional liberation analyses to provide a more nuanced dataset to characterize induced cracking. The void spaces (cracks) were defined as a phase in order to be accounted for in the subsequent calculations. By conducting a liberation analysis for the crack “phase,” it is possible to quantify the percentage of crack boundaries in contact with a particular mineral phase (e.g. this analysis could show that 50% of the crack boundaries are in contact with quartz in a hypothetical specimen). These results can be used to generate statistics on preferential spatial association between cracks and particular minerals in the specimen, as well as crack locations. This modified type of liberation analysis is useful because quantitative data on cracks can be rapidly generated for an entire scanned area. However, an important limitation is that no distinction can be made between an intragranular crack that breaks an individual grain, and an intergranular crack that occurs between two grains of the same composition.
2.4.6.3. Crack Classification

Prior studies have calculated linear crack density by drawing equally spaced horizontal lines across an image of a specimen and counting the number of times an open crack crosses a line, and then dividing this number by the line length (Underwood 1970; Fredrich and Wong 1986; Wang et al. 1989; Menéndez et al. 1999). The method can be extended or repeated to assess the cracking density in various directions and obtain a preferred orientation for cracking in a rock specimen (Menéndez et al. 1999). Line method point-counting (Underwood 1970, Galehouse 1971) can be paired with mineralogical data to characterize the boundaries of a crack.

The present study employed simultaneous point-counting of automated mineralogy false-color maps and cross-polarized thin section images. This method makes it possible to determine the frequency with which cracks occur within a single grain, between two grains of the same composition, and between two grains of different compositions. Individual grains of the same composition can be differentiated using transmitted light petrography, and grains with similar optical properties but distinct compositions can be differentiated using electron microscopy. If used in isolation, the limitations of each microscopy technique would preclude accurate crack boundary classification. In the simultaneous point-counting method, matching pairs of cross-polarized and automated mineralogy false-color images were imported into the software Jmicrovision (v.1.27). In order to provide a visually consistent demarcation of crack locations and geometries, the crack phase was delineated in red in the false-color map, and then this red crack phase was overlain on the cross-polarized image using Adobe Photoshop CC 2017. The cross-polarized image was used to identify grain boundaries, and the false-color map was used to definitively identify compositions. The matching pairs of images were divided into 10 equal areas by drawing parallel equidistant lines across each image at 2.5 mm spacing in Jmicrovision. Ten categories were developed to classify the possible boundaries on either side of a crack segment based on the three most abundant minerals identified through automated mineralogy: quartz, feldspar and biotite (Table 2.2). At each intercept between a crack and a point-counting line on the image, the boundary type was classified and recorded in Jmicrovision (Figure 2.2).

2.5. Results

The following sections demonstrate how the above described suite of methods was used to evaluate the effects of a rock cracking treatment.
Figure 2.2  Schematic diagram depicting how the point-counting method (Underwood 1970, Galehouse 1971) can be adapted to quantify the crack boundary mineralogy. The crossed polarized thin section image (left) and false color mineral map (right) are paired using Jmicrovision software, and equidistant parallel lines are drawn (at 2.5 mm spacing for the present study). For every crack intersecting a parallel line, the user manually attributes the crack boundary to the appropriate category (Bt-Bt, Bt-Qz, Qz-Qz); Bt: biotite, Qz: quartz.

2.5.1. Microwave Heating Characteristics

Microwave irradiation lead to the heating behavior described in Figure 2.3 and Table 2.3 The data describe an almost straight line up to 180 s of irradiation. The values at 360 s, however, are only slightly higher and the curve is suspected to flatten out. The scatter appears to increase with irradiation time. The maximum temperature reading was 427°C after 360 s of microwave irradiation.

2.5.2. Changes in Mechanical Properties

The mechanical property effects of microwave irradiation are shown in Table 2.3 and Figures 2.4, 2.5 and 2.6. Four core specimens crumbled into many small (0.25 to 5 cm$^3$) pieces after approximately 150 seconds of microwave irradiation and could not be subjected to UCS testing or the post-irradiation P-wave velocity testing. The UCS and $v_1$ values of these specimens were set to 0, representing their non-existing bearing capacity and inability to transmit P-waves.
Figure 2.3 Graph showing the maximum temperature reached by the specimen surface versus the duration of exposure to microwave irradiation (in seconds).

Figure 2.4 Graph showing UCS test results of untreated and irradiated specimens, plotted by irradiation time (in seconds). Untreated specimens were irradiated for 0 seconds. Specimens which were destroyed during the irradiation were assigned a UCS value of 0 MPa.
Table 2.3 Temperature, UCS and P-wave velocity collected for Boulder granodiorite specimens.

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Irrad = irradiation, std dev = standard deviation, \( \nu_0 \) = P-wave velocity prior to irradiation, \( \nu_1 \) = P-wave velocity after irradiation, \( \nu_{rel} \) = \( \nu_1 / \nu_0 \)

Means and standard deviations were calculated for specimens subjected to the same duration of irradiation; note that there is a substantial risk of underestimating population standard deviation for sample sizes under 30 (e.g. Lau and Carson, 2017).
Figure 2.5 Graph showing the relative P-wave velocity as function of microwave exposure time (in seconds). 100 % = initial P-wave velocity of each specimen ($v_{p0}$), relative velocity = $v_{p\text{after irradiation}} / v_{p0}$. The relative P-wave velocity is defined by the relation $v_{p\text{after irradiation}} / v_{p0}$, $v_{p0}$ being the P-wave velocity of the specimen prior to microwave irradiation (100%) and $v_{p\text{after irradiation}}$ the P-wave velocity of the specimen after microwave irradiation treatment.

Figure 2.6 Graph showing UCS test results (as presented in Table 2.3), plotted as a function of P-wave velocity after irradiation ($v_{p\text{after irradiation}}$).
For both UCS and P-wave velocities, there is scatter between specimens that were irradiated for the same length of time (Table 2.3). This scatter appears to represent pre-existing heterogeneity between specimens and is represented by the large standard deviations calculated for replicates. The UCS test results do not show a clear trend with microwave exposure time (Figure 2.4). The UCS values for duplicate untreated specimens (no irradiation) are 86 and 103 MPa, respectively. The UCS values after 90 s of irradiation are both higher: 122 and 148 MPa. However, the lowest measured value (4.6 MPa) was obtained for a specimen heated for the longest duration (360 s). Figure 2.5 shows the relative P-wave velocity along the z-axis of the irradiated Boulder granodiorite specimens as function of the irradiation time. Up to 60 s of irradiation time, the P-wave velocity is not significantly reduced relative to the pre-irradiation measurements. For irradiation times between 60 and 180 s, a negatively sloped trend can be observed, with minimum values of 57 %. Increasing the irradiation time above 120 s may lead to two different effects. Some specimens broke apart and completely disintegrated, represented by relative P-wave velocities of 0. The specimens that could be irradiated for 360 s show that the longer irradiation time does not necessarily lead to a decrease in P-wave velocity. This is similar to Figure 2.3, where the longer irradiation times did not lead to further temperature increases of the specimens. Figure 2.6 shows that there is a relationship between UCS and post-irradiation P-wave velocity $v_1$; specimens with more cracks are weaker.

### 2.5.3. Crack Characteristics

#### 2.5.3.1. Crack Detection

Figure 2.7 shows microCT images of a Boulder granodiorite specimen prior to treatment, compared to the same specimen after 45 seconds of irradiation. This technique clearly shows the location and morphology of the cracks. However, the small attenuation contrast between quartz and feldspar makes it challenging to differentiate minerals using this technique, precluding analysis of the crack boundary mineralogy.

Figure 2.8 shows plane and cross-polarized thin section images of untreated and irradiated granodiorite. In plane-polarized light, the blue epoxy helps show the locations of microwave-induced cracks in the irradiated rock. The cracks occur as a complex network of intragranular and intergranular cracks of varying apparent apertures from 5 µm to 1.5 mm width. The cross-polarized
light images can be used to identify grain boundaries, but cracks are much less obvious than in plane-polarized light.

Figure 2.9 shows 22 x 31 mm false-color mineral maps generated by automated mineralogy of the thin sections shown in Figure 2.8. The automated mineral mapping cannot distinguish grain boundaries within aggregates of a single mineral phase, so the false-color mineral maps show compositional regions rather than individual mineral grains. The data processing technique employed in this study enabled pore spaces (including cracks) to be shown as a phase in the false-color maps. Little pore space exists in the untreated specimen, whereas the automated mineral mapping clearly delineates microwave-induced cracks in the irradiated specimen. Quantitative analysis of the false-color mineral maps shows that the modal percent of each mineral phase decreased as a result of the heat treatment, in accordance with the increase in porosity (Table 2.2).

2.5.3.2. Mineral-Crack Associations

Liberation analysis was applied to the automated mineralogy data using the TIMA3 software, in order to quantify the spatial relationship between pore space and each mineral phase. Preferential cracking of particular mineral phases can be assessed by comparing the porosity-normalized modal compositions of irradiated specimens and the percentage of crack boundaries in contact with each mineral. Prior to irradiation the rock contains almost no porosity (Table 2.2, column A), so the 17.0 % void space in irradiated specimen B17 is attributed to the microwaving treatment with some potential dilation of microwave-induced cracks by the epoxy (Table 2.2, column B). The void space can be excluded from the modal abundance calculations by normalizing the data to a total of 83.0 rather than 100.0 (Table 2.2, column C). This is necessary because there is some variability among thin sections or specimens (Table 2.2 column D), so the statistical analysis of crack boundaries in contact with each mineral should be compared to the mineral modal abundances in the same thin section. The results of this comparison (Table 2.2, columns C and E) demonstrate that although most of the cracks are in contact with quartz and feldspar, the proportions do not match the porosity-normalized modal abundances of those minerals in the thin section. The percentage of cracks associated with feldspar is lower (46.7%) than the normalized percentage of feldspar in the specimen (53.6%), indicating that feldspar grains are fractured at a lower percentage than their modal abundance in the specimen. The percentage of cracks in contact with biotite is higher (15.6%) than the normalized percentage of biotite.
Figure 2.7 MicroCT images of (A) a specimen prior to irradiation and (B) the same specimen after 45 seconds of irradiation with arrows showing crack locations; (C) and (D) show the same images with cracks highlighted in red.
Figure 2.8 Thin section microphotographs of (A) whole thin section of untreated specimen B0 in cross polarized light and (B) whole thin section of irradiated specimen B17 in cross polarized light; arrows point to cracks. Microphotographs show the absence of cracks in (C) B0 untreated specimen in plane polarized and (D) cross polarized light, and the development of cracks in B17 after irradiation (E) in plane polarized and (F) in cross polarized light. Blue epoxy highlights the cracks in microphotographs of irradiated specimen B17 in (G) plane polarized and (H) cross polarized light. Bt: biotite, Fd: feldspar, qtz: quartz.
Figure 2.9 False-color mineral maps generated by electron microscopy and automated mineralogy of the entire thin section for (A) untreated specimen B0, corresponding to the area shown in Figure 2.8A, and (B) specimen B17 after irradiation for 146 seconds, corresponding to the area shown in Figure 2.8B. Figure 2.9 (C) shows the same area of uncracked specimen B0 as Figure 2.8C, and Figure 2.9 (D) corresponds to the same area of cracked specimen B17 shown in 2.8E.
present in the specimen (9.4%). The percentage of cracks in contact with quartz (31.8%) is similar to the normalized percentage of quartz in the specimen (33.5%); the similarity between these numbers suggests that the cracks have no preferential association or dissociation with quartz. Together these data suggest that cracks occur preferentially within or adjacent to biotite grains as compared to quartz or feldspar.

2.5.3.3. Crack Classification

Although the liberation analysis shows the statistical association between cracks and minerals, it cannot be used to determine whether the cracks occur within or between grains of a particular composition (e.g. within a grain of biotite, between two grains of biotite, or between biotite and another mineral). These data can be obtained by point-counting of crack-mineral boundaries, which allows one to distinguish among intragranular cracks, intergranular cracks between grains of identical composition, and intergranular cracks between grains of different compositions. Figure 2.10 shows how cracks were classified in a selected area of irradiated specimen B17. For example, for cracks that involve biotite there is a fairly even distribution among those that occur within biotite grains, between adjacent biotite grains, between biotite and feldspar, and between biotite and quartz.

2.6. Discussion

The goal of the present study is to provide suggested methods to characterize cracking induced in rock, rather than to determine why or how cracks are generated. This study also helps establish guidelines for the setup of future experimental work on rock breakage by various alternative
methods, particularly microwave irradiation. If crack characterization is well executed, the resulting datasets will provide a robust basis for theoretical studies on the mechanisms of cracking, as well as other applications such as geotechnical investigations on the damaged rock, or assessments of the efficacy of the breakage technique. The following sections provide suggested workflows that combine different methods in order to achieve results appropriate for four highlighted applications.

Figure 2.10 Point-counting workflow for a selected area of irradiated specimen B17. The false-color mineral map generated by automated mineralogy (right image; for color legend see Figure 7) allows one to determine the mineralogy of the crack boundaries. As in the schematic example shown in Figure 2.2, equidistant lines are drawn at 2.5 mm spacing. The cross polarized optical microscopy image (center) allows one to identify whether the cracks are intragranular or intergranular; this image has been overlaid with the cracking map generated by automated mineralogy in order to better locate the cracks. Each intersection between a crack and a line is manually assigned to one of ten categories of cracks, defined by the mineralogy of the specimen (left panel). Bt: biotite, Fd: feldspar, Qz: quartz.

During the design of any experiment, it is important to determine how many specimens should be tested for each treatment (e.g. for microwave-induced cracking, how many replicates from each irradiation time should be examined using each method). According to ASTM standard D7012 for UCS testing, the number of specimens should be determined using standard E122, “Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process” (ASTM 2017b). The sample size \( n \) (number of specimens to be tested at each condition) is determined according to the equation \( n = \left( \frac{3 \sigma_0}{E} \right)^2 \), where \( E \) is the maximum acceptable difference between the sample average (the average test result for \( n \) specimens comprising the statistical sample) and the true average, and \( \sigma_0 \) is an advance estimate of the lot or process standard deviation. Following these guidelines, UCS data from the present study (Table 2.3) can be used to
estimate the number of Boulder granodiorite specimens that should be subjected to UCS testing at each irradiation time. If an $E$ of 30 is acceptable, two specimens should be tested; if $E$ is 20, four specimens should be tested, and if $E$ is 10 then 16 specimens should be tested. Because the tolerance for $E$ is different for every testing method and individual experiment, researchers investigating rock cracking are urged to calculate the number of specimens according to the specific goals of the individual study. The following sections provide guidance on the testing methods suitable for four different study goals, focusing on the advantages and disadvantages of each method, rather than the number of specimens to be tested.

**Example 1: Quick assessment of damage and influence of pre-treatment conditions**

The results of this study confirm that *P*-wave velocity measurements can be used to provide a quantitative indication of cracking, as shown by previous authors (Table 2.1). *P*-wave velocity measurements are quick and cost-effective, and these data can be gathered from a large number of specimens relatively efficiently. This technique can be used to assess heterogeneity between specimens prior to application of a breakage technique. Individual *P*-wave velocity measurements can be compared to the average for a population of specimens in order to assess whether individual specimens have more pre-existing cracks than average.

Whereas studies on pre-existing cracks can employ destructive or non-destructive techniques to characterize the rock, studies attempting to assess induced cracking present a particular challenge because the pre-existing characteristics of the rock need to be compared to those after a fracturing treatment is applied. *P*-wave velocity measurements are non-destructive and enable one to compare the same specimen before and after a treatment. The Boulder granodiorite results show that relative *P*-wave velocity is a useful metric which can be used to quickly identify specimens that have been damaged (Figure 2.5). The non-destructive aspect is also especially suitable if only few specimens are available, because they remain intact for further study. However, *P*-wave velocity data do not allow one to study why or how the cracking occurred.

**Example 2: Geotechnical investigations**

A combination of UCS and *P*-wave velocity measurements can be used to assess geotechnical parameters of pre-existing cracks and new damage induced in the rock by a breakage treatment. The merits of *P*-wave velocity measurements are discussed above; for geotechnical
applications the relative P-wave velocity can be used to approximate the strength reduction resulting from a breakage treatment applied to the rock.

*Uniaxial compressive strength* testing is rapid, relatively cost-effective, and widely used for geotechnical purposes. This technique provided an efficient first-pass assessment of the effects of microwave irradiation on rock strength for the test specimens used in the present study (Figures 2.4 and 2.6). Most of the irradiated specimens showed a strength reduction relative to the untreated specimens, and the cracking recorded by P-wave velocity generally causes a reduction in strength. However, more nuanced conclusions could not be drawn given the pre-existing heterogeneity of the specimens. If the acceptable $E$ is high, UCS may serve as a useful approximation of the mechanical property effects of cracking. If the acceptable $E$ is low and few specimens are available, UCS must be used in conjunction with P-wave velocity measurements or another strength test. This technique may serve as a good indicator for studies on other types of induced cracking, particularly for more homogeneous specimen suites. However, the nature of the cracks cannot be determined from the changes in mechanical properties recorded by UCS or P-wave velocity, so these results will not add to the understanding of the processes of cracking.

**Example 3: Mineralogical investigations**

Data on the mineralogy of crack boundaries and cracked fragments are needed for studies on mineral liberation for ore processing, as well as for investigations on the causative mechanisms of cracking. For these applications, a combination of destructive and non-destructive microscopy techniques is recommended.

The existing non-destructive testing methods produce limited information and have practical constraints governing the resulting data outputs (Table 2.1). Of these techniques, *microCT* is probably the most applicable to investigations of crack mineralogy, which require data on the associations between cracks and particular minerals. However, the mineralogy of individual grains can only be resolved for some rock types. For the granodiorite test specimens used in the present study, the two most abundant minerals (feldspar and quartz) could not be differentiated from one another. MicroCT may also have some utility for studies on the mechanisms of cracking by providing data on crack morphology or crack apertures. Crack aperture measurements were not attempted using tomograms in the present study. Such measurements would be of limited utility because the thin section microscopy revealed that some apertures had apparent widths considerably smaller than detectable
with the microCT. It is unclear what size can be considered representative of a study material with
grain sizes in the mm-range, but analyses of a 5 mm$^3$ specimen would not likely provide useful data
for a specimen with 1 mm diameter grains. Toifl et al. (2017), Hartlieb and Grafe (2017) and Hartlieb
et al. (2017) demonstrated that damage induced by microwave irradiation can stretch out over tens of
centimeters in a radial pattern. Therefore the specimen size constraints of microCT instruments also
preclude the detection of cracks that may occur at a larger scale.

The combined approach of *thin section optical light microscopy and automated mineralogy*
can be used to classify cracks according to their mineralogical association. Previous studies have
used either thin section petrography (by optical light or SEM) or liberation analysis by automated
mineralogy, but the resulting datasets are insufficient if only one of these techniques is used in
isolation. Fredrich and Wong (1986) used transmitted light petrography to identify grain boundaries
between multiple grains of the same composition during point-counting of thermally induced rock
cracks, but they reported difficulties in distinguishing between different types of feldspars (i.e. grains
of similar but distinct composition). These authors reported that SEM backscatter electron images
provided sufficient compositional information to distinguish between intergranular and intragranular
.cracks. However, Wang et al. (1989) encountered challenges differentiating individual grains of the
same composition using SEM, precluding accurate identification of intergranular cracks between
multiple grains of the same composition. Previous studies on microwave-assisted grinding of ores
employing automated mineralogy have employed liberation analysis to determine the percentage of
ore mineral grains that have been broken free from the matrix (e.g. Kingman et al. 2004a, Scott 2008,
Singh et al. 2017), but this technique cannot differentiate between intergranular and intragranular
cracks in grains of the same composition.

The results of the present investigation demonstrate that *optical light microscopy* and
*automated mineralogy* should both be applied to obtain complete mineralogical information on the
cracks. The integration of these techniques enabled the quantification of the pore space generated by
cracking, liberation of the mineral phases, qualitative assessment of crack morphology, and
quantitative assessment of mineral-crack associations. Optical microscopy could be used to
determine mineralogy, whereas electron microscopy showed compositional boundaries only. The
pairing of these techniques allows one to differentiate individual mineral grains even for adjacent
grains with similar optical properties or identical compositions. Mineral-crack associations could only
be qualitatively determined using optical microscopy. The blue epoxy in the thin sections most clearly
demarcates the cracks in plane-polarized light, but cross-polarized light must be used to determine
mineralogy in many cases. The plane-polarized and cross-polarized datasets might be combined
using image-processing software for semi-quantitative classification of crack boundaries by point-
counting, but more reliable data are provided by point-counting of paired cross-polarized optical and
false color electron microscopy images using the assignment of pore space to a unique species
definition via automated mineralogy. Quantification of the induced pore space and statistical
determination of crack boundary mineralogy via liberation analysis required the application of
automated mineralogy to the electron microscopy data. Crack morphologies and apparent apertures
could be assessed using either optical or electron microscopy.

There are several drawbacks to the presented microscopy approach. First, it is important to
impregnate the specimens with epoxy so that they do not disintegrate during thin section preparation,
but the epoxy may cause some dilation of the cracks. This may be problematic for investigations of
crack aperture or total porosity, although it is not an issue for mineralogical characterization. Second,
the thin section microscopy data are limited to two dimensions. Crack aperture measurements will
record the apparent aperture rather than the true aperture. Although granodiorite is isotropic, any
anisotropy in the cracks would not be captured in the two-dimensional thin section data. Some three-
dimensional information could be obtained by pairing analyses of thin sections taken from orthogonal
planes, and this approach is recommended in particular for anisotropic rocks. Three-dimensional
information could also be obtained by examining a stacked series of thin sections from a single
specimen. The final drawback is that the microscopy techniques presented here are time consuming
and expensive, so the investigations can only be performed on a few specimens.

**Example 4: Comprehensive evaluations of cracking**

The entire suite of presented methods should be used in detailed investigations on cracking,
as well as studies evaluating the efficacy of an induced cracking procedure. No single non-destructive
or destructive test is known which can provide information on the characteristics of rock cracks, such
as aperture, location, morphology and associated mineralogy, as well as the effects of cracking on the
rock’s mechanical properties. However, by integrating all of the testing methods presented here for
the Boulder granodiorite specimens, a comprehensive dataset can be obtained which will provide
both types of data. This dataset can be used to characterize the effects of cracking on bulk rock
properties and mineral-scale microstructure, or to evaluate and optimize a cracking procedure. For example, the comprehensive dataset enables one to compare a) cracks induced in different rock types or specimens that were subjected to the same cracking procedure, or b) the efficacy of different cracking procedures applied to specimens of the same rock type. The testing will involve quantitative measurement of bulk-rock parameters (e.g. P-wave velocity, liberation analysis), as well as quantitative and qualitative analysis of single phenomena (e.g. crack boundary mineralogy, crack opening, interaction of cracks). The application of destructive tests will require a comparably large number of specimens. In order to assess a cracking treatment by destructive testing, each method must be applied to untreated and treated specimen pairs. The influence of pre-treatment conditions can only be investigated in control specimens, and such comparisons are only valid if there is no significant inter-specimen heterogeneity.

2.7. Conclusions
The present contribution demonstrates that a combination of destructive and non-destructive techniques can be used to provide a rich dataset on rock property changes and crack characteristics generated by an applied cracking treatment. A suite of methods were applied to microwave irradiated granodioritic specimens to demonstrate the strengths and weaknesses of each technique, as well as the utility of integrating multiple techniques. Ultimately, the selection of methods for characterization of induced cracks will depend on the specific research questions, as well as the amount of material and budget available.

Four example protocols are presented to provide guidance on the selection of analytical methods for different research questions: 1. Quick assessments of induced cracking can employ relative P-wave velocity measurements as non-destructive indicators of damage; 2. Geotechnical investigations can use both UCS and relative P-wave measurements to characterize the changes in strength caused by cracking; 3. Investigations on crack mineralogy can employ an integrated suite of microscopy techniques to gather data for ore processing or to examine the causative mechanisms of cracking, including simultaneous point-counting of optical and electron microscopy imagery, as well as liberation analysis of a specially designated crack phase using automated mineralogy. MicroCT analyses may be of utility if conducted before and after the cracking treatment, depending on the specific research question and mineralogy of the rock; 4. Comprehensive evaluations of cracking
should include data on the bulk rock property changes caused by cracking, as well as the characteristics of the cracks, including aperture, location, morphology and mineralogy. No single test can provide all the required data, but the entire suite of analytical methods presented here can be integrated in the workflow demonstrated for the Boulder granodiorite specimens, in order to provide a comprehensive dataset on cracks induced in rock.

2.8. Acknowledgments

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CHAPTER 3
TEXTURAL AND MINERALOGICAL CONTROLS ON MICROWAVE-INDUCED CRACKING IN GRANITES

A paper submitted to the Journal of Rock Mechanics and Rock Engineering
Marion Nicco¹,², Elizabeth A. Holley¹, Philipp Hartlieb³, Katharina Pfaff⁴

3.1. Abstract
Microwave irradiation has been considered as a potential method for weakening rock in mining and civil engineering applications. The technique has been mainly applied to ores containing strong microwave absorbers. However, most rocks in Earth’s crust are predominantly composed of silicate minerals which are weak microwave absorbers. This study investigates the textural and mineralogical controls on microwave-induced cracking of three types of granite primarily composed of weak microwave absorbers (albite, amphibole, biotite, orthoclase, and quartz) in varying proportions, textures (perthitic, granophyric and oikocrystic) and grain sizes (fine and coarse grained). Microwave irradiation experiments at 3.2 kW and 2.45 GHz led to a reduction in rock strength that was quantified using P-wave velocity measurements. The microwave-induced cracks were characterized qualitatively and quantitatively using a combined approach of optical microscopy and automated mineralogical analysis of scanning electron microscopy images. Coarse grained (1-5 mm) granites developed extensive networks of narrow cracks. Fine grained (<1 mm) granites of similar composition developed relatively few cracks. In all the specimens, cracking appears to be driven by contrasts among the chemical, mineralogical, thermal and microwave properties of the constituent minerals. The microwave energy is absorbed by the grains with the highest relative dielectric constant and transferred to neighboring grains via dielectric loss and thermal conductivity. Intragranular cracks develop along cleavage planes in response to thermal expansion of brittle grains such as albite or amphibole, and intergranular cracking occurs adjacent to thermally conductive or highly expansive.

¹ Department of Mining Engineering, Colorado School of Mines, 1600 Illinois Street, Golden, Colorado 80401, USA
² Primary researcher and author, corresponding author
³ Chair of Mining Engineering and Mineral Economics, Montanuniversitaet Leoben, Erzherzog-Johann-Straße 3, A-8700, Leoben, Austria
⁴ Department of Geology and Geological Engineering, Colorado School of Mines, 1516 Illinois Street, Golden, Colorado 80401, USA
grains such as quartz and biotite. The data indicate that granitoid rocks are potential targets for application of microwave-induced cracking in mining and civil excavation.

Keywords: Microwave, Crack, Microscopy, SEM, Granite, Mineralogy, Texture, Dielectric Properties

3.2. Introduction

Microwave irradiation has been proposed as a potential energy-saving method of rock fracturing for civil and mining applications (Didenko et al. 2005; Peinsitt et al. 2010; Hartlieb et al. 2012; Hartlieb et al. 2016; Hartlieb et al. 2017; Somani 2017). The thermal properties of minerals have long been known (e.g., Skinner 1966; Robertson 1988 and references therein), and a number of early studies measured the dielectric properties of minerals that govern their behavior during irradiation (e.g., Heiland 1951; Haalck 1958; eller, 1966, Parkhomenko 1967; Schuch 1967; Church et al. 1988). Studies on microwave-induced rock damage have focused on macro-scale characterization of cracks (Hartlieb et al. 2012; Hartlieb et al. 2016; Hartlieb et al. 2017), reduction of the rock mass strength (Peinsitt et al. 2010, Toifl et al. 2017, Zheng 2017, Reinosa et al. 2019), and particle size reduction of ores (Walkiewicz et al. 1991; Salsman et al. 1996; Kingman 2004; Scott 2006; Scott et al. 2008; Guo et al. 2011; Barani et al 2011; Rizmanoski 2011; Li et al. 2016; Singh et al. 2017, Zhong et al. 2018). However, it has been difficult to link macro-scale observations of microwave-induced rock fracturing to the mineral-scale causative processes. Without a fundamental understanding of the underlying mechanisms, researchers have been unable to target geologically appropriate rock types for microwave-induced fracturing, so the specimens and potential industrial applications investigated thus far have been limited.

3.2.1. Compositional Controls On Microwave-Induced Rock Fracturing

Although the thermal and dielectric properties of many individual minerals are known, most rocks contain multiple mineral phases. Numerical modeling studies have demonstrated that the microwave response of a polymineralic rock cannot necessarily be predicted by averaging the properties of the constituent minerals. This has been demonstrated in numerous studies of binary (two-mineral) systems (Whittles et al., 2003; Jones et al., 2005, 2007; Wang et al., 2008; Ali and Bradshaw, 2009, 2010, 2011; Wang and Djordjevic, 2014), as well as for more complex rock types such as granite and basalt (Meisels et al. 2015).
Microwave-induced fracturing is thought to be most effective if the mineral phases have contrasting properties. This has been established by empirical studies of particle size reduction in relatively simple particulate material, such as two or three mineral ores containing high proportions of sulfide minerals (good microwave absorbers) in a matrix of quartz or other non-ore minerals (Walkiewicz et al. 1991; Salsman et al. 1996; Harrison 1997; Kingman et al. 2000; Rizmanoski 2011; Batchelor et al. 2015). The above studies have attributed the macro-scale observations of cracking and particle size reduction to contrasting degrees of microwave absorption between the sulfide minerals and the matrix minerals such as quartz and feldspar. Without microscopic investigations of the response of individual grains, these studies have been unable to definitively attribute the observed cracks to particular mineral properties or textures. Significantly, many of these experiments applied microwave treatment to a pre-concentrated particulate ore containing more sulfides than commonly found in intact or unprocessed rock. However, most of Earth’s crust comprises silicate rocks such as granites, predominantly containing low dielectric loss minerals which are weak microwave absorbers.

Several studies have suggested that dielectric loss may not be the only mineral property that is predictive of microwave behavior. Ford and Pei (1967) showed that dark-colored metal compounds heated faster and typically attained higher temperatures than light-colored metal compounds. That study was conducted on single metal oxides, sulfides and charcoal and may not be representative of the reaction of the same materials when part of a rock assemblage. Similarly, Harrison (1997) postulated that dark colored minerals are better microwave absorbers than lighter minerals. Robinson et al. (2014) showed that the presence of hydrated clays (kaolinite, illite and others) lead to a higher maximum temperature of the microwaved specimen of oil sands, and Harrison (1997) also showed that clay minerals heat much faster than silica in sand. Lu et al. (2017) postulated that the iron content is a significant driver of microwave irradiation; this assertion has been questioned by Zheng (2017). Nicco et al. (2018) showed that microwave-induced fractures were disproportionately associated with hydrous minerals (biotite and chlorite) in granodiorite.

Although various properties of individual mineral grains and the contrast in properties between grains appear to be important, how individual mineral grains interact under microwave irradiation to generate cracks is not well understood. The present study addresses this knowledge gap by relating empirical observations of microwave-induced cracks in polymineralic granitic rocks to the constituent textures, mineralogical compositions, and mineral properties. The selected specimen suite was
specifically chosen to allow for comparisons between rock types with different modal abundances of the same minerals, as well as comparisons between rock types of similar compositions but different textures. The results offer important conclusions on the influence of grain size on crack network patterns, as well as the role of color, iron content, hydrous mineral water content, thermal expansion and dielectric loss in initiating and propagating cracks.

3.3. Methods

3.3.1. Core Specimen Preparation

Large cubic blocks of rock approximately 300 mm per side were obtained from three units in the Pike's Peak granitic suite, Colorado: the Pike's Peak biotite-bearing granite (PPB), the Mount Rosa granite (MR), and the Mount Rosa amphibole-bearing granite (MRA). The mineralogical descriptions are given in Section 3. The specimens were collected from exposures that had no macroscopically visible pre-existing weaknesses. The blocks were drilled at the Colorado School of Mines laboratory to produce 50 mm diameter by 100 mm length cores. A total of 29 specimens were prepared: 8 PPB, 8 MR, and 13 MRA.

3.3.2. Microwave Experiments

Microwave irradiation tests were conducted on individual core specimens with durations from 60 to 300 seconds at 2.45 GHz and 3.2 kW using a Panasonic NE-3240 microwave in multi-mode configuration. By varying the irradiation time, specimens with varying degrees of cracking were obtained, from initiation of individual single-grain scale microcracks to coalescence into through-going cracks. P-wave velocity testing was used as a first-pass, non-destructive assessment of the cracking. This method has been used in other studies to effectively record the presence of cracks induced in rocks (Wang and Simmons, 1978; Wang et al. 1989; Menéndez et al. 1999; Darot and Reschlé, 2000; Sone and Condon, 2017). In the present study, P-wave measurements were obtained before and after microwaving, in order to indicate the presence of newly induced cracks. The relative P-wave velocity is negatively correlated with maximum temperature of the bulk specimen as well as irradiation time (Peinsitt et al. 2010; Hartlieb et al., 2012; Nicco et al. 2018). Of the 30 specimens, only 20 retained their integrity and allowed a measure of P-wave velocity after irradiation. The surface temperature of irradiated specimens was measured during treatment with an Ahlborn MR721420
infrared thermometer with adjustable focus and emissivity $\varepsilon$, according to the specific emissivity of the rock type calibrated with a standard thermocouple.

### 3.3.3. Specimen Preparation After Irradiation

For specimens showing a decrease in P-wave velocity after microwaving, the core was impregnated with colored epoxy in order to visually identify and preserve the microcracks. This step also prevents the specimen from disintegrating during cutting of thin section billets. The epoxy was applied passively at low temperature (55°C) rather than by vacuum impregnation, in order to prevent post-experimental dilation or extension of cracks. Polished thin sections were prepared perpendicular to the core axis in the middle of each core specimen, including one unirradiated specimen of each rock type. For irradiated specimens showing macroscopically visible cracking, additional thin sections were prepared perpendicular to the core axis at one quarter of the length and one eighth of the length of the core axis. In total 16 thin sections were prepared, including 6 PPB, 6 MR, and 4 MRA. The thin sections were 48 by 27 mm and 30 microns thick.

### 3.3.4. Crack Characterization Methods

The methods chosen to describe the cracks in this study were tested and explained at length in Nicco et al. (2018). In the following sections, short descriptions of each technique accompany a workflow for the present study shown in Figure 3.1.

Traditional transmitted light petrography was conducted on the thin sections in plane and cross-polarized light in order to assess the specimen mineralogy and the geometry of the cracks. Automated mineralogy analysis was conducted at the Colorado School of Mines using a TESCAN-VEGA-3 Model LMU VP-SEM platform equipped with the control program TIMA3. Backscatter electron brightness and EDS spectra were collected at a beam stepping interval (i.e. spacing between acquisition points) of 15 µm, an accelerating voltage of 25 keV, and a beam intensity of 14. The results were output by the TIMA3 software as a spreadsheet giving the area percent of each composition and cracks, respectively. Compositional assignments were grouped appropriately. Mineral association tables were generated, which calculate the number of contacts between any two compositional phases assigned in the software. This allows for the quantification of minerals associated with an identified crack phase as described in Nicco et al. (2018).
This type of analysis is particularly useful for determining the mineral phases associated with and hosting cracks where textures are complex.

In order to quantify intragranular versus intergranular cracks, the automated mineralogy false-color maps and cross-polarized thin section images were point-counted in tandem. The method is based on Underwood (1970) and detailed in Nicco et al. (2018). Equidistant lines were drawn on each pair of images. The line spacing was 100 pixels defined based on the automated mineralogy images. Depending on the size of the area selected for point counting, the total number of lines varied from 20 to 28. At each intercept between a fracture and a point counting line on the image the boundary type was classified and recorded in Jmicrovision (v.1.27) as shown in Figure 3.2. Particular textures were differentiated in order to identify any preferential association with cracks.

3.4. Material

The suite of specimens comprised three types of granite: the Pikes Peak biotite granite (PPB), the Mount Rosa amphibole granite (MRA) and the Mount Rosa granite (MR). These granitic rocks were collected in Mount Rosa area of Teller and El Paso counties, Colorado (Figure 3.3).
Figure 3.2 Point-counting workflow for a selected area of a specimen of PPB. The false-color mineral map generated by automated mineralogy (left image; for color legend see Figure 3.7) allows one to determine the mineralogy of the crack boundaries. Equidistant lines are drawn at 100 pixel (~0.25 mm) spacing. Each intersection between a crack and a line is manually assigned to one of ten categories of cracks, defined by the mineralogy of the specimen (left image). The cross polarized optical microscopy image (right image) allows for better identification of the nature of the cracks (intra- or intergranular); this image has been overlain with the cracking map generated by automated mineralogy in order to better locate the cracks.

Figure 3.3 Simplified geological map of the specimen area (adapted from Persson 2016). Markers show where the specimens were collected.
The three different rock types were described in detail by Gross and Heinrich (1965) and Persson (2017). Even though all three rock types are geologically related and mineralogically similar, their mineral modal abundances vary (i.e., biotite versus amphibole versus a lack of a hydrous mineral phase), as well as contrasting textures as shown in Table 3.1.

The Pikes Peak Biotite granite (PPB) is a white to pink, medium to coarse-grained biotite granite. It is characterized by roughly equal modal abundances of albite (27-44%), K-feldspar (20-25%) and quartz (36-44%), with approximately 4% biotite (Table 3.1). Minor to trace Fe-Ti oxides, apatite, zircon, fluorite and monazite can also be observed. The grain size ranges from 1 to 5 mm. The texture of the feldspars is perthitic as seen on Figure 3.4, characterized by visible segregation of potassium- and sodium-rich feldspar.

Table 3.1 Modal percent and textural characteristics of major minerals in PPB, MR, and MRA specimens

<table>
<thead>
<tr>
<th></th>
<th>PPB</th>
<th>MR</th>
<th>MRA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>43.9</td>
<td>39.8</td>
<td>35.5</td>
</tr>
<tr>
<td>Albite</td>
<td>27.3</td>
<td>23.7</td>
<td>34.1</td>
</tr>
<tr>
<td>Orthoclase</td>
<td>28.8</td>
<td>36.6</td>
<td>30.4</td>
</tr>
<tr>
<td>Biotite</td>
<td>4.4</td>
<td>0.1</td>
<td>1.5</td>
</tr>
<tr>
<td>Amphibole</td>
<td>0.0</td>
<td>5.6</td>
<td>8.0</td>
</tr>
<tr>
<td>Astrophyllite</td>
<td>0.0</td>
<td>2.4</td>
<td>0.3</td>
</tr>
<tr>
<td>Porosity</td>
<td>0.0</td>
<td>0.1</td>
<td>0.2</td>
</tr>
<tr>
<td>other minerals (less than 1%)</td>
<td>1.3</td>
<td>1.2</td>
<td>1.7</td>
</tr>
<tr>
<td>Grain size</td>
<td>1-5 mm</td>
<td>&lt;1 mm</td>
<td>0.5-2 mm</td>
</tr>
<tr>
<td>Texture</td>
<td>Perlitic feldspars</td>
<td>Granophyric quartz and alkali feldspars</td>
<td>Oikocrystic amphiboles</td>
</tr>
</tbody>
</table>

The Mount Rosa granite hosts two facies which have almost identical composition (Persson 2017). The mineralogy of both facies is dominated by albite (24-34%), orthoclase (30-37%), and quartz (35-40%), with lesser amphibole and mica, and trace fluorite, REE minerals, zircon, clays, and Fe-Ti oxides (Table 3.1). The first facies of the Mount Rosa granite (MR) is fine-grained (<1 mm), and
the texture is characterized by elongated amphibole randomly oriented in an albite-quartz-microcline matrix; quartz and alkali feldspar occur in a granophyric intergrowth, as seen on Figure 3.4. Specimens of MR contains 6% Na-Fe amphibole, 2% astrophyllite, and almost no biotite mica. The second facies of the Mount Rosa amphibole granite (MRA) has similar mineralogy but contains slightly different proportions of the hydrous minerals (8% Na-Fe amphibole, 2% biotite mica, and no astrophyllite; Table 3.1). The grain size of MRA ranges from 0.5 to 2 mm, and the texture is oikocrystic. This texture is common in granites and refers to large phenocrysts (oikocrysts) that envelop smaller crystals as shown in Figure 3.4.

Table 3.2 presents the chemical, mineralogical, thermal, and microwave properties of the five primary minerals in PPB, MR, and MRA. The dielectric constant is the capacity of a mineral to capture and store the microwave energy in the form of heat, whereas the dielectric loss number represents the capacity of the mineral to release that heat. The thermal conductivity translates the ability of the mineral to transfer heat. The thermal expansion relates to the behavior of a mineral when heated; a high thermal expansion coefficient means the mineral will tend to expand whereas a low thermal expansion coefficient means the mineral will tend to crack rather than expand. Depending on the shape of the mineral it may expand preferentially in a certain direction (axis), which is called linear thermal expansion; the total expansion is referred to as volumetric thermal expansion.

The five main minerals (amphibole, albite, biotite, quartz and orthoclase) in the PPB, MR and MRA granites are low dielectric loss minerals compared to ore minerals such as iron oxides or sulfides (Campbell and Ulrichs 1969; Chen et al. 1984; Church et al. 1988; Nelson et al. 1989b; Shannon et al. 1991; Shannon et al. 1992; Harrison 1997). However, there are differences in dielectric properties between the constituent minerals observed in the granitic samples, as well as variation in their other properties, such as thermal expansion (volumetric and linear), thermal conductivity, color, habit, crystal system tenacity and cleavage. Although some of the properties of the minerals are well documented, other properties relevant to this study (e.g., thermal expansion and dielectric loss) are less agreed upon. The data in Table 3.2 presents data were gathered from multiple sources (see Table 3.2 references) under varying experimental conditions. Furthermore, properties have not been studied for arfvedsonitic amphibole nor albitic plagioclase which are present in PPB, MR, and MRA. Therefore the table presents data for the closest possible compositions: amphibole- group minerals for arfvedsonite, and albite for the albitic plagioclase feldspar.
Figure 3.4 Cross polarized photomicrographs showing a representative area of PPB (A), a detailed view of the perthitic texture in PPB (B), a representative area of MR (C), a detailed view of the granophyric texture in MR (D), a representative area of MRA (E), and a detailed view of the oikocrystic texture in MRA (F). Ab: albite, Amp: amphibole, Fsp: feldspar, Qz: quartz, Or: orthoclase.
Table 3.2 Chemical, mineralogical, thermal, and microwave properties of the primary minerals in MR, MRA, and PPB specimens.

<table>
<thead>
<tr>
<th>Chemical properties</th>
<th>Biotite</th>
<th>Amphibole</th>
<th>Quartz</th>
<th>Feldspar - Albite</th>
<th>Feldspar - Orthoclase</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Empirical Formula</strong></td>
<td>KMg$<em>{2.5}$Fe$^{3+}$.5A$</em>{0.5}$Si$<em>{3}$O$</em>{10}$(OH)$_{2.75}$</td>
<td>Na$<em>{3}$Fe$^{3+}$.4Fe$^{3+}$.(Si$</em>{6}$O$<em>{22}$)(OH)$</em>{2}$</td>
<td>SiO$_{2}$</td>
<td>Na (AlSi$<em>{3}$O$</em>{8}$)</td>
<td>K(AlSi$<em>{3}$O$</em>{8}$)</td>
</tr>
<tr>
<td><strong>H$_{2}$O content (%)</strong></td>
<td>8.33-29.97</td>
<td>8.29</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td><strong>Ferrous content (%)</strong></td>
<td>3.64</td>
<td>1.88</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Mineralogical properties</th>
<th>Biotite</th>
<th>Amphibole</th>
<th>Quartz</th>
<th>Feldspar - Albite</th>
<th>Feldspar - Orthoclase</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Color</strong></td>
<td>Dark brown</td>
<td>Black</td>
<td>Colorless to white</td>
<td>White to grey</td>
<td>Colorless to pale pink</td>
</tr>
<tr>
<td><strong>Habit</strong></td>
<td>Lamellar</td>
<td>Fibrous</td>
<td>multiple</td>
<td>Blocky granular or striated</td>
<td>Blocky granular or prismatic</td>
</tr>
<tr>
<td><strong>Crystal system</strong></td>
<td>monoclinic</td>
<td>Monoclinic - prismatic</td>
<td>trigonal</td>
<td>triclinic</td>
<td>monoclinic</td>
</tr>
<tr>
<td><strong>Tenacity</strong></td>
<td>Elastic</td>
<td>brittle</td>
<td>brittle</td>
<td>brittle</td>
<td>brittle</td>
</tr>
<tr>
<td><strong>Cleavage</strong></td>
<td>{001} Perfect</td>
<td>{110} Perfect</td>
<td>Poor/Indistinct</td>
<td>Perfect on {001}, good on {010}, imperfect on {110}</td>
<td>Perfect on {001}, good on {010}</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Thermal properties</th>
<th>Biotite</th>
<th>Amphibole</th>
<th>Quartz</th>
<th>Feldspar - Albite</th>
<th>Feldspar - Orthoclase</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Volumetric thermal expansion coefficient 10$^{-5}$/°C</strong></td>
<td>x</td>
<td>2.27-2.52$^a$</td>
<td>4.52 (inversion at 573°C)$^b$</td>
<td>2.68$^c$</td>
<td>0.97$^b$</td>
</tr>
<tr>
<td><strong>Highest magnitude linear thermal expansion 10$^{-5}$/°C</strong></td>
<td>perpendicular to c-axis: 411 - 5,552$^a$</td>
<td>parallel to a-axis: 1.40 - 1.53$^b$</td>
<td>perpendicular to c-axis: 1.75$^b$</td>
<td>parallel to a-axis: 0.83$^b$</td>
<td>parallel to a-axis: 0.98$^b$</td>
</tr>
<tr>
<td><strong>Thermal conductivity at room temperature</strong></td>
<td>1.17</td>
<td>2.85</td>
<td>7.69</td>
<td>2.31</td>
<td>2.31</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Microwave properties</th>
<th>Biotite</th>
<th>Amphibole</th>
<th>Quartz</th>
<th>Feldspar - Albite</th>
<th>Feldspar - Orthoclase</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Dielectric constant</strong></td>
<td>5.9$^f$</td>
<td>7.37$^g$</td>
<td>3.89$^f$</td>
<td>5.45-7.24$^f$</td>
<td>4.34$^f$</td>
</tr>
<tr>
<td><strong>Dielectric loss</strong></td>
<td>3.46*10$^{-4}$</td>
<td>0.026$^g$</td>
<td>1.37*10$^{-4}$</td>
<td>x</td>
<td>0.43*10$^{-4}$</td>
</tr>
<tr>
<td><strong>Microwave absorbing capacity</strong></td>
<td>+</td>
<td>++</td>
<td>-</td>
<td>--</td>
<td>+/-</td>
</tr>
</tbody>
</table>

---

$^a$ Oberti et al. (2018), amphibole = riebeckite, single crystal from temperature 25 to 425 °C
$^b$ Skinner (1966) on single crystal from 20°C to 400°C
$^c$ Prewitt et al (1976) from 25°C to 1105°C
$^d$ Hildert and Dickinson (1945) on multiple crystals from 20°C to 700°C under 30lb/in$^2$ load
$^e$ Cermack and Rybach (1982) amphibole = hornblende
$^f$ Church et al. (1988): room temperature; 1GHz
$^g$ Zheng 2017 at room temperature and 2.45GHz, from Nelson et al. 1989 (amphibole = richterite)
$^h$ Keller 1966 1MHz to 1000GHz
$^i$ Comparative ranking, from Zheng 2017
x: no data available
3.5. Results
3.5.1. Physical Properties

Figure 3.5 shows the maximum surface temperature reached for the three types of specimens as a function of irradiation time. Specimens of PPB and MR show linear relationships between irradiation time and the maximum attained temperature. Specimens of PPB reached a maximum surface temperature of 415°C after 240 seconds of irradiation, and MR reached a maximum surface temperature of 275°C after 300 seconds of irradiation. The MRA specimens produced scattered surface temperature results, including one specimen that achieved a surface temperature of 240°C after 180 seconds of irradiation; visual inspection showed that the specimen melted at the center. In general, the actual temperature reached at the center of the core is higher than the measured maximum temperature on the outside of the core (Hartlieb 2012).

Figure 3.6 shows the relative p-wave velocity along the z-axis for the three types of specimens (PPB, MRA, and MR) as a function of irradiation time. Ten of the thirty specimens broke apart and completely disintegrated during microwave irradiation, represented by relative p-wave velocities of 0. The 8 PPB specimens which retained their integrity show a negative linear correlation between irradiation time and relative p-wave velocity; relative p-wave velocities are approximately 0.9 after 40 seconds of irradiation and approximately 0.4 after 240 seconds of irradiation. The 6 MR
specimens show a similar negative linear correlation, but the relative p-wave velocities do not
decrease below 70% even for irradiation times over 300 seconds. The MRA specimens show
scattered results.

![Graph showing the relative p-wave velocity as function of microwave exposure time. 100% = initial p-wave velocity of each specimen (vp₀), relative velocity = vp_after_ireadiation / vp₀.]

3.5.2. Microscopic Observations

The following paragraphs present the characteristics of the microwave-induced cracks
observed in PPB, MR, and MRA. Details are provided on crack abundance, morphology, apparent
aperture and length, as well as mineralogical associations of the cracks.

The irradiated specimens of PPB developed numerous cracks of narrow apparent aperture
(15 to 30 microns; Figure 3.7). The cracks form complex and intricate networks that appear to
delineate most of the grain boundaries in the specimen (intergranular cracks), although numerous
intragranular cracks are also present within grains. Some wider cracks (30 to 100 microns) occur but
are uncommon. The high abundance of cracks correlates with the observation of low relative p-wave
velocities for PPB. The cracks are equally distributed throughout the specimens and have no
Figure 3.7 Photomicrograph panorama of representative PPB specimen after microwave irradiation. Cross polarized light image (A) is overlain with the cracking map generated by automated mineralogy in order to better locate the cracks. The false color mineral map generated by electron microscopy and automated mineralogy of the same area is shown in (B), and the crack phase is shown in isolation in (C).
preferential association with the perthitic texture in alkali feldspar. Within alkali feldspar, intragranular cracks occur both parallel and perpendicular to the perthitic exsolution lamellae. Visual inspection did not reveal a preferential mineralogical association of the cracks.

In contrast, irradiated MR specimens developed relatively few cracks which are mostly of large apparent aperture (100 to 700 microns; Figure 3.8). These sparse cracks appear to be through-going, extending from one side or end of the core specimen to the other. The cracks in MR typically occur along grain boundaries. Rare cracks of narrower aperture are also present and contain a higher proportion of intragranular segments. The cracks in MR do not appear to show any preferential relationship with zones of the granophyric quartz-feldspar intergrowth texture. The fine grain size of the specimens precludes assessments of the mineralogical association of the cracks by visual inspection.

Specimens of MRA host three types of cracks (Figure 3.9). The most volumetrically abundant cracks are of relatively wide apparent aperture (60 to 300 microns). The wide cracks have complex morphology that typically follow grain boundaries. Where they encounter oikocrystic textures, the wide cracks are dominantly intragranular within the amphiboles. The specimens also developed numerous narrow (<15 microns) microcracks of very short apparent length (<100 microns, more typically <50 microns) within individual amphibole grains. It is not possible to determine where the cracks originate or terminate, but the two-dimensional thin section data suggest that these microcracks may be uncoalesced precursors of the first crack type in MRA. Finally, the MRA specimens host rare through-going, straight cracks of narrow apparent aperture (10 to 30 microns) that are commonly intragranular.

The mineralogical affinity for cracks can be quantitatively determined using automated mineralogy data. Designation of a separate crack phase during the automated mineralogy analysis allows for quantification of a mineral’s spatial association with cracks; the mineral’s crack affinity can then be calculated by comparison with the modal abundance of the mineral. If the cracks are equally distributed among all the minerals in a specimen, the ratio of crack association to modal abundance will be 1 for each mineral. A ratio greater than 1 indicates that a particular mineral is disproportionately cracked, whereas a ratio smaller than 1 indicates that a mineral is disproportionately not cracked. For example, in specimen PPB8C, the modal abundance of albite is 36%, but 46% of the cracks are in contact with albite, giving a ratio of 1.3 (table 3.3): therefore the
Figure 3.8 Photomicrograph panorama of representative MR specimen after microwave irradiation. Cross polarized light image (A) is overlain with the cracking map generated by automated mineralogy in order to better locate the cracks. The false color mineral map generated by electron microscopy and automated mineralogy of the same area is shown in (B), and the crack phase is shown in isolation in (C).
Figure 3.9 Photomicrograph panorama of representative MRA specimen after microwave irradiation. Cross polarized light image (A) is overlain with the cracking map generated by automated mineralogy in order to better locate the cracks. The false color mineral map generated by electron microscopy and automated mineralogy of the same area is shown in (B), and the crack phase is shown in isolation in (C).
cracks are preferentially associated with albite. The automated mineralogy data produce spatial associations between compositional areas (e.g., areas of crack porosity and areas of quartz), rather than mineral grains.

Therefore it is not possible to differentiate between intergranular and intragranular cracks, although all three groups of specimens developed both types. On average for PPB specimens, table 3.3 shows that cracks tend to be preferentially associated with albite (1.2) rather than biotite (0.7), quartz (0.8) or orthoclase (0.9). For MR specimens, table 3.4 shows that cracks tend to be preferentially associated with albite (ratio 1.4) and amphibole (1.8) rather than orthoclase (0.7) or quartz (0.7). For MRA specimens, table 3.5 shows that cracks tend to be preferentially associated with amphibole (ratio 2.7) rather than albite (0.9), orthoclase (0.6) or quartz (0.7). This analysis cannot differentiate between the three crack types present in MRA. In general, according to the automated mineralogy data cracks seem to be more associated with amphibole and albite, and less with biotite, quartz and orthoclase.

Table 3.3 Mineral association table for PPB from automated mineralogy analyses.

<table>
<thead>
<tr>
<th></th>
<th>PPB1B - Modal analysis</th>
<th>PPB1C - Modal analysis</th>
<th>PPB8C - Modal analysis</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>modal abundance normalized to exclude porosity</td>
<td>% crack boundaries in contact with each mineral</td>
<td>rati o</td>
<td>% crack boundaries in contact with each mineral</td>
<td>rati o</td>
</tr>
<tr>
<td>Quartz</td>
<td>24.67</td>
<td>21.33</td>
<td>0.9</td>
<td>37.18</td>
</tr>
<tr>
<td>Albite</td>
<td>37.12</td>
<td>42.61</td>
<td>1.1</td>
<td>32.29</td>
</tr>
<tr>
<td>Orthoclase</td>
<td>26.49</td>
<td>23.66</td>
<td>0.9</td>
<td>26.11</td>
</tr>
<tr>
<td>Biotite</td>
<td>9.13</td>
<td>5.76</td>
<td>0.6</td>
<td>3.30</td>
</tr>
<tr>
<td>other</td>
<td>2.59</td>
<td>6.64</td>
<td>1.12</td>
<td>2.31</td>
</tr>
</tbody>
</table>

Point counting results are presented in Table 3.6 for 4 thin sections of PPB, 2 thin sections of MR, and 1 thin section of MRA. These results allow for comparison of intragranular cracks within grains of each composition, as well as intergranular cracks between grains of the same or different compositions. The point counting results do not differentiate between the orthoclase and albite which occur as exsolution lamellae in the alkali feldspar. For all rock types, approximately 60% of the
microwave-induced cracks are intergranular between grains, and about 40% of the microwave-induced cracks are intragranular within grains. For PPB the results are consistent and have a low standard deviation among the 4 thin sections (<5%). In PPB, most intragranular cracks occur within feldspars, and most intergranular cracks occur between quartz and feldspar. For MR, most intergranular cracks occur between quartz and feldspar, or within the granophyric intergrowth zones of quartz and feldspar (designated as “other” for MR in the table). In MRA, intragranular microwave-induced cracks are mostly in feldspar and amphibole grains, or intergranular along feldspar and amphibole grain boundaries.

Table 3.4 Mineral association table for MR from automated mineralogy analyses.

<table>
<thead>
<tr>
<th></th>
<th>MR5B - Modal analysis</th>
<th></th>
<th>MR5C - Modal analysis</th>
<th></th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>modal abundance</td>
<td>% crack boundaries in contact with each mineral</td>
<td>ratio</td>
<td>modal abundance</td>
<td>% crack boundaries in contact with each mineral</td>
</tr>
<tr>
<td>Quartz</td>
<td>34.29</td>
<td>21.82</td>
<td>0.6</td>
<td>32.54</td>
<td>21.79</td>
</tr>
<tr>
<td>Albite</td>
<td>25.96</td>
<td>39.12</td>
<td>1.5</td>
<td>27.58</td>
<td>38.34</td>
</tr>
<tr>
<td>Orthoclase</td>
<td>31.86</td>
<td>24.8</td>
<td>0.8</td>
<td>32.10</td>
<td>20.14</td>
</tr>
<tr>
<td>Amphibole</td>
<td>5.86</td>
<td>8.83</td>
<td>1.5</td>
<td>6.11</td>
<td>13.08</td>
</tr>
<tr>
<td>other</td>
<td>2.03</td>
<td>5.43</td>
<td>1.66</td>
<td>6.65</td>
<td></td>
</tr>
</tbody>
</table>

Table 3.5 Mineral association table for MRA from automated mineralogy analyses.

<table>
<thead>
<tr>
<th></th>
<th>MRA4F - Modal analysis #2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>modal abundance</td>
</tr>
<tr>
<td>Quartz</td>
<td>28.65</td>
</tr>
<tr>
<td>Albite</td>
<td>32.27</td>
</tr>
<tr>
<td>Orthoclase</td>
<td>32.60</td>
</tr>
<tr>
<td>Amphibole</td>
<td>4.17</td>
</tr>
<tr>
<td>other</td>
<td>2.31</td>
</tr>
</tbody>
</table>
3.5.3. Summary

Table 3.7 synthesizes the observations made on the three types on granite.

3.6. Discussion
3.6.1. Textural Controls On Cracking

Despite similar mineralogical compositions of PPB and MR, there are distinct differences in the cracking patterns between MR and PPB. The main difference between samples PPB and MR seems to be their overall grain size. The MR specimens are fine grained (0.5 to 2 mm) and have very few cracks which are relatively wide. In contrast, the PPB specimens are coarser grained (1 to 5 mm) and display a complex network of fine cracks. The PPB cracking patterns are similar to those observed in microwave-irradiated Boulder granodiorite specimens of similarly coarse grain sizes (Nicco et al. 2018). These observations are also in accordance with Zheng (2017), who subjected three types of rocks to microwave irradiation: a gabbro with a grain size of 3 mm to 7 mm, a granite with grain size of about 2 mm, and a monzonite with grain size <1 mm. The finer-grained granite and

Table 3.6 Average point counting results for PPB (4 specimens), MR (2 specimens) and MRA (1 specimen). Amp: amphibole, Bt: biotite, Fsp: Feldspar, Qz: quartz.

<table>
<thead>
<tr>
<th></th>
<th>PPB</th>
<th>MR</th>
<th>MRA</th>
<th>MR wave-induced cracks</th>
<th>Pre-existing crack</th>
</tr>
</thead>
<tbody>
<tr>
<td>Intra Fsp</td>
<td>31% 1.9%</td>
<td>Intra Fsp</td>
<td>10.1% 1.3%</td>
<td>Intra Fsp 22.6% 17.5%</td>
<td>36.6%</td>
</tr>
<tr>
<td>Inter Fsp</td>
<td>16.7% 0.4%</td>
<td>Inter Fsp</td>
<td>10% 2.3%</td>
<td>Inter Fsp Fsp 7.0% 9.8%</td>
<td></td>
</tr>
<tr>
<td>Intra Qz</td>
<td>7.5% 1.3%</td>
<td>Intra Qz</td>
<td>5.2% 0.2%</td>
<td>Intra Qz 13.6% 6.1% 34.1%</td>
<td></td>
</tr>
<tr>
<td>Inter Qz</td>
<td>5.3% 1.7%</td>
<td>Intra Qz</td>
<td>7.1% 1.9%</td>
<td>Inter Qz 2.6% 2.6% 2.4%</td>
<td></td>
</tr>
<tr>
<td>Qz-Fsp</td>
<td>25.7% 1.2%</td>
<td>Qz-Fsp</td>
<td>23% 4.6%</td>
<td>Qz-Fsp 12.3% 12.3% 0%</td>
<td></td>
</tr>
<tr>
<td>Intra Bt</td>
<td>1.9% 0.2%</td>
<td>Intra Amp</td>
<td>12% 1.5%</td>
<td>Intra Amp 15.5% 20.2% 2.4%</td>
<td></td>
</tr>
<tr>
<td>Inter Bt</td>
<td>0.6% 0.3%</td>
<td>Inter Amp</td>
<td>2.7% 0.4%</td>
<td>Inter Amp 0.0% 0% 0%</td>
<td></td>
</tr>
<tr>
<td>Fsp - Bt</td>
<td>3.7% 0.7%</td>
<td>Fsp-Amp</td>
<td>8.5% 0.2%</td>
<td>Fsp-Amp 18.1% 23.7% 0%</td>
<td></td>
</tr>
<tr>
<td>Qz-Bt</td>
<td>5.7% 1.4%</td>
<td>Qz-Amp</td>
<td>3.8% 0.4%</td>
<td>Qz-Amp 7.1% 9.7% 0%</td>
<td></td>
</tr>
<tr>
<td>Other</td>
<td>2.2% 0.8%</td>
<td>Other</td>
<td>18% 8.1%</td>
<td>Other 0.7% 0.9% 0%</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>40.1% 2.9%</td>
<td>39% 7.5%</td>
<td>51.6% 43.6%</td>
<td>73.2%</td>
<td></td>
</tr>
</tbody>
</table>

Table 3.6 Average point counting results for PPB (4 specimens), MR (2 specimens) and MRA (1 specimen). Amp: amphibole, Bt: biotite, Fsp: Feldspar, Qz: quartz.
monzonite developed relatively few cracks which typically crosscut the entire specimen, and most of the granite specimens broke in half along these cracks. In contrast, the coarser grained gabbro showed a complex network of cracks (Zheng 2017). In another study on specimens containing minerals of heterogeneous grain size, larger sulfide grains were spatially associated with cracks whereas smaller sulfide grains were not (P. Hartlieb, unpublished data).

Table 3.7 Summary of observations by specimen type.

<table>
<thead>
<tr>
<th>Characteristics of Crack</th>
<th>PPB</th>
<th>MR</th>
<th>MRA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain Size</td>
<td>1-5 mm</td>
<td>&lt;1 mm</td>
<td>0.5-2 mm</td>
</tr>
<tr>
<td>Apparent Aperture</td>
<td>Narrow (0.08 to 0.25 mm)</td>
<td>wide (0.1 to 0.7 mm)</td>
<td>wide (0.06 to 0.32 mm)</td>
</tr>
<tr>
<td>Morphology</td>
<td>Network around the grains</td>
<td>Across the specimen</td>
<td>irregular morphology and intergranular</td>
</tr>
<tr>
<td>Association</td>
<td>Affinity for albite and amphibole</td>
<td>Affinity for amphibole</td>
<td>Affinity for quartz</td>
</tr>
</tbody>
</table>

The relationship between grain size and cracking patterns is likely explained by the influence of grain size on the heating mechanisms of the specimens during microwave irradiation. Good microwave absorbers with high dielectric constants facilitate the heating, but the spatial distribution of those grains is crucial. In a fine-grained specimen such as MR, the grains are intercalated, and each small grain of a good microwave absorber likely lies in close spatial proximity to other good absorbers. This proximity may lead to better heat redistribution, which was also described by Toifl et al. (2017). This phenomenon may lead to the thermal runaway described by Peinsitt et al. (2010) and Jerby et al. (2013), causing strong specimen-scale thermal gradients through efficient heat
redistribution. The center of the irradiated samples reached temperatures much higher than the rims, resulting in melting, as well as the formation of small numbers of large-aperture cracks. In MR, the close spacing of small grains may have enabled efficient heat redistribution at the centers of the specimens. This could result in a strong thermal gradient at the specimen scale, which would produce the observed sparse, large-aperture, through going cracks. In coarse-grained rocks such as PPB, inefficient heat redistribution between more spatially dispersed microwave absorbers may cause high thermal gradients to develop between grains, leading to heterogeneous stresses which generate complex networks of intergranular cracks throughout the specimen.

The oikocrystic texture in MRA may have led to a thermal runaway similar to that described above. The texture in this rock type comprises patches of amphiboles that are clustered around other grains. This geometric arrangement of the amphiboles likely led to efficient heat redistribution in these zones, causing the melting observed in the specimen center of the specimen.

There are key differences between the microwave behavior of biotite in PPB and the biotite in Boulder granodiorite specimens studied by Nicco et al. (2018). In the previous study, the cracks were preferentially associated with biotite as shown in table 3.8, whereas in the present study the cracks were preferentially disassociated with biotite. This difference may be explained by contrasting biotite textures in the two types of specimens. In the Boulder granodiorite, the biotite is represented by clusters of small biotite minerals: each grain is 200 to 600 microns, and the clusters are 2 to 3 millimeters (Figure 3.10), whereas biotite in the PPB samples occurs as larger, isolated grains of 1.2 to 2.3 millimeters in a matrix of quartz and feldspar. The intragranular biotite and intergranular biotite-biotite cracks in the Boulder granodiorite appear to link cleavage planes among adjacent biotite grains. When one biotite grain undergoes linear thermal expansion perpendicular to the cleavage planes, it puts stress on the surrounding biotite grains, causing the crack to propagate along the cleavage planes of the biotites in the immediate vicinity. The cracks that are associated with biotite in the PPB specimens occur between biotite and other grains, where biotite grains are not adjacent to one another. Therefore thermal expansion of biotite is more likely to create stress at the grain boundaries with other minerals, causing intergranular biotite-quartz and biotite-feldspar cracks. The morphology of crack patterns also appears to be correlated with the cracking mechanisms. The interpretation is that the relatively straight and predominantly intragranular cracks are most likely pre-existing, related to loading or unloading. In contrast, microwave-induced cracks display irregular
morphology and are dominantly intergranular rather than intragranular. This can be observed in specimen MRA4F (Figure 3.9): the large, through-going, pre-existing crack is relatively straight and links numerous intragranular cracks as shown in table 6 in quartz, whereas the microwave-induced crack has an irregular shape because it links grain boundaries. Comparisons of crack morphology could be used to determine which cracks were present prior to the application of a rock fracturing treatment. However, there are relatively few non-destructive analytical techniques which can be applied before and after treatment to determine which cracks are new, and these methods tend to be time consuming and costly (e.g., microcomputed tomography; Nicco et al. 2018).

Table 3.8 Mineral association table for Boulder granodiorite specimens automated mineralogy analyses¹ (Nicco et al. 2018).

<table>
<thead>
<tr>
<th></th>
<th>Boulder18</th>
<th>Boulder17</th>
<th>average ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>modal abundanc e</td>
<td>% crack boundaries in contact with each mineral</td>
<td>rati o</td>
</tr>
<tr>
<td>Quartz</td>
<td>32.43</td>
<td>36.68</td>
<td>1.1</td>
</tr>
<tr>
<td>Albite</td>
<td>37.53</td>
<td>30.98</td>
<td>0.8</td>
</tr>
<tr>
<td>K-Fsp</td>
<td>19.47</td>
<td>18.6</td>
<td>1.0</td>
</tr>
<tr>
<td>Biotite</td>
<td>8.05</td>
<td>10.94</td>
<td>1.4</td>
</tr>
</tbody>
</table>

¹ Analyses were conducted at 30 micron intervals, in contrast to the 15 micron interval used in the present study.

3.6.2. Mineralogical Controls On Cracking

The specimens selected in this study allow for comparisons between two general categories of minerals (Table 3.2): amphibole and biotite are both Fe- and water-bearing, dark colored minerals. Quartz and feldspars are light colored and do not contain any water or iron. However, significant differences were observed in microwave behavior within each group: the feldspars and amphibole crack preferentially compared to the quartz and biotite.

These results show that the color of the mineral does not correlate with microwave irradiation behavior, in contrast to the assertions by Ford and Pei (1967) and Harrison (1997). Those studies postulated that darker minerals would be better microwave absorbers. In the present study, biotite (dark brown to black-ish) and amphibole (black) are the darkest minerals. Even though their dielectric constants are high relative to the other minerals present, their reaction to microwaves is very different: biotite does not show cracks whereas amphibole is disproportionately associated with cracks. This may relate to the fact that they do not have the same dielectric loss.
Figure 3.10 Photomicrograph panoramas in cross polarized light, overlain with the cracking map generated by automated mineralogy showing the biotite texture in specimens of Boulder granodiorite (A) and PPB (B). Higher magnification images of the inset areas are shown in cross polarized light in (C) and (D). False color mineral maps are shown in (E) and (F). The Boulder granodiorite images (A, C, E) show the association of cracks with cleavage planes in the biotite, whereas the PPB images (B, D, F) show the disseminated texture of biotite which is not selectively cracked. Bt: biotite Fsp: feldspar, Qz: quartz. See Figure 3.7 for automated mineralogy legend.
The present study also does not support the related hypothesis developed by Lu et al (2017) that higher iron content drives effective irradiation. Dark-colored silicate minerals commonly contain iron, and if iron content strongly influenced microwave behavior then color could be used as a proxy. Both amphibole and biotite contain iron; however the microwave behavior of these minerals is not easily discernible from minerals lacking iron, such as orthoclase or quartz. Finally, the present study does not support the hypothesis of Harrison (1997), Robinson (2014) and Nicco et al. (2018) that hydrous minerals are strongly responsive to microwave irradiation. Amphibole and biotite both contain water in their mineral structure (bound water); however they behave differently when exposed to microwave.

No single characteristic presented in Table 3.2 explains the cracking behavior of all minerals in the specimens. For example, minerals identified in literature as good microwave absorbers (e.g., biotite and amphibole) are not consistently cracked. Plagioclase feldspars are weak microwave absorbers with low dielectric constants (Zheng 2017 and Batchelor 2016) but were preferentially cracked during microwaving. Therefore, the microwave response must be explained by a combination of thermal and physical properties of a mineral, as well as the properties of the other minerals present in the specimen. The following interpretation focuses on the mineralogical properties, dielectric constant, dielectric loss, thermal expansion, and thermal conductivity of each mineral to explain the observed microwave responses of the specimens.

### 3.6.2.1. Feldspar

In the granitic specimens examined in this study, albite is disproportionately associated with cracks, whereas orthoclase is disproportionately disassociated with cracks, relative to the modal abundances of these minerals. This is the case in PPB specimens with perthitic texture (exsolution lamellae of albite and orthoclase; Table 3.3), as well as in MR where the feldspars occur separately (Table 3.4). Both types of feldspar are affected by intragranular and intergranular cracks (Table 3.6). Relative to the other minerals in the specimens, feldspars have moderate thermal conductivity and low linear thermal expansion coefficients. Similar to amphibole, feldspars are brittle and also exhibit one perfect cleavage direction. In contrast, biotite exhibits one direction of perfect cleavage but is not brittle, and quartz is brittle but does not exhibit cleavage. When a feldspar grain expands, it will crack, most likely along a cleavage plane. The differential behavior between the two feldspar types may be
explained by the dielectric constant and volumetric thermal expansion coefficient, which are higher for albite than orthoclase. Albite more effectively absorbs microwave irradiation and expands more significantly than orthoclase, leading to cracking. The dielectric loss of orthoclase is moderate but is unknown for albite, precluding comparisons of how the two feldspars transfer energy to neighboring grains.

3.6.2.2. Amphibole

Amphibole is disproportionately associated with cracks in MR (Table 3.4) and MRA (Table 3.5) relative to its modal abundance; it is not present in PPB specimens. In MR and MRA, amphibole grains are affected by both intragranular and intergranular cracks (Table 3.6). The intragranular cracking behavior may be explained by the high dielectric constant relative to neighboring minerals, as well as the brittle tenacity and perfect cleavage. Microwave energy is efficiently absorbed by the amphibole grains, and they respond brittlely by cracking. The high dielectric loss permits amphibole to transfer the microwave energy to neighboring grains, and differential thermal expansion relative to the amphibole (either in magnitude or orientation) likely causes the intergranular cracks.

3.6.2.3. Quartz

Quartz is disproportionately disassociated with cracks relative to its modal abundance in all three types of specimens tested in this study (Table 3.3, 3.4 and 3.5). Quartz grains are rarely affected by intragranular cracks or intergranular cracks between neighboring quartz grains, but the grain boundaries between quartz and other minerals are commonly cracked (Table 6). This may be explained by the fact that quartz has a low dielectric constant and low dielectric loss compared to the other minerals in the specimens, meaning that it is relatively unresponsive to microwave irradiation. However, quartz has a high volumetric thermal expansion coefficient and is the most thermally conductive of the minerals in the specimens. Energy lost from high dielectric loss grains such as amphibole will be transmitted efficiently by quartz, which itself will expand. Differential expansion between quartz and neighboring grains will generate cracking at the grain boundaries.
3.6.2.4. Biotite

Biotite is disproportionately unassociated with cracks relative to its modal abundance in PPB specimens, exhibiting few intragranular or intergranular cracks (Table 3.3). The modal abundance of biotite is very low in MR and MRA, precluding assessments of cracking behavior in those rock types. Biotite absorbs and transmits microwave energy relatively well, based on its high dielectric constant and high dielectric loss compared to the other minerals present. The linear expansion coefficient of biotite is dramatically larger than the other minerals; because biotite is elastic and expands perpendicular to cleavage planes it does not crack, but instead transfers heat stress to neighboring minerals, leading to cracking of other grains rather than biotite. In contrast, in rock types containing numerous adjacent biotite grains such as the Boulder granodiorite (Figure 10), the cracks propagate in biotite zones by linking cleavage planes, or along the boundaries of biotite zones due to high differential expansion.

3.7. Conclusions and Recommendations

Microwave-induced cracking of granites is driven by contrasts in textural, mineralogical, thermal, and microwave properties among the constituent minerals. The data demonstrate that no single criterion can explain the behavior of polymineralic rocks during microwave irradiation. Even for rocks such as granite containing only relatively weak microwave absorbers, contrasting properties cause the development of stresses between grains. The mechanisms by which the cracking occurs include heating of the specimen via the absorption of microwave energy by grains with the highest relative dielectric constant, transfer of this energy to neighboring grains via dielectric loss and thermal conductivity, intragranular cracking along cleavage planes in response to thermal expansion of brittle grains such as albite or amphibole, and intergranular cracking adjacent to thermally conductive or highly expansive grains such as quartz and biotite.

From a textural point of view, this study shows that coarse grained rocks develop complex networks of narrow cracks during microwave irradiation. Clusters of minerals with high thermal expansion coefficients and perfect cleavage such as biotite can enhance the cracking by linking cracks in cleavage planes. From a mineralogical point of view, this study shows that the color, water and iron content of constituent minerals cannot be used to predict the microwave behavior of a rock. The presence of adjacent grains with contrasting properties appears to be more critical.
This study suggests that even rocks containing only weak microwave absorbers are suitable targets for industrial applications of microwave-induced cracking. In such cases, the ideal candidates are either (1) coarse grained (1-5 mm) rocks containing randomly disseminated minerals with contrasting physical and microwaving properties, such as PPB, or (2) coarse grained (1-5 mm) rocks containing clusters of minerals with physical and microwaving properties contrasting with those in the surrounding rock, such as MRA. Ore deposits hosted in granitoids would be suitable targets for weakening by microwave irradiation prior to comminution. For example, porphyry copper deposits, some skarn deposits (tin, tungsten, and molybdenum), some epithermal deposits, intrusion-related gold deposits, and rare earth element-bearing intrusions may be hosted in granitoids that meet the two criteria defined above. Microwave pre-treatment of the rock should also be considered for civil excavations in granitoids that meet the same criteria.

3.8. Acknowledgments
This work was supported by the National Science Foundation (CMMI award #1550307). The authors thank Jae Erickson at Colorado School of Mines for assistance with specimen preparation.

3.9. References


Heiland GA (1951) Resistivities and dielectric constants of minerals, ores, rocks and formations.


4.1. Abstract

The mineralogical and textural controls on microwave-induced rock cracking are not completely understood. This case study investigates the behavior of biotite under microwave irradiation in specimen of Pikes Peak granite, in order to determine whether this process creates differential thermal expansion thereby generating cracks. Micro-computed tomography (microCT) is used on a specimen of granite before and after irradiation, and machine learning in imaging software is used to segment the cracks and identify the grains of biotite. This contribution documents these methods. Scanning electron microscopy-based automated mineralogy is used to assess the preferred mineralogical affinity of the cracks. The results of this study show that the homogeneous repartition of hydrated minerals (e.g., biotite) in the specimen seem to be more important drivers of cracking than their presence. Through observation of the microCT dataset before and after microwaving treatment, the present study also indicates that the shape of the specimen governs the cracking behavior more than the presence of pre-existing fine cracks.

Keywords: microwave, crack, microscopy, microCT

4.2. Introduction

Mineralogy and microtextures are known to exert important controls on rock fracture initiation and propagation (Eberhardt et al., 1999a, b). However, few rock mechanics studies incorporate sufficient mineralogical detail to shed light on the processes of fracturing at the grain scale. Rock types in such studies are commonly lumped into broad categories that do not account for subtle mineralogical or textural variations, e.g. granite or sandstone. This study utilizes state-of-the-art...
microanalytical techniques to illuminate the mineralogical and microtextural controls on rock fracturing, using a case study of microwave-induced cracks. Recent advances in micro-computed tomography (microCT) allow the examination of micron scale features in drill core-sized specimens. Automated mineralogy allows for quantification of micron scale mineralogy, textures, and void spaces in rocks subjected to fracturing treatments (e.g., Nicco et al., 2018). In concert, microCT and automated mineralogy provide a powerful suite of data at a resolution and scale suitable for detailed analysis of cracks in two and three dimensions.

Crack characteristics are known to reveal causative fracturing processes (e.g. Simmons and Richter, 1976). However, observation of those characteristics has been constrained by the limitations of the available analytical techniques. It has historically been difficult to capture data on crack morphology and mineralogy in two dimensions, and only very limited analyses have been possible in three dimensions. Recent studies have quantified microcracks in two dimensions, but the conclusions were limited by the small area of study and the lack of data in the third dimension (Charikinya et al. 2015, Griffiths et al. 2017). Fredrich and Wong (1986) extended their two-dimensional observations of thermally cracked granite to three dimensions using a geometric probability equation (Underwood, 1970), assuming isotropy of the granite mineralogy and the microfracture network. However, Wang et al. (1989) noted that this method is invalid because thermally-induced crack networks can be anisotropic even in isotropic rocks such as granite. Menéndez (1999) reconstructed a network of thermally-induced granite microfractures in three dimensions using orthogonal two-dimensional sections; each section was analyzed using confocal scanning laser microscopy of progressively deeper focal planes in the specimen. This laborious method allows for examination of crack morphology and aperture but does not provide mineralogical data. By refining microanalytical methods for characterizing microfractures in polymineralic rocks, the present study paves the way for investigations on microfractures generated by other mechanisms, such as conventional heating, loading, and hydrofracturing. The goal of this study is to show how recent advances in microCT and automated mineralogy can be used to study the mechanisms of rock fracturing. To illustrate the methods, the present study investigates the cracking of granite and expansion of biotite caused by microwave irradiation.
4.2.1. Mechanisms of Microwave-Induced Cracking

It has been postulated that differential thermal expansion is the cause for crack initiation and propagation in polymineralic rocks (Jones et al. 2005, Batchelor et al. 2015). For microwave-induced cracking, Harrison (1997), Kingman and Rowson (1998) and Batchelor et al. (2015) concluded that differential thermal expansion is promoted by a mix of strong and weak microwave absorbing minerals. However, most crustal rocks are composed of weak microwave absorbers (such as the silicate minerals quartz, feldspar and mica) with few strong absorbers if any (such as sulfides and oxides). Silicate rocks have not been extensively investigated even though they represent a large portion of the material encountered in civil excavations (e.g., tunneling) and mining (host rock, barren from mineralization). Kombusheshe (2010) showed that cracks were associated with biotite in kimberlite specimens lacking strong microwave absorbers. Kombusheshe (2010) postulated that this could either be due to the presence of interlayered water between the sheets of biotite, or the reaction of hydroxyl (−OH) groups in the structure of the biotite that liberate water if exposed to sufficient thermal energy (e.g., Grim 1968). Lu et al. (2017) showed that free grains of biotite expand up to 2.5 times when submitted to a microwave treatment, and other studies have shown that biotite expands perpendicularly to the cleavage plane under conventional heating (Hidnert and Dickson 1945, Cartz and Tooper 1964). However it has not been proven that the biotite contained in a rock specimen expands the same way.

Nicco et al. (2018) showed that microwave-induced cracks were disproportionately associated with biotite in Boulder granodiorite specimens; in contrast, cracks were disproportionately disassociated with biotite in a study of Pikes Peak granite specimens (Nicco et al. in review). This case study examines the thermal expansion of biotite in the Pikes Peak granite, in order to determine whether this process creates differential thermal expansion and controls cracking in granitic rocks. In order to investigate this question, a non-destructive 3D method is required that allows the observation of biotite grains before and after microwave treatment.

4.3. Material and Experimental Set Up

4.3.1. Material

The material chosen for this experiment is the Pikes Peak Biotite granite (PPB) from the Mount Rosa Complex in Teller and El Paso Counties, Colorado. The petrology of the Mount Rosa
Complex was described in detail by Gross and Heinrich (1965) and Persson (2017). The Pikes Peak Biotite granite is a white to pink, medium to coarse-grained biotite granite with grain size ranging from 1 to 5 mm (Figure 4.1).

It is characterized by roughly equal modal abundances of albite (27-44%), K-feldspar (20-25%) and quartz (36-44%), with approximately 4% biotite. Minor to trace Fe-Ti oxides, apatite, zircon, fluorite and monazite can also be observed. The texture of the feldspars is perthitic as seen in Figure 4.2, characterized by visible segregation of potassium- and sodium-rich feldspar. Large cubic blocks of Pikes Peak biotite granite (PPB) were collected from exposures that had no macroscopically visible pre-existing weaknesses. The blocks were drilled at the Colorado School of Mines laboratory to obtain cores 50 mm long by 50 mm diameter.

Figure 4.1 Photographs of the PPB specimen before (A) and after (B) microwave treatment. The rectangles indicate the area of the specimen captured in the microCT analyses.

4.3.2. Microwave Experiments

Microwave irradiation tests were conducted on the core specimens for 110 seconds at 2.45 GHz and 3.2 kW using a Panasonic NE-3240 microwave in multi-mode configuration. P-wave velocity testing was used as a preliminary method to assess the cracking. This method has been used in other studies on induced cracking (Wang and Simmons, 1978; Wang et al. 1989; Menéndez et al. 1999;
Figure 4.2 Cross polarized photomicrographs showing a representative area of PPB (A) and a detailed view of the perthitic texture (B). Ab: albite, Bt: Biotite, Fsp: feldspar, Qz: quartz, Or: orthoclase.

Darot and Reschlé, 2000; Sone and Condon, 2017; Nicco et al., 2018). The P-wave measurements in the present study were obtained before and after microwave irradiation, in order to identify new cracks. The surface temperature of the irradiated specimens was measured during treatment with an Ahlborn MR721420 infrared thermometer with adjustable focus and emissivity $\varepsilon$, according to the specific emissivity of the rock type calibrated with a standard thermocouple.

4.4. Crack Characterization Methods

4.4.1. Thermogravimetry Analysis (TGA)

In order to assess the amount of water present in the biotite mineral structure, Thermogravimetry Analysis (TGA) was conducted on grains of biotite that were handpicked from PPB specimens. TGA measures weight changes in a material subjected to temperature changes. The analyses were conducted at the Colorado School of Mines using a Setaram Setsys thermal analyzer. All experiments were run using synthetic air (20.9% $O_2$ with balance $N_2$) at a flow rate of 50 sccm. Heating was done at 20K/min from room temperature to 950°C, held for ten minutes, and then cooled at 40K/min.

4.4.2. X-ray Microcomputed Tomography (microCT)

X-ray computed tomography (CT) is a non-destructive technique that can provide three-dimensional information on the same rock specimen before and after a treatment. The technology relies on the acquisition of radiographs for different positions that are then computationally
reconstructed (Mees et al. 2003). This technique has been used to examine cracks induced by loading (Sellers et al. 2003; Jia et al. 2014; Weerakone et al. 2017), and by hydrofracturing (Renard et al. 2009, Zhang and Sheng 2017 and references therein). Numerous studies have measured apertures of induced or pre-existing rock cracks using CT (e.g. Johns et al. 1993, Keller 1998, Bertels et al. 2001, Van Geet and Swennen, 2001, Vandersteen et al. 2003, Charikinya et al. 2015, Weerakone et al. 2017). However microCT presents two main limitations. Firstly, the attenuation contrast between minerals in the specimen is governed by the atomic numbers of the mineral-forming elements. If the minerals do not have enough attenuation contrast between them they are hard to identify as individual phases. Secondly, there is a tradeoff between the specimen size (and its density) and the maximum resolution achievable. In Nicco et al. (2018), a proof of concept study was conducted on a small specimen (5mm³), and microwave-induced cracks were able to be detected. This study extends the field of view up to 50 mm core specimen.

For the present study, microCT analyses were conducted on specimens of PPB granite before and after irradiation. The analyses were conducted at the Colorado School of Mines using a ZEISS Xradia 520 Versa 3D X-ray microscope. The analyses were run at 140 kV and 10 W, Bin 1 objective 0.4 (wide view), filter HE3 (high energy) and an exposure time for acquisition of 12 seconds, 1601 projections with 360 rotation. After being scanned a first time the specimen was microwaved for 110 seconds then run again using the same settings in order to achieve the closest pixel resolution (24 microns). The two scans (before and after microwave irradiation) show the same volume of the core (bottom two thirds) as shown on Figure 4.2. As the induced damage is thought to behave symmetrically around the center of the core (Hartlieb et al. 2012) this approach was chosen to guarantee for a proper trade-off between the resolution of the scan (the larger the specimen, the lower the resolution) and the area of observation.

4.4.3. SEM Based Automated Mineralogy

After both microCT scans were completed, the core was impregnated with colored epoxy in order to visually identify and preserve the cracks. This step also prevents the specimen from disintegrating during cutting of thin section billets. The epoxy was applied passively at low temperature (55°C) rather than by vacuum impregnation, in order to prevent post-experimental dilation or extension of cracks. Three polished thin sections (48 by 27 mm and 30 microns thick) were
prepared perpendicular to the core axis of the core specimen. PPB9A, B and C were made 13 mm, 19 and 25 mm from the bottom of the specimen, respectively.

Automated mineralogy analysis was conducted at the Colorado School of Mines using a TESCAN-VEGA-3 Model LMU VP-SEM platform equipped with the control program TIMA3. Backscatter electron brightness and EDS spectra were collected at a beam stepping interval (i.e. spacing between acquisition points) of 15 µm, an accelerating voltage of 25 keV, and a beam intensity of 14. The results were output by the TIMA3 software as a spreadsheet giving the area percent of each composition and cracks, respectively. Compositional assignments were grouped appropriately. Mineral association tables were generated, which calculate the number of contacts between any two compositional phases assigned in the software. This allows for the quantification of minerals associated with an identified crack phase as described in Nicco et al. (2018). The maps can be used to quantify the mineralogy in the microCT results.

4.4.4. Image Processing Software for 3D Data Reconstruction

Data acquired by the microCT were reconstructed and visualized using the 3D visualization and analysis software Dragonfly (v4.1). This type of software allows for image processing and data manipulation in order to obtain quantitative data from the microCT scans. Two manipulations were realized for this study and are explained in the following sections.

4.4.4.1. Using SEM Generated Mineralogy Maps to Isolate Selected Minerals

In microCT data it is not possible to distinguish between minerals with similar attenuation contrasts, which are determined by the atomic numbers of the mineral-forming elements. Figure 4.3 shows that two types of phases can be observed based on attenuation contrast: bright phases that encompass atomically heavier minerals such as biotite, chlorite Fe-Ti oxides, apatite, zircon, fluorite and monazite; and darker phases including quartz, orthoclase, and albite. In this study, a protocol was developed to identify grain mineralogy in the microCT data using the SEM-based automated mineralogy maps. The mineral composition maps were imported into the Dragonfly software and aligned with the microCT scan data to identify a category of grains of the same mineralogy throughout the specimen.
Dragonfly users who want to apply this method will need to download the plugin `convertRGBtoROI` from the infinite toolbox (available at infinite toolbox>deep learning>convertRGBtoROI). The false colored automated mineralogy image (png extension) is imported as a file in the software with spacing parameters X and Y corresponding to the pixel size (beam step) of the automated mineralogy image. The Z component should be 1 pixel because it is a 2D image. However, the Z value can be exaggerated in order to be able to manipulate the data in the 3D window more easily. The file RGB output should be three different channels. Next the automated mineralogy image is transformed into a multi Region Of Interest (ROI) using the function which converts RGB channels to a multiROI tool. The newly created ROI is then aligned at the correct position in the scan. Once the false color automated mineralogy image and scan are aligned, the grains of interest can be selected in 3D using the paintbrush (3D) and added to a new ROI in the software (see Figure 4.4).
One complicating factor is that thin section preparation is destructive and therefore the SEM analysis can only be conducted on a single piece of rock one time: either before or after microwave treatment. The resulting automated mineralogy map can be overlain on the post-treatment microCT scan. However in the case the automated mineralogy generated map does not correlate to a slice of the microCT dataset (which is most likely to happen due to the making of the thin section) it can be difficult to align the whole map.

The above method was used to identify biotite grains in the microCT scans after microwaving. In order to evaluate the changes in biotite caused by the microwave irradiation treatment, the same grains were identified in the pre-treatment scan by screening both scans along the z-axis and using the paintbrush (3D) tool to select the grains that match the pre-treatment biotite grains that were identified in the previous step. This allowed the comparison between the volume occupied by the biotite before and after, providing data on the biotite expansion. Only the biotite grains that were visible on the automated mineralogy images were taken into account, as the rest of the bright phases in the scans could be accessory minerals.
4.4.4.2. Crack Segmentation Using Deep Learning

The cracks in the untreated specimen (before microwave irradiation) were too fine to be segmented using the general tools provided by the Dragonfly software. In order to be able to segment the cracks an algorithm was developed that uses neural network training. The first step consists of creating a Region of Interest (ROI) and segmenting the cracks on one slice of the dataset as shown in Figure 4.5. This was accomplished using the smart grid and the path tools; then another ROI was created to be used as a mask over the segmented portion of the slice, as shown in Figure after irradiation. The finest cracks segmented were a pixel wide (24.8 microns). This step was repeated on two additional slices representative of the crack morphology throughout the specimen. The second step is to train the neural network on the training data just created. This was done using the deep learning toolbox of Dragonfly (4.1). The neural network used is based on a U-net. U-net is a convolutional network architecture that allows for fast and precise segmentation of images. The architecture of a U-Net consists of a contracting path to capture context of the image followed by a symmetric expanding path that generates precise localization (Ronneberger et al 2015). The training was done using the parameters presented in Table 1. The last step consists of segmenting the dataset of interest using the neural network; this step is done in the image processing toolbox.

The same process was followed to train a second neural network to detect cracks in the scan of the specimen after irradiation. This was accomplished using the same parameters presented in Table 4.1.

4.5. Results

Microwaving resulted in cracks that were clearly visible in hand specimen as shown in Figure 4.1. The main cracking plane is located in the middle of the core. The creation of cracks throughout the specimen is confirmed by a definitive reduction of the p-wave velocity from 3408 m/s for the specimen before irradiation to 1725 m/s after irradiation, which shows a relative ratio of 50%. The maximum temperature obtained on the outside of the core was 360°C.
Figure 4.5 Screenshot showing how the cracks are segmented in the software using two different Regions Of Interest (ROI) to serve as training data and mask for the deep learning program.

Table 4.1 Parameters used for training the deep learning tool using a U-Net neural network to segment the cracks in a specimen of PPB

<table>
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<th>Parameter</th>
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</tr>
<tr>
<td>Batch size</td>
<td>64</td>
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</tbody>
</table>
4.5.1. Microscopic Observations

The thin section observations show that there are more cracks in the section closest to the center of the core (PPB9C as shown in (E) and (F) in Figure 4.6), compared to the thin sections from nearest to the top of the core (PPB9A as shown in (A) and (B) in Figure 4.6).

Table 4.2 shows the association between cracks and minerals, using the SEM based automated mineralogy data. A ratio of greater than 1 indicates that the cracks are disproportionately associated with a mineral phase, whereas a ratio less than 1 indicates that the cracks are disproportionately disassociated with that mineral phase.

The results are consistent with the previous findings for the Pikes Peak Biotite granite in Nicco et al. (in review); cracks tend to be associated with albite (average ratio 1.2) and disassociated with K-feldspar (0.8) and biotite (0.5). The low ratio observed in the present experiment confirms the disassociation between cracks and biotite. The cracks are slightly more associated with quartz towards the middle of the specimen (ratio 1.1 in PPB9C) where the highest temperature is inferred to occur.

<table>
<thead>
<tr>
<th>Mineral</th>
<th>PPB9A</th>
<th>PPB9B</th>
<th>PPB9C</th>
<th>Average</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>25.13</td>
<td>18.31</td>
<td>0.7</td>
<td>27.96</td>
<td>23.33</td>
</tr>
<tr>
<td>Albite</td>
<td>38.04</td>
<td>48.12</td>
<td>1.3</td>
<td>36.20</td>
<td>45.16</td>
</tr>
<tr>
<td>K-Fsp</td>
<td>32.54</td>
<td>27.82</td>
<td>0.9</td>
<td>32.21</td>
<td>26.81</td>
</tr>
<tr>
<td>Biotite</td>
<td>3.04</td>
<td>1.91</td>
<td>0.6</td>
<td>2.07</td>
<td>1.19</td>
</tr>
<tr>
<td>Other</td>
<td>1.25</td>
<td>3.84</td>
<td>1.56</td>
<td>3.31</td>
<td></td>
</tr>
</tbody>
</table>

4.5.2. TGA Results

The results of the TGA are shown in Figure 4.7. The specimens of biotite from PPB only showed a total of 0.63% weight reduction. The first weight loss event occurred between 0 and 200°C. This is inferred to correspond to the evaporation of ambient water and is about 0.2% of the total specimen weight. The second weight loss event occurred between 650 and 750°C and is about 0.43% of the total specimen weight. After the 800°C mark the specimen starts regaining weight back to the point it was at once the ambient water was evaporated (99.8%). Around 700°C the oxygen
Figure 4.6 Photomicrograph panorama of representative PPB specimen after microwave irradiation. The false color mineral map generated by electron microscopy and automated mineralogy of PPB9A, B and C are shown in (A) (C) and (E) respectively, and the crack phase is shown in isolation in (B) (D) and (F) respectively.
Figure 4.6 Continued
partial pressure presents a similar behavior with a drop and gain back to normal. During the TGA, the specimen of biotite changed color from black to golden copper.

The weight loss demonstrates similar behavior to that reported by Kombusheshe (2010). That author attributed to the liberation of water from the hydroxyls groups present in biotite following the equation $2\text{OH}^- = \text{H}_2\text{O} + \text{O}^2-$ (Grim 1968). However in the present results the weight is gained back around 950°C. If the weight was gained back this could be associated with the OH groups being reincorporated into a stable mineral structure.

Figure 4.7 Results of the TGA analysis.

4.5.3. MicroCT Results

4.5.3.1. Crack Segmentation Results

Without conducting any data processing on the scans, it is possible to observe that the post-irradiation scan shows a significant amount of cracking relative to the pre-irradiation scan. The cracks can be visually isolated by changing the contrast in the scan. This is a visual confirmation that cracks
were created during the microwaving treatment. In order to quantify the cracks that were created during the treatment, the cracks were segmented using the neural network approach presented in section 3.4.2. The deep learning method was used to segment the cracks from the scans before and after as shown on Figure 4.7. This step showed that there were already cracks present along planes in the specimen, as shown in the first scan (pre-treatment; Figure 4.8).

![Figure 4.8 Result of the crack segmentation using the U-Net network on the before (left) and after (right) microwave treatment of the PPB specimen. Note that for ease of the 3D view the rest of the dataset was made more transparent than the segmented cracks.](image)

The processed scans facilitate observation of entire cracks, due to the relatively large volume of scanned rock. This type of display of the results provides information on the crack location and morphology, allowing for interpretation on cracking mechanisms. The data show that pre-treatment cracks tend to be planar, cutting directly through the core. Observation of the post-treatment cracks in their entirety inside the core shows that the cracks are concentrated at the center of the core. In contrast to the pre-treatment crack morphology, the post-treatment cracks appear to step around features in the core. These features are interpreted to be grain boundaries that cannot be directly observed in the scan data. Finally Figure 4.9 shows the two segmented ROIs representing the cracks before (green) and after (pink) microwaving overlaid on one another.
Figure 4.9 Cracks segmented using the U-Net network on the before (left) and after (right) microwave treatment. The two datasets of segmented cracks were overlaid to show crack evolution. Note that the datasets were made transparent to allow for better 3D view.

Figure 4.10 Biotite grains identified as separate phase on the datasets before (left) and after (right) microwave irradiation. Note that the matrix composed of quartz and feldspar was made transparent to allow for better 3D view.
4.5.3.2. Biotite Segmentation Results

Figure 4.10 shows the result of the biotite segmentation using the method described in Section 3.4.1. The results show an expansion in volume of approximately 1% of the total voxel occupied by the segmented biotite grains.

4.6. Discussion

4.6.1. Crack Segmentation Discussion

The main advantage of microCT analysis is its non-destructive capability to provide data on the morphology of the cracks and how they are connected in 3 dimensions. The thin section petrography on PPB9C shows a large planar crack, but only in 2D. In Figure 4.9, it appears that the main planar (horizontal) crack seems to be building on a preexisting crack as seen in the pre-treatment scan. This suggests that the microwave-induced crack exploited a pre-existing weakness in the rock. However the other pre-existing cracks were not significantly expanded or extended according to observations of Figure 4.8. As shown on Figure 4.2 the main horizontal crack is located in the middle of the 50 mm long core specimen; this is where the most cracking should be expected according to results from Hartlieb et al. (2012).

It is significant that most of the pre-existing cracks in the specimen were not impacted by the microwaving treatment. Only one pre-existing crack which was perpendicular to the core axis slightly expanded during irradiation. This means that pre-existing weaknesses do not entirely govern the way the rock will crack during microwave irradiation. This interpretation comes in contrast with a recent study by Huang et al. (2019) that showed that pre-existing cracks in core specimens of coal expanded during microwave irradiation, whereas pre-existing cracks perpendicular to the main core axis did not change or shrank. In that study, the pre-existing cracks along the core axis were macroscopically and were wider than a millimeter. In the samples of PPB in the present study, the cracks were only visible by using the microCT analysis and they were smaller than a millimeter. For the samples of PPB, it appears that the shape of the specimen (core) was a more important driver of the cracking behavior than the presence of pre-existing cracks. Indeed it has been shown that for core specimens the hottest point will be the center, and the main damage plane will be perpendicular to the axis of the core (Hartlieb 2012). This suggests that if the rock is fairly intact with minor pre-existing weaknesses (cracks with apertures <1 mm) it is the shape of the sample that will mostly govern the cracking,
whereas if the specimen shows significant pre-existing cracks (apertures >1 mm) they will expand, controlling the cracking pattern in the specimen. From the perspective of an industrial application of microwave-induced cracking, this would mean that the condition of the material to be microwaved does not entirely control the way it cracks under microwave irradiation. To test the microwaving treatment prior to application, the samples should be representative of the actual shapes of the material for the most accurate prediction of the material’s behavior.

The microwave-induced cracks also present a jagged morphology compared to the planar pre-existing cracks. This supports the observation made in Nicco et al. (in review) where pre-existing cracks could easily be segregated from the microwave induced cracks due to their morphology and mineral association.

4.6.2. Biotite Discussion

Lu et al. (2017) showed that grains of biotite submitted to microwave irradiation can expand up to 2.5 times their size along the axis perpendicular to the cleavage plane. Other studies (Hidnert and Dickson 1945 and Cartz and Tooper 1964) show that different types of biotite show substantial expansion perpendicular to the cleavage plane. One of the goals of using the microCT technology in this study was to assess whether the biotite grains of biotite in the specimen expand due to microwave irradiation. The grains of biotite were segmented in the 3D scan using the automated mineralogy maps. The results show that biotite grains expanded to about 101% of their initial volume after microwave irradiation. Given the subjectivity of the method used, a 1% expansion is below the margin of error. It has been mentioned in the literature (Hidnert and Dickson 1945) that biotite expansion is a reversible transformation and biotite will tend to take its original volume back during cooling. Given the error, it cannot be proven that the biotite in the PPB specimens expanded during the microwaving, but expansion (or expansion and contraction) could have occurred.

Since microwaves heat a material from the inside, it can be assumed that the outside temperature value does not represent the highest temperature in the interior. The interior temperature is significantly higher as demonstrated by Hartlieb et al. (2012). That study demonstrated that the outside of a core specimen of basalt is 200°C cooler than the center of the core. Therefore it is possible to postulate that while the outside temperature recorded on the Pikes Peak specimen is
360°C, the center of the specimen is much hotter. This means that the biotite at the center of the specimen may have reached temperature closer to the biotite dehydration temperature.

The results from the biotite segmentation in the before and after 3D scans and the results from the DTA analysis of biotite specimens from Peaks Peak indicate that dehydration was not achieved. A total loss of only 0.2% of the specimen weight was observed; corresponding to the loss of ambient water and not the water contained in the structure of the biotite. However, the data show a drop then gain of 0.43% in specimen weight between 500 and 900°C and a similar behavior in the oxygen partial pressure. This could mean that the hydroxyl groups are being separated from the mineral structure. If one assumes that the biotite present in the specimen is constituted of 3.64% weight of bound -OH groups based on ideal biotite composition, the observed specimen weight loss of 0.43% between 500 and 900°C means that the biotite lost 11% of the water contained in the mineral structure. The regain may be explained by a return to a more stable state of the structure.

4.6.3. Methods Discussion

This study employs state of the art technology and imaging processing. The results show that these techniques can be used to study mineralogical behavior during a rock cracking treatment. This approach could greatly improve the understanding of crack inducing mechanisms such as microwaving. However, there are still some issues to be resolved in the data manipulations. First, in order to align the SEM images properly on the scan, the thin section needs to correspond to a perfect slice of the microCT, and it is difficult to achieve the required level of precision when cutting a thin section. Secondly, biotite could be successfully identified and segmented biotite using the mineralogy maps because biotite has a highly different X-ray absorption behavior compared to the other minerals present in the specimen. For example, it would be more challenging to differentiate quartz or feldspar. However, the use of neural networks and deep learning opens new possibilities for mineral identification. It is possible that the SEM-generated mineralogy maps could be used as the training data for a deep learning approach which would segment quartz from feldspars. Lastly, in order to refine results, other users attempting to segment cracks in natural materials may want to go through the step by step explanation presented in section 3.4 to create a more customized segmentation tool.
4.7. Conclusions And Recommendations

This study successfully used microCT analysis on a 50 mm long by 50 mm diameter core of granite with a resolution of 25 microns to observe microwave-induced cracks. The methods presented in this study can be applied to a wide array of studies in different engineering fields. For example:

1) Fundamental investigation of cracking mechanisms, whether induced by microwaves or another damage method (e.g., heating, cooling, tension, stress, thermal shock). The results of this study show that microCT can provide qualitative and quantitative data on crack location and morphology. This method would be particularly interesting to investigate the influence of specimen geometry in relation to microwave-induced cracking.

2) The deep learning method based on the U-Net neural network detailed in this study can be applied to segment cracks in other rock types or materials. By following the steps explained in section 3.4.2 it is possible to create training data for the deep learning that is specific to the study material. If handling multiple datasets, it is possible the user will have to create a set of training data for each dataset as it was the case during this study.

3) The approach presented in this study would be useful for investigations of geological processes requiring larger specimen sizes, since up until now microCT analyses were mostly done on small specimens.

Regarding the mineralogical controls on microwave-induced cracking, this study showed that the biotite present in PPB specimens tends to be disassociated with the cracks. It was not possible to determine whether the biotite grains expanded (or expanded and then contracted), as the observed 1% expansion post-treatment was within error of the method used. The TGA analysis suggests that the PPB biotite does not have interlayered water, and that the only loss during microwaving is due to dehydration of the -OH hydroxyl groups. Therefore the loss is minimal and only occurs at high temperatures around 700°C, which may not have been attained during microwave of the specimen. Therefore the observed cracking of the specimen could not be clearly linked to the thermal expansion or water loss of biotite.

The present study showed that pre-existing cracks can easily be distinguished from microwave-induced cracks. The former will tend to be straight and cut through grains, whereas the latter will tend to be sinuous and occur along grain boundaries. These findings reinforce the two-
dimensional observations made in Nicco et al. (under review). Through observation of the microCT dataset before and after microwaving treatment, the present study also indicates that the shape of the specimen (core) seems to govern microwave behavior more than the presence of fine (<1 mm aperture) pre-existing cracks in the specimen.

4.8. Acknowledgments
This work was supported by the National Science Foundation (CMMI award 1550307). The authors thank Jae Erickson for specimen preparation, Dr Katharina Pfaff for SEM analysis, Dr Michael Sanders for TGA analysis; as well as the ORS Dragonfly technical team for their help.

4.9. References


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5.1. Summary of Results

Table 5.1 shows the main results from chapters 2, 3 and 4. The results are classified based on the type of information: material selection, method development, and mineralogical or textural controls on fracturing. Subsequent sections integrate this information to generate holistic conclusions addressing each of the scientific questions raised in this dissertation.

Table 5.1 Main results from this study

<table>
<thead>
<tr>
<th>Chapter</th>
<th>Conclusion</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Not all rock types are suited for a microwave study</td>
<td>Material selection</td>
</tr>
<tr>
<td>2</td>
<td>An integrated suite of destructive and non-destructive methods is required in order to characterize cracks</td>
<td>Methods</td>
</tr>
<tr>
<td>2</td>
<td>In Boulder granodiorite, biotite is preferentially associated with cracks</td>
<td>Mineralogical controls</td>
</tr>
<tr>
<td>3</td>
<td>In Pikes Peak Biotite granite, biotite is preferentially disassociated with the cracks</td>
<td>Mineralogical controls</td>
</tr>
<tr>
<td>3</td>
<td>Amphibole is disproportionately associated with cracks in both Mount Rosa rock types where they are present</td>
<td>Mineralogical controls</td>
</tr>
<tr>
<td>3</td>
<td>Amongst feldspar albite is disproportionately associated with cracks whereas orthoclase is disassociated with cracks</td>
<td>Mineralogical controls</td>
</tr>
<tr>
<td>3</td>
<td>Quartz is disproportionately disassociated with cracks for all rock types tested in the study</td>
<td>Mineralogical controls</td>
</tr>
<tr>
<td>3</td>
<td>Color, water, and iron content of constituent minerals cannot be used to predict the microwave behavior of a rock</td>
<td>Mineralogical controls</td>
</tr>
<tr>
<td>3</td>
<td>Presence of adjacent grains with contrasting properties appears to be critical</td>
<td>Textural controls</td>
</tr>
<tr>
<td>3</td>
<td>Coarse grained rocks develop complex networks of narrow cracks during microwave irradiation whereas fine grained rocks will develop few wider cracks throughout the samples</td>
<td>Textural controls</td>
</tr>
<tr>
<td>3</td>
<td>Straight and predominantly intra granular cracks are most likely pre-existing whereas microwave induced cracks are inter granular mostly</td>
<td>Textural controls</td>
</tr>
<tr>
<td>4</td>
<td>MicroCT of a larger core of granite (50 mm by 50 mm diameter) opens new possibility for study of geological processes</td>
<td>Methods</td>
</tr>
<tr>
<td>4</td>
<td>Use of U-Net neural network to segment cracks in a microCT scan</td>
<td>Methods</td>
</tr>
<tr>
<td>4</td>
<td>Segmentation of isolated minerals on scans before and after microwaving treatment allows for study of thermal expansion of grains in a mineral assemblage</td>
<td>Methods</td>
</tr>
<tr>
<td>4</td>
<td>Biotite grains do not seem to have expanded during the microwaving treatment</td>
<td>Mineralogical controls</td>
</tr>
<tr>
<td>4</td>
<td>Biotite is disassociated with cracks in PPB</td>
<td>Mineralogical controls</td>
</tr>
</tbody>
</table>
Table 5.1 Continued

| 4  | Preexisting cracks tend to be straight whereas microwave induced cracks are sinuous around grain boundaries | Textural controls |
| 4  | Main damage is a plane at the middle of the core perpendicular to the axis of the core | Textural controls |
| 4  | Preexisting cracks do not seem to be significant drivers of microwave induced damage | Textural controls |

5.1.1. Integrated Conclusions on Material Selection for Microwave Studies

Throughout the work conducted for this dissertation, a number of potential rock types were considered but were not pursued. The following is a non-exhaustive list of key characteristics that should be taken into account when selecting the material for a study on microwave-induced rock damage.

The first aspect to take into account is the general geology, location, and accessibility of the study area. The material needs to be present in large enough quantities to allow for repetitive testing. This requires an easily accessible location that permits the retrieval of large numbers of large blocks of rock.

The second aspect is the homogeneity of the material. The sample area needs to be homogeneous from a textural and mineralogical point of view if the goal of the study is a fundamental study on the mechanisms of microwave induced cracking. If the goal of the study is to assess the potential of microwave induced cracking as a strength weakening method, samples should be taken that are the most representative of the material to be treated. Alteration and weathering are typically distributed heterogeneously; ideally the samples should be fresh and not too weathered if mineralogical controls are to be investigated. In addition, the material should be strong enough to be cored.

The last aspect to take into account is the mineralogical content of the material. In order to create a high enough thermal gradient between the minerals and generate cracks, the material needs to be polynmineralic. The mineralogy should be homogeneous enough to be captured in a representative way for making thin sections. The grain size needs to be fine enough to allow for representative thin section study, not exceeding 10 mm if standard thin sections are to be used. However, this is an analytical constraint, and coarser grain sizes may crack better under irradiation.
Lastly, the choice of material will highly depend on the goal of the study. Table 5.2 shows the ideal characteristics of the material depending on the type of study.

Table 5.2 Material characteristics depending on the type of microwave related study.

<table>
<thead>
<tr>
<th>Material characteristic</th>
<th>Type of study</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fundamental study on microwave induced cracking mechanisms</td>
</tr>
<tr>
<td>Location</td>
<td>Accessibility</td>
</tr>
<tr>
<td></td>
<td>Large quantities</td>
</tr>
<tr>
<td>Textural aspect</td>
<td>Homogeneous</td>
</tr>
<tr>
<td></td>
<td>Fresh</td>
</tr>
<tr>
<td>Mineralogy</td>
<td>Grain size</td>
</tr>
<tr>
<td></td>
<td>Hardness</td>
</tr>
<tr>
<td></td>
<td>Polymineralic</td>
</tr>
<tr>
<td></td>
<td>Homogeneous</td>
</tr>
</tbody>
</table>

X indicates important

5.1.2. Integrated Conclusions on Method Development

The integrated suite of methods developed in Chapter 2 provides a dataset on microwave-induced changes in rock strength, fracture mineralogy and morphology; these data can be used to investigate the mechanisms causing the fracturing, as well as how the fracturing treatment could be optimized. In Chapter 4, new microCT methods were presented that enable comparison of individual cracks and mineral grains before and after microwave irradiation, as well as machine learning to identify small aperture cracks. These techniques could be integrated into the suite of methods presented in Chapter 2 for a richer dataset. A table summarizing all methods used for this work is presented in table 5.3. The main conclusion regarding all the methods and methods combinations used in this study is that they are highly versatile and can be used in different combinations depending on the goal of the study and the type of data needed.

Table 5.3 Summary table of the methods used in this dissertation showing their strength and weaknesses.

<table>
<thead>
<tr>
<th>Rock properties</th>
<th>Microfracture characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Destructive</td>
<td></td>
</tr>
<tr>
<td>UCS</td>
<td><em>Thin section optical light microscopy</em></td>
</tr>
<tr>
<td>Advantages: data show strength changes in rock. Cheap.</td>
<td>Advantages: provides data on fracture location, morphology and mineralogy. Can be quantitative if point counting is conducted. Cheap.</td>
</tr>
<tr>
<td>Non-destructive</td>
<td>P-wave velocity</td>
</tr>
<tr>
<td>----------------</td>
<td>----------------</td>
</tr>
<tr>
<td><strong>Advantages:</strong> data show rock structure. Cheap and fast.</td>
<td><strong>Advantages:</strong> 3D. Provides location and morphology of fractures.</td>
</tr>
<tr>
<td><strong>Disadvantages:</strong> Only qualitative. Can be made quantitative if using large number of specimens</td>
<td><strong>Disadvantages:</strong> Tradeoff between specimen size and resolution. Visual and qualitative only. Mineralogy data may not be possible. Expensive and time consuming.</td>
</tr>
</tbody>
</table>

The following paragraphs highlight some of the drawbacks and uncertainties that arose when using the methods described in this dissertation and that should be taken into account when selecting the approach.

### 5.1.2.1. Electron Microscopy and Automated Mineralogy

The methods used in this study are highly reliant on electron microscopy and automated mineralogy data acquisition. These methods provide quantitative data on crack location, morphology, mineralogical association and liberation. The main drawback is that the methods are somewhat time consuming.
consuming because the samples need to be prepped, and the analytical time is relatively long at the small beam step sizes required to characterize narrow aperture cracks. In addition, data analysis of the results can take time. The crack location identification is limited by the fact that electron microscopy is a chemical analysis and therefore cannot differentiate individual mineral grains of the same composition.

Mineral association statistics are generated during the automated mineralogy analysis. However, these statistics may over-represent or over-simplify the relationships between cracks and mineral phases. For example if a crack bifurcates along the cleavage planes in a biotite grain, the mineral association statistics will include each pixel touching the sub-cracks because the automated mineralogy analysis does not recognize that these sub-cracks are related. The same issue applies to point counting, where each crack encountering a line is counted as a separate unit. However, when looking at the big picture it is observable that some cracks seem to “extend” following cleavage patterns or path of least resistance in certain minerals but form only a single large crack outside of these minerals. This is observable in the sample shown in Figure 5.1, where a single large crack (labeled 1 on Figure) bifurcates within biotite grains (labeled 2 on Figure) but is only one crack elsewhere. How this issue should be resolved depends on the goal of the study. For studies focused on the application of microwave technology, it may be important to count the sub-cracks separately because they show the actual outcomes of the cracking treatment. From a mechanistic point of view, the sub-cracks formed via the same process and should be considered as a single entity. For this study they were counted individually.

A second and related point is that the mineral association statistics do not allow for segregation of certain features such as pre-existing cracks. This may result in non-representative results that can only be corrected by manual point counting. For example, the ratios presented for specimen MRA4F (Chapter 3) account for both pre-existing and microwave-induced cracks in the statistics generated by the automated mineralogy analysis. Therefore it may be beneficial to use automated mineralogy and manual point counting in concert to obtain the most accurate description of the crack mineralogy.
5.1.2.2. Crack Classification Using Visualization Software Jmicrovision (v1.27)

The results of the present investigation demonstrate that optical light microscopy and automated mineralogy should both be applied to obtain complete mineralogical information on the cracks. The integration of these techniques enabled the quantification of the pore space generated by cracking, liberation of the mineral phases, qualitative assessment of crack morphology, and quantitative assessment of mineral-crack associations. The method is explained in detail in Chapter 2. Optical microscopy could be used to determine mineralogy, whereas electron microscopy showed compositional boundaries only. The pairing of these techniques using image-processing software allows for semi-quantitative classification of crack boundaries by point-counting, and reliable data are provided by point-counting of paired cross-polarized optical and false color electron microscopy images (using the assignment of pore space to a unique species definition via automated mineralogy). Quantification of the induced pore space and statistical determination of crack boundary mineralogy via liberation analysis requires the application of automated mineralogy to the electron microscopy data. Crack morphologies and apparent apertures can be assessed using either optical or electron microscopy. As shown in Chapter 3, this method also seems to be the best way to recognize the presence of pre-existing weaknesses if the study does not use microCT.
This approach of crack classification has the advantage that it can work for any type of study on cracks; it proves to be the most complete way to describe the crack morphology, and quantitative assessment of mineral-crack associations. However there are several drawbacks to the method.

First, the method requires a lot of data from different microscopy techniques and equipment, thin sections microphotographs must be obtained in plain polarized light and cross polarized light. This step is time consuming unless the microscope is equipped with a moving stage that allows for automated panorama acquisition. Even if the cross polarized microphotographs typically suffice for the research purpose, it is still important to record the plain polarized microphotographs because it is not possible after the thin section is carbon coated for SEM analysis. A mineral map generated by an SEM equipped with automated mineralogy helps with identification of minerals and is necessary to generate the map of the cracks. For this step it is important to set the SEM acquisition brightness detection to zero, otherwise the cracks will not show as their own separate phase and no quantitative analysis will be possible. Once all microphotographs and mineral maps are obtained, the next step is to align them and prepare them for the image processing software in terms of size, alignments, overlaying etc.

The second important consideration is the choice of the grid spacing and orientation. For this study, the grid was created parallel to the short axis of the thin section, and the spacing was constant throughout all thin sections (100 pixels). In a comparative study, a wider and a smaller spacing did not show significant variation in results. However the influence of orientation of the thin section was not tested. It is important to keep the method as constant as possible throughout thin section analysis to obtain the most comparable results.

The last drawback of the method is that it is time consuming. The data acquisition using the SEM can take 5 hours for a typical study using a beam stepping interval of 15 microns. The point counting preparation and alignment takes another 2 to 3 hours. In addition, point counting in itself is a long task. The method also requires having knowledge of basic mineralogy to be able to recognize grains, minerals, and make decisions of classification when the automated mineralogy data and optical microscopy images do not provide enough information.

5.1.2.3. MicroCT and Image Processing

In this study, the use of microCT and image processing software allowed for new perspectives and better observations of crack patterns in 3D. In this dissertation, two separate microCT
experiments were conducted. The first was a proof of concept study (presented in Chapter 2), demonstrating that cracks could be observed before and after microwaving on a small sample of 5 mm by 5 mm by 5 mm. In Chapter 4, a more detailed study was presented, comprising a scan of a core sample 50 mm diameter by 50 mm long, in which a resolution of 25 microns was achieved. The success of the latter study opens up many possibilities for the use of microCT methods in studies on crack generation and propagation for various purposes including relatively large scale samples.

The microCT analyses presented in Chapter 4 also were used to examine the potential expansion of biotite grains due to microwave irradiation. This grain size comparison is novel and opens new possibilities in understanding how microwave treatment affects the minerals in a rock. Similar research questions have only been previously addressed using mineral separates.

The approach presented in Chapter 4 also involved machine learning using the microCT data. This approach also allowed for successful identification of the cracks in the entire dataset by only manually segmenting a small number of cracks; these were used as training data for the software to identify more cracks.

Despite all the capabilities of microCT, there are some drawbacks to be aware of when choosing to use this method. Important observations can be made directly from the unprocessed data generated by the analysis. However, further manipulation of the dataset is time consuming in order to get any quantitative information using image processing software such as Dragonfly. It is a tedious task to learn how to use the software to its full potential. In addition, microCT analysis is relatively expensive compared to the other methods employed in this study; therefore microCT should only be included in a study if the results will add new information.

### 5.1.2.4. Rock Property Testing Methods

The two methods for rock properties testing used in this dissertation were chosen for their speed and low cost. However both methods have some limitations to keep in mind when building a methods flow chart for a study on cracks.

The UCS tests done in this study, but also other standard tests such as the Brazilian disc test for tensile strength test require a core sample prepared to specific dimensions. While this is not a problem for fundamental study of cracking mechanisms, it may create issues for studies applying microwaving at a bigger scale. For example, in a mining environment the run of mine material will
most likely be composed of rock pieces of different shapes and sizes, and some may not be of
dimensions suitable for preparation of a UCS sample. In Chapter 2 the number of samples needed for
a statistical study was presented. According to ASTM standard D7012 for UCS testing, the number of
specimens should be determined using standard E122, “Practice for Calculating Sample Size to
Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process” (ASTM
2017b). The sample size \( n \) (number of specimens to be tested at each condition) is determined
according to the equation \( n = \left( \frac{3 \sigma_0}{E} \right)^2 \), where \( E \) is the maximum acceptable difference between the
sample average (the average test result for \( n \) specimens comprising the statistical sample) and the
true average, and \( \sigma_0 \) is an advance estimate of the lot or process standard deviation. While this study
mostly focused on proof of concepts for using new methods to study cracks, the number of specimens
needed for a representative statistical study should be calculated using the ASTM standards in case
of an industrial application.

So far, the studies at a pilot scale have been focused on separating the ore from the barren
host rock (e.g., Batchelor et al. 2016). In that study, the porphyry ore was already crushed and milled
prior to microwaving. The resulting material was grains less than 50 mm wide. However, in order to
use microwave as a pre-treatment before comminution to weaken the rock and therefore reduce the
amount of energy needed for crushing and grinding, the samples would be much larger than 50 mm
and vary in size and shape. Hartlieb et al (2012) proved that a core sample will tend to break in a
plane perpendicular to the axis of the core and at about the middle of the sample during microwaving,
which is the behavior that was observed in the present work. However no work has shown in detail
how the shape of the material will influence the breakage. Therefore it may not be representative to
use UCS testing on cores if the material will not have this shape in the actual application of the
microwaving.

Relative P-wave is a qualitative method and is used as way of quickly determining if cracks
were generated on a sample per sample basis. Therefore it can be used in many situations. However
only using relative P-wave measurements will not give any information on the amount of cracks
generated, or the strength reduction generated by the cracking method, whether it is microwaving or
another crack inducing method.
5.1.3. Integrated Conclusions on the Mechanisms of Microwave-Induced Cracking

5.1.3.1. Textural Controls

There are distinct differences in the cracking patterns between the different rocks presented in this dissertation. Fine grained specimen such as Mount Rosa (MR) (0.5 to 2 mm) have very few cracks which are relatively large. In contrast, coarse grained specimens such as Pikes Peak Biotite (PPB) and Boulder granodiorite (1 to 5 mm) display a complex network of fine cracks.

The relationship between grain size and cracking patterns is likely explained by the influence of grain size on the heating mechanisms of the specimens during microwave irradiation. In a fine-grained specimen such as MR, the grains are intercalated, and each small grain of a good microwave absorber likely lies in close spatial proximity to other good absorbers, leading to better heat redistribution and potential thermal runaway (Chapter 3). The center of the irradiated samples reach temperatures much higher than the rims, in some cases resulting in melting as well as the formation of small numbers of large-aperture through going cracks. In coarse-grained rocks such as PPB, inefficient heat redistribution between more spatially dispersed microwave absorbers may cause high thermal gradients to develop between grains, leading to heterogeneous stresses which generate complex networks of intergranular cracks throughout the specimen.

The morphology of crack patterns also appears to be correlated with the cracking mechanisms. The interpretation of observations from the point counting study (Chapter 3) and the visual observations on the microCT dataset (Chapter 4), is that the relatively straight and predominantly intragranular cracks are most likely pre-existing, related to loading or unloading. In contrast, microwave-induced cracks display irregular morphology and are dominantly intergranular rather than intragranular. This can be observed in specimen MRA4F (Chapter 3): the large, through-going, pre-existing crack is relatively straight and links numerous intragranular cracks in quartz, whereas the microwave-induced crack has an irregular shape because it links grain boundaries. Comparisons of crack morphology could be used to determine which cracks were present prior to the application of a rock fracturing treatment when the microCT analysis was not available (or not carried out for budgetary reasons).

It can also be observed for PPB (Chapter 4) that the pre-existing cracks do not seem to be impacted by the microwaving treatment, indicating that pre-existing weaknesses in the specimens are not necessarily affected by the microwaving treatment.
The microwave-induced cracks also present a jagged morphology compared to the planar pre-existing cracks. This supports the previous point that pre-existing cracks could easily be segregated from the microwave induced cracks due to their morphology and mineral association. From an application point of view, this would mean that the condition of the material to be microwaved does not entirely control the way it cracks under microwave irradiation.

5.1.3.2. Mineralogical Controls

The specimens selected in the granite suite in Chapter 3 allow for comparisons between two general categories of minerals: amphibole and biotite are both Fe- and water-bearing, dark colored minerals. Quartz and feldspars are light colored and do not contain water or iron. However, significant differences were observed in microwave behavior within each group: the feldspars and amphibole crack preferentially compared to the quartz and biotite.

No single chemical, physical or microwaving related characteristic presented in Chapter 3 explains the cracking behavior of all minerals in the specimens. For example, minerals identified in literature as good microwave absorbers (e.g., biotite and amphibole) are not consistently cracked: the biotite is consistently associated with cracks in specimens of Boulder granodiorite (Chapter 2) but also consistently disassociated from cracks in samples of PPB (Chapters 3 and 4). Plagioclase feldspars are weak microwave absorbers with low dielectric constants (Zheng 2017 and Batchelor 2016) but were preferentially cracked during microwaving. Therefore, the microwave response must be explained by a combination of thermal and physical properties of a mineral, as well as the properties of the other minerals present in the specimen. The following interpretation focuses on the mineralogical properties, dielectric constant, dielectric loss, thermal expansion, and thermal conductivity of each mineral to explain the observed microwave responses of the specimens. The behavior of hydrated minerals amphibole and biotite will be treated more in depth in Section 5.1.3.3.

5.1.3.2.1. Feldspar

In Boulder granodiorite specimens (Chapter 2), both feldspar types (albite and orthoclase) appear to be very slightly disassociated with the cracks according to the mineralogy table generated using automated mineralogy. In Mount Rosa Amphibole (MRA) specimens, the statistics show that both feldspar types are disassociated with the cracks. However, the latter results include both the preexisting cracks and the microwave induced cracks and may not be representative of microwave-
induced changes. In the PPB and MR specimens, albite is disproportionately associated with cracks as shown in Chapter 2 and 4, whereas orthoclase is disproportionately disassociated with cracks, relative to the modal abundances of these minerals. This is the case in PPB specimens with perthitic texture (exsolution lamellae of albite and orthoclase), as well as in MR where the feldspars occur separately. Both types of feldspar are affected by intragranular and intergranular cracks. Relative to the other minerals in the specimens, feldspars have moderate thermal conductivity and low linear thermal expansion coefficients. They are brittle and also exhibit one perfect cleavage direction, which means the cracking mostly happens along cleavage planes when the grains expand. The differential behavior between the two feldspar types observed in Chapter 3 may be explained by the dielectric constant and volumetric thermal expansion coefficient, which are higher for albite than orthoclase.

5.1.3.2.2. Quartz

Quartz is disproportionately disassociated with cracks relative to its modal abundance in PPB, MR and MRA specimens (Chapter 3) and is neutral (ratio 1.1) in Boulder granodiorite specimens (Chapter 2). For the PPB specimen presented in Chapter 4, the study of the three thin sections from different locations in the same core showed a slightly higher ratio when moving towards the middle of the specimen where the highest temperature is inferred to occur. This could be due to thermal expansion of quartz where temperatures are higher (>400°C) and cause stress between neighboring minerals. For all rock types, quartz grains are rarely affected by intragranular cracks or intergranular cracks between neighboring quartz grains, but the grain boundaries between quartz and other minerals are commonly cracked. Quartz is relatively unresponsive to microwave irradiation (low dielectric constant and low dielectric loss) but has a high volumetric thermal expansion coefficient and thermal conductivity, which leads to high differential expansion between quartz and neighboring grains, and cracking at the grain boundaries when the temperature of the specimen rises.

5.1.3.2.3. Amphibole

As shown in Chapter 3, amphibole is disproportionately associated with cracks in MR and MRA relative to its modal abundance (note that amphibole is not present in PPB specimens or in the Boulder granodiorite). The intragranular cracks are likely explained by the high dielectric constant, the brittle tenacity and perfect cleavage of amphibole grains. The intergranular cracks are explained by
the high dielectric loss of amphibole that allows for differential thermal expansion forces with neighboring grains.

5.1.3.2.4. Biotite

Biotite is disproportionately disassociated with cracks relative to its modal abundance in PPB specimens (Chapters 3 and 4), exhibiting few intra- or intergranular cracks. Even though biotite absorbs microwave energy relatively well (high dielectric constant and loss), its elastic properties and high linear expansion suggest that biotite transfers heat stress to neighboring minerals when they are homogeneously distributed within the sample. In contrast, in rock types containing numerous adjacent biotite grains such as the Boulder granodiorite (Chapter 2), the cracks propagate in biotite zones by linking cleavage planes, or by extending along the boundaries of biotite zones. This is likely due to differential expansion of the clusters of biotite grains relative to the other minerals, therefore inducing a higher ratio that shows crack being disproportionately associated with biotite. This textural control of biotite is detailed in the following section.

5.1.3.3. Contribution of Hydrated Minerals

The three papers presented in this dissertation have addressed different aspects of the contribution of hydrated minerals (biotite and amphibole) to rock fracturing during microwave irradiation. The results show that microwave behavior of biotite and amphibole is not uniquely governed by their dark color (theorized by Ford and Pei (1967) and Harrison (1997)), higher iron content (theorized by Lu et al (2017)) or higher water content (theorized by Harrison (1997), Robinson (2014) and Nicco et al. (2018)). Those criteria may be used as indicators of potential good microwaves absorbers; however they cannot be used to anticipate the behavior of biotite and amphibole under microwave irradiation.

Chapter 2 examined specimens of Boulder granodiorite and documented that biotite was disproportionately associated with cracks. This observation led to the hypothesis that the water contained in the minerals contributed to the specimen’s behavior during microwave irradiation. The second set of experiments presented in Chapter 3 was designed to test that hypothesis. The experiments utilized a suite of rocks from the Mount Rosa complex containing different proportions of the same hydrated mineral observed in the Boulder specimens (biotite) in a matrix of the same minerals (quartz and feldspar) but with different textures. Observations from Chapters 2 and 3 show
that there are key differences between the microwave behavior of biotite in PPB and the biotite in Boulder granodiorite specimens. In the Boulder granodiorite, the cracks were preferentially associated with biotite, whereas in PPB the cracks were preferentially disassociated with biotite. This difference may be explained by contrasting biotite textures in the two types of specimens. In the Boulder granodiorite, the biotite is represented by clusters of small biotite minerals: with 200 to 600 micron grain size, and clusters of 2 to 3 millimeters, whereas biotite in the PPB samples occurs as larger, isolated grains of 1.2 to 2.3 millimeters in a matrix of quartz and feldspar. The intragranular biotite and intergranular biotite-biotite cracks in the Boulder granodiorite appear to link cleavage planes among adjacent biotite grains. When one biotite grain undergoes linear thermal expansion perpendicular to the cleavage planes, it puts stress on the surrounding biotite grains, causing the crack to propagate along the cleavage planes of the biotite grains in the immediate vicinity. The cracks that are associated with biotite in the PPB specimens occur between biotite and other grains, where biotite grains are not adjacent to one another. Therefore, thermal expansion of biotite is more likely to create stress at the grain boundaries with other minerals, causing intergranular biotite-quartz and biotite-feldspar cracks. This hypothesis that biotite may expand under high temperatures (proven by Cartz and Tooper 1964) caused by conventional heating and by microwaves irradiation (as suggested by Lu et al 2017) was tested in the third experiment documented in Chapter 4. The results show that biotite grains expanded to about 101% of their initial volume after microwave irradiation. Given the subjectivity of the method used, a 1% expansion is below the margin of error. In order to resolve this issue, the biotite was tested using a TGA analysis. The results confirmed that grains of biotite from PPB samples in the Mount Rosa Complex only lost about 1% of weight when heated at 950°C using synthetic air (20.9% O₂ with balance N₂) at a flow rate of 50 sccm with heating done at 20K/min from room temperature to 950°C, held for ten minutes, and cooled at 40K/min.

Lastly, the possibility was evaluated that the biotite in the Boulder granodiorite contains more water than the biotite in the PPB granite samples. This would explain why the biotite from the granodiorite was disproportionately associated with the cracks (Chapter 2) whereas the same phenomenon was not observed in the Mount Rosa granite specimens (Chapter 3). A TGA analysis was conducted on samples of biotite from the Boulder granodiorite using the same protocol and parameters presented in Chapter 4. The results presented in Figure 5.2 show that the sample lost 0.6% of its weight. The drop in oxygen partial pressure around 800°C is similar to that observed in the
TGA analysis of the biotite from the Pikes Peak granite. This means that it is unlikely that the cracking in the biotite observed in Boulder granodiorite samples is due to the liberation of the hydroxyl groups.

The proposed explanation is as follows: despite being hydrated minerals, the water contained in the biotite for both sample types (PPB granite and Boulder granodiorite) was most likely too strongly bonded to the mineral structure to allow for evaporation. Therefore the only difference between the two biotite lies in the texture as discussed in Chapter 3. Biotite present in PPB samples were large isolated grains, whereas the biotite present in Boulder granodiorite were small grains aggregated together.

![Figure 5.2 TGA analysis results of the grains of biotite from Boulder granodiorite and Pikes Peak Biotite (PPB).](image)

This suggests that for granite type specimens the texture of the rock is a more important driver of microwave irradiation behavior than the presence of bound water in the structure of the mineral.

As for amphibole, the experiment presented in Chapter 3 showed that amphibole grains tend to be disproportionately associated with the cracks. The oikocrystic texture in MRA may have led to a thermal runaway similar to that described in Peinsitt et al. (2010). The texture in this rock type comprises patches of amphiboles that are clustered around other grains. This geometric arrangement of the amphiboles likely led to efficient heat redistribution in these zones, causing the melting observed in the specimen center of a couple of specimens. Therefore it is not possible to say with
certainty whether the disproportionate association of cracks with amphibole was due to the texture or the presence of water.

5.2. Applications

The suite of methods developed in this dissertation is applicable to various types of studies on rock cracking in addition to microwave-induced cracking. However the type of study will dictate the methods to use, and in which combination. The following paragraphs describe different types of studies and the recommended materials and suite of methods that would be relevant.

5.2.1. Study of Large Scale Microwave Pretreatment In Mining

As presented in Chapter 1, the most likely set up (as of 2019) for large scale implementation would be a vertical flow configuration, with the material going through a feed hopper into a processing tube which is transferred to the microwave are applied for material that has already been crushed in order to ease the ore-gangue separation during the processing phase (Buttress et al. 2017). However, implementation at an earlier stage before grinding could be even better in terms of energy savings. For this type of study, the mineralogical association of the cracks does not seem as important as particle size reduction or rock strength reduction. Therefore it is not necessary to spend the time and money doing further microscopy analyses once the rock type has been chosen and characterized. However the choice of the rock is the most important step. For that purpose, representative rocks from the mining operation need to be fully described using microscopy analyses. The material can be described using a combination of optical microscopy and automated mineralogy, in order to get an accurate description of both the mineralogy and the texture of the rock. Here is a list of the characteristics of the rock to be included are:

- Grain size
- Mineralogy and the dielectric properties and microwave behavior of the minerals using available literature. Chen et al. (1984), Church et al. (1988) and Haque (1998) provide a good starting point for common minerals.
- Texture of the rock: are the grains homogeneously distributed? Are the stronger absorbers agglomerated or isolated?

In terms of rock candidates for applied microwave irradiation in the mining industry, this work demonstrates that strong microwave absorbers are not a necessary prerequisite for microwave-
induced fracturing. Instead, it is the contrast in properties that is important. It was shown in Chapter 1 that monomineralic rocks (e.g., quartzite and marble) were not good candidates because the lack of contrasting properties resulted in longer times of irradiation to achieve even small degrees of damage. Minerals relatively transparent to microwaves (e.g. quartz, feldspar, calcite) commonly form the gangue in ore deposits. Minerals with higher dielectric constant and higher dielectric loss (better microwave absorbers such as amphibole or biotite) or contrasting thermal properties (e.g. biotite’s abnormal thermal expansion) commonly form the “ore” phase. The term ore in this context relates to grains with contrasting properties compared to the gangue, not to the mining definition of ore as economically valuable mineral (e.g., sulfide or oxide minerals).

The present study shows that a mix of properties enhances microwave irradiation. This corroborates the work of Harrison (1997), Kingman et al. (2000), Jones et al. (2005), and Batchelor et al. (2015) that microwaving is most effective in rocks with a mix of ore and gangue minerals, and that the larger the ore gangue contact surface, the more efficient the microwaving treatment. For example as found in the present study, in PPB samples the presence of biotite (a better microwave absorber with greater thermal expansion than the other minerals) led to an extensive network of fine cracks at the grain boundaries. The grain size of the rock is also important: the results of Chapter 3 show that coarse grained rocks (1-5 mm) fracture better under microwave irradiation than do fine grained rocks. The results from those experiments also indicate that the effectiveness of microwaves is higher for the minerals with contrasting properties that are either randomly disseminated as individual grains, or in aggregated clusters that are randomly disseminated.

Based on these constraints, ore deposits hosted in granitoids would be suitable targets for weakening by microwave irradiation prior to comminution. For example, porphyry copper deposits, some skarn deposits (tin, tungsten, and molybdenum), some epithermal deposits, intrusion-related gold deposits, and rare earth element-bearing intrusions may be hosted in granitoids that meet the two criteria defined above. IOCG (Iron Oxide-Copper-Gold deposits) hosted in breccias could also be viable candidates. The material chosen needs to be easily available in large quantities and not weathered.

For this type of application where the main goal is strength reduction by generation of microfractures, the most quantitative results will be given by UCS testing. One could consider using relative P-wave velocity measurements; however that method is qualitative and will not give any
information about the efficacy of the microwaving. UCS testing will allow for efficient quantification of the optimal microwave settings (duration of exposure, power level, etc.). Tensile Strength testing methods such as Brazilian Disc Test (presented in Section 1.4.2.2) could also be used to quantify the optimal microwave settings. However UCS testing requires a specific shape of sample (core that is at least 2 times longer than its diameter) and it has not yet been proven that the cracks induced by microwaving core samples are representative of the outcomes in bigger samples of different shapes. Whichever strength test method is used, the number of samples needed for a statistical result should be calculated using the standard E122, “Practice for Calculating Sample Size to Estimate, With Specified Precision, The Average for a Characteristic of a Lot or Process” (ASTM 2017b) as discussed in Chapter 2.

5.2.2. Study of Large Scale Microwave Treatment in Civil Engineering

The application of microwaving methods to civil engineering is more likely to be for material mainly composed of weak absorbers (any rock mainly composed of quartz, calcite, feldspar, and other microwave transparent minerals), such as the silicate or carbonate rocks which are common at tunneling and excavation sites around the world. This work demonstrates that even rocks containing only weak microwave absorbers are suitable targets for industrial applications of microwave-induced cracking. In such cases, the ideal candidates are either (1) coarse grained (1-5 mm) rocks containing randomly disseminated minerals with contrasting physical and microwaving properties, such as PPB, or (2) coarse grained (1-5 mm) rocks containing clusters of minerals with physical and microwaving properties contrasting with those in the surrounding rock, such as MRA.

Based on these criteria, suitable rock types for microwave applications in civil excavations include intrusive igneous rocks such as granites, granodiorites, diorites, and gabbros, as well as possibly pegmatites. Suitable sedimentary rocks include conglomerates, breccia, as well as coarse sandstone. Suitable metamorphic rocks include those without small-scale fabrics, such as hornfels, eclogites, and some gneisses.

Similar to above, the material characterization is important during site selection in order to determine whether microwaving could be applied given the local rock types. Once the rocks have been well characterized, it will be critical to determine the microwave-induced strength reduction of the material. The previous discussion of P-wave and UCS testing applies here as well.
5.2.3. Fundamental Investigations of Cracking Mechanisms

Whether cracks are induced by microwaves or another damage method, it is critical to understand the fundamental cracking mechanisms. For this type of study, three aspects are important in choosing the rock type and the methods:

1) It is important to choose material that is easily available in large quantities, fresh and homogeneous at both outcrop scale and mineral scale. 2) The grain size should be less than 5mm on average to allow for representative thin sections to be made. 3) Finally the material should be polymineralic if the study is focused on microwave-induced cracks because it is the difference in dielectric properties between the minerals that will enable efficient microwaving. However if the study focuses on other crack inducing treatments such as loading or conventional heating, the material may not need to be polymineralic.

Comprehensive evaluations of cracking should include data on the bulk rock property changes caused by cracking, as well as the characteristics of the cracks, including aperture, location, morphology and mineralogy. If strength reduction is not the main focus of the type of study, there is no need for the time consuming UCS testing. Use of the quick relative P-wave assessment presented in this dissertation would be informative. The relative P-wave may provide information on whether or not cracks were created in a specimen. Microscopy analyses are very time consuming and could be costly, whereas P-wave testing is quick and inexpensive.

The results of the present investigation demonstrate that optical light microscopy and automated mineralogy should both be applied to obtain complete mineralogical information on the cracks. The integration of these techniques enabled the quantification of the pore space generated by cracking, liberation of the mineral phases, qualitative assessment of crack morphology, and quantitative assessment of mineral-crack associations. The most reliable data is provided by point-counting paired with cross-polarized optical and false color electron microscopy images using the assignment of pore space to a unique species definition via automated mineralogy. The details of the method can be found in Chapter 2.

Finally the use of microCT analysis is a useful tool in this type of study. As shown in Chapter 4, microCT can provide qualitative and quantitative data on crack location and morphology. The method is non-destructive and allows for the investigation of crack evolution over time. The 3D component of this analysis is really the only way to get a sense of the crack morphology inside a
sample. However, the method is costly and time consuming. This method is not sufficient in itself and needs to be paired with other techniques. Even though the size limitation and tradeoff between resolution and sample size can be accommodated to allow for representative results (as shown in Chapter 4), the mineralogy of individual grains can only be resolved for certain rock types. Minerals with similar X-ray absorbance capacity will not be differentiated on the CT scan. It may also be difficult to observe grain boundaries.

5.3. Recommendations for Future Work

The following paragraphs detail different questions that arose during this investigation and that the author finds it would be interesting to pursue.

5.3.1. Refining the Methods

Some of the methods used in this dissertation are cutting-edge, and even during the three years of this study, significant technological advances occurred. At this point in time the following refinements to the methods would be the most useful for similar studies.

It is theoretically possible to compare the cracks on two microCT scans generated before and after microwave scans to obtain information on crack evolution. However, in the present study the two scans could not be fully aligned with one another (because of the slight bending of the top of the sample after microwaving). This made that analysis impossible at the time. Using before- and after-scans, it would be very interesting to find a way to quantitatively analyze the behavior of the cracks. This would indicate where the most transformation in the sample has happened, where most cracks were created or if pre-existing cracks expanded.

Another interesting idea to be developed using microCT image processing technology would be to align multiple SEM automated mineralogy generated maps on the microCT dataset in an image processing software. This study attempted this manipulation (Figure 5.3), however the slight angle that was produced when making the thin sections made it impossible to align the data completely. Ideally those three images could be used as training data for the deep learning tool, and potentially project the mineralogy of the entire sample based on the training data using a U-net neural network.

This would open the path to extract even more information from the microCT dataset: measurements regarding the mineral abundances in the whole sample, not just the thin sections,
information on spatial distribution, and the texture of the sample. Going further with this idea, one could use the 3D scan and projected mineralogy to extract planar data at the location of thin sections to conduct separate point counting studies. This would give more information on the mineralogical associations of the cracks (minus the boundaries component unless they are visible in the microCT dataset) and advance the understanding of how cracks are induced and propagate in natural rocks. This could be used for applied research in engineering but also to study natural geological processes.

Figure 5.3 Capture from sample PPB9 in Dragonfly showing the alignment of SEM generated mineralogy maps. Note on this example the maps could not be perfectly aligned and therefore could not be used in a neural network to train the software to recognize the mineralogy.

A study on how the specimen shape influences the microwaving response would be of great interest in order to implement the technology as a pre-treatment to comminution to weaken the rock before crushing. Thus far, studies on the implementation of microwave technology in mining have been focused on separating the ore from the gangue, and the material used was already crushed down to 50 mm (Batchelor et al. 2016). If a correlation between the shape of the material and its behavior under microwave irradiation could be made, it would allow for a better prediction of microwave treatment efficacy.
5.3.2. Microwave-Induced Cracking Mechanisms

As mentioned in the previous section, the development of microCT and image processing tools such as deep learning opens the possibility of more quantitative assessments of crack quantity, morphology, and mineral association.

As mentioned in Section 5.1.2.2, point counting for crack characterization was done only at one orientation of the thin section (perpendicular to the long axis). This could be done at different angles to see how representative/accurate one orientation is in various rock types. Menendez et al. (2001) addressed this issue briefly; however they did not provide quantitative data on how much the rotation of the grid influences the point counting result.

From the point of view of understanding crack morphology, it would be interesting to apply the point counting technique to large numbers of samples of the same rock. This would help assess how various parameters affect the cracking pattern and mineralogical association: Do longer times of microwave exposure or different microwave settings lead to wider cracks? More cracks? Between which minerals?

A recent study by Huang et al. (2019) showed that, for coal ore, the presence of preexisting cracks perpendicular to the axis of the core led to widening of those cracks during microwaving treatment. In the present study such a change in preexisting weaknesses was not observed. Therefore it may be interesting to pursue a study specifically focused on the influence of preexisting weaknesses (cracks) depending on the type of rock.

The present study did not focus on anisotropic samples. If the sample presents an anisotropy or grain orientation (for example the foliation present in a schist, or compositional banding in a gneiss) how would that affect the microwaving? Especially when doing UCS tests, it would be interesting to see how the orientation of the foliation (perpendicular or parallel to the core axis) has an impact on the strength reduction due to microwaving.

If microwaving is industrially applied at a mine, it would be interesting to test different ores from the same deposit to determine if local differences in mineralogy and texture would lead to needing a different microwaving setting (longer time of exposure, stronger power etc.). This would be useful for any application of microwave technology to an ore that has spatially heterogeneous mineralogy (e.g., skarns, epithermal deposits, porphyry deposits).
5.4. References


