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**Thermal Cracking in Lac du Bonnet Granite During
Slow Heating to 205°C**

**Fissuration à chaud dans le granit de Lac du Bonnet
au cours du chauffage lent jusqu'à 205°C**

P.J. Chernis and P.B. Robertson

AECL RESEARCH

THERMAL CRACKING IN LAC DU BONNET GRANITE DURING
SLOW HEATING TO 205°C

by

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1993

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FISSURATION À CHAUD DANS LE GRANIT DE LAC DU BONNET
AU COURS DU CHAUFFAGE LENT JUSQU'À 205°C

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RÉSUMÉ

On a enregistré les émissions acoustiques à mesure qu'on a chauffé lentement des carottes de forage provenant du granit de Lac du Bonnet à des températures entre 66 et 205 °C pour évaluer les effets de la température sur les propriétés des échantillons de roche. On a mesuré les vitesses d'ondes longitudinales et transversales à travers les échantillons et calculé le module de Young, le module d'élasticité transversale et le coefficient de Poisson. On n'a décelé aucunes émissions acoustiques importantes jusqu'à ce que les températures atteignent approximativement 73-80 °C. Au-dessus de ce "seuil" de température, les propriétés de la roche ont diminué et, à 205 °C, le module de Young, le module d'élasticité transversale et le coefficient de Poisson calculés, ont été réduits respectivement de 30, 26 et 29 %.

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ABSTRACT

Acoustic emissions (AE) were recorded as drill core samples of Lac du Bonnet granite were slowly heated to between 66 and 205°C to evaluate the effects of temperature on the properties of rock samples. Longitudinal and shear velocities of the samples were measured, and Young's moduli, shear moduli and Poisson's ratios were calculated. No significant AE activity was detected until temperatures reached approximately 73-80°C. Above this "threshold" temperature, calculated rock properties decreased, and at 205°C calculated Young's modulus, shear modulus, and Poisson's ratio were reduced by 30, 26, and 29% respectively.

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1. INTRODUCTION

AECL is currently assessing the feasibility and safety of disposing of nuclear fuel waste deep in plutonic rock bodies of the Canadian Shield. Nuclear fuel waste generates a decreasing amount of heat, due to the decay of the contained radionuclides, over time. The effects of increased temperatures on the properties of the rock are, therefore, very important. One such effect is the creation of additional microcracks in the rock in response to the stresses caused by differences in the thermal expansion rates of neighbouring mineral grains. An increase in the microcrack population in the rock could alter the mechanical and mass transport properties that may be important in disposal vault design.

Laboratory studies in other crystalline rocks have shown that there is a threshold temperature below which there is no microcracking due to increased rock temperature. For example, the threshold temperature of Charcoal granodiorite is 75°C (Bauer and Johnson 1979), and for Westerly granite it is 60-70°C (Yong and Wang 1980).

Temperatures in excess of 60°C have been shown to affect the properties of rock. Potter (1978) found that, when heated, the permeability of Westerly granite initially decreased by 15% and reached a minimum at temperatures between 60 and 103°C; it then increased exponentially when heated to 200°C, reaching a value of almost 10 times the initial permeability. Upon cooling to ambient temperature, the permeability remained almost four times higher than the initial value. Bauer and Handin (1983) measured a negative coefficient of linear thermal expansion for the Charcoal granite over the range 25-100°C. Annor and Jackson (1986) measured, at temperatures up to 200°C, permeability, linear thermal expansion, and strength of drill core samples from the Lac du Bonnet granite and achieved results similar to those of Potter (1978): permeability first decreased, reached a minimum at 75-100°C, then increased. The coefficient of linear thermal expansion increased in the temperature range 75-100°C. Strength was not significantly affected by temperatures of 100°C, but was reduced by 15% at 200°C.

To develop expertise within the Nuclear Fuel Waste Management Program in the detection and evaluation of the effects of thermal cracking on the properties of crystalline rocks, a thermal cracking experiment was performed. The objectives of the experiment were threefold:

- (1) to determine the threshold temperature of thermal cracking in the Lac du Bonnet granite sampled from the Underground Research Laboratory (URL) site;
- (2) to calculate the effects of thermal cracking on physical properties that may be important for disposal vault design; and
- (3) to provide data used in evaluating models that estimate microcrack population changes caused by temperature changes.

The threshold temperature of Lac du Bonnet granite was determined by monitoring acoustic emissions (AE) during slow heating of samples to various temperatures up to 205°C. Acoustic emissions are elastic waves generated

when energy is rapidly released, principally by the formation and propagation of cracks. The effect of thermal cracking on rock properties was determined from measurements of P- and S-wave velocities before and after heating. Changes in the dynamic elastic constants, Young's modulus, shear modulus or rigidity, and Poisson's ratio, were calculated. The experiment provided a fast, inexpensive method of evaluating the thermal cracking potential of rocks. This report outlines the experimental method used and presents the results obtained. The equipment and experimental methodology are also described.

2. THRESHOLD TEMPERATURE MEASUREMENT

The threshold temperature, or the temperature at which thermal cracking commences, was determined by monitoring acoustic emissions as granite drill-core samples were slowly heated to a maximum of 205°C. The threshold temperature has been found to be independent of the heating rate (Yong and Wang 1980), although the number of emissions detected is dependent on the heating rate and the monitoring system. The heating rates were kept below 2°C/min to minimize cracking caused by thermal gradients within the samples (Richter and Simmons 1974).

The instrumentation for the thermal cracking experiment consisted of three parts (Figure 1): the furnace and temperature controller, the AE detection instrumentation, and the data storage instrumentation.

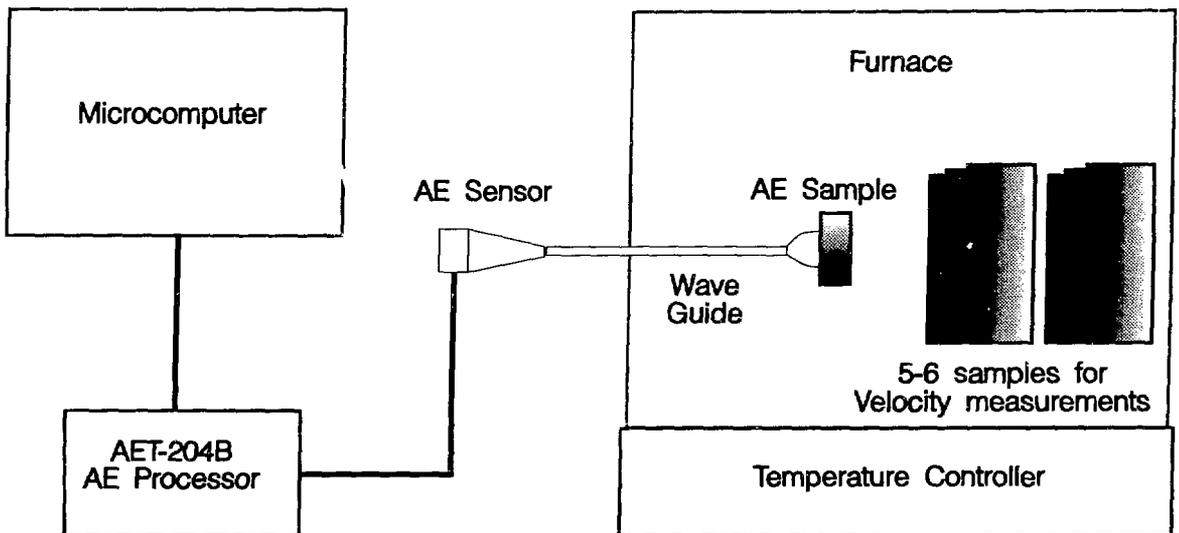


FIGURE 1: Schematic Diagram of Thermal Cracking Experiment Set-Up

2.1 FURNACE AND TEMPERATURE CONTROLLER

Samples were heated in a Lindberg box furnace (model 51442) with a Lindberg 7-segment programmable temperature-slope controller (model 59544-E1). The initial and final temperatures and the duration of each of the 7 segments could be set independently. The temperature of the furnace was monitored with an Omega Engineering Inc. Model 199 digital readout thermometer with a type K (Ni-Cr/Ni-Al) thermocouple and linearized analog output (1 mV/°C). Large temperature gradients within the furnace were unlikely because of the slow heating rates used and the large thermal mass of the furnace.

2.2 ACOUSTIC EMISSION DETECTION INSTRUMENTATION

Acoustic emissions were monitored with an Acoustic Emission Technology (AET) Corporation model 204B, single-channel AE detection system. The total instrument bandwidth is 6.5 kHz to 2.8 MHz (-3 dB). The detector was an AET corporation model FC500 sensor, a PZT crystal with sensitivity of -90 dB \pm 3 dB over a frequency range of 300 kHz to 2.1 MHz (-100 dB at 100 kHz). A bandpass filter restricted the detected energy band to 125 kHz-2 MHz to avoid interference by mechanical noise (Dunegan et al. 1968). The system was operated in the COUNTS mode at a gain of 102 dB, with floating threshold (1.35 V) and a sampling period of 1 s.

2.3 DATA STORAGE INSTRUMENTATION

Voltages representing the temperature (from the digital thermometer), AE counts and rms (root-mean-square) signal level (from the 204B system) were read by an Omega Engineering Inc. analog interface card (model WB-AIO-B) within an IBM-PC microcomputer. Signal level was not used in the analysis of the results. A BASIC program (ASUBR.BAS) that was supplied with the analog card to read the eight analog and digital inputs was modified to collect data on only three analog channels, and to write data to diskette at appropriate time intervals. The resolution on the card was set to 12 bits and the filter delay to 16 ms. Cycle time to read all three channels was approximately 1.1 s.

Since the service temperature of the AE sensor was -50 to 107°C, it was placed outside the furnace, and a wave guide assembly was employed between the sensor and the sample. The wave guide assembly consisted of a half-sphere-shaped mounting stub of Sauereisen No. 29 low-expansion cement on the end of a silica glass rod (Corning Vycor). A drill core stub, 45 mm in diameter by 20 mm long, was attached to the cement mounting stub of each wave guide assembly, using a thin layer of low-expansion cement. Thermal expansion of the cement closely matches that of the rock in the temperature range 20-300°C (0.25-0.3%). As well, shear strengths of cement-rock bonds that were heated were not significantly different from unheated bonds (Table 1), and therefore thermally induced cracking of the bond between the rock and the cement was judged to be insignificant.

TABLE 1
STRENGTHS OF CEMENT-ROCK BONDS
(kN/mm² x 1000)

Heated Samples	Unheated Samples
328	369
717	440
307	212
493	618
Average 461 ± 190	Average 410 ± 168

The thermal expansion of the Vycor glass rod was lower than that of the thermal cement mounting stub. This caused cracks to form in the stubs surrounding the rods, principally at 60-80°C and 180-209°C, and generation of acoustic emissions. This cracking of the mounting stubs during the experiment was eliminated at low temperatures (<170°C), and reduced to an insignificant amount at temperatures above 180°C, by heating the wave guide assemblies (cement stubs attached to glass rods) to 230°C before the rock samples were attached to the guides.

A type K thermocouple and wave guide assembly with a 20-mm-long drill-core stub were placed through ports in the furnace door. The tip of the thermocouple was positioned near the sample. At specified time intervals, time and voltages representing the following quantities were recorded:

- (1) temperature,
- (2) total number of AE ringdown counts with energy exceeding the threshold voltage, and
- (3) rms signal level.

The time interval between measurements was 300 s, but the computer was programmed to record the above data if the count rate exceeded background, or if the temperature rose more than 7.5°C before the end of the 300-s time period. The background emission rate, approximately 200 counts/s, was taken as the maximum emission rate at the start of each run.

3. ACOUSTIC WAVE VELOCITY MEASUREMENTS

Stubs approximately 60 mm in length were cut from a section of drill core, and the ends were ground flat to within ±0.03 mm and parallel to within

0.15 mm. Dimensions were measured with steel vernier calipers with a resolution of 0.01 mm. The samples were weighed with a Metler 2000 electronic scale with a resolution of 0.01 g. Densities of the samples were calculated from their dimensions and weights. The samples were divided into groups of five or six "thermal" samples that were heated, and groups of five or six "standards" that were not heated. The wave velocities, V_p and V_s , of the standards and the thermal samples were measured prior to and following heating of the thermal samples (after cooling to ambient temperature). Wave velocities were measured at atmospheric pressure using two piezoelectric transducers, one acting as a signal source, the second as a receiver. A pulse generator applied an electrical impulse to the transmitting crystal, which converted the electrical energy to a mechanical wave. The mechanical wave travelled through the sample, was detected by the receiving transducer and converted to an electrical signal that was displayed on an oscilloscope. The instrument was calibrated by measuring the wave velocity of steel cylinders of known length. The repeatability (1 standard deviation over the mean) determined from 55 measurements was 0.95%.

The P-wave arrivals were easily observed. Their velocities (V_p) were calculated from the time taken for the mechanical wave to travel the length of the sample. The S-wave arrival was taken where a phase reversal appeared on the displayed waveform. Some ambiguity was encountered in locating the exact position of the S-wave arrival. Determinations of V_s of standard samples were repeatable to $\pm 6\%$, and repeatability of V_p was $\pm 7\%$.

Some variation in wave velocity is attributable to differences in water content of the samples, which varied with the relative humidity in the laboratory. Drying samples at 46°C for 67 h reduced V_p by 1-5%. The amount of water removed by drying was 0.02-0.06%, approximately 10-25% of the porosity. The humidity effects were systematic, that is, a change in water content of the samples tended to raise or lower the velocities of all samples. The effects of humidity on the velocities can be compensated for by adjusting the post-heating velocities of the thermal samples.

The (unadjusted) change in P- and S-wave velocity is

$$\Delta V_{p,s} = V_{pre} - V_{post} \quad (1)$$

where

$$\begin{aligned} V_{pre} &= \text{pre-heating velocities of each sample, and} \\ V_{post} &= \text{post-heating velocities of each sample.} \end{aligned}$$

The adjusted post-heating velocities of each thermal samples are

$$V_{(adj)} = V_{post}(\text{thermal}) - (V_{post}(\text{thermal}) \times A) \quad (2)$$

where

$$A = \left[\frac{\sum [V_{pre}(\text{standard}) - V_{post}(\text{standard})]}{V_{pre}(\text{standard})} \right] \times 100 \quad (3)$$

A is the average difference of the pre-experiment and post-experiment velocities of the standards, normalized to the pre-experiment velocity and expressed as a percentage.

V_{post} (thermal) = post-heating velocity of each thermal sample,
 V_{pre} (standard) = pre-experiment velocity of each standard,
 V_{post} (standard) = post-experiment velocity of each standard, and
N = number of standard samples = 5 or 6.

The dynamic elastic constants G (shear modulus or rigidity), ν (Poisson's ratio) and E (Young's modulus) were calculated using the following equations (Gyenge and Herget 1977):

$$G = rV_s^2 \quad (4)$$

$$\nu = [V_p/V_s]^2 - 2] / [2(V_p/V_s)^2 - 1] \quad (5)$$

$$E = 9K\nu / (3K + \nu) \quad (6)$$

where

$$K, \text{ the bulk modulus} = r(V_p^2 - 4/3V_s^2) \quad (7)$$

r = density (g/cm³)

4. DESCRIPTION OF SAMPLES

The samples were unfractured, 45-mm-diameter drill core taken from borehole URL-2, 200 m from the collar. They were grey (unaltered), inequigranular, medium- to coarse-grained, and massive. Minor inhomogeneities, such as aplite stringers and schleiren, were avoided. The modal composition of thin sections of other samples from borehole URL-2 is compared in Table 2 with the modes of other granitic rocks for which elastic properties have been measured. The Charcoal granodiorite is fine- to medium-grained and inequigranular, and the well-known Westerly granite is an inequigranular, but generally fine-grained granodiorite (Krech et al. 1974). The URL samples in thin section exhibit a hypidiomorphic-granular texture, and grain size ranges from microscopic to 15 mm. Prismatic albite-oligoclase (<3.5 x 4.5 mm) and microcline up to 10 x 20 mm form a framework in a contiguous fine- to medium-grained quartz groundmass. Quartz displays a variety of deformation and recrystallization structures: mild undulatory extinction, sutured grain boundaries, polygonized grains and bubble trails. Muscovite, biotite, opaques and other accessory minerals occur at the quartz-feldspar contacts.

TABLE 2

AVERAGE MODE OF LAC DU BONNET SAMPLES AND COMPARABLE GRANITIC ROCKS

	Westerly Granite ^{1,2,3}	Charcoal Granodiorite ^{2,3}	Lac du Bonnet Granite ⁴
Quartz (%)	22.5 - 29.3	16.7 - 21	30.6 (3.9)*
K-feldspar (%)	22 - 32	20 - 26	27.3 (6.8)
Plagioclase (%)	31.3 - 43.0	38 - 40.8	37.5 (5.7)
Biotite (%)	3.8 - 6.9	3 - 9.5	3.5 (1.5)
Muscovite (%)	0.7 - 3.0	-	0.5 (0.3)
Amphibole (%)	-	11.7 - 12	-
Opagues (%)	0.8 - 0.9	1.2	0.4 (0.3)
Density (g/cm ³)	2.64	2.71 - 2.73	2.61 - 2.62
Grain Size	Fine	Fine to coarse inequigranular	Medium to coarse inequigranular
Young's Modulus GN/m ²	44.3	48.4	64.7
Shear Modulus GN/m ²	19.4	23.9	-
Poisson's Ratio	0.21	0.25	0.27
Threshold Temperature	60-70°C	75°C	68-83°C**

¹ Richter and Simmon (1974)

² Bauer and Johnson 1979)

³ Krech et al. (1974)

⁴ Chernis (1985)

* Standard deviation in brackets

** This paper

5. DESCRIPTION OF EXPERIMENT

Six heating runs were performed in the experiment. In each run, one AE sample and up to six velocity samples were heated to various temperatures as outlined below:

Run 1: One AE sample and 6 velocity samples were heated to 126°C, then cooled slowly to room temperature.

Run 2: In part A, 6 samples were heated to 205°C. In part B, one AE sample was heated to 199°C. Monitoring was not done during the cooling period.

Run 3: One AE sample was heated to, and held for 11 h, at 115°C.

Run 4: One AE sample and 5 velocity samples were heated to, and held for 10 h, at 91°C, then slowly cooled to room temperature.

Run 5: One AE sample was heated to, and held for 11 h at, 116°C, then slowly cooled to room temperature.

Run 6: One AE sample and 5 velocity samples were heated to, and held for 11 h at 66°C, then slowly cooled to room temperature.

The series of runs was planned so that temperatures near and well above the threshold temperatures of similar rocks (see Table 2) were reached. The intention was to use linear heating rates in all runs, but the furnace controller produced initial logarithmic rates in run 1 and run 3. The cause for the initial non-linear rates is not known. In these runs, the slope of the temperature-time curves decreased as temperature increased. The maximum heating rate for run 1 was 1.0°C/min at approximately 50°C, and for run 3, 0.6°C/min at 45°C. The rates of cooling were similar to the rates of heating, to avoid thermal shocking.

6. RESULTS

6.1 ACOUSTIC EMISSION DATA

Plots of the acoustic emission activity detected during heating are presented in Figure 2. Time (s) is plotted on the horizontal axes; the vertical axes shows the emission or count rate (count/s) exceeding background, and temperature (°C). Minor irregularities in the heating curves are not correlatable with increased emissions. Figure 2 illustrates the following:

- (1) the emission rate above background generally increases with the temperature;
- (2) for runs in which the maximum temperature was held constant for a number of hours, the emission level remained high for 20-50 min after reaching T_{max} , then returned to background levels with only sporadic AE activity; and
- (3) few emissions were produced during cooling.

The temperature at which the acoustic emissions first exceed background is difficult to determine on the plots of count rate versus temperature, so the data were replotted as cumulated emission rate above background versus temperature (Figure 3). The curves for all the data plotted in this manner (except for run 6) can be divided into three segments:

- (1) a low-level background at temperatures of less than about 60°C (AB, Figure 3);
- (2) a region between approximately 60 and 80°C (BC, Figure 3), where a few counts above background occur; and
- (3) a segment above about 80°C (point C, Figure 3) where emissions above background become almost continuous. This point is the threshold temperature.

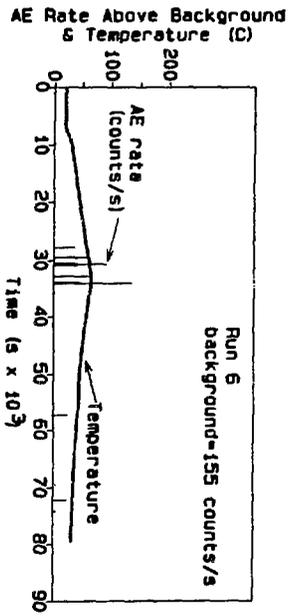
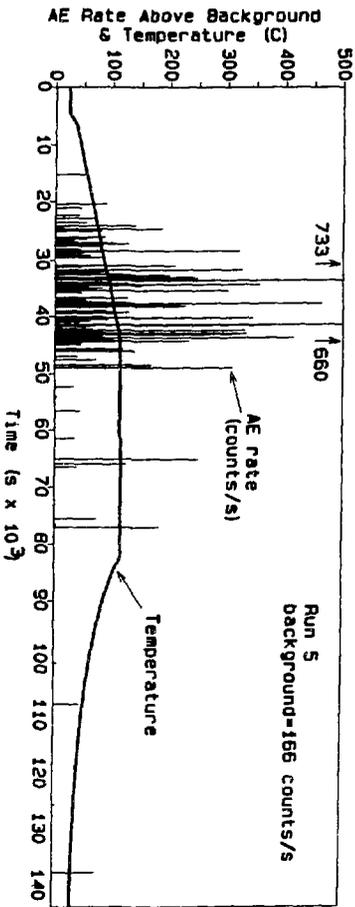
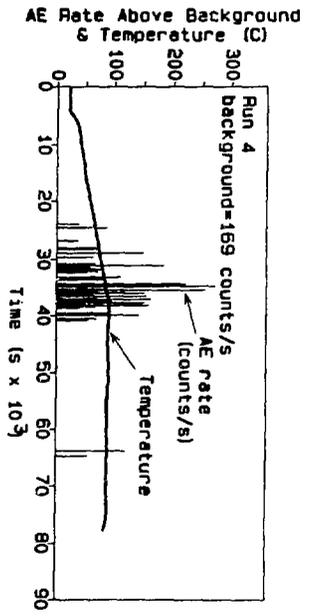
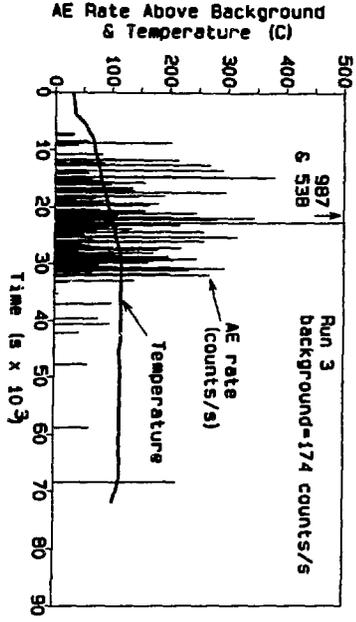
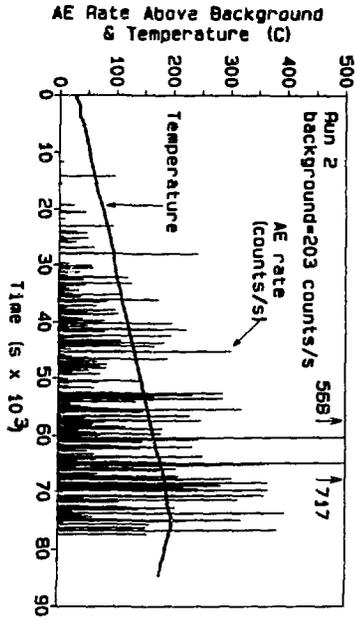
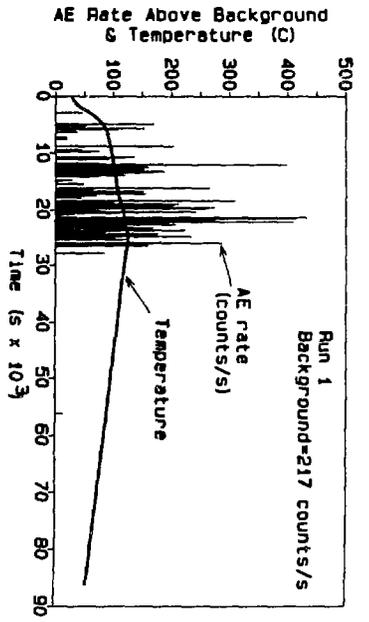


FIGURE 2: Acoustic Emissions and Temperature Versus Time. Emissions above background are plotted.

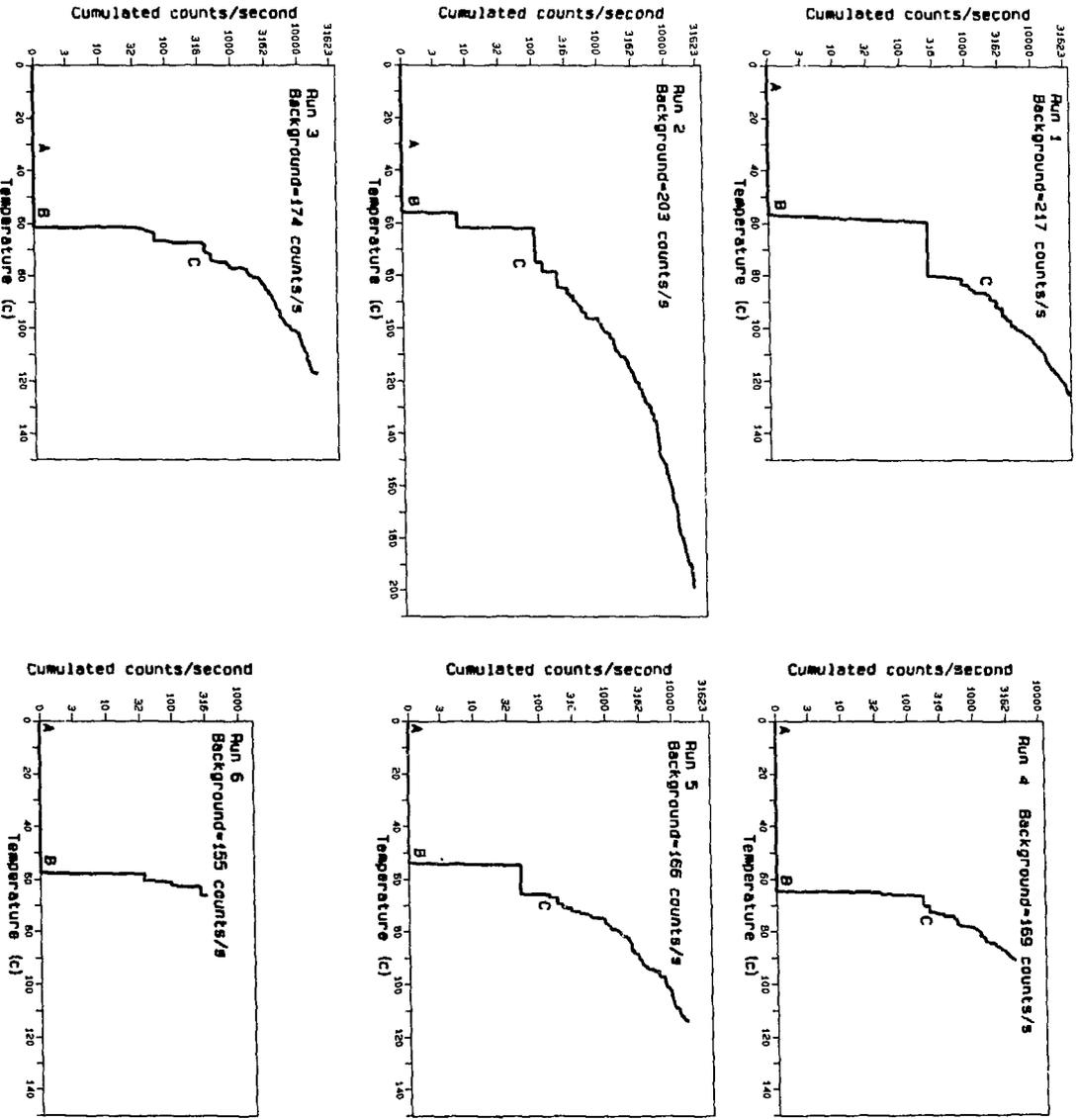


FIGURE 3: Cumulative Acoustic Emissions Versus Temperature. Background count rate has been subtracted from the plots. Background emissions occur between A and B. Sporadic emissions occur between B and C. Continuous emissions occur to the right of C. Point C was not reached in run 6.

Point B is reached between 55 and 70°C. Point C is reached between 68 and 83°C. The temperature in run 6 was not high enough to reach point C.

6.2 WAVE VELOCITIES AND MECHANICAL PROPERTIES

The effects of temperature on wave velocities are illustrated in Figure 4, which plots the reduction of velocities and calculated mechanical properties with increasing temperature. The velocities increase slightly after heating to 66°C. Above 66°C, however, velocity decreases with increasing temperature, although velocities of samples heated to 91 and 126°C are similar. Young's modulus calculated using $\Delta V_{p(adj)}$ and $\Delta V_{s(adj)}$ are lower by 12% at 91 and 126°C, and by 31% at 205°C. Shear modulus calculated using $\Delta V_{p(adj)}$ and $\Delta V_{s(adj)}$ decreased by 11% at 91 and 126°C, and by 26% at 205°C. The calculated Poisson's ratio decreased by 2% at 91°C, 6% at 126°C, and by 29% at 205°C.

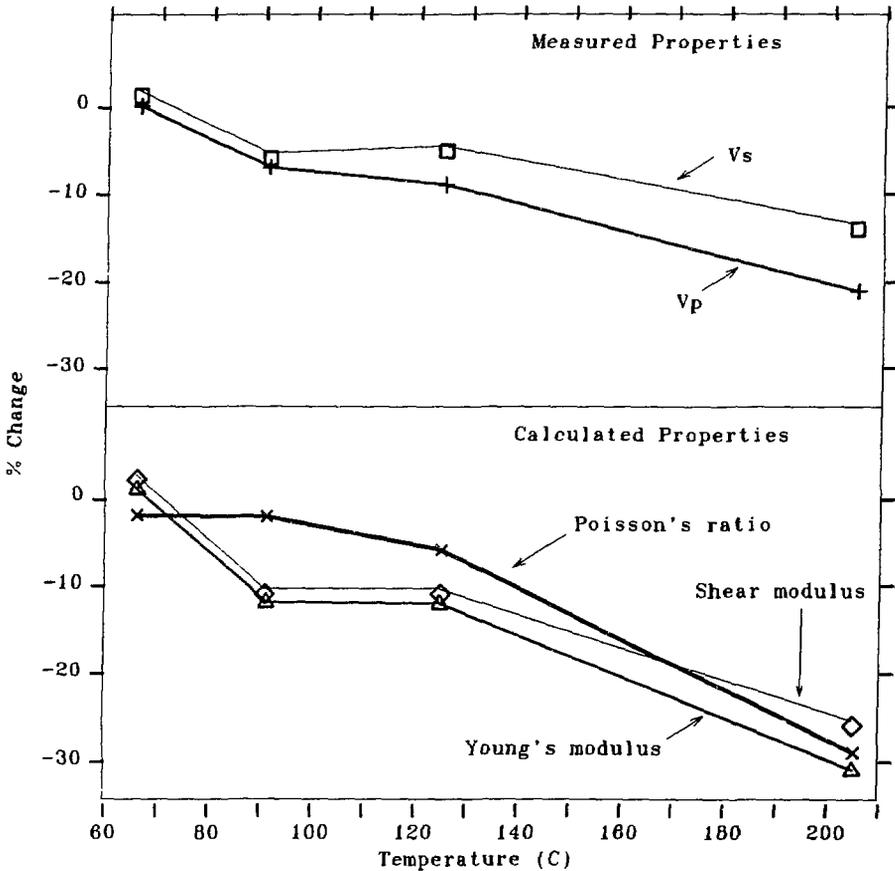


FIGURE 4: Change in Measured and Calculated Properties with Temperature. Change is calculated relative to properties of unheated samples. V_s and V_p are adjusted velocities calculated using Equation (2).

7. DISCUSSION

Although some minor acoustic events occurred at temperatures as low as 54°C, no significant rise above background occurred in the samples until temperatures reached approximately 73-80°C, slightly higher than the threshold temperature of the fine-grained Westerly granite (60-75°C, Yong and Wang 1980, Bauer and Johnson 1979) and medium-grained, more mafic Charcoal granodiorite (75°C, Bauer and Johnson 1979).

The observed reductions in V_p of the samples heated to 205°C are consistent with the results of others. Bauer and Johnson (1979) observed reductions of 15 and 19% in the V_p of Charcoal granodiorite and Westerly granite respectively, and also a more than twofold increase in crack density after slow heating to 200°C. The V_p of Remiremont (fine-grained) and Senones (medium- to coarse-grained) granites were reduced by 15 and 18% respectively after heating to 200°C (Homand-Etienne and Troalen 1984). Poisson's ratio of Charcoal granodiorite (Lehnhoff and Scheller 1975) fell from 0.20 at 24°C to 0.15 at 122°C (25% reduction) and to 0.03 at 260°C (85% reduction). Potter (1978) and Annor and Jackson (1986) observed initial decreases in permeability, followed by increases.

These behaviours are attributed to the differential thermal expansion of adjacent minerals in the rocks, particularly quartz, which has large coefficients of expansion that vary with crystallographic orientation. The initial decrease in permeability is postulated to be the result of expansion of minerals into, and reduction of, the pore and microcrack space in the rock (Annor and Jackson 1986). Above approximately 125°C, increases in the coefficient of thermal expansion and decreases in strength and permeability are the result of microcrack formation. The thermal cracking runs produced little AE activity below 80°C (Region BC, Figure 3); therefore it can be concluded that few cracks are formed in this region of microcrack closure. This is confirmed by the retention of normal wave velocities following heating to 66°C, within the regime where AE are first sporadically generated. With a continued temperature increase above about 80°C, stresses between and within grains increase. When the stresses exceed the local strength of the rock, they are relieved by crack extension and formation, which generate substantial numbers of AE. Upon cooling, newly formed or extended cracks remain partially open, resulting in reduced wave velocities, as reported here. Potter's (1978) results indicate that some cracks close when the rocks cool. The reductions in modulus calculated using velocities measured at temperature may therefore be slightly greater than those calculated here.

8. CONCLUSIONS AND RECOMMENDATIONS

Thermal cracking in the Lac du Bonnet granite occurs during slow heating when temperatures rise above approximately 80°C. Beyond this threshold temperature, microcracks form at increasing rates, permanently altering the physical properties of the rocks. In samples heated to 90-126°C and then cooled, the calculations suggest that dynamic Young's modulus and shear modulus are reduced by 12 and 11% respectively, and for samples heated to

205°C and then cooled, they are reduced by 31 and 26% respectively. Potter's (1978) results indicate that some cracks may close when the rocks cool; therefore, the actual reductions in properties may be greater than those calculated in this report using ambient temperature velocities.

When temperature is held constant for a period of time, acoustic emissions are detected for up to 50 min after reaching the maximum temperature, indicating that time-dependent thermal cracking occurs. These runs were conducted over fairly short periods of time. The effect on elastic properties, of temperature sustained for long periods of time slightly below the threshold temperature, should also be studied.

The runs were conducted in an unconfined stress state, and therefore the results are likely valid only for the near-field rock, where an annulus of blast damage and stress-relief cracking may exist. Such induced cracks may raise the threshold temperature by allowing the rock to expand into a greater microcrack space, delaying the buildup of intergranular stresses. Confining pressure will reduce the effects of temperature, but its effect on threshold temperature and the amount of cracking that occurs at a given temperature are not precisely known. Handin et al. (1977) found that a confining pressure of 50 MPa reduced the effects of thermal cracking at 400°C by 50%. Kern (1978) estimated that the minimum pressure to prevent microcrack formation at a given temperature is 100 MPa/100°C. Confining pressures may lower the threshold temperature by reducing the pore space into which the rock expands during the initial stages of heating, resulting in buildup of intergranular stresses at lower temperatures (Annor, personal communication, 1986).

These runs support the conclusion of Wilkins et al. (1987) that cracking due to temperature increases in the rock surrounding a vault would be negligible below a rock temperature of 80°C. If the temperatures were kept below about 60°C, the only effect of differential thermal expansion would likely be the closing of existing cracks, reducing the pore space.

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