# THERMAL DIFFUSIVITY OF ROCKS AS A FUNCTION

OF PRESSURE AND TEMPERATURE

By

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iii

# TABLE OF CONTENTS

Chapte	er		Page
I.	INTRODUCTION	•	1
	Thermal Diffusivity as a Function of Temperature Thermal Diffusivity as a Function of Pressure .	•	3 6
II.	EXPERIMENTAL PROCEDURE	•	9
	Thermal Diffusivity Measurements Set-Up (Apparatus) Measurement Procedures Problems in Performing the Measurements	• • •	9 10 13 17
III.	RESULTS	•	18
IV.	DISCUSSION	•	51
	Thermal Diffusivity as a Function of Pressure . Thermal Diffusivity as a Function of Temperature	•	51 55
V.	SUMMARY, CONCLUSIONS AND SUGGESTIONS	•	59
BIBLIOG	КАРНУ	•	62
APPENDI	KES	•	64
1	APPENDIX A - WHY AND HOW THE ROCKS BROKE	•	64
1	APPENDIX B - SAMPLE HOLDER FOR THE SAW	•	67
1	APPENDIX C - TEMERATURE CONTROLLER - WIRING SCHEME .	•	68
	APPENDIX D - CONTROL, SAMPLING AND DATA ACQUISITION PROGRA	MS	69

# LIST OF TABLES

Table											Page
I.	Setting of	the Exp	eriments	•	•			•	•	•	18
II.	Summary of and Pres	Thermal sure	Diffusi	vity	as a	ı Func	tion	of ?	Tempeı	cature	49

# LIST OF FIGURES

Figu	re	Page
1.	Block Diagram of Experimental Apparatus	12
2.	The Temperature Gradient in a Dolomite Stone	16
3.	Thermal Diffusivity of a Dolomite Stone	16
4.	Thermal Diffusivity as a Function of Pressure for Rock BSH1	19
5.	Porosity as a Function of Pressure for Rock BSH1	20
6.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock BSH1	21
7.	Thermal Diffusivity as a Function of Pressure for Rock BSH4.	22
8.	Porosity as a Function of Pressure for Rock BSH4	23
9.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock BSH4	24
10.	Thermal Diffusivity as a Function of Pressure for Rock BSV1.	25
11.	Porosity as a Function of Pressure for Rock BSV1	26
12.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock BSV1	27
13.	Thermal Diffusivity as a Function of Pressure for Rock C22 .	28
14.	Porosity as a Function of Pressure for Rock C22	29
15.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock C22.	30
16.	Thermal Diffusivity as a Function of Pressure for Rock L150.	31
17.	Porosity as a Function of Pressure for Rock L150	32
18.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock L150	33
19.	Thermal Diffusivity as a Function of Pressure for Rock L165.	34

# Figure

20.	Porosity as a Function of Pressure for Rock L165	35
21.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock L165	36
22.	Thermal Diffusivity as a Function of Pressure for Rock L166.	37
23.	Porosity as a Function of Pressure for Rock L166	38
24.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock L166	39
25.	Thermal Diffusivity as a Function of Pressure for Rock MCA362.	40
26.	Porosity as a Function of Pressure for Rock MCA362	41
27.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock MCA362	42
28.	Thermal Diffusivity as a Function of Pressure for Rock MXN1.	43
29.	Porosity as a Function of Pressure for Rock MXN1	44
30.	Thermal Diffusivity as a Function of Temperature, at Various Pressures for Rock MXN1	45
31.	Thermal Diffusivity as a Function of Pressure for Rock MXN4.	46
32.	Porosity as a Function of Pressure for Rock MXN4	47
33.	Thermal Diffusivity as a Function of Temperature, at Various Pessures for Rock MXN4	48
34.	Thermal Diffusivity Multiplied by Temperature, Rock MXN1 .	57
35.	Thermal Diffusivity Multiplied by Temperature, Rock C22 .	57
36.	Thermal diffusivity at different pressures normalized with the one at 14.5 PSI, Rock L166	58
37.	Thermal diffusivity at different pressures normalized with the one at 14.5 PSI, Rock BSV1	58

# Figure

38.	Limestone After 1500 PSI was Applied to the Long Axis.		64
39.	Sandstone After 1500 PSI was Applied to the Long Axis.		66
40.	Dolomite After 2500 PSI was Applied to the Long Axis .	•	66
41.	The Sample Holder for the Saw	•	67
42.	Temperature Controller Wiring to the System		68

Page

#### CHAPTER I

#### INTRODUCTION

The thermal energy in a solid may be present in a variety of forms corresponding to the mode of motion of its fundamental particles. Among these, one can find the vibration of the atoms around an equilibrium position, rotation of functional groups in a molecule, and translation of electrons. The lattice vibrations, i.e. the phonons, are directly involved in typical properties such as thermal conductivity and thermal diffusivity.

The phonons are scattered by any lattice defect as well as by other phonons. The actual mobility of a phonon depends upon its velocity of propagation and the mean free path. At ordinary temperatures, most of the energy is associated with short-wavelength phonons and the structural scattering factor is almost independent of temperature.

The knowledge of the thermal diffusivity is required for the prediction of the response of a rock body to a transient heat, source or sink. The thermal diffusivity is important in developing predictive models of the history of sedimentary basins and can be useful in thermal oil recovery process. The thermal diffusivity of a rock is not uniform. Within any rock unit there may be both random and systematic variations, depending on the degree of chemical differentiation, crystal or particle size, orientation and nature of crystal boundaries, and cooling history of the rock. A real rock is built from small crystals of lower purity

1

and crystalline quality with intercrystal boundary zones of which the properties and behavior are little known. The sandstones, for example, are generally rich in quartz (up to 60%) which can be found in small crystallites as well as in amorphous form. All these facts may change significantly the physical properties of the rock from those of the pure crystal.

The thermal resistance (1/K) is the result of processes that allow the interchange of energy between the lattice waves (phonons). Real crystals contain irregularities of the lattice (impurities, vacancies, etc.) which can scatter the lattice waves.

Fourier's law states the relationship between the heat current density, the temperature and the thermal conductivity [1].

$$\vec{J} = -K \nabla T \tag{1}$$

Let's choose a point P(X,Y,Z,t) where we want to know the temperature and draw a rectangular parallelepiped around it with edges parallel to the coordinate axes and 2dX, 2dY, 2dZ in length. The rate at which heat will flow into the parallelepiped through the plan X-dX can be written as  $4(J_X - \partial J_X / \partial X \, dX) dY dZ$  were  $J_X$  is the X component of the heat flux at point P(X,Y,Z,t).

The heat flow into the parallelepiped through the plan X+dX can be written in the same way as:

The net heat flux that will enter the box from the X direction will therefore be the sum of heat flow through the two planes.

Using the same arguments on the other axes (Y and Z) will result in the same type of equation. Therefore the net heat entering the parallelepiped will be:

$$-8(\partial J_{x}/\partial X + \partial J_{y}/\partial Y + \partial J_{z}/\partial Z) dX dY dZ = -8 divJ dX dY dZ$$
(2)

The rate at which heat is gained in the paralleleiped can be written as:

$$8C \ \partial T/\partial t \ dX \ dY \ dZ \tag{3}$$

where C is the specific heat per unit volume. Since the flux of heat at a point across any surface is  $-K\partial T/\partial n$  ( $\partial/\partial n$  denotes differentiation in the direction of the outward normal). Therefore we can write:

$$J_{x} = -K \frac{\partial T}{\partial X}$$
$$J_{y} = -K \frac{\partial T}{\partial Y}$$
$$J_{z} = -K \frac{\partial T}{\partial Z}$$

In vector notation we get Fourier's law:

$$\mathbf{J} = -\mathbf{K} \text{ grad}\mathbf{T}$$
 (4)

By equating the heat gained to that entering the paralleleipeped (3) with (2) we get:

-8 divJ dX dY dZ = 8C 
$$\partial T/\partial t$$
 dX dY dZ  
Or: C  $\partial T/\partial t$  dX dY dZ + divJ dX dY dZ = 0 (5)

Substituting  $\vec{J}$  from (4) into (5) and assuming the solid to be homogeneous and isotropic gives the thermal diffusion equation:

$$\nabla^2 \mathbf{T} - \mathbf{C}/\mathbf{K} \ \partial \mathbf{T}/\partial \mathbf{t} = 0 \tag{6}$$

#### Thermal Diffusivity as a Function of Temperature

Investigations done by Eucken [2],[3] showed that the thermal conductivity of dielectric solids is usually inversely proportional to the absolute temperature T. According to the kinetic theory, the thermal conductivity of a gas can be written as:

$$K = 1/3 C V L$$
 (7)

where K is the thermal conductivity, C is the specific heat per unit volume, V is the mean velocity of the molecules and L is their mean free path. Debye [4] treated the lattice vibrations of the solid as a gas of phonons and expressed the thermal conductivity of a dielectric crystal as:

$$K = 1/3 \sum C_{y} V L_{y}$$
 (8)

where  $C_{\nu}$  is the specific heat of those phonons with frequency  $\nu$ , V is the velocity of sound in the crystal and  $L_{\nu}$  is the mean free path of the of frequency  $\nu$ .

According to Debye's model the sound velocity (V) is considered to be constant for all polarizations. Provided that the temperature is about Debye's temperature or higher the specific heat approaches a constant value. Therefore the thermal conductivity is proportional to the phonon mean free path (L). It is easy to show that the mean-freepath for phonon-phonon scattering should go as 1/T and therefore K $\propto$ 1/T.

The mean-free-path, L, is inversely proportional to the density of the scattering centers, n. Since the scattering centers are other phonons, the scattering density centers will be proportional to the number of the phonons:

$$n \propto 1/(e^{(\hbar\omega/kT)}-1) = kT/\hbar\omega$$
 for  $T >> \hbar\omega/k$ 

therefore, n is proportional to T. L is inversely proportional to T and so is the thermal conductivity.

The three phonon process in a crystal can be written as:

$$\vec{q}_1 + \vec{q}_2 = \vec{q}_3 + \vec{G}$$
 (9)

where  $\vec{q}_1$ ,  $\vec{q}_2$  and  $\vec{q}_3$  are wavevectors of the phonons and  $\vec{G}$  is reciprocal lattice vector.

When G=O the process is called normal; otherwise (G≠O) it is called Umklapp process. The three phonon process can be helpful in explaining the thermal properties of the crystal. The normal process does not change the momentum of the phonon and therefore does not change the heat flow through the crystal, i.e., it does not produce thermal resistivity. The Umklapp process does not conserve the total momentum of the phonon, therefore changing the heat flow through the crystal. The Umklapp process is capable of bringing the distribution of phonons into local thermal equilibrium. At high temperature (room temperature and above) most phonons have enough energy for the Umklapp process to take place. Therefore, this is the dominant process that affects the thermal resistivity (1/K) of the crystal.

The three phonon process is caused by the anharmonic coupling between different phonons. The theory of the anharmonic coupling predicts that the mean free path is proportional to 1/T at high temperature. The total number of the exited phonons at high temperature is proportional to T. The collision frequency,  $1/\tau$ , (for a given phonon) is proportional to the number of phonons that it can collide with.

Since  $L = V\tau$  and V is constant

5

 $L \propto 1/T$ 

Therefore, the thermal conductivity of a crystalline substance is expected to be proportional to 1/T.

On the other hand in amorphous substances the heat conduction is due to localized phonons. By increasing the temperature the number of the local phonons increases. The local phonons can hop from one site to another and thus increase the thermal conductivity of the material. Therefore, as opposed to the inverse T dependence of K in a perfect crystal, mechanism in amorphous substances lead to dependence proportional to T.

In a real rock the heat conduction can be affected by both the three phonon process (Umklapp process) as well as the localized phonons.

The thermal conductivity is related to the thermal diffusivity by:

$$\eta = K/C = 1/3 V L$$
(10)

where  $\eta$  is the thermal diffusivity, K is the thermal conductivity and C is the specific heat per unit volume.

Since the specific heat per unit volume of sedimentary rocks is about constant in the range 25-100  $^{\circ}$ C, the thermal diffusivity is predicted to vary as 1/T.

# $\eta \propto 1/T$

Thermal Diffusivity as a Function of Pressure

Petroleum reservoir rocks sometimes are found in great depths. Therefore, they are subjected to the pressure of the overlying rocks. The pressure on the rock at a depth of 1000 m will be about 230 At (assuming an average density of 2300 Kg/m<sup>3</sup> for the overlying rocks). Therefore, it is important to measure the thermodynamical properties of rocks under stress. Measurements of thermophysical properties of a sample under pressure are complicated. In nature the pressure on the rock is axial, but the rock is supported from its sides. In the laboratory when applying an axial pressure the sides of the rock have no support. The rock is composed of several minerals with pores between them. When pressure is applied to the rock the grains start to move toward each other, closing the pores. The mechanism is probably elastic but only to a certain point leading to micro cracks in the rock when additonal stress is applied. Applying more pressure leads to the collapse of the rock.

The thermal diffusivity of a given rock depends, at a constant temperature and pressure, on the mineralogical composition as well as on its porosity and pore-filling [5]. It is customary to subdivide the bulk into contributions due to the rock matrix (pure crystalline) and due to the pore space. It can depend on the type of pore fill (air, water, hydrocarbons, etc.) and on the geometrical configuration of the pores.

According to Ulrich [5] the porosity dependence of the thermal conductivity can be written as:

$$K = K_{p}^{\phi} K_{m}^{(1-\phi)}$$
(11)

where K is the bulk thermal conductivity,  $K_p$  is the pore thermal conductivity,  $K_m$  is the rock matrix (crystalline) thermal conductivity and  $\phi$  is the porosity.

This equation (11) was derived by Woodside and Messmer [6] assuming that the rock contains a volume fraction  $\phi$  of pores with thermal conductivity K<sub>p</sub> and 1- $\phi$  pure crystalline grains with thermal conductivity of K<sub>m</sub>. Since K<sub>p</sub> < K<sub>m</sub> we get K<sub>p</sub> < K < K<sub>m</sub>. By considering two phase distributions, the series and parallel, other limits may be set on K. These distributions correspond to minimum and maximum values of K.

In the series distribution, the two phases are thermally in series with respect to the direction of the heat flow. Therefore, the minimum value of the effective conductivity will be the weighted harmonic mean of the crystal and the pores.

$$K_{\min} = K_{m} K_{p} [K_{m} \phi + K_{p} (1-\phi)]^{-1}$$
(12)

In the parallel distribution, the phases are in parallel with respect to the heat flow direction. Therefore the weighted arithmetic mean of the two is the maximum value of the effective conductivity.

$$K_{\max} = K_p \phi + K_m (1 - \phi)$$
(13)

Taking the first derivative of K with respect to  $K_m$  (at the point where  $K_m = K_p$ ) for both equations gives the same result.

$$[dK/dK_m] = 1 - \phi \tag{14}$$

The fact that equation (14) is satisfied by the two distributions means that the conductivity equation for any phase distribution must also be satisfied by (14).

Woodside [6] chose as an intermediate distribution (11) the weighted geometric mean to represent the contribution of the crystalline and the pore to the whole conductivity. Equation (11) satisfies the required condition expressed by (14) and therefore can be a valid equation for the thermal conductivity.

Increasing the pressure on the rock will close the pores and decrease the thermal resistivity to heat flow, resulting in an increase in the thermal conductivity. Ulrich [5] also presented the same relation (11) between the thermal conductivity and the porosity. He showed a decrease in the thermal conductivity as the porosity increased.

#### CHAPTER II

## EXPERIMENTAL PROCEDURES

## Thermal Diffusivity Measurement

The thermal diffusivity can be measured directly using either one of two basic techniques: the steady-state [7] or the transient method [8], [9], [6]. The steady state method requires simultaneous measurements of the steady state heat flux and the temperature gradient across the sample. The divided-bar or thermal comparator is an example to this method [7]. The measurement requires relatively long times in order to get the steady state. In the transient method the sample is initially set in thermodynamic equilibrium. A pulse of heat (thermal energy) is supplied to one side of the sample creating a temperature gradient in the sample. The temperatures are measured at three different points and are used to calculate the thermal diffusivity. This method is much faster and has less restrictions on the sample. An example to this method is the needle-probe method [6] and the pulse method [8], [9].

The thermal diffusivity governs the temperature profile of a sample through the thermal diffusion equation (6). In our experiment we applied heat and checked the temperatures in one axis. Therefore, the thermal diffusion equation can be reduced to one dimension.

$$dT/dt = \eta d^2 T/dX^2$$

Using the pulse method [8], [9] the thermal diffusivity is determined. The heat losses through the lateral surface of the sample,

9

in the region of interest (about 10 mm) has little effect on the temperature distribution of the sample in the case of low thermal diffusion. Therefore, there was no need to correct for heat losses through radiation or convection.

In the pulse method there are two possible sources of error: 1. Systematic error due to the error in measuring the distances between the thermocouples. It can be of the order of 10% (measuring 5 mm with accuracy of 0.5 mm). This affects only the absolute value of the diffusivity and does not change with temperature or pressure. 2. Random errors due to the scattering of the experimental data with respect to the least square regression line. These can affect both, the relative and absolute values of the thermal diffusivity, and changes with temperature and pressure. The value of the random error (Sigma) was calculated for each experiment. The Sigma was 1% to 5% for the various experiments.

## Set-Up (Apparatus)

The experimental set up, as illustrated in Fig. 1, includes:

- An H.P. Integral Personal Computer to set the measurement parameters, start and control the experiment, collect, reduce, store and plot the data.
- 2. Digital Multimeter (x3) (Model 3478A) to measure the voltage produced by the thermocouples and output it, in digital form, to the computer (upon request). These D.V.M. allow measurement as sensitive as 0.1mV (0.003 K)
- 3. D.C. Power Supply (H.P. Model 16271B) to give the energy to the heater.

- 4. Data Acquisition Control Unit (H.P. Model 4321A) to time the heater.
- 5. Computerized Temperature Controller (Omega CN-2000) to set and control the temperature of the ambient , around the sample.
- Hydraulic hand pump press (ENERPAC Model P-141) to set load on the sample.
- 7. Thermocouples (x3) type K to measure the temperatures gradient at three different point on the sample.
- Electronic Ice Points (X3, Omega Model MCJ) to set an electronic reference point to the thermocouples.
- 9. Cylindrical Furnace to heat the ambient (around the rock) and set the temperature of the whole rock.
- 10. Heater to provide a pulse of heat to the sample and enable the thermal diffusivity measurement.
- 11. Sample holder and housing.
- 12. The sample itself.



Figure 1: Block Diagram of the Experimental Apparatus

#### Measurement Procedure

#### A. System Preparation

The system was built from two basic components: hardware and software. The hardware, as described in the previous section, contains the setups (hydraulic press, temperature controller) and the measuring devices (D.V.M., computer). The system described by French [9] was designed to measure the thermal diffusivity at atmospheric pressure only. For the present study it was necessary to modify the system and allow pressure to be applied to the sample with an hydraulic press. Heat conduction through the press head limited the temperature that could be safely reached on the sample to about 100 <sup>o</sup>C. The hydraulic press was brought to a mode were it can hold the pressure required to put on the sample for the measuring cycle. The temperature controller was also modified so that it could be remotely programmed using the computer. The temperature controller was wired to the computer and the heater as illustrated in Appendix C. In order to set and check the ambient temperature the operating program and several subroutines were modified and a subroutine (CONTSET) was added to it. Some other changes were made in the programs in order to simplify the names of the experiments, allow stronger interaction with the experiment conducted and record bigger records of information about the experiments performed. The programs and subroutines used in order to perform the measurement and reduce the data are added as Appendix D.

13

#### B. Sample Preparation

The samples were sedimentary rocks in the shape of cylinders, 3 to 5 cm in length and 2.5 cm in diameter. The bases of the cylinders were cut in parallel using a special holder designed to get parallel bases (Appendix B). Three thermocouple wells 0.9 mm in diameter and 8 mm deep were drilled at 5 mm intervals along the long axis of the rock. The distance between the thermocouples was chosen to be 5 mm in order to allow response to the pulse of heat by all three thermocouples, when measuring thermal diffusivity of the order of 0.01 cm<sup>2</sup>/s. A heat shrink sleeve (CLEAR FPS-096-4005-CLR) was used to support the sample and hold the rock to its place. The heat shrink sleeve, 2 inches in diameter, was cut 5 mm longer then the rock, set over the rock and heated (150 °C for 25 minutes) in a furnace . Three holes were drilled in the heat-shrink above the wells in the rock.

#### C. The Measurement

The main program (TDFM1) was loaded and run. The parameters for the experiment (control and working set-ups) were chosen and set. The rock was brought to thermodynamic steady state at the temperature and pressure where the thermal diffusivity was requiered. The computer triggered the D.V.M and recorded the initial temperatures of the rock. The heater, located at one end of the rock, was turned on causing a heat flow through the rock. After a delay of 2 to 10 seconds a series of simultaneous temperature readings was collected for 40 s, in intervals of 0.5 s, and stored in the computer. When the heater was turned on a pulse of 2 W was applied to the heater causing a raise of 10 to 14  $^{\circ}$ C in

the reading of the thermocouple nearest to the heater. Then the heater was turned off allowing equilibrium to be re-established in the rock. While awaiting equilibrium, the computer reduced the data of the temperatures, calculated the thermal diffusivity to be displayed, plotted (upon request) and stored them on a diskette.

The data reduction process started by converting the voltages to temperatures. To eliminate mismatch between the thermocouples, the initial temperature was subtracted from each point. Then the thermal diffusion equation, in one dimension, was numerically solved. The temperatures and the thermal diffusivity were stored on the diskette and we were able to see and plot them. Fig. 2,3 represent the temperature gradient in the sample and the thermal diffusivity calculated based on it.

15



Figure 2. The Temperature Gradient in a Dolomite Stone



Figure 3. Thermal Diffusivity of a Dolomite Stone

#### Problems In Performing The Measurements

The first problem we faced was the hand press not holding the pressure it was supposed to set on the sample. We found that within an hour the pressure dropped by about 50% from the initial pressure. To overcome this problem a needle valve, which can hold pressures up to 10000 PSI (ENERPAC V-8), was added to the system. It was added in series with the hydraulic press lines. By adding the valve the pressure set on the rock could be locked.

The second problem was due to the heat we applied to the system. By heating the ambient around the sample the sample holder and part of the press expended. As a result the pressure on the sample increased. To overcome this problem the pressure needed to be reduced each time before making the measurements. This problem caused, once, the pressure to exceed the limits of the rock resulting in its collapse.

The main problem we faced was the resistance of the rock to uniaxial pressures. Most of the rocks broke at pressures less than 200 At. To protect the rocks and enable measurements, at limited pressures, up to 1500 PSI (without breaking the rock) we prepared the rocks in the following way:

1. Cut the bases of the cylinders parallel.

2. Cover the rock with heat shrink sleeve to support the outer walls and hold the rock to its base on the heater.

These two steps, mentioned above, were done for each rock in the preparation phase. More detail in Appendix A.

# CHAPTER III

# RESULTS

The thermal diffusivity of 10 different rocks was measured as a function of temperature and pressure. The settings of the various experiments is detailed in Table I.

## TABLE I

# SETTING OF THE EXPERIMENTS

ROCK DESCRIPTION		MEASUREMENT						RANGES			
		RANGE OF	PRESSURES WHERE TEN					4P. (25-100 °C)			
SYMBOL	TYPE	At 25 <sup>o</sup> C	14.5	400	500	600	700	800	1000	1300	
BSH1	Berea Sandstone Horizontal bed	14.5-1500	+	+					+		
BSH4	Berea Sandstone Horizontal bed	14.5-1500	+	+			+				
BSV1	Berea Sandstone Vertical bed	14.5-1500	+	+				+			
C22.1	sandstone	14.5-1500	+			+					
L150	Limestone	14.5-1500	+					     			
LI65	Limestone	14.5-1500	+		+			+	+	+	
L166	Limestone	14.5-1500	+	+					+		
MCA362	Dolomite	14.5-2500	+	+				   +	+	+	
MXN01	Dolomite	14.5-2500	+	+	+	+			+	+	
MXN04	Dolomite	14.5-2500	+			   					

## Rock BSH1

The thermal diffusivity of rock BSH1 as a function of pressure (14.5-1500 PSI) was measured at room temperature in two consecutive cycles (Fig. 4). The thermal diffusivity increased from 0.0081 to 0.0090 cm<sup>2</sup>/s as the pressure increased and recovered to its initial value upon reducing the pressure. The results maintained the same value in all the cycles.



Figure 4. Thermal Diffusivity as a Function of Pressure for Rock BSH1

Using equation (11) and assuming that the rock contains crystalline grains of  $SiO_2$  and air at the pores the porosity was calculated. It decreased from 27% to 25% while the pressure increased from 14.5 to 1500 PSI, and increased back to 27% upon reducing the pressure (Fig. 5).



Figure 5. Porosity as a Function of Pressure for Rock BSH1

At three pressures (14.5, 400 and 1000 PSI) the thermal diffusivity was measured as a function of temperature (25-100  $^{\circ}$ C). The thermal diffusivity decreased by 24%-28%. These slopes (Fig. 6) are slightly bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. Normalizing the thermal diffusivity as function of temperature at 400 and 1000 PSI with the one measured at 14.5 PSI gave a negative slope of about 20% (400 PSI) for the first cycle and 10% for the other two (1000, 400 PSI). The bias factor decreased from 1.18 to 0.95 in the last two .



Figure 6. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock BSH1

# Rock BSH4

The thermal diffusivity of rock BSH4 was measured as a function of pressure (14.5-1500 PSI) at room temperature in two consecutive cycles (Fig. 7). The thermal diffusivity increased from 0.012 to 0.016 cm<sup>2</sup>/s. By reducing the pressure back to 14.5 PSI the thermal diffusivity recovered to its initial value (0.012 cm<sup>2</sup>/s).



Figure 7. Thermal Diffusivity a as Function of Pressure for Rock BSH4

Using equation (11) and assuming that the rock contains crystalline grains of  $SiO_2$  and air at the pores the porosity was calculated. It decreased by about 30% changing from 20% to 14% and recovered to 14% upon releasing the pressure (Fig. 8).



Figure 8. Porosity as a Function of Pressure for rock BSH4

At three pressures (14.5, 400 and 700 PSI) the thermal diffusivity as a function of temperature (25-100  $^{\circ}$ C) was checked. It decreased by 27% to 30% (Fig. 9). These slopes are bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. Normalizing the thermal diffusivity as a function of temperature at 400 and 700 PSI with the one got at 14.5 PSI gave a negative slope of 10% to 20%. The normalization factor was found to change from 1.4 to 1.2 for 400 PSI and from 1.3 to 1.2 for 700 PSI.



Figure 9. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock BSH4

#### Rock BSV1

The thermal diffusivity of rock BSV1 as a function of pressure (14.5-1500 PSI) at room temperature was measured in 3 sequential cycles (Fig. 10). The thermal diffusivity increased as the pressure increased and decreased to its initial value, upon reducing the pressure. The thermal diffusivity increased by 20% from 0.0115 cm<sup>2</sup>/s at atmospheric pressure to 0.0138 cm<sup>2</sup>/s at 1500 PSI.



Figure 10. Thermal Diffusivity as a Function of Pressure for Rock BSV1

Using equation (11) and assuming that the rock contains crystalline grains of  $SiO_2$  and air at the pores, the porosity was calculated. It decreased by about 20% (Fig. 11), from 21% to 17%.



Figure 11. Porosity as a Function of Pressure for Rock BSV1

At three pressures (14.5, 400 and 800 PSI) the thermal diffusivity was checked as a function of temperature (25-100  $^{\circ}$ C). The thermal diffusivity decreased by about 30%. These slopes (Fig. 12) are bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. Normalizing the thermal diffusivity as a function of temperature at 400 and 800 with the one at 14.5 PSI gave a constant with normalization factor of 1.1.



Figure 12. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock BSV1
#### Rock C22

The thermal diffusivity of rock C22 as a function of pressure (14.5-1500 PSI) was measured at room temperature. Four cycles of pressure were applied to the rock (Fig. 13). The first 3 cycles were performed one immediately after the other, but the fourth was applied after 4 months. In all the cycles the thermal diffusivity increased as the pressure increased, but the increasing rate decressed in the sequent cycles (12%, 6% and 3% in the third). In the last cycle which was performed after four months the rate came back to 10%. The thermal diffusivity at atmospheric pressure, increased in the subsequent cycles from 0.0103  $cm^2/s$  in the first cycle to 0.0128  $cm^2/s$  in the last one.



Figure 13. Thermal Diffusivity as a Function of Pressure for Rock C22

Using equation (11) and assuming that the rock contains crystalline grains of  $SiO_2$  and air at the pores, the porosity was calculated. It decreased by less than 10% each cycle, changing from 22.4% to 20.4% in the first cycle, from 20.9% to 19.8% in the second and from 20.8% to 20.4% in the third cycle. In the fourth cycle the porosity decreased from 18.5% to 16.8% and was found to recover to 18% when the load was released (Fig. 14).



Figure 14. Porosity as a Function of Pressure for Rock C22

At two pressures (14.5 and 600 PSI) the thermal diffusivity was checked as a function of the temperature (25 to 100 °C). It decreased by 26%-27%. These slopes (Fig. 15) are slightly bigger than 1/T at that range. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. Normalizing the thermal diffusivity as a function of temperature at 600 PSI with the one got at 14.5 PSI gave a constant with normalization factor of 1.1.



Figure 15. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock C22.

## Rock L150

The thermal diffusivity of rock L150 as a function of pressure (14.5-1500 PSI) was measured at room temperature in two consecutive cycles (Fig. 16). The thermal diffusivity increased from 0.0088 to 0.012  $\rm cm^2/s$  in the first cycle and from 0.010 to 0.013  $\rm cm^2/s$  in the second cycle, as an "hyperbola". At the end of the second cycle (after measuring the thermal diffusivity three times) at 1500 PSI the sample broke.



Figure 16. Thermal Diffusivity as a Function of Pressure for Rock L150

Using equation (11) and assuming that the rock contains crystalline grains of  $CaCO_3$  and air at the pores the porosity was calculated. It decreased from 20% to 13% in the first cycle and from 17% to 12.5% in the other (Fig. 17).



Figure 17. Porosity as a Function of Pressure for Rock L150

The thermal diffusivity as a function of temperature (25-100  $^{\circ}$ C) was checked at atmospheric pressure. It was found to decrease by 36% (Fig. 18). This slope is bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. It was found to decrease from 3.2 to 2.7.



Figure 18. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock L150

## Rock L165

The thermal diffusivity of rock L165 as a function of pressure (14.5-1500 PSI) was measured at room temperature in three consecutive cycles (Fig. 19). The thermal diffusivity increased from 0.0071 to 0.0113 cm<sup>2</sup>/s in the first cycle, from 0.0091 to 0.0111 cm<sup>2</sup>/s in the second and from 0.0096 to 0.0111 cm<sup>2</sup>/s in the last one. The shape of the change was like an "hyperbola". The values of the thermal diffusivity at atmospheric pressure increased in each sequent cycle (hysteresis). It increased by 28% between the first and the second cycles and by 5% between the second and the last cycle.



Figure 19. Thermal Diffusivity as a Function of Pressure for Rock L165

Using equation (11) and assuming that the rock contains crystalline grains of  $CaCO_3$  and air at the pores the porosity was calculated. The porosity decreased from cycle to cycle, at atmospheric pressure, and within each cycle. It decreased, as the pressure increased, from 24.4% to 15.3% in the first cycle, from 19.6% to 15.6% in the second and increased in the third cycle to 18.5% upon releasing the pressure (Fig. 20).



Figure 20. Porosity as a Function of Pressure for Rock L165

At five pressures (14.5,500,800,1000 and 1300 PS1) The thermal diffusivity was checked as a function of temperature (25-100  $^{\circ}$ C). The thermal diffusivity decreased by 24% to 33%. These slopes (Fig. 21) are bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. The factor as function of temperature changed from 2.2 to 2.1, for 14.5, and from 3.5 to 2.7 for 1300 PS1. All the thermal diffusivities were normalized relativly to the thermal diffusivity at atmospheric pressure. The normalization factor changed from 1.4 to 1.6 as increasing the pressure but the slopes maintained about the same value for all pressures. The normalization factor



Figure 21. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock L165

# Rock L166

The thermal diffusivity of rock L166 was measured as a function of pressure (14.5 to 1500 PSI) at room temperature (Fig. 22). The thermal diffusivity increased from 0.0065 to 0.00925 cm<sup>2</sup>/s like an "hyperbola" while the pressure increased, and decreased to 0.00755 cm<sup>2</sup>/s (with an hysteresis) upon releasing the pressure.



Figure 22. Thermal Diffusivity as a Function of Pressure for Rock L166

Using equation (11) and assuming that the rock contains crystalline grains of  $CaCO_3$  and air at the pores the porosity was calculated. The porosity decreased from 26.2% to 19.2% when the pressure cycle was set and reduced to 23.2% upon reducing the stress (Fig. 23).



Figure 23. Porosity as a Function of Pressure for Rock L166

At three pressures (14.5, 400 and 1000 PSI) the thermal diffusivity as a function of the temperature (25-100  $^{\circ}$ C) was measured. The thermal diffusivity decreased by 32% to 38% as the temperature was increased. These slopes (Fig. 24) are bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. The slopes were about constant but the bias changed with the pressure from 2.3 to 2.8. Normalizing the thermal diffusivity as a function of temperature at 400 and 1000 PSI with the one got at 14.5 PSI resulted in two slopes. For 400 PSI the normalization factor changed from 1.1 to 0.97 and for 1000 PSI it changed from 1.22 to 1.15.



Figure 24. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock L166

#### Rock MCA362

The thermal diffusivity of rock MCA362 was measured as a function of pressure (14.5-2500 PSI) at room temperature in 3 consecutive cycles (Fig. 25). The thermal diffusivity increased from 0.0115 cm<sup>2</sup>/s at atmospheric pressure to 0.013 cm<sup>2</sup>/s (at 2500 PSI). By reducing the pressure to 14.5 PSI the thermal diffusivity recovered to its initial value.



Figure 25. Thermal Diffusivity as a Function of Pressure for Rock MCA362

Using equation (11) and assuming that the rock contains crystalline grains of  $CaCO_3$  and air at the pores the porosity was calculated. Upon increasing the pressure to its maximum value (2500 PSI) the porosity decreased from 15% to 13% and recovered to 15% as the pressure was released (Fig. 26).



Figure 26. Porosity as a Function of Pressure for Rock MCA362

At six pressures (14.5,400,800,1000,1300 and 1500 PSI) the thermal diffusivity as a function of temperature (25-100  $^{\circ}$ C) was checked. The thermal diffusivity decreased by 25% to 32%. These slopes (Fig. 27) are bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. It decreased from 3.6 to 3.3 cm<sup>2</sup>/(s K) for 14.5 PSI and from 3.7 to 3.3 for 1000 PSI. Normalizing the thermal diffusivity as a function of temperature at the higher pressures with the one got at 14.5 PSI gave a normalization factor which changed from 1.1 to 0.9.



Figure 27. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock MCA362

## Rock MXN1

The thermal diffusivity of rock MXN1 as a function of pressure (14.5-2500 PSI) was measured at room temperature in 3 sequential cycles (Fig. 28). The thermal diffusivity changed by about 5% when pressure was applied to the sample. It increased from 0.0229 to 0.0235 cm<sup>2</sup>/s in the first cycle, slightly decreased from 0.0180 to 0.0177 cm<sup>2</sup>/s in the second and increased from 0.0211 to 0.0224 cm<sup>2</sup>/s in the last cycle.



Figure 28. Thermal Diffusivity as a Function of Pressure for Rock MXN1

Using equation (11) and assuming that the rock contains crystalline grains of  $CaCO_3$  and air at the pores the porosity was calculated. It decreased from 1.4% to 0.9% in the first cycle, increased from 6.2% to 6.5% in the second and decreased again from 3.0% to 1.9%. It raised to 2.8% when the load was released (Fig. 29). Within the experimental error we can say that it maintained the value of 4%, regardless of the pressure.



Figure 29. Porosity as a Function of Pressure for Rock MXN1

At seven pressures (14.5, 400, 500, 600, 1000, 1300 and 1800 PS1) the thermal diffusivity as a function of temperature (25-100  $^{\circ}$ C) was measured. The thermal diffusivity decreased by 30% to 36%. These slopes (Fig. 30) are bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. The multiplication resulted in a slope of 10% around the value of 6 to 6.5. Normalizing the thermal diffusivities as a function of temperature at 400, 500, 600, 1000, 1300 and 1800 PSI with the one got for 14.5 PSI resulted in values from 1 to 1.3.



Figure 30. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock MXN1

#### Rock MXN4

The thermal diffusivity of rock MXN4 was measured as a function of pressure (14.5-2500 PSI) at room temperature in two consecutive cycles (Fig. 31). The thermal diffusivity decreased in the first cycle from 0.0238 to 0.0216 cm  $^2$ /s and from 0.0235 to 0.022 cm $^2$ /s in the second and maintained about the same value upon decreasing the pressure to 500 PSI.



Figure 31. Thermal Diffusivity as a Function of Pressure for rock MXN4

Using equation (11) and assuming that the rock contains crystalline grains of  $CaCO_3$  and air at the pores the porosity was calculated. It increased from 0.9% to 2.5% in the first cycle and from 1.2% to 2.7% in the last one (Fig. 32).



Figure 32. Porosity as a Function of Pressure for Rock MXN4

At a pressure of 14.5 PSI the thermal diffusivity as a function of the temperature (25-100  $^{\circ}$ C) was measured (Fig.33). The thermal diffusivity decreased by 24% with increasing the temperature to 100  $^{\circ}$ C. This slope (Fig. 33) is bigger than 1/T. Plotting the function  $\eta$ T also showed that the thermal diffusivity decreased faster than 1/T. The result of the multiplication was a constant of 7.2.



Figure 33. Thermal Diffusivity as a Function of Temperature, at Various Pressures, for Rock MXN4

The results are summarized and represented in the following Table.

#### TABLE II

# SUMMARY OF THERMAL DIFFUSIVITY AS A FUNCTION OF TEMPERATURE AND PRESSURE

ROCK Symbol	Thermal Diff. as a function of Pressure 14.5 to Max.	% Porosity as a function of Pressure 14.5 to Max.	Thermal Diff. as a function of Temperature 25 to 100 <sup>O</sup> C	Values of Thermal Diff. Multiplied by Temperature
BSH1	.00810090 .00820090(B) .0082	26.9-24.9 26.6-24.9 26.6	.00980074 .01040075 .00890070 .00830063	2.9-2.7 (14.5) 3.1-2.8 (400) 2.7-2.6 (1000) 2.5-2.4 (400)
BSH4	.01190160 .01180160(B)	19.8-14.4 20.0-14.4	.0157011 .01200098 .01440108	4.7-4.2 (700) 3.6-3.3 (14.5) 4.3-4.0 (400)
BSV1	.01140135 .01130138 .01160138(B)	20.6-17.5 20.8-17.1 20.3-17.1	.01140080 .01200083 .01210084	3.4-3.1 (14.5) 3.5-3.1 (400) 3.6-3.1 (800)
C22.1	.01030115 .01120119 .01130115 .01280140 .0134	22.4-20.4 20.9-19.8 20.8-20.4 18.5-16.8 17.6	.01120083 .01280093 .01350097	3.4-3.1 (14.5) 3.8-3.5 (14.5) 4.0-3.7 (600)
L150	.00880125 .01020130 Sample broke	20.2-13.3 17.3-12.5	.01040071	3.1-2.6 (14.5)

Table II (continued)

ROCK Symbol	Thermal Diff. as a function of Pressure 14.5 to Max.	% Porosity as a function of Pressure 14.5 to Max.	Thermal Diff. as a function of Temperature 25 to 100 <sup>O</sup> C	Values of Thermal Diff. Multiplied by Temperature
L165	.00710113 .00910111 .00960111(B)	24.4-15.3 19.6-15.6 18.5-15.6	.00950072 .01070075 .01100075 .01110078 .00970065	2.9-2.6 (500) 3.2-2.8 (800) 3.4-2.8 (1000) 3.4-2.9 (1300) 2.9-2.5 (14.5)
L166	.006500925 .00755	26.3-19.2 23.2	.00790049 .00930060 .00760052	2.4-1.8 (400) 2.8-2.3 (1000) 2.3-1.9 (14.5)
MCA362	.01160128 .01150133 .0119 .01140128	14.8-12.8 15.0-12.1 14.3 15.1-12.8	.01160079 .01250088 .01190089 .01170089 .01210090 .01210091 .01180093 .01220091	3.5-2.9 (14.5) 3.7-3.3 (1000) 3.6-3.3 (14.5) 3.5-3.3 (400) 3.6-3.4 (800) 3.6-3.4 (1000) 3.5-3.5 (1300) 3.7-3.4 (1500)
MXN1	.02290235 .01800177 .02110224 .0214	1.4-0.9 6.2-6.5 3.0-1.9 2.8	.022014 .021016 .021015 .022016 .022017 .023016	6.6-5.2 (14.5) 6.3-6.0 (400) 6.3-5.6 (600) 6.6-6.0 (1000) 6.3-6.3 (1300) 6.9-6.0 (1800)
MXN4	.02380220 .02400224	0.9-2.5 1.2-2.7	.024019	7.2-7.1 (14.5)

#### CHAPTER IV

#### DISCUSSION

Thermal Diffusivity as a Function of Pressure

The thermal diffusivity as a function of pressure was checked for 10 sedimentary rocks: sandstones (Berea: BSH1, BSH4, BSV1 and C22), 1imestones (L150, L165, L166) and Dolomites (MCA362, MXN1, MXN4) (Table II). In general the thermal diffusivity positively followed the changes in the pressure. Similar response was also observed by Ashworth [10] who found in a limestone rock an increase of 6% (0.0257-0.02727 W(cm K)<sup>-1</sup>) in the thermal conductivity upon increasing the pressure from 177 to 2653 PSI.

The thermal diffusivity of three Berea Sandstones (BSH1, BSH4, BSV1) was measured as a function of pressure at room temperature. All these rocks responded similarly. As the pressure increased the thermal diffusivity increased and decreased to the initial value upon reducing the pressure. The thermal diffusivity increased by 10% to 40% in the various rocks (BSH1, BSV1, BSH4) as the pressure increased to 1500 PSI. The value of the thermal diffusivity at atmospheric pressure was about the same  $(0.011, 0.012 \text{ cm}^2/\text{s})$  for BSH4, BSV1 and about 30% smaller  $(0.081 \text{ cm}^2/\text{s})$  for BSH1. These differences in the response of the rocks can be related to the different histories they probably experienced. The different magnitude in the response of BSH4 and BSV1 is probably due to experimental errors. In rock C22 the thermal diffusivity in the first

cycle increased from 0.0103 to 0.0115  $cm^2/s$  by increasing the pressure to 1500 PSI, but reduced to 0.0112 upon reducing the pressure. This hysteresis was found to decrease and disappear in the consecutive cycles. The rate of increase (slope) of the thermal diffusivity decreased and disappeared in the sequential cycles. These two findings indicate the closing of the pores. Therefore, the thermal diffusivity did not change as the pressure on the rock was increased in the sequential cycles. In an experiment that was conducted after four months, the hysteresis and the change in thermal diffusivity as function of pressure were the same as in the first cycle. These findings may point to a recovery mechanism in the rock.

In the group of Dolomites three rocks (MCA362, MXN1 and MXN4) were checked. In the MCA362 rock the thermal diffusivity increased from  $0.0116 \text{ cm}^2/\text{s}$  by 10% when the pressure was increased to 2500 PSI and reduced to the initial value upon decreasing the pressure to atmospheric pressure. In the sequential cycles the same results were obtained. The same result was also obtained in the last experiment conducted five months later. These findings may point out that the motion of the grains within the rock was elastic up to 2500 PSI and that no plastic deformation or micro-cracks occured. The results for the MXN type were not consistent. The thermal diffusivity increased by 3% in the first cycle, decreased by 1% in the second and increased again by 5% in the last cycle. With rock MXN4 the thermal diffusivity decreased from 0.024 $cm^2/s$  by 7% at 2500 PSI. The changes are in the experimental error of the measurement and, therefore, we can say that the thermal diffusivity is about  $0.023\pm0.001 \text{ cm}^2/\text{s}$  (within the experimental error) regardless of the pressure.

The thermal diffusivities of 3 limestone rocks: L150, L165 and L166 were measured. Essentially the rocks responsed in a similar way. Two phenomena were observed: the thermal diffusivity increased as an "hyperbola" upon increasing the pressure and decreased to a higher value than the initial, when the pressure was reduced (hysteresis). The size of the hysteresis decreased in the sequential cycles from 30% to less than 5%. The thermal diffusivity at maximum pressure (1500 PSI) was similar in all cycles. Upon increasing the pressure to 1500 PSI the thermal diffusivity increased by 40% in the first cycle and 30% in the following cycles. The apparent differences in the thermal diffusivity measured for the three rocks may result from experimental errors. Assuming that the three rocks are from the same batch (place, depth and sharing the same history), the thermal diffusivity at standard conditions (room temperature and atmospheric pressure) was 0.0075±0.0009  $cm^2/s$ . One of the rocks, L150, broke during the experiment after being held under the high pressure (1500 PSI) for about 2 hours. Using all these facts one can try and explain what happens in the rock when pressure is applied. The grains are probably condensed by the pressure closing the pores between them and thereby decreasing the thermal resistance to heat flow. This process is limited by the size of the pores. Increasing the pressure causes the grains to start touching each other. This process is probably elastic to a certain point where microcracks start to appear. The elasticity of the process is, probably decreasing in the consecutive cycles. Additional increase in the pressure will not change the thermal diffusivity, but will result in breaking the rock, as happened in the case of L150. The decrease in the

elasticity can explain why by reducing the pressure the thermal diffusivity reduced to a higher value than the initial one. Repeated pressure cycles probably closed most of the gaps and therefore, the hysteresis in the thermal diffusivity was reduced, the changes in the thermal diffusivity become smaller and finally vanished.

The crystalline  $SiO_2$  is the main mineral (60% to 70%) in sandstone. In limestones and Dolomites  $CaCO_3$  is the main mineral (70%-80%). Both have about the same thermal conductivity [11]. The porosity was calculated by assuming that the main filling of the pores is air, using equation (11) and values from [11]. In general, the porosity changed in the opposite direction to the thermal diffusivity. The porosities in the first cycle at room temperature and atmospheric pressure were 20%-27% for the sandstones, 20%-26% for the limestones and 1%-15% for the Dolomites. Woodside [6] obtained similar results for the porosity of Berea Sandstone (22%). By increasing the pressure the porosity of all the rocks, except MXN1 and MXN4, decreased as predicted. The rate and shape were different for the various rocks. In the Berea Sandstones the porosity decreased: from 20% to 14% for BSH4, from 21% to 17% for BSV1 and from 27% to 25% for BSH1. These results lead to the previous conclusion: the history of BSH1 is probably different (location, depth) from that of BSH4 and BSV1. In rock C22 the porosity decreased from 22% by 8% in the first cycle and increased to 21% upon releasing the pressure. In the following cycles the porosity maintained the same value, 20.5%, (within the error limits) regardless of the pressure . In the Dolomite, MCA362, the porosity decreased from 15% to 12% upon increasing the pressure. For the MXN the porosity changed around 4%, within the error limit of the measurements, with no distinct direction.

In the limestones the porosity in the first cycle, at room temperature and atmospheric pressure, varied from 20% to 26%. By increasing the pressure the porosity decreased with an hysteresis and increased to a lower value (than the initial) upon releasing the pressure. In the sequential cycles the rate of decrease in the porosity was smaller. These results indicate a decrease in the volume fraction of the pores as pressure is applied.

## Thermal Diffusivity as a Function of Temperature

The thermal diffusivity was found to decrease as the temperature increased from 25 to 100  $^{\circ}$ C. It decreased by 24% to 36% according to the rock and pressures. The mean-free-path in the solid can be approximated by the equation:

# $L = 3 \eta/V$

where L is the mean-free-path,  $\eta$  is the thermal diffusivity and V is the wave velocity in the rock ( $5 \times 10^5$  cm/S in quartz [12]). Using this equation and thermal diffusivities of  $1 \times 10^{-2}$  cm<sup>2</sup>/s one can obtain the mean-free-path to be  $0.6 \times 10^{-7}$  cm. The grain size (10-30  $\mu$ m) is 4 orders bigger, therefore, we can assume that most of the scattering occurs inside the crystal grains. Therefore, we can predict the thermal diffusivity to decrease as 1/T when raising the temperature. In the range 25-100 °C 1/T decreases by 20% linearly. We found the thermal diffusivity to decrease, in the range 25-100 °C, by 24% to 36% for the different rocks and pressures. No simple relation was found between the the rate of decrease (slope) to the pressure set on the rock. The thermal diffusivity in all the rocks decreased as 1/T or faster. In an effort to understand the relationship between the thermal diffusivity and the temperature we plotted the thermal diffusivities multiplied by the temperature as a function of the temperature (Fig. 34,35). The slopes were negative, indicating that  $\eta$  changes faster than 1/T, and varied from rock to rock and for each rock at the various pressures. No simple relation between the slopes and the pressure applied to the rock was found.

Another relation we looked for was the nature of the change in the thermal diffusivity as a function of the pressure normalized with the value got at 14.5 PSI (Fig. 36,37). The slopes were found to be negative and to vary from 3% to 15%. No simple relation was found in this study.

Another function we tried to fit to the set of data was

$$\eta(T) = \eta(300) [300/T]^{\gamma}$$

looking for a relation between  $\gamma$  and the pressure. Sweet [13] made measurements on rocksalt (NaCl) and found the value of  $\gamma$  to be 1.2. For L165 we got  $\gamma$  to increase from 1.47 (at 14.5 PSI) to 1.77 (at 1000 PSI). For L150  $\gamma$  was found to be 1.5 (at 14.5). For other rocks the value of the  $\gamma$  did not show a simple relation to the pressure set.

These findings lead to the conclusion that in all the rocks the grains were of the crystalline type (rather than amorphous) and that the thermal diffusivity was limited within the grains.







Figure 35. Thermal Diffusivity Multiplied by Temperature, Rock C22



Figure 36. Thermal diffusivity at different pressures normalized with the one at 14.5 PSI, Rock L166



Figure 37. Thermal diffusivity at different pressures normalized with the one at 14.5 PS1, Rock BSV1

#### CHAPTER V

#### SUMMARY, CONCLUSIONS AND SUGGESTIONS

The thermal diffusivity of ten sedimentary rocks: sandstones (BSH1, BSV1, BSH4, C22), limestones (L150, L165, L166) and Dolomites (MCA362, MXN1, MXN4) was measured as a function of pressure and temperature (25-100 °C), using the pulse method [8], [9]. The pressure on the rock was increased (up to 1500 PSI for sandstones or limestones and up to 2500 PSI for the Dolomites) and the thermal diffusivity checked. For all the rocks (except MXN1, MXN4) the thermal diffusivity increased as the pressure increased. In the Dolomites MXN type it maintained the same value (within the experimental error) 0.023±0.001 cm<sup>2</sup>/s regardless of the pressure (14.5-2500 PSI). In the other Dolomite, MCA362, it increased from 0.0116  $cm^2/s$  by 10% while the pressure increased to 2500 PSI. In the Berea Sandstones the thermal diffusivity increased from  $0.008 \text{ cm}^2/\text{s}$  by 12% in BSH1 and from  $0.012 \text{ cm}^2/\text{s}$  by 33% (upon increasing the pressure to 1500 PSI) in the other rocks. In the limestones the thermal diffusivity increased in the first cycle from an average value of 0.0075±0.0009 cm<sup>2</sup>/s by about 50% as an "hyperbola" upon increasing the pressure to 1500 PSI. The process was found to have an hysteresis upon releasing the pressure. The hysteresis decreased and disappeared in the sequential cycles.

The porosity of the rocks was calculated assuming a simple structure (pure crystalline grains and pores filled with air) for the

rocks. The porosity changed oposite to the thermal diffusivity. For all the rocks, except the MXN type, the porosity decreased as the pressure increased. The porosity at room temperature and atmospheric pressure was 4% for the MXN type, 15% for the MAC362 and 20% to 30% for all the other. In the limestones and C22 an hysteresis effect was found upon releasing the pressure in the thermal diffusivity as well as in the porosity.

The thermal diffusivity as a function of the temperature (25-100 <sup>o</sup>C) was checked at several pressures. For all the rocks the thermal diffusivity decreased as 1/T or faster, regardless of the pressure. No consistent corrolation between the pressure and the slope was found. This finding indicates that the grains are crystalline type (rather than amorphous) and that the heat conduction is limited within the grains.

The results are reproducible within a random error (sigma) of less than 5%.

#### Suggestions for Future Research:

- Perform additional experiments with larger quantities of samples from the same type and batch (sharing the same history). This way the results and conclusions can be established to a greater accuracy. For each sample the results should be related to the history (location, depth, bed, etc.) of the sample.

- Perform the measurements on the rocks as close as possible (within a month) to the time they were taken out from their natural place, before relaxation or recovery process can take place.

- Conduct experiments of the thermal diffusivity as a function of the pressure at the same temperatures, for the different rocks and study the

thermal diffusivity as a function of the pressure at the various temperatures. Check how the thermal diffusivity as a function of pressure varies with the temperature.

- Measure the thermal diffusivity as a function of temperature at higher temperatures (up to 300  $^{\rm O}$ C). Study the response of thermal diffusivity as a function of temperature and pressure at elevated temperatures. Check whether the thermal diffusivity at these higher temperatures still decreases faster than 1/T.

- Conduct measurements on rocks saturated with liquid (water, oil, etc.) in the pores. Under these conditions the porosity can be calculated independently and more accurately by weighing the sample before and after setting pressure on it. This will allow us to test the results gotten by measuring the thermal diffusivity (Eq. 11).

- In order to set and hold a desired pressure on the sample more accurately some work must be done to improve the press.

- In order to set, maintain and record the pressure set on the sample during the experiment computer controlled devices are required. It can be done by setting strain gauges, for example, on the rocks (to check the pressure) and using an electric pump, controlled by the computer, to add or reduce the pressure on the rock accordingly.

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## APPENDIX A

### WHY AND HOW THE ROCK BROKE

The high pressure applied on the long axis of the rock caused it to break. The rock is build from layers of grains and pores. When load is applied to rock the grains tend to move toward each other closing the pores between them. This mechanism is probably elastic to a certain point were the forces between the grains does not hold them anymore. By increasing the pressure more the elasticity of the bond between the grains is lost and micro-cracks start to appear. Increasing the pressure a little more results in breaking the rock, as can be seen in Fig. 38-40.



Figure 38. Limestone After 1500 PSI was Applied to the Long Axis

The various rocks broke at different pressures. The sandstone could not stand pressures bigger than 1500 PSI. The limestone broke at 2000 PSI, after being at that point for about two hours. The Dolomite broke at about 4000 PSI. To improve the resistance of the rock to the pressure and enable maximum load on the rock we insured that the pressure is equally spread on the rock bases. We did it by cutting the bases of the rocks in perpendicular to the long axis of the cylinder. In order to support the external walls of the cylinder, and hold the rock to its base we used a plastic sleeve. The sleeve was a heat shrink tube 2 inches in diameter and about 5 mm longer than the rock. The tube was set on the rock and heated to 150 °C. When the sleeve cooled back to room temperature it gained strength and supported the external walls of the rock. The elasticity and strength of the sleeve enabled us to raise the pressure a little bit, but the rock continued to break (tearing the tube as can be seen in Fig. 39).

Various types of heat shrink tubes were checked, but the results were about the same. Even the heavy-duty heat shrink from 3M did not help.

As a result we decided to limit the range of the pressures checked to 1500 PSI for the rocks we believed to be limestones or sandstones and to 2500 PSI for the harder rocks like Dolomites.



Figure 39. Sandstone After 1500 PSI was Applied to the Long Axis



Figure 40. Dolomite After 2500 PSI was Applied to the Long Axis

# APPENDIX B

## SAMPLE HOLDER FOR THE SAW.

In order to cut the rock in parallel bases perpendicular to the long axis of the cylinder a holder was designed. The holder is sketched in Fig. 41.



Figure 41. The Sample Holder for the Saw

67

### APPENDIX C

## TEMPERATURE CONTROLLER - WIRING SCHEME

In order to set and control the ambient temperature a temperature controller, OMEGA Model CN-2000, was used. This controller was designed to be computer controlled. It senses the temperature of the furnace (it is supposed to control) and supplies high voltage (120 V) to the heater when it's temperature is smaller than the set point. The set point can be set manually (with switches on the controller) or by the computer through the bus. The controller was wired to the System (Fig. 42).



Figure 42. Temperature Controller Wiring to the System

APPENDIX D

# CONTROL, SAMPLING AND DATA ACQUISITION PROGRAMS

```
10 1 2/2/88 PROGRAM FROM THE HARD DISC (D040)
20 ! THE CHACHES WERE DONE IN THE PROGRAM " trange "
30 !the name of the program is " TDFM1 "
40 ! THERMAL DIFFUSIVITY -- MAIN PGM
50 COM t1(200),t2(200),t3(200),dtdt(200),d2tdx(200),tdfs(200)
50 COM eta(200),xs(200),ys(200),tc(50),tbar(200)
70 COM runum, jr, tint, deti, dlay, x12, x23, t1, th, dt, NT, runtyp, etabar, sd(200)
80 COM tau
90 COM T1$(200),T2$(200)
100 DIM subtitle$[80]
110 DISP "Enter a descriptive title for the experiment"
120 INPUT title$
130 DISP "Enter additional information" @ INPUT subtitle$
140 !
150 DISP "Choose a file name for data storage."
160 DISP " USE THE SPECIAL FUNCTION KEYS"
170 DD$=DATE$ @ TT$=TIME$
180 GOSUB 1710
190 D$=DD$[7,8]&DD$[4,5]&DD$[1,2]
200 ON KEY# 1,"CONOCO" GOTO 590
210 ON KEY# 2, "OTHER" GOTO 640
220 KEY LABEL
230 GOTO 230
240 OFF KEY#
250 ! 200-299 are reserved for alarms
260 WAIT 2000
270 CLEAR
280 DISP "*** CHOOSE THE TYPE OF EXPERIMENT *** "
290 DISP " "
300 DISP "
             <fl> Single temperature only"
310 DISP "
              <f2> N specified temperatures"
320 DISP "
               <f3> Range of temperatures at regular intervals"
330 DISP "
              <f4> Several temperatures, manual control"
340 DISP "
               <f5> Auto"
350 ON KEY# 1,"SINGLE" GOTO 440
360 ON KEY# 2,"N TEMP" GOTO 530
370 ON KEY# 3,"T RANGE" GOTO 560
380 ON KEY# 4, "MANUAL" GOTO 470
390 ON KEY# 5, "AUTODATA" GOTO 500
400 KEY LABEL
410 GOTO 410
420 OFF KEY#
430 GOTO 700
440 runtyp$="singlet"
450 runtyp=1
450 GOTO 420
470 runtyp$="singlet"
480 runtyp=4
490 GOTO 420
500 runtyp$="AUTO"
```

510 runtyp=5 520 GOTO 420 530 runtyp\$="ntemp" 540 runtyp=2 550 GOTO 420 560 runtyp\$="trange" 570 runtyp=3 580 GOTO 420 590 ! set up Conoco file 600 !D\$=VAL\$(DATE) 610 L\$="CONOCO"&D\$ 620 DISP "The data will be stored as ";L\$ 630 GOTO 240 640 ID\$=VAL\$(DATE) 650 DISP "Enter the file name (up to 5 characters)" @ INPUT L\$ 660 L\$=L\$&D\$ 670 DISP " The data will be stored as ";L\$ 680 GOTO 240 690 ! 700 ! Enter the experimental parameters 710 CLEAR @ DISP "SET THE PARAMETERS FOR THE DIFFUSIVITY MEASUREMENT" 720 DISP " " 730 DISP "Time between temperature measurements (msec)" @ INPUT tint 740 DISP "Total time for a diffusivity measurement (msec)" @ INPUT deti 750 DISP "Time delay before starting data acquisition (msec)" @ INPUT dlay 760 DISP "Distances between thermocouples: x12,x23 (cm)" @ INPUT x12,x23 770 runum=0 780 tau=2\*tint 790 ! 800 IF runtyp\$="singlet" THEN 840 ELSE 970 810 IF runtyp=1 THEN 840 820 CLEAR @ DISP " INSTRUCTIONS FOR A MANUAL RUN" 830 DISP " " @ GOTO 850 840 CLEAR @ DISP "INSTRUCTIONS FOR A SINGLE TEMPERATURE RUN " @ DISP " " 850 DISP " 1. Set the temperature controller manually to the desired " 860 DISP " temperature. NOTE THAT THE SETTINGS ARE IN DEG C." 870 DISP " " 880 DISP " 2. Wait for the system to equilibrate. 890 DISP " " 900 DISP " 3. Press CONT  $\langle f4 \rangle$  when equilibrium has been reached." 910 DISP " " 920 DISP " 4. If the desired temperature is room temperature, you may", 930 DISP " press CONT at once." @ DISP " " 940 PAUSE 950 runs=1 960 GOTO 1200 970 IF runtyp\$="ntemp" THEN 980 ELSE 1060 980 CLEAR @ DISP " INSTRUCTIONS FOR SETTING N TEMPERATURES " 990 DISP " " 1000 DISP " 1. Enter the number of temperatures. " @ INPUT NT

```
1010 DISP " 2. Enter the ";NT;" temperatures as prompted" @ DISP " "
1020 FOR jt*1 TO NT @ DISP " Temp #";jt;" (deg C) "; @ INPUT tc(jt)
1030 NEXT jt
1040 runs=NT
1050 GOTO 1200
1060 IF runtyp$="trange" THEN 1070 ELSE 1170
1070 CLEAR @ DISP " INSTRUCTIONS FOR SETTING A RANGE OF TEMPERATURES "
1080 DISP " "
1090 DISP " 1. Enter the lowest temperature. (deg C)" @ INPUT tl
1100 DISP " "
1110 DISP " 2. Enter the highest temperature. (deg C)" @ INPUT th
1120 DISP " "
1130 DISP " 3. Enter the temperature interval between points. (deg C)"
1140 INPUT dt
1150 runs=(th-tl)/tint+1
1160 GOTO 1200
1170 runs=1
1180 GOTO 1200
1190 !
1200 ! Set up storage for the experiment.
1210 NR=6
1220 IF runtyp$="ntemp" THEN NR=NR+NT+1
1230 IF runtyp$="trange" THEN NR=NR+3
1240 LL$=L$&".TX"
1250 MASS STORAGE IS "/data"
1260 ON ERROR GOTO 1480
1270 CREATE LL$,NR,256
1280 ASSIGN# 1 TO LL$
1290 PRINT# 1 ; runs
1300 PRINT# 1 ; title$
1310 PRINT# 1 ; subtitle$
1320 PRINT# 1 ; runtyp$
1330 PRINT# 1 ; L$
1340 PRINT# 1 ; tint,deti,dlay
1350 PRINT# 1 ; ×12,×23
1360 IF runtyp$="trange" THEN PRINT# 1 ; tl,th,dt
1370 IF runtyp$="ntemp" THEN 1380 ELSE 1400
1380 PRINT# 1 ; NT
1390 FOR jt=1 TO NT @ PRINT# 1 ; tc(jt) @ NEXT jt
1400 ASSIGN# 1 TO * @ ! Close the file
1410 MASS STORAGE IS "/disc"
1420 ASSIGN# 1 TO "name"
1430 PRINT# 1 ; L$
1440 ASSIGN# 1 TO *
1450 ICLEAR @ DISP USING 1810
1460 IMAGE " TEMP " ,3X," DFSVTY ",3X," ETA ",3X,"STD DEV"
1470 CHAIN runtyp$
1480 OFF ERROR
1490 IF ERRN=63 THEN 1500 ELSE 1610
1500 ASSIGN# 1 TO LL$
```

72

```
1510 READ# 1 ; oldruns
1520 READ# 1 ; oldtitle$
1530 ASSIGN# 1 TO *
1540 DISP " File: ";L$
1550 DISP title$
1560 DISP oldruns
1570 DISP "Increment number of runs? (y/n)" @ INPUT qm$
1580 IF gm$="y" THEN 1590 ELSE 1610
1590 runs=runs+oldruns
1600 IF title$=oldtitle$ THEN 1280
1610 ERRM
1620 IF ERRN<>63 THEN 1680
1630 DISP "Old title: ";oldtitle$
1640 DISP "New title: ";title$
1650 DISP "Use old title? (y/n)" @ INPUT qm$
1660 IF qm$="y" THEN title$=oldtitle$ ELSE 1680
1670 GOTO 1280
1680 DISP "This action will DESTROY data."
1590 DISP "Execution of this program is terminated."
1700 BEEP @ GOTO 1700
1710 | CODING TABLE FOR MONTHS.
1720 AAA=VAL(DD$[4,5])
1730 ON AAA GOTO 1740,1750,1760,1770,1780,1790,1800,1810,1820,1830,1840,1850
1740 DD$[4,5]="JA" @ GOTO 1860
1750 DD$[4,5]="FB" @ GOTO 1860
1760 DD$[4.5]="MS" @ GOTO 1860
1770 DD$[4,5]="AP" @ GOTO 1860
1780 DD$[4,5]="MY" @ GOTO 1860
1790 DD⊈[4,5]="JN" @ GOTO 1860
1800 DD$[4,5]="JL" @ GOTO 1860
1810 DD$[4,5]="AG" @ GOTO 1860
1820 DD$[4,5]="SP" @ GOTO 1860
1830 DD$[4,5]="OC" @ GOTO 1860
1840 DD$[4,5]="NO" @ GOTO 1860
1850 DD$[4,5]="DE" @ GOTO 1860
1860 RETURN
```

10 ! THE NAME OF THIS PROGRAM IS trange. Last chande 19/5/88 H.Y 20 COM t1(200),t2(200),t3(200),dtdt(200),d2tdx(200),tdfs(200) 30 COM eta(200),xs(200),ys(200),tc(50),tbar(200),sd(200) 40 COM runum,jr,tint,deti,dlay,x12,x23,tl,th,dt,NT,runtyp,etabar 50 COM tau 60 COM T1\$(200),T2\$(200) 70 DIM subtitle\$[60] 80 ASSIGN 7 TO "hpib" 90 ASSIGN 9 TO "serial.b" 100 ASSIGN# 1 TO "name"

```
110 READ# 1 ; L$
120 ASSIGN# 1 TO *
130 LL$=L$&".TX" @ MASS STORAGE IS "/data"
140 ASSIGN# 1 TO LL$
150 READ# 1 : runs
160 READ# 1 ; title$
170 READ# 1 ; subtitle$
180 READ# 1 ; runtyp$
190 READ# 1 : L$
200 READ# 1 ; tint,deti,dlay
210 READ# 1 ; x12,x23
220 READ# 1 ; t1,th,dt
230 ASSIGN# 1 TO *
240 DISP "SAMPLE # ";title$
250 PRINT "SAMPLE # ",L$,TIME$
260 PRINT ""
270 PRINT ""
280 DISP "STORED AS ";L$
290 runum=0
300 DISP "DO YOU WANT TO SEE EVRY STEP ? IF YES TYPE Y"
310 INPUT CON$
320 DISP "SET TIME IN MINUTES BETWEEN OBSERVATIONS"
330 INPUT TOBS
340 TOBS=TOBS*60000
350 MASS STORAGE IS "/disc"
360 FINDPROG "TAKEDATA"
370 FINDPROG "CONTSET"
380 FINDPROG "TDREDUCE"
390 FINDPROG "TDSTORE"
400 NN=(INT((th-t1)/dt)+1)*3 @ DISP "total # of runs will be ===>",NN
410 OFF TIMER# 1
420 CALL "CONTSET"
430 DISP TIME$," YOU HAVE TO WAIT", TOBS/60000, "MINUTES"
440 NNIT=0
450 NBB=0
460 GOTO 640
470 OFF TIMER# 1 @ OFF TIMER# 2
480 runum=runum+1
490 rnt$=VAL$(runum) @ LTB$=L$&"."&rnt$
500 PRINT USING 510 ; TIME$,LTB$,TOB5/50000
510 IMAGE K,1X,",",4X,K1X,",",4X,"DELAY TIME WAS ",DD," MINUTES"
520 DISP TIMES @ CALL "TAKEDATA" @ tim1s=TIME$
530 IF NNIT=NN THEN GOTO 560
540 DISP TIME$ @ CALL "CONTSET" @ tim1$=TIME$
550 DISE TIME: "# OF RUN", runum, "YOU HAVE TO WAIT", TOBS/60000, "MINUTES"
550 TB$=TIME$
570 'DISP "point!"
590 DISP TIME$ @ CALL "TDREDUCE"
590 !DISF "point2"
600 DISP TIME$ @ CALL "TDSTORE" (L$)
```

```
610 IDISP "point3"
620 MASS STORAGE IS "/disc"
630 OFF KEY# @ tim2$=TIME$ @ DISP tim2$
540 IF NNIT=NN+1 THEN GOTO 1480 @ TIM$=TIME$ @ DISP TIM$
650 NTBS=0 @ IF NNIT=0 THEN TOBS1=TOBS ELSE GOSUB 2770
550 ON TIMER# 1, TOBS1 GOTO 470 @ NNIT=NNIT+1
670 ON TIMER# 2,300000 GOTO 810 @ !CHECK
580 IF NNIT=NN+1 THEN GOTO 1480 @ TIMS=TIMES
590 IF CON$#"Y" THEN GOTO 800 @ !CHECK
700 IF NNIT=1 THEN GOTO 800 @ !CHECK
710 OFF KEY# @ ON KEY# 1,"PARMS" GOTO 860
720 ON KEY# 3,"CONT" GOTO 790
730 ON KEY# 16, "STOP PROG" GOTO 1480
740 BEEP
750 IF NBB=0 THEN GOTO 740
760 GOTO 760
770 OFF KEY#
780 GOTO 780
790 OFF KEY#
800 GOTO 800
810 NTBS=NTBS+1
820 DISP TIME$,"YOU HAVE TO WAIT",TOBS/60000-NTBS*5,"MINUTES"
830 OFF TIMER# 2
840 ON TIMER# 2,300000 GOTO 810
850 GOTO 850
860 OFF KEY#
870 ON KEY# 1,"PLOT" GOTO 950
880 ON KEY# 2, "TOBS" GOTO 1290
890 ON KEY# 3,"TINT" GOSUB 1350
900 ON KEY# 4, "DETI" GOSUB 1390
910 ON KEY# 5, "DELAY" GOSUB 1440
920 ON KEY# 8, "RESUME" GOTO 2630
930 ON KEY# 16, "STOP PROG" GOTO 1480
940 GOTO 940
950 rn$=VAL$(runum) @ LL$=L$&"."&rn$
960 ! Set plotting options
970 tbar#tbar(runum) @ tdfs#tdfs(runum) @ OFF KEY#
980 LT$=L$&".TX"
990 MASS STORAGE IS "/data"
1000 ASSIGN# 1 TO LT$
1010 READ# 1 ; runs
1020 READ# 1 ; title$
1030 ASSIGN# 1 TO *
1040 ON KEY# 1, "TMP/TIM" GOTO 1150
1050 ON KEY# 2,"DERIVS" GOTO 1190
1060 ON KEY# 8,"RETURN" GOTO 860
1070 ON KEY# 16,"STOP PROG" GOTO 1600
1080 KEY LABEL
1090 CLEAR @ DISF " SELECT THE TYPE OF PLOT USING THE FUNCTION KEYS"
1100 DISP " "
```

1110 DISP "<f1> Temperatures vs. time "1120 DISP "<f2> dT/dt vs. d2T/dx2 " 1130 DISP " " @ DISP " <f8> Return to main menu." 1140 GOTO 1140 1150 ymax=INT(t1(jr)) @ xmax=deti/1000 @ fl=1 1160 IF ymax=0 THEN ymax=1 1170 x1b1\$="TIME (sec)" @ y1b1\$="TEMP. CHANGE (C)" 1180 GOTO 1800 1190 ymax=MAXAB(ys) @ xmax=MAXAB(xs) @ xmax=INT(xmax)+1 @ fl=2 1200 IF ymax<1 THEN 1210 ELSE 1250 1210 za=1 1220 za=10\*za 1230 IF za\*ymax<1 THEN 1220 1240 ymax=INT(za\*ymax+1)/za 1250 GOTO 1270 1260 ymax=INT(ymax+1) 1270 x1b1\$="d2T/dx2" @ y1b1\$="dT/dt" 1280 GOTO 1800 1290 DISP "SET TIME IN MINUTES BETWEEN OBSERVATIONS" 1300 INPUT TOBS 1310 TOBS=TOBS+60000 1320 tim2\$=TIME\$ 1330 GOSUB 2770 1340 OFF KEY# @ GOTO 690 1350 DISP "SET TINT" 1360 DISP "OLD TINT = ";tint 1370 INPUT tint 1380 RETURN 1390 DISP " SET DETI -- TIME FOR EXPERIMENT" 1400 DISP "OLD DETI = ";deti 1410 INPUT deti 1420 RETURN 1430 MASS STORAGE IS "/disc" @ END 1440 DISP "SET DELAY" 1450 DISP " OLD DELAY = ";dlay 1460 INPUT dlay 1470 RETURN 1480 CLEAR @ DISP "SAMPLE # ";title\$ 1490 PRINT "STORED AS ";L\$ 1500 PRINT "TIME BETWEEN MEASUREMENTS ";TOES/60000 1510 PRINT "LAST MEASUREMENT ENDED AT "; TB\$ 1520 PRINT USING 2690 1530 FOR JU=1 TO runum 1540 ON ERROR GOTO 1560 1550 PRINT USING 2750 ; JU,tbar(JU);tdfs(JU);eta(JU);sd(JU) 1560 OFF ERROR 1570 ×tdfs=×tdfs+tdfs(JU) 1580 NEXT JU 1590 PRINT "AVERAGE VALUE IS", tdfs/runum 1600 MASS STORAGE IS "/disc" @ ASSIGN# 1 TO "name" @ READ# 1 ; L\$ 1610 ASSIGN# 1 TO \* 1620 LL\$≃L\$&".TX" @ MASS STORAGE IS "/data" @ ASSIGN# 1 TO LL\$ 1630 READ# 1 ; runs 1640 READ# 1 : title\$ 1650 READ# 1 ; subtitle\$ 1660 READ# 1 ; runtyp\$ 1670 READ# 1 ; L\$ 1680 READ# 1 ; tint,deti,dlay 1690 READ# 1 ; ×12,×23 1700 READ# 1 ; t1,th,dt 1710 PRINT# 1 ; NNIT-1 1720 PRINT# 1 ; TIME\$ 1730 PRINT# 1 ; TOBS/60000 1740 DISP TIME\$ 1750 t1=0 1760 dt=0 1770 CALL "CONTSET" 1780 ASSIGN# 1 TO \* 1790 GOTO 1430 1800 ! plotting routine 1810 GCLEAR @ DEG @ CSIZE 4.0.6 1820 LOCATE 0,200,0,100 1830 SCALE (-0.5)\*xmax,1.05\*xmax,(-0.2)\*ymax,1.15\*ymax 1840 CLIP 0,xmax,0,ymax 1850 AXES xmax/5,ymax/5,0,0 @ AXES xmax/5,ymax/5,xmax,ymax 1860 LDIR 0 @ LORG 2 1870 y=(-0.05)\*ymax 1880 FOR x=0 TO xmax STEP xmax/5 1890 MOVE x,y 1900 LABEL x 1910 NEXT × 1920 MOVE xmax/2,(-0.12)\*ymax 1930 LABEL ×161\$ 1940 x=0.32\*xmax @ LORG 8 1950 FOR y=0 TO ymax STEP ymax/5 1960 MOVE x,y 1970 LABEL y 1980 NEXT y 1990 LDIR 90 @ LORG 5 2000 MOVE (-0.1)\*xmax,0.6\*ymax 2010 LABEL y1b1\$ 2020 LDIR 0 @ LORG 2 2030 MOVE 0.05\*xmax,1.04\*ymax 2040 LABEL title\$ 2050 MOVE 0.05\*xmax,0.9\*ymax 2050 LABEL "T = " 2070 MOVE 0.1+Amax,0.9+ymax 2080 tbar=INT(100\*tbar)/100 2090 LABEL than 2100 IF f1=1 THEN 2130

```
2110 GOTO 2490
2120 ! Plot the temperature vs. time data
2130 FOR j=1 TO jr
2140 x=j*tint*0.001
2150 y=t1(j)
2160 MOVE x,y @ GOSUB 2570
2170 y=t2(j)
2180 MOVE x,y @ GOSUB 2610
2190 y=t3(j)
2200 MOVE x,y @ GOSUB 2570 @ GOSUB 2610
2210 NEXT j
2220 ! Add additional data to the plot
2230 LDIR 0 @ LORG 2 @ CSIZE 4,0.6
2240 MOVE (-0.4)*xmax,1.04*ymax
2250 LABEL "DATA"
2260 CSIZE 3.0.6
2270 MOVE (+0.5)*xmax,0.9*ymax @ LABEL "DELAY = "
2280 MOVE (-0.4)*xmax,0.9*ymax @ LABEL dlay
2290 MOVE (-0.5)*xmax.0.8*ymax @ LABEL "T-INT = "
2300 MOVE (-0.4)*xmax,0.8*ymax @ LABEL tint
2310 MOVE (-0.5)*xmax,0.7*ymax @ LABEL "TDFSVTY = "
2320 tdfs=INT(tdfs*100000)/100000
2330 MOVE (-0.38)*xmax,0.7*ymax @ LABEL tdfs
2340 MOVE (-0.5)*xmax,0.6*ymax @ LABEL "L$ = "
2350 MOVE (-0.42)*xmax,0.6*ymax @ LABEL LL$
2360 MOVE (-0.5)*xmax,0.5*ymax @ LABEL "x12 ="
2370 MOVE (-0.4)*xmax,0.5*ymax @ LABEL x12
2380 MOVE (-0.5)*xmax,0.4*ymax @ LABEL "x23 = "
2390 MOVE (-0.4)*xmax,0.4*ymax @ LABEL x23
2400 MOVE (-0.5)*xmax,0.3*ymax @ LABEL "TIME CONST. ="
2410 MOVE (-0.3)*xmax,0.3*ymax @ LABEL tau
2420 MOVE (-0.5)*xmax,0.2*ymax @ LABEL "STD. DEV. ="
2430 sd=INT(sd(runum)*1000000)/1000000
2440 MOVE (-0.3)*xmax,0.2*ymax @ LABEL sd
2450 MOVE (-0.5)*xmax,0.1*ymax @ LABEL "ETA ="
2460 etabar=INT(etabar*100000)/100000
2470 MOVE (-0.4)*xmax,0.1*ymax @ LABEL etabar
2480 CLEAR @ GOTO 960
2490 ! dtdt vs d2tdx2 plots
2500 FOR jp=2 TO jr-1
2510 MOVE xs(jp),ys(jp) @ GOSUB 2570
2520 NEXT jp
2530 | Draw the regression line
2540 MOVE 0,0 0 y=tdfs*xmax
2550 LINE TYPE 1 @ DRAW xmax,y
2560 GOTO 2230
2570 / plus symbol
2580 SETGU @ IDRAW 1,0 @ IDRAW -2,0 @ IDRAW 1,0 @ IDRAW 0.1
2590 IDRAW 0,-2 @ IDRAW 0,1
2600 SETUU @ RETURN
```

```
2610 ! x symbol
2620 SETGU @ IDRAW 1,1 @ IDRAW -2,-2 @ IDRAW 1,1 @ IDRAW -1,1
2630 IDRAW 2,-2 @ IDRAW -1,1
2640 SETUU @ RETURN
2650 JUPDATE THE DISPLAY
2660 CLEAR @ DISP "SAMPLE # ";title$
2670 DISP "STORED AS ";L$
2680 DISP "TIME BETWEEN MEASUREMENTS ";TOBS/60000
2690 DISP "LAST MEASUREMENT ENDED AT ";TB$
2700 DISP USING 2710
2710 IMAGE " RUN
                     TEMP ",3X," DFSVTY ",3X," ETA ",3X,"STD DEV"
2720 FOR JU=1 TO runum
2730 ON ERROR GOTO 2750
2740 DISP USING 2770 ; JU,tbar(JU);tdfs(JU);eta(JU);sd(JU)
2750 OFF ERROR
2760 NEXT JU
2770 IMAGE 1X,DD,4X,DDDD.DD,3(3X,DD.5D)
2780 GOTO 700
2790 tm1=VAL(tim1$[1,2])*3600+VAL(tim1$[4,5])*60+VAL(tim1$[7,8])
2800 tm2=VAL(tim2$[1,2])*3600+VAL(tim2$[4,5])*60+VAL(tim2$[7,8])
2810 dts=(tm2-tm1)/60
2820 DISP tml,tm2,dts
2830 TOBS1=(TOBS/60000-dts)*60000 @ DISP TOBS1
2840 ON TIMER# 1, TOBS1 GOTO 470
2850 RETURN
10 SUB "CONTSET"
20 104-02-88 CONTROLER SET POINT SUBRUTINE
                                              **** H. Y. ****
30 COM t1(200),t2(200),t3(200),dtdt(200),d2td.(200),tdfs(200)
40 COM eta(200),xs(200),ys(200),tc(50),tbar(200)
50 COM runum, jr, tint, deti, dlay, x12, x23, t1, th, dt, NT, runtyp, etabar
60 COM tau
70 COM T1$(200),T2$(200)
80 INTEGER NN,N1,NCA
90 DIM B$[100]
100 NN=(INT(th-t1)/dt+1)*3
110 N2=N2+1
120 DISP "N2=",N2,"N1=",N1
130 IF N2<4 THEN GOTO 160
140 N2=1
150 N1=N1+1
160 ASSIGN 9 TO "serial.b"
170 CONTROL 9,4 ; 62
180 CONTROL 9,3 ; 5
190 STATUS 9,10 ; A10
200 IF A10>31 THEN 210 ELSE 190
210 CA=INT(t1+dt*N1)
220 V$=VAL$(CA) @ IJ=LEN(V$) @ IF IJ<2 THEN V$=VAL$(0)&V$
230 IF IJ<3 THEN V$=VAL$(0)&V$
240 B$="*00:WCSP/111:"
250 B$[10,12]=V$ @ B$=B$&CHR$(13)
260 DISP TIMES, "THE NEW SET POINT IS =", CA
270 OUTPUT 9 ; B$
280 SUBEND
```

```
10 SUB "TDREDUCE"
20 ! tdreduce
30 COM t1(200).t2(200).t3(200).dtdt(200).d2tdx(200).tdfs(200)
40 COM eta(200),xs(200),ys(200),tc(50),tbar(200),sd(200)
50 COM runum.jr,tint.deti,dlay,x12.x23.tl,th,dt.NT,runtyp,etabar
50 COM tau
70 COM T1$(200),T2$(200)
80 xbar=0.5*(x12+x23)
90 at=25.33 @ bt=-0.352 @ ct=0.032
100 ! Convert voltages to temperatures
110 FOR j=0 TO jr
120 tt=t1(j)*1000 @ t1(j)=tt*(at+tt*(bt+tt*ct))
130 tt=t2(j)*1000 @ t2(j)=tt*(at+tt*(bt+tt*ct))
140 tt=t3(j)*1000 @ t3(j)=tt*(at+tt*(bt+tt*ct))
150 NEXT (
160 ! Calculate the time derivatives of t2
170 n=8
180 sj=0 0 sj2=0 0 st=0 0 sjt=0
190 FOR j=1 TO 2*n+1
200 sj=sj+j @ sj2=sj2+j*j
210 st=st+t2(j) @ sjt=sjt+j*t2(j)
220 NEXT j
230 dtdt(n+1)=1000*((2*n+1)*sjt-sj*st)/(tint*((2*n+1)*sj2-sj*sj))
240 FOR j=n+2 TO jr+n
250 sj=sj+n+n+1 @ sj2=sj2-(j-n-1)*(j-n-1)+(j+n)*(j+n)
260 st≈st-t2(j-n-1)+t2(j+n) @ sjt=sjt-(j-n-1)*t2(j-n-1)+(j+n)*t2(j+n)
270 dtdt(j)=1000*((2*n+1)*sjt-sj*st)/(tint*((2*n+1)*sj2-sj*sj))
280 NEXT j
290 ! Get the delta t's and the laplacian
300 FOR j=1 TO jr
310 t1(j)=t1(j)-t1(0) @ t2(j)=t2(j)-t2(0) @ t3(j)=t3(j)-t3(0)
320 d2tdx(j)=((t3(j)-t2(j))/x23-(t2(j)-t1(j))/x12)/xbar
330 NEXT j
340 ! Get the thermal diffusivity by least squares
350 sx=0 @ sx2=0 @ sy=0 @ sxy=0 @ st=0 @ set=0 @ sy2=0
360 FOR i=n+1 TO ir-n
370 x=(d2tdx(j-1)+d2tdx(j)+d2tdx(j+1))/3
380 xs(j)=x
390 y=dtdt(j)
400 ys())=y
410 sxy=sxy+x*y
420 5×2=5×2+×*×
430 sy2=sy2+y*y
440 st=st+t2(j)
450 eta())=y/x
460 set=set+y/x
470 NEXT j
480 tdfs(munum)=sky/sk2
490 tbar(runum)=st/(jr-2*n)+t2(0)
500 etabar=set/(jr-2*n)
```

```
510 sd=SQR((sy2-sxy*sxy/sx2)/(jr-2))/x @ sd(runum)#sd
520 ON ERROR GOTO 550
530 DISP USING 540 ; tbar(runum);tdfs(runum);etabar;sd(runum)
540 IMAGE DDDD.DD,3(3X,DD.5D)
550 OFF ERROR
560 eta(runum)=etabar
570 SUBEND
10 SUB "TAKEDATA"
20 !last adjestment at 14/07/88 ,GOING BACK TO THE NEW SYS.
30 ! 19/1/88 ADJESTING THE PROG. TO THE FACILITIES
40 COM t1(200),t2(200),t3(200),dtdt(200),d2tdx(200),tdfs(200)
50 COM eta(200),×s(200),ys(200),tc(50),tbar(200),sd(200)
60 COM runum, jr, tint, deti, dlay, x12, x23, t1, th, dt, NT, runtyp, etabar
70 COM tau
80 COM T1$(200),T2$(200)
90 DIM T3(100), T4(100)
100 INTEGER dummy, Tint
110 LOADBIN "timer"
120 Tint=tint
130 ON KYBD dummy, ""027T" GOSUB TIMEDOUT
140 GOTO 230
150 GOTO 150
160 TIMEDOUT: TRIGGER vm1,vm2,vm3 @ jr=jr+1
170 ENTER vm1 ; t1(jr) @ ENTER vm2 ; t2(jr) @ ENTER vm3 ; t3(jr)
180 RETURN
190 OFF TIMER# 3 @ GOTO 390
200 CALLBIN "offtimer" (0)
210 OFF TIMER# 1
220 OUTPUT dac ; "cpn 0" @ 60T0 420
230 ! Set up instruments
240 dac=709 @ vm1=726 @ vm2=727 @ vm3=728 @ !hpib addresses
250 ! set up voltmeters
260 FOR ji=0 TO 2 @ vm=vm1+ji @ OUTPUT vm ; "F1R-2T4Z0N5" @ NEXT ji
270 OUTPUT dac ; "opn 0 "
280 ! get the temperature baseline
290 TRIGGER vm1, vm2, vm3
300 ENTER vm1 ; t1(0)
310 ENTER vm2 ; t2(0)
320 PRINT "START TIME=",TIME$,TIME
330 ENTER vm3 ; t3(0)
340 in=0
350 BUTPUT dae ; "cls 0"
360 IF dlay<10 THEN 390
370 ON TIMER# 3,dlay GOTO 190
380 GOTO 380
390 ON TIMER# 1,deti GOTO 200
400 CALLBIN "on timer" (500)
410 LOOF: GOTO LOOF
420 FOR ji≠0 TO 2 @ vm=vml+ji @ OUTPUT vm ; "T1" @ NEXT ji
430 IDT21=VAL(T2$(1))-VAL(T1$(1))
440 IDT31=T3(1)-VAL(T1$(1)) @ DT32=T4(1)-T3(1) @ DT33=VAL(T2$(1))-T4(1)
450 IDISP USING 490 ; runum,jr
460 !IMAGE "Run number", 3X, 2D, 3X, "Has ", 3X, 3D, 3X, "Sampling points."
470 SUBEND
```

```
10 SUB "TDSTORE" (L$)
20 ! last attach at 04/02/88
30 | last attach at 25/1/88
40 ! tdstore
50 COM t1(200),t2(200),t3(200),dtdt(200),d2tdx(200),tdfs(200)
60 COM eta(200),xs(200),ys(200),tc(50),tbar(200)
70 COM runum, jr, tint, deti, dlay, x12, x23, t1, th, dt, NT, runtyp, etabar, sd(200)
80 COM tau
90 COM T1$(200),T2$(200)
100 DIM SUBTITLE$[80]
110 sd=sd(runum)
120 rns=VALs(runum)
130 LL$=L$&"."&rn$
140 MASS STORAGE IS "/data"
150 CREATE LL$, jr+8,40
160 ASSIGN# 1 TO LL$
170 PRINT# 1 ; LL$
180 PRINT# 1 ; tbar(runum),tdfs(runum),etabar,sd
190 PRINT# 1 ; x12,x23
200 PRINT# 1 ; tint,deti,dlay,tau
210 PRINT# 1 ; jr
220 FOR js=0 TO jr
230 PRINT# 1 ; t1(js),t2(js),t3(js),xs(js),ys(js)
240 NEXT js
250 !MASS STORAGE IS "/data"
260 ASSIGN# 1 TO *
270 LL$=L$&".TX"
280 ASSIGN# 1 TO LL$
290 READ# 1 ; RUNS
300 READ# 1 ; TITLE$
310 READ# 1 ; SUBTITLE$
320 READ# 1 ; RUNTYP$
330 READ# 1 ; L$
340 READ# 1 ; TIT, DETI, DELY
350 READ# 1 ; X12,X23
360 READ# 1 ; T1,T2,T3
370 ASSIGN# 1 TO *
380 ASSIGN# 1 TO LL$
390 PRINT# 1 ; runum
400 PRINT# 1 ; TITLE$
410 PRINT# 1 : SUBTITLE$
420 PRINT# 1 ; RUNTYP$
430 PRINT# 1 ; L$
440 PRINT# 1 ; TIT, DETI, DELY
450 PRINT# 1 ; X12,X23
450 PRINT# 1 : T1,T2,T3
470 PRINT# 1 ; TITLE$
480 ASSIGN# 1 TO *
490 SUBEND
```

# VITA'

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### Master of Science

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