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Ultra Sound Absorption Measurements in Rock Samples at Low Temperatures

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Abstract. A new technique, comparable with the reverberation method in room acoustics, is described. It allows Q -measurements at rock samples of arbitrary shape in the frequency range 50 to 600 kHz in vacuum (10^{-4} torr) and at low temperatures ($+20^{\circ}\text{C}$ to -180°C). The method was developed in particular to investigate rock samples under "lunar conditions". Ultrasound absorption has been measured at volcanics, breccia, gabbros, feldspar and quartz of different grain size and texture yielding the following results: Evacuation raises Q mainly through lowering the humidity in the rock. In a "dry" compact rock the effect of evacuation is small. With decreasing temperature generally Q increases. Between $+20^{\circ}\text{C}$ and -30°C Q does not change much. With further decrease of temperature in many cases distinct anomalies appear, where Q becomes frequency dependent. Rock samples of equal or similar mineralogical composition show a qualitatively similar $Q(T)$ - curve. The absolute level of Q , however, is essentially determined by the rock texture, i.e. grain size, porosity, and grain boundary structure. Rapid cooling with liquid nitrogen causes irreversible lowering of Q apparently by increase of the microcrack density in the sample. This observation seems to be relevant to Q values obtained from lunar surface rock samples.

Key words: Q -Ultra Sound Absorption — Internal Friction — Lunar Rock Samples — Rock Physics.

1. Introduction

The unexpected long duration of lunar seismic signals can be explained by scattering of elastic waves in a layer of extremely low absorption i.e. of high Q . Natural rocks do not show sufficiently high Q -values under terrestrial conditions. Since the mineralogical composition of lunar material does not differ considerably from terrestrial rocks, the reason for high Q -values was suspected to be found in the extreme lunar environmental conditions i.e. absence of air and water in pores and fissures of the rocks and a rather low temperature of -20°C to -40°C close to the moon's surface. Fairly little was known about the influence of evacuation on the ultra sound absorption in silicate rocks and nothing about the influence of low temperatures. Although our measurements did not result in extremely high Q -values, they might contribute to a better understanding of the very complex absorption processes of elastic waves in polycrystalline silicates.

Techniques hitherto used for laboratory measurements of Q required test samples of special shape and size, usually of bar shape. To avoid any destructive treatment in particular in view of the very valuable lunar rock samples, a new method was developed; its physical principles, its technical realization, and some results being discussed in the following sections. The theoretical foundation of the method will be treated in detail in a later paper by J. Drisler and P. Antony-Spies. Results on shock induced ultrasound absorption in lunar anorthosite will separately be published by the present authors. An attempt to interpret the measurements in terms of solid state physics in particular of dislocation theory will be undertaken after some additional experiments have been carried out.

2. The Basic Principles of the Method

The quality factor Q is a measure of unelasticity and defined by $Q = 2\pi E / \Delta E$, where E is the energy density of an elastic wave and ΔE the energy loss per cycle τ . In an oscillating system Q is related to the coefficient α of amplitude decay by $Q = 2\pi / (1 - \exp(-2\alpha\tau))$. If $Q \gg 2\pi$, then $Q \approx \pi f / \alpha$ where f the frequency of the oscillation. This relation is used furtheron to determine Q from the experimental data.

The method described here for Q -measurements in rock samples at ultrasonic frequencies has some similarity with the well known reverberation measurements in room acoustics. The rock sample having arbitrary shape has to be acoustically insulated from its surroundings. Experimental tests show that the assumption of an arbitrarily shaped body is sufficiently fulfilled, if the ratio of longest to shortest dimension is not more than 5 to 1. A piezoceramic transducer, fed by a pulse generator, causes the sample to vibrate and serves as an acoustic receiver for the exponentially decaying

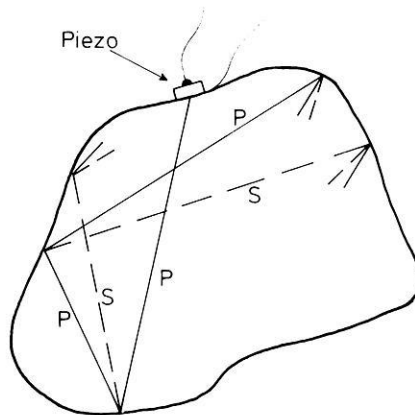


Fig. 1. Multiple reflections in a body of arbitrary shape

reverberations. Before displayed on the screen of the oscillograph the signal passes a variable bandpass filter for frequency selection.

Speaking in terms of ray theory each surface reflection divides the incident wave energy into a reflected P - and S -wave according to the angle of incidence (Fig. 1). After some 10^{-4} seconds a stationary random distribution with a definite energy partition between P - and S -wave is achieved.

Neglecting absorption for the moment, in case of energy equilibrium in space and time, the production rate of converted waves must be equal, i. e.

$$\left(\frac{dE_P}{dt}\right)_{P \rightarrow S} + \left(\frac{dE_S}{dt}\right)_{S \rightarrow P} = 0.$$

On the other hand these quantities are certainly proportional to the respective energies E_P and E_S in the sample. A threedimensional statistical model, based on these principles, leads to the following energy distribution within the sample

$$\frac{E_P}{E_S} = \frac{V_S}{V_P} \cdot \phi$$

where V_P and V_S are the wave velocities for P - and S -waves and ϕ is a function of the ray geometry in the sample. For different statistical models ϕ was found to be certainly larger than 0.5 but probably less than one. This means that for Poissons ratio 0.25 the energy ratio becomes $0.3 < E_P/E_S < 0.6$. For the derivation of this relation and for model calculations reference is made to the above mentioned paper by Drisler and Antony-Spies.

While travelling through the sample the elastic waves lose energy by various nonelastic processes. Q , as determined from the amplitude decay factor α of the random oscillation, is in fact a mixture of the Q -factors of P - and S -waves, according to $Q^{-1} = Q_P^{-1} E_P/E + Q_S^{-1} E_S/E$, where $E = E_P + E_S$ is the total energy.

The specific features of this method are:

1. The sample has not to be altered in shape which is most important for investigating lunar rock samples but also for applications in technical material testing.
2. Q can be measured in a frequency range of about one decade. This allows studies of frequency dependent absorption processes.
3. If measurements are made at lunar rock samples, the values of Q obtained by this method are the most representative ones for an interpretation of lunar seismograms, because these consist also more or less of a statistical mixture of different wave types.
4. The separate determination of Q_P and Q_S is not possible a priori but requires the knowledge of the energy partition in P - and S -waves which can usually only be estimated.

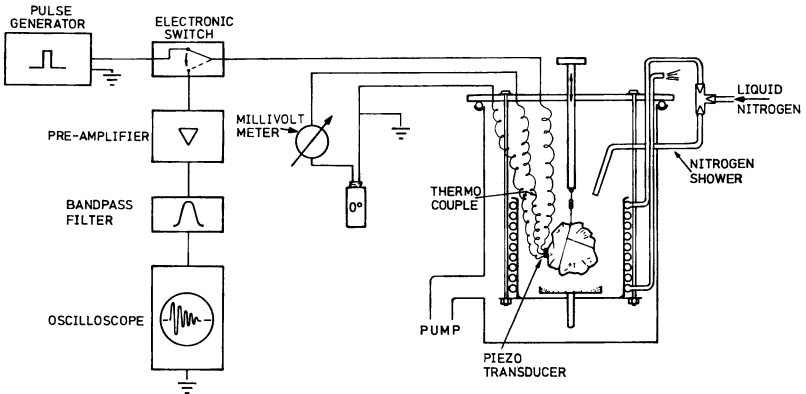


Fig. 2. Scheme of equipment for Q -measurements

3. Measurement Apparatus

In order to measure the influence of evacuation and low temperature on the absorption of elastic waves, a vacuum equipment has been developed, which allows Q -measurements at a pressure of less than 10^{-4} torr within the temperature range of $+20^\circ$ to -180°C . The recipient is evacuated by a two stage rotation pump followed by a three stage oil diffusion pump. Water- and liquid nitrogen cooled baffles prevent the diffusion of oil vapor into the vacuum chamber. A cylindrical copper cryostat, which can be cooled by liquid nitrogen to -196°C is attached to the cover of the recipient.

The rock sample is suspended inside this cryostat at a chain of high acoustic impedance, consisting of nylon and brass links, to prevent acoustic energy flow from the sample to the case. The sample can be moved up and down in the chamber by a manipulator and brought in contact with the cold metal of the cryostat. By this way after several hours the temperature of the sample is reduced to -100°C . In order to reach even lower temperatures and to study the effect of temperature shocks, the sample can directly be sprayed with liquid nitrogen. Repeated liquid nitrogen showers cool the rock to the minimum temperature of -196°C . Vacuum is restored within a few minutes. After the cooling process the sample slowly warms up and reaches its initial temperature of $+20^\circ\text{C}$ after approximately 10 hours. During the whole time Q -measurements are taken.

To measure Q , a lead-zircon-titanate piezo ceramic element of 3 mm diameter and 1 mm thickness together with three electric leads is glued to the sample by a two component cement E-Solder Silver Epoxy. Two thin copper wires connect the piezo element with the electronic circuit. The third wire consists of constantan and forms together with one of the copper

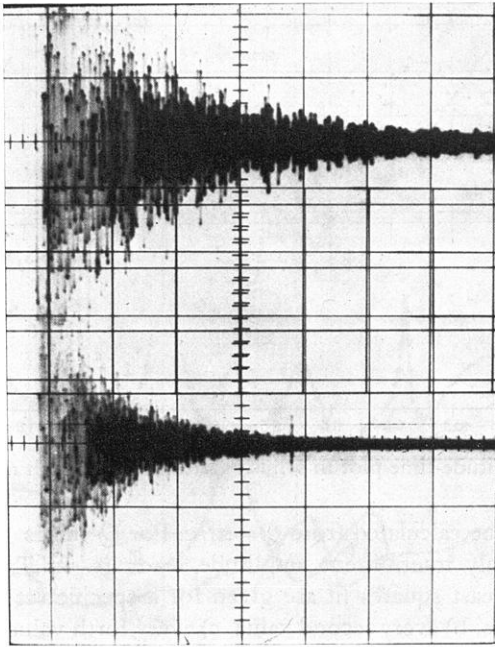


Fig. 3. Ultra-sound reverberations of an obsidian sample

wires a thermocouple to measure the temperature of the sample (Fig. 2). The piezo element serves as pulse transmitter and as signal receiver according to the position of the electronic switch. A pulse of 90 volts and a duration of 1 to 5 μ sec is produced by the pulse generator and applied to the piezo element, which causes the rock to vibrate. Some μ sec later for recording the vibrations, the switch connects the piezo element via preamplifier and variable band pass filter with the oscilloscope. This is repeated 50 to 100 times per second to obtain a standing image. The exponential amplitude decay of the curve is not influenced by changes in strength or duration of the pulse. Signals of sufficient amplitude can be obtained within the frequency range 50 to 600 kHz.

4. Determination of Q

Photographs are taken from the oscilloscope screen with a polaroid camera (Fig. 3). Plotted on logarithmic scale the amplitude values read from the photographs show a linear decrease with time. The slope of the regression line is proportional to the absorption coefficient α . An example is shown in Fig. 4. The corresponding frequency is controlled by the filter setting but should be determined from the oscillogram directly.

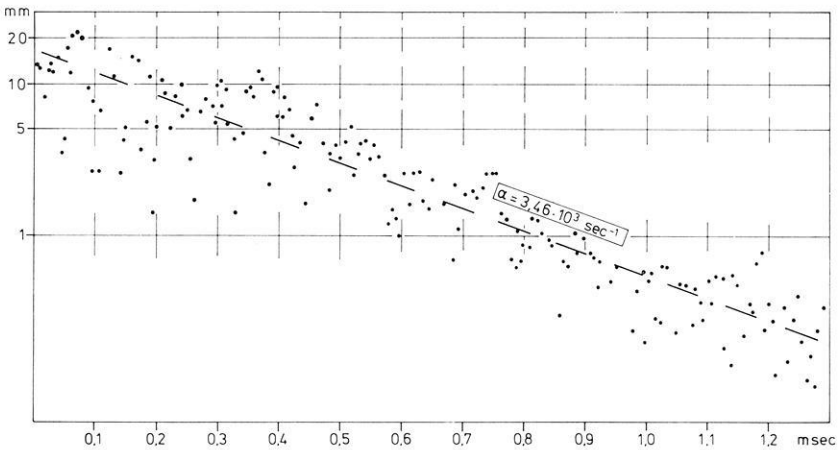


Fig. 4. Amplitude-time plot in semi-logarithmic scale with regression line

Q can then be calculated from $Q = \pi f / \alpha$. For Q -values > 200 it is sufficient to use only intermittent amplitude readings. In Table 1 Q -values obtained by a least squares fit are given for a specific set of data if a) all amplitude values, b) every second value, c) every fourth value d) every eighth value is used. The result demonstrates the permission of this procedure and gives also some estimate of the precision of the method. The standard error of Q -values determined by this method is 2 to 8% for $100 < Q < 500$ and less than 5% for $Q > 500$. A more sophisticated data acquisition system using a mean-square rectifier and logarithmic amplification is intended to be developed.

Table 1. Standard error of Q for different selections of amplitude values

Q	standard error	interval selection (for explanation see text)
148,26	$\pm 3,71$	a
148,53	$\pm 3,05$	b
147,30	$\pm 3,04$	c
144,60	$\pm 3,58$	d

To check whether systematic errors could be introduced by energy loss from the sample to its surroundings, measurements were taken with an additional piezo-transducer and three additional connecting leads, but no detectable difference was found for $100 < Q < 1000$ and samples of a weight between 30 and 200 grams. This is taken as the proof for sufficient acoustical insulation.

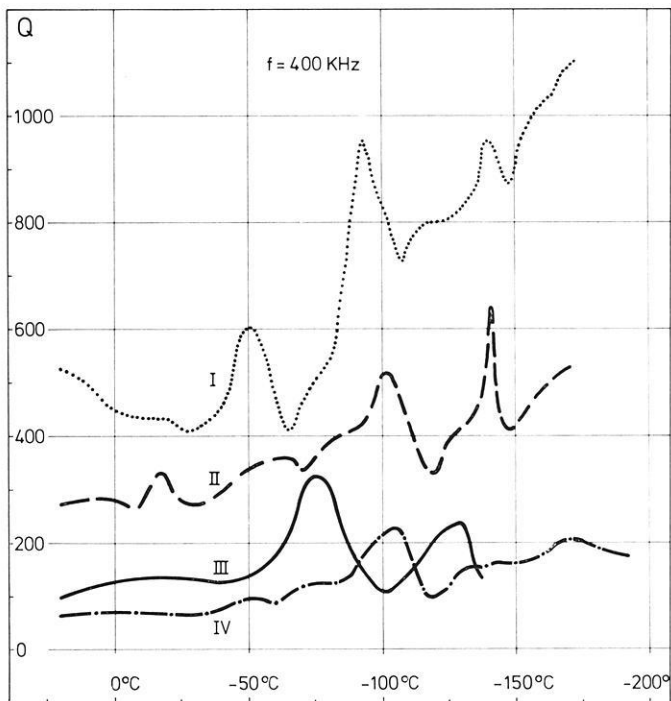


Fig. 5. Q as a function of temperature

$f = 400$ kHz

- I = limestone-breccia
- II = alkalibasalt
- III = olivinbasalttuff
- IV = suevite

5. Results

For a general orientation Fig. 5 shows $Q(T)$ in the range $+20 < T < -180$ °C at 400 kHz for four rocks of different composition and texture. The curves are typical for most of the rock samples investigated. Between $+20$ ° and -40 °C Q does not change much. At lower temperatures there is a general trend for an increase in Q , superimposed by more or less pronounced peaks and troughs. The higher Q the more pronounced are the variations of $Q(T)$ which are in amplitude and probably also in their position in the curve frequency dependent. In order to bring some systematics into the very complex phenomena of ultrasound absorption in rocks, specimens of different mineralogical composition and different grain-structure were treated in several ways.

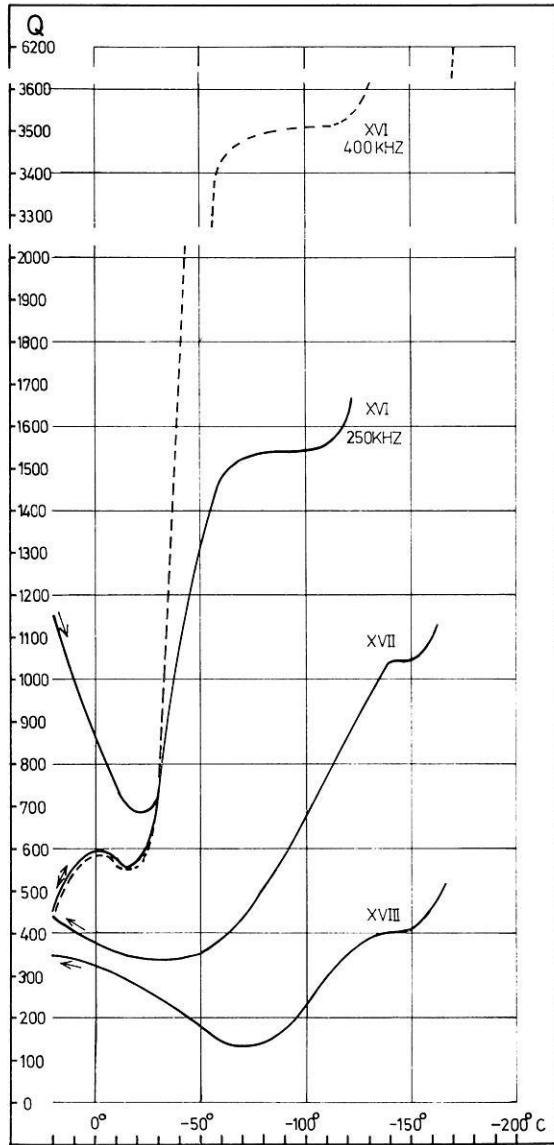


Fig. 6. Q as a function of temperature for SiO₂-samples
 XVI quartz, $f = 400$ kHz, 250 kHz
 XVII quartzite, $f = 275$ kHz
 XVIII chalcedon, $f = 400$ kHz

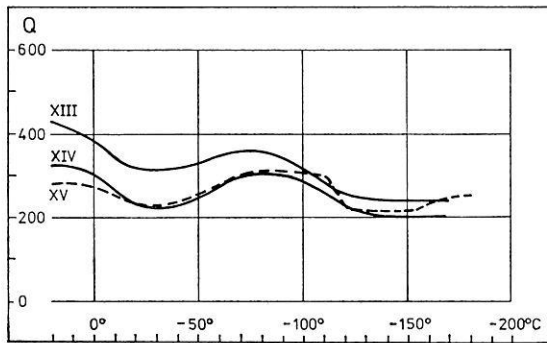


Fig. 7. Q as a function of temperature for gabbroic rock samples
 XIII = anorthosite, $f = 450$ kHz
 XIV = leukogabbro, $f = 325$ kHz
 XV = norit, $f = 400$ kHz

Influence of Mineral Composition and Texture on Q

The very important influence of grain size and texture is most clearly demonstrated in Fig. 6, which shows $Q(T)$ of three monomineral samples consisting exclusively of SiO_2 . XVI belongs to a natural single quartz crystal measured at 250 and 400 kHz, XVII to a quartzite rock with tightly connected mineral grains of a typical dimension of 0.5 mm and XVIII to a chalcedon of tufty grown crystals of large specific grain surface. The highest Q -value obtained is 6200 for the single crystal at -170°C and 400 kHz and the lowest 140 for the very fine grain chalcedon at -70°C and the same frequency. There is a qualitative similarity of all four curves showing a minimum, a steep ascent, a plateau, and again an ascent shifting to higher temperatures with increasing textural complexity.

In Fig. 7 $Q(T)$ is shown for three gabbroic rock samples (anorthosite, leukogabbro, norit) of similar grain size. Although somewhat different in composition the major mineral constituent is plagioclase (71–95%). As one might have already expected from the experience made before, because of the similar texture Q in all three curves is of the same order of magnitude. The qualitative similarity with a flat minimum near -30°C and a maximum at -80°C apparently reflects the dominating influence of the plagioclase component. Q of a lunar anorthosite described elsewhere (Herminghaus and Berckhemer 1974) fits well into this group.

$Q(T)$ for several basalt samples with a considerable variation in density is shown in Fig. 8. While a loosely packed and porous rock has a low Q and a denser and more compact one of the same composition a higher Q , the qualitative character of $Q(T)$ for both is very similar. This systematic relation between ρ and Q does not hold for rocks of different composition.

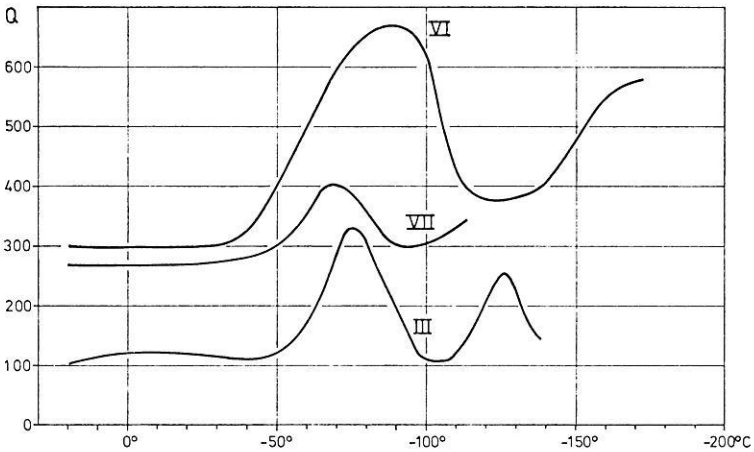


Fig. 8. Q as a function of temperature for volcanic rocks

$f = 400$ kHz

VI = feldspar-basalt, $\rho = 3,0$ gr/cm³

VII = basaltuff, $\rho = 1,98$ gr/cm³

III = olivinbasaltuff, $\rho = 1,36$ gr/cm³

Q as a Function of Water Content and Air Pressure

As suspected the presence of water in pores and fissures of a rock sample has a strong influence on its ultrasound absorption. To show this, a sample of pumice had completely been saturated with water.

After Q was measured at 20 °C and 760 torr, the wet sample was gradually exsiccated by shortly reducing the air pressure in the vacuum chamber. Measurements taken at 400 kHz are shown in Fig. 9. With decreasing water content Q increases almost exponentially. The Q -value of the saturated sample is only $1/6$ the value of the dry rock. It should be noticed that already small quantities of water lower Q considerably. Gordon and Davis (1968) have found a similar behaviour for wet granite. They believe that his effect is due to stress-induced fluid flow through thin, interconnecting channels in the rock and therefore increasing proportional to frequency.

Evacuation from atmospheric pressure to 10^{-1} torr caused in a very porous pumice sample an increase in Q by a factor of two, but no further increase when the pressure was lowered to 10^{-4} torr. Very similar results were reported by Jones (1972) for Q -measurements with standing waves of 6 Hz at lunar rock powder samples.

In compact rocks, however, evacuation from atmospheric conditions to 10^{-4} torr caused an increase in Q of 10 to 30 % only. Filling the recipient with dry, pure nitrogen gas did not affect Q of compact rocks. After the rock was exposed again to air, however, Q was brought back slowly to its lower initial value. It seems, therefore, that Q is influenced more by very

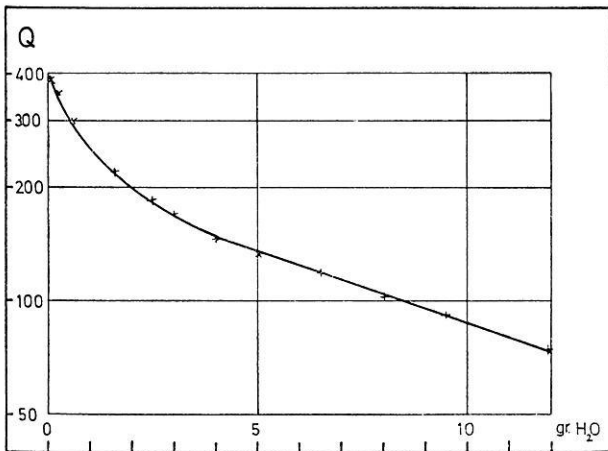


Fig. 9. Q as a function of water content in a pumice sample at $f = 400$ kHz. 12 gr H_2O corresponds to saturation

small amounts of adsorbed atmospheric vapors, predominantly water, than by dry gas. This is supported by another experiment in which a quartzite specimen was heated to $+200^\circ C$ at atmospheric pressure and thereafter slowly cooled down to room temperature in vacuum. Q at 10^{-4} torr had increased by 25%. Then the recipient was filled with dry nitrogen gas of 760 torr which did not effect Q . When the sample was exposed again to atmospheric air, however, it reached, after several hours, its initial lower Q -value. These observations are qualitatively comparable with those of Tittmann and Housley (1973)

Shock Cooling

When the sample slowly cools down by heat radiation or conduction to $-100^\circ C$ and thereafter warms up again to room temperature $Q(T)$ is reproducible. Curves IX a and IX b in Fig. 11, obtained from an obsidian sample during slow cooling and warming are therefore practically identical.

When, however, to lower the temperature in the sample to -150 or $-180^\circ C$, the sample had been exposed to a more or less intensive shower of liquid nitrogen as described in chapter 3, Q assumed considerably lower values in the following warming up period. Curve IX c (Fig. 10) shows a reduction of Q by 10 to 20% after the obsidian sample had been showered with liquid nitrogen. Shock cooling tests were undertaken at different materials (Fig. 11). The higher Q initially and the more compact the specimen the more pronounced is the irreversible reduction after shock cooling. The largest drop of 60% had been observed for a quartz crystal. Additional nitrogen showers, however, did not change Q any more.

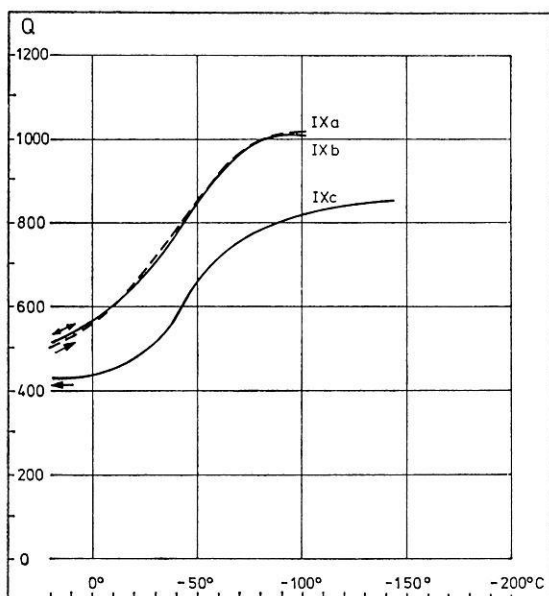


Fig. 10. Obsidian-sample during slow cooling down (IXa) and warming up (IXb) and after shock-cooling (IXc)

The irreversible drop of Q after shock cooling is apparently caused by friction loss at microcrack surfaces in crystal grains and along grain boundaries created by thermally induced stress. Since Q seems to be a quite sensitive measure for microcrack density which, in turn, is related to the strength of matter, the method of Q determination described here might be applicable to problems of technical material testing.

Microscopic studies have shown a high microcrack density in lunar surface rock samples (Chung, 1972). These cracks may have been caused either by the multitude of temperature cycles between lunar day (+ 130 °C) and night (- 160 °C) or by shock waves of meteoritic impacts. The lunar anorthosite 60015.33 studied by us showed, indeed, a surprisingly low and flat $Q(T)$ -curve which could not be lowered anymore by rapid cooling with a spray of liquid nitrogen. This behaviour is very unusual for terrestrial materials and can only be understood if the rock had already a high microcrack density and therefore an insensitivity to strong temperature variations.

6. Discussion

As indicated in the introduction no attempt will be made here for a detailed interpretation of the results in terms of solid state physics but a few remarks should be made to summarize the results of these studies. The

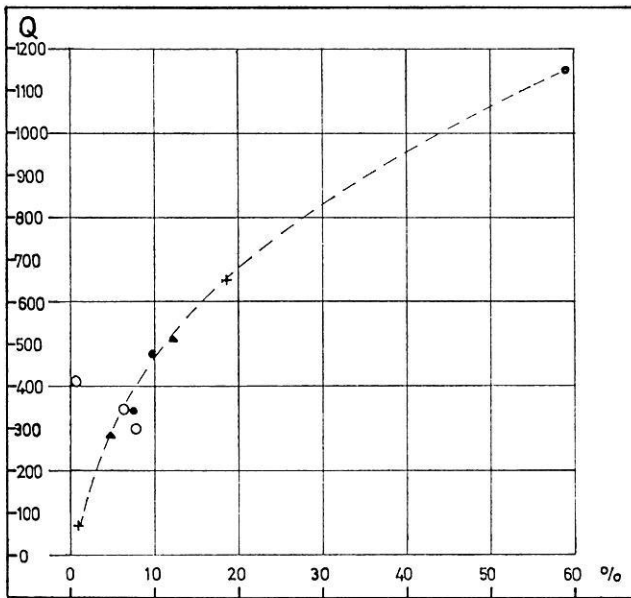


Fig. 11. Relative drop of Q (in %) after temperature shock for rocks of different initial Q -values

absorption of elastic waves is caused by several mechanisms which alone or superimposed are responsible for a frequency dependent or independent Q .

From the strong correlation between texture and Q we conclude that grain boundary effects play a dominating role in sound absorption. It seems that the general background level of the absorption $1/Q$ which might be attributed to sliding at microcrack surfaces and ultra low energy dislocation processes in the grain boundary region is less frequency dependent than the peaks in the $Q(T)$ curve. The trend of a Q growing with decreasing temperature is expected for all processes in which an activation term of the type $\exp - E/KT$ is involved. The shock-cooling experiments in particular emphasize the importance of friction processes at crack surfaces but of course this process must stop if a rock is under sufficiently high hydrostatic pressure.

Friction-type mechanisms with a frequency independent Q have proposed e.g. Knopoff (1964) and Walsh (1966). A special type of structurally controlled absorption is that one by fluid motion in pores and fissures. This is an important process for ultrasound absorption in wet rocks but because of its frequency dependency probably of little significance in the seismic frequency range.

Another important result is the fact that rocks of equal or similar mineralogical composition but of different texture have qualitatively similar $Q(T)$ curves. This characteristic behaviour has also been found in a quartz crystal with no noticeable texture. This means that there must be a group of intrinsic absorption mechanisms typical for a specific micro lattice structure and binding force system. Also here only attenuation mechanisms with extremely low activation energy can be relevant, such as dislocation kink movement (Bordoni effect) or certain low energy atom displacement processes. The existence of peaks and troughs in the $Q(T)$ curve suggests that most of these processes are some kind of relaxation process with a certain, but temperature dependent, relaxation frequency. The observation that the peaks in $Q(T)$ shift somewhat with texture may indicate that also these mineral specific attenuation processes are somehow related to the grain boundary.

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