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DYNAMIC FRACTURE PRESSURE OF QUARTZ

by

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JDKnapton/ICStobie/nlg
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January 1969

DYNAMIC FRACTURE PRESSURE OF QUARTZ

ABSTRACT

The dynamic fracture pressure of quartz when mounted in a simple pressure transducer was measured based on pressure steps with rise times of 0.5 to 1 msec. Maximum measured dynamic fracture pressures were about 4 kbar (52 to 62 kpsi). Both natural and synthetic quartz samples were studied. The apparatus consists of a gas operated pressure vessel which utilizes the stretched stem principle for achieving fast valve opening times.

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I. INTRODUCTION

The continuing requirement of the U. S. Army for measuring pressure in guns and rockets prompted the present investigation. Evaluations of new weapons have required improved pressure transducers. The purposes of this investigation were the following:

1. Design and build a pressure step generator suitable for delivering reproducible and positive pressure steps with rise times less than 1 msec and pressures up to 80 kpsi on axially loaded samples with areas of a few square millimeters,

2. Determine a value for the dynamic fracture pressure of small quartz samples when mounted in a simple pressure transducer,

3. Modify the pressure step generator for use from cryogenic temperatures up to around 400° C,

4. Evaluate piezoelectric and other pressure sensitive materials over the above temperature range[1*] for possible use as pressure transducers, and

5. Subject Group II - VI compounds to pressure steps followed by an examination of the compound for bulk variations in electron mobility and lifetime by using a short pulse electron beam system.

This report summarizes the preliminary results on the first two objectives. Although more data would be desirable concerning the dynamic fracture pressure of quartz, the data presented here will at least represent a lower limit at the loading rates and the sample preparation and mounting methods which were used. An evaluation of samples obtained from different sources was also considered worth while due to the general trend in materials improvement in recent years and since the fracture pressure is dependent on the degree of sample perfection.

The pressure step generator represents an extension to higher pressures of gas operated systems used earlier. The apparatus provides a simple method for subjecting samples to reproducible pressure steps with rise times of about 0.5 msec.

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* References are found on page 22

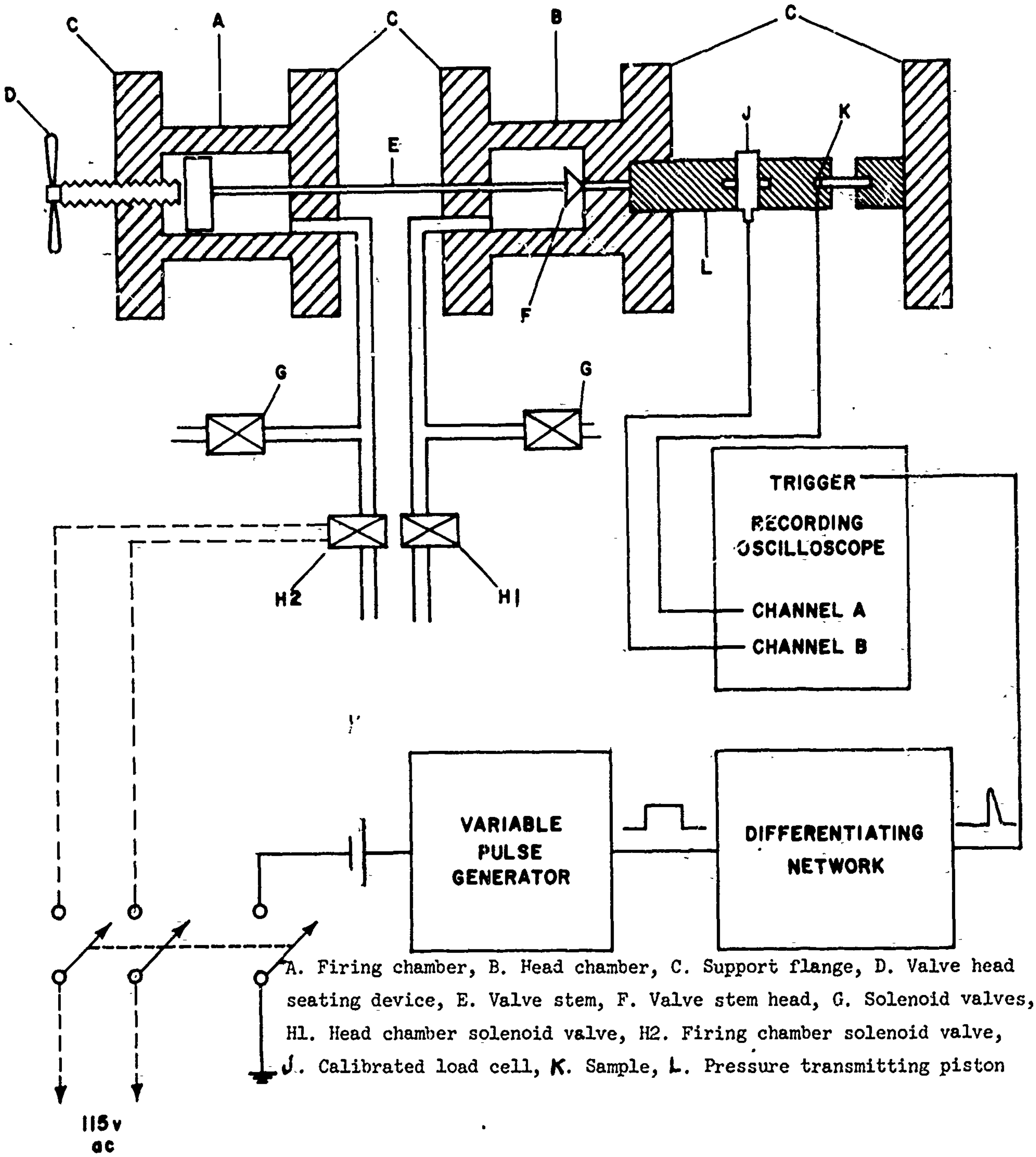
II. EXPERIMENTAL PROCEDURE

A. Pressure Step Generator

The pressure step generator was designed after the work of Lederer[2], Aronson and Waser[3], and Dykstra[4]. The device is based on the stretched stem principle. A valve stem is constrained at the pressure sealing end and is pulled at the opposite end. Potential energy is stored in the stem while it is stretched. The pulling force on the constrained end is continually increased until the constraining force plus frictional retarding forces are overcome. At this instant the entire stem moves, and simultaneously, the initially strained end snaps back due to the stored potential energy. The result is a fast acting valve giving pressure rise times of less than 1 msec.

The sealing end of the valve stem is a conically shaped valve stem head of soft material to insure a proper pressure seal and of low mass to minimize inertial effects. A pressure tight seal is made by forcing the soft valve head onto the steel edge of a cylindrical cavity. The valve stem head is made of nylon and threaded (0-80 screw thread) onto the valve stem.

A head chamber (B, in Figure 1) pressure of 2000 psi operating on a 0.244 sq in. piston was considered adequate for delivering pressures up to 80,000 psi on samples of a few square millimeters. In actual operation the head chamber is closed off by seating the valve stem head, F. This step is accomplished by simply rotating the valve head seating device, D, which forces the nylon valve head onto the valve seat. The head chamber is then pressurized to the desired pressure by opening solenoid valve, H1. The valve stem head is opened by first withdrawing the valve head seating device and then pressurizing the firing chamber, A, by opening a second solenoid valve, H2. The sudden contraction and displacement of the valve stem allows the gas in the head chamber to enter a relatively small cavity where the gas imparts a force on a pressure transmitting piston, L, which is mounted adjacent to a calibrated load cell, J, and the sample, K, under study.



A. Firing chamber, B. Head chamber, C. Support flange, D. Valve head seating device, E. Valve stem, F. Valve stem head, G. Solenoid valves, H1. Head chamber solenoid valve, H2. Firing chamber solenoid valve, J. Calibrated load cell, K. Sample, L. Pressure transmitting piston

Figure 1. Schematic, Pressure Step Generator

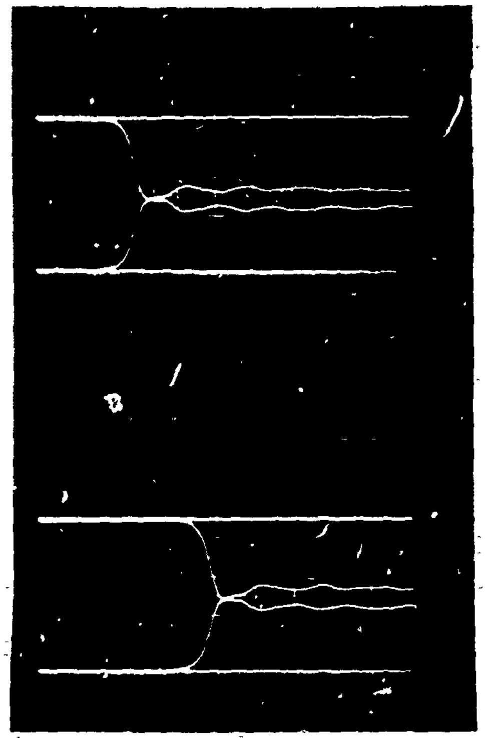
The charge developed across the load cell and the sample was recorded on a chopped dual sweep oscilloscope with a camera attachment. A recording of the desired event was usually taken at 1 msec/cm sweep speed in order to reveal rise time detail. Proper synchronization was obtained between the opening of the solenoid, H2, and the trigger pulse for initiating the oscilloscope sweep by using a variable pulse length generator and a simple differentiating circuit. A recording of two pressure steps is shown in Figure 2 which indicates rise times of about 0.7 msec. The recordings also show the degree of reproducibility.

In general, the rise time probably depends on the amount of stem elongation (given in Table 1), the rate of loading of the unconstrained end of the valve stem, and the number of components mounted between the pressure transmitting piston and the rear support flange, C. For example, by recording the charge output with only the load cell mounted, it was found that the rise time depended somewhat on the firing chamber pressure. The rise time reached a minimum of 0.4 msec with about 400 psi (or greater) applied to the firing chamber. Figure 3 shows a pressure step recording with only the load cell mounted. The rise time from the load cell is approximately 0.4 msec.

The two pressure chambers were machined from Elastoff 44 and sealed with buna N "O" rings and back-up rings. The valve stem is 1/16 in. diameter drill rod and is supported between the chambers and also in the head chamber. It was found that a minimum pressure of 200 psi in the head chamber is required to effect a seal by the valve stem head when the valve head seating device is withdrawn. Additional characteristics of the pressure step generator are given in Table I.

B. Sample Preparation

Natural x-cut quartz crystals used earlier in pressure gages were cut into squares 2mm on a side and also 4mm on a side. The thickness of the crystals was 1mm. Some of the crystals were cut with a diamond saw while others were cut with a wire cutter. The crystals obtained by both cutting procedures were investigated "as cut" and after annealing at about 1130°C.

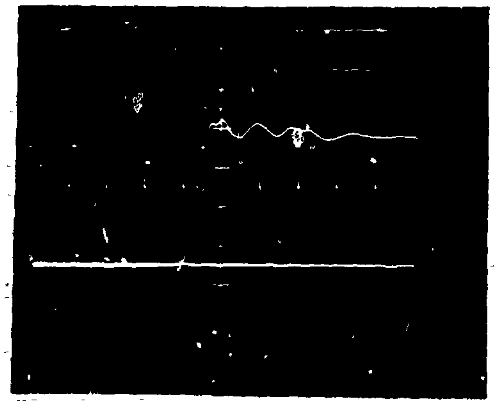


A

B

A typical recording showing 2 pressure steps indicated by A and B. For each pressure step the upper trace represents the unknown quartz sample and the lower trace represents a calibrated load cell. The pressure increase on the quartz sample is 25 kpsi and the sweep speed is 1 msec/cm with time going from left to right.

Figure 2.



A pressure step showing a rise time of 0.4 msec., the fastest rise time achieved. The firing chamber pressure was 420 psi and the valve head chamber pressure was 639 psi. The sweep speed is the same as in Figure 2.

Figure 3.

Table I. Characteristics of the Gas Operated Pressure Step Generator

Pressure Range	0.2 to 2kpsi	minimum rating limited by lowest pressure seal attainable in valve head chamber (see Text); maximum rating limited by solenoid valves
Pressure Transmitting piston	diam 0.558 in.	
Head Chamber reservoir	i.d. 1.626 in.	
	inside length 3.5 in.	
	volume 7.27 cu. in.	
Valve head stem	seal diam 0.25 in.	
Stem diam	0.0625 in.	
Stem area	0.00307 sq. in.	
Stem length	11.5 in.	
Force on valve head	9.8 to 98 lbs.	Based on above pressure range
Stem stress	3.2 to 32 kpsi	Based on above pressure range
Stem elongation	0.001 to 0.01 in.	Based on above pressure range, neglecting frictional forces, and assuming a modulus of elasticity of 3×10^7 psi

Additional quartz crystals, of synthetic origin, were obtained from the Valpey Corporation[5]. These crystals were cylindrical, x-cut, and 1mm in thickness. Two groups were obtained: 2mm in diameter and 4mm in diameter. The crystals were specified as being optically polished on a 1 wave length flat and with parallel surfaces to 0.00005 in. Additionally, the edges were fine ground with 1 - 3 micron powder, annealed and without strains or edge defects.

III. DYNAMIC FRACTURE PRESSURE OF QUARTZ

A pressure of about 15 kpsi[6] is usually given as the practical working stress of quartz. This working pressure is considerably lower than the typical crushing pressure of quartz at one atmosphere. References[7,8] indicate a crushing pressure for small quartz samples of about 22 kbar (320 kpsi). Graham,[9] studying synthetic quartz under high transient stress and impacted by projectiles, obtained a linear charge release up to 25 kbar (362 kpsi) for + X orientated crystals (following the sign convention of Graham) and a linear charge release up to 8 kbar (116 kpsi) for -X orientated crystals. For the +X orientation a discontinuity in charge release vs pressure occurred at 34 kbar (493 kpsi). This discontinuity was attributed to gross mechanical yield. Since these crystals were destroyed on impact,[10] a determination of the actual fracture pressure would be difficult. The fracture pressure could possibly be inferred from the charge release data assuming the period of observation is of sufficient duration to include dislocation line motion, or other effects, leading up to fracture.

The rather low working pressure of quartz is not surprising since quartz at room temperature remains "brittle and elastic" under mechanical deformation up to the moment of fracture.[11] Furthermore, the presence of unavoidable microscopic surface line defects (for synthetic crystals, 10-10^4 per cm^2)[12] may produce stresss concentrations capable of initiating early fracture in the larger specimens.

An attempt was made in the present investigation to reduce the effect of early fracture by mounting the crystal between two easily deformable electrodes. The crystal was cemented onto a copper electrode using a cellulose nitrate type cement. The initial thickness of the copper electrodes was 1/64 in. The opposing copper electrodes and the mounted crystal were then simply mounted in a nylon sample holder.

Each crystal was subjected to two pressure steps, and each pressure step was recorded as shown in Figure 2. The crystal was inspected for possible fracture after the second pressure step. If no fracture was evident, the free electrode was repolished with 4-0 emery paper (about 10 - 15 micron particle diameter) and the components reassembled in the sample holder for additional testing. Pressure step rise times generally varied between 0.5 and 1 msec. A series of such recordings, in addition to providing fracture pressure data, also provided charge output vs pressure data from which plots such as Figure 4 were made. It is interesting to note, as illustrated in Figure 4, that the linear response of the gage continued beyond the fracture point. This was a general observation and is due to the continued alignment of the fractured sections which remained in their initial positions.

Extrapolating back to the abscissa in Figure 4 gives a value for the initial loading pressure. This value represents a small correction to the total fracture pressure and was added onto the experimentally observed fracture pressures. Given in Table II is a summary of the dynamic fracture pressures obtained from the various types of quartz samples. The fracture pressure is given as a range between two values: the upper value represents the pressure where fracture actually occurred; the lower value represents the pressure in the preceding test where fracture did not occur.

It is apparent from Table II that dynamic fracture pressures approaching 40 kpsi can be expected for the smaller crystals and for the

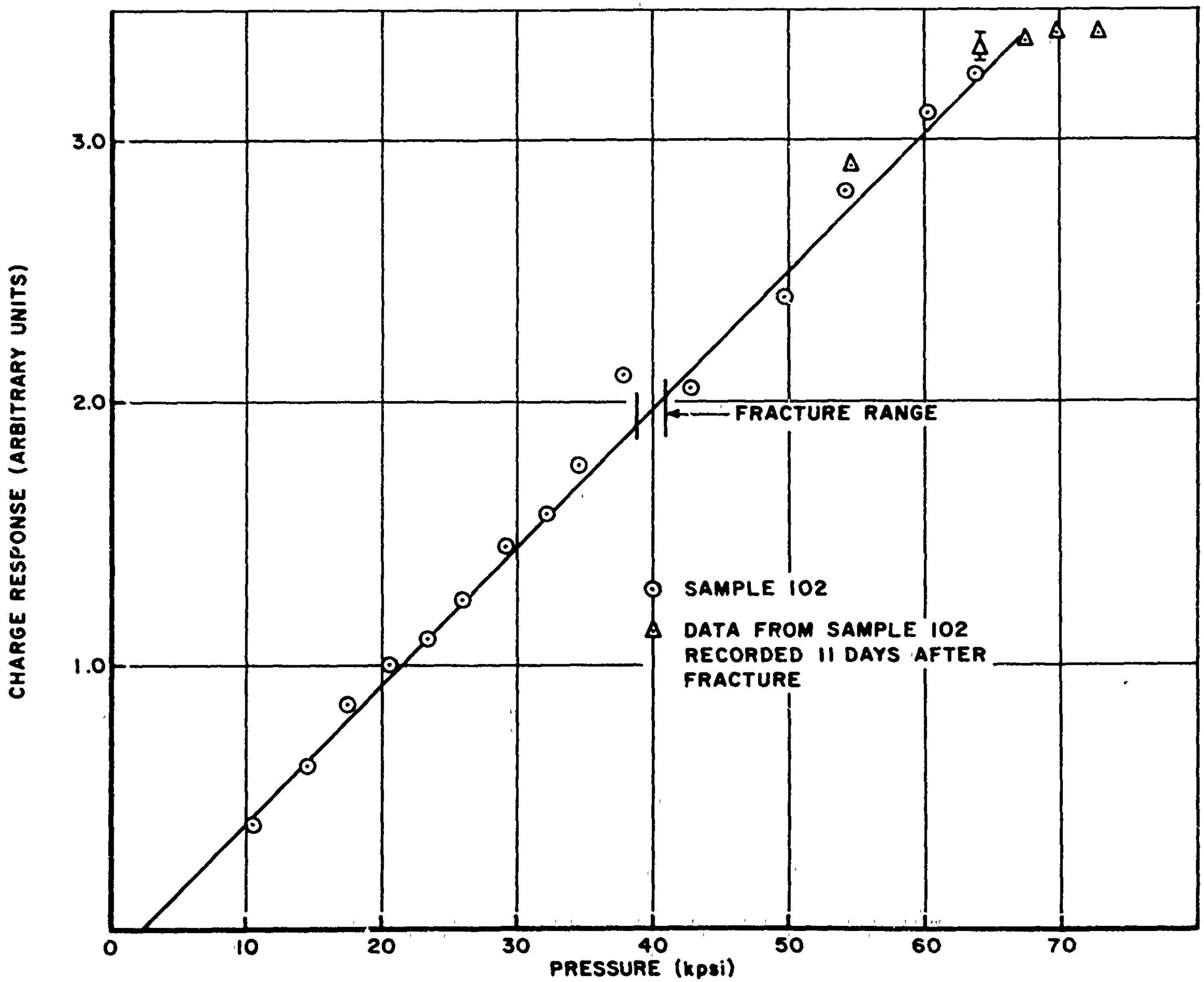


Figure 4 - Charge Response vs Pressure

Table II. Dynamic Fracture Pressure of Quartz

Sample Origin	Size (mm)	Cutting method	Heat Treatment	Fracture Pressure (kpsi)
natural	4x4	diamond	not annealed	< 5.5
natural	4x4	diamond	not annealed	7.3 - 8.4
natural	4x4	diamond	not annealed	11.9 - 12.8
natural	2x2	diamond	not annealed	< 19.1
natural	2x2	diamond	annealed	20.9 - 24.2
natural	2x2	diamond	annealed	58.4 - 62.2
natural	2x2	diamond	annealed	25.1 - 29.1
natural	4x4	wire	not annealed	< 7.2
natural	4x4	wire	not annealed	< 5.3
natural	2x2	wire	not annealed	22.7 - 25.3
natural	2x2	wire	not annealed	10.7 - 14.7
natural	2x2	wire	not annealed	19.3 - 25.7
natural	4x4	wire	annealed	4.7 - 5.3
natural	2x2	wire	annealed	29.2 - 32.4
natural	2x2	wire	annealed	37.6 - 40.8
natural	2x2	wire	annealed	38.5 - 41.2
synthetic	4 (diam)			10.5 - 11.6
synthetic	4 (diam)	all synthetic crystals tested		15.4 - 17.7
synthetic	4 (diam)	as received (see text).		10.2 - 11.3
synthetic	2 (diam)			52.0 - 57.0
synthetic	2 (diam)			34.0 - 39.0

loading rates employed. Of the limited samples investigated, the wire cut natural crystals generally fractured at higher pressures than the diamond cut natural crystals. One exception was an annealed diamond cut crystal which fractured at approximately 60 kpsi. Annealing both the wire and diamond cut crystals at 1130°C resulted in higher fracture pressures. Reducing the size of the crystals, for both the natural and synthetic quartz, increased the fracture pressure which suggests the part that surface line defects may have in controlling the fracture pressure.

IV. GAGE APPLICATIONS

The use of short circuited quartz gages for providing data from single shot (gage destruction) tests under high dynamic shock loading is well documented by Graham and others[13] at Sandia Laboratory. The advantages of their short circuited current generating gage lie in a fast time response ($\sim 10^{8}$ sec) and in a capability for measuring high pressure shocks (up to 25 kbar). However, the short circuited gage may produce pulse distortion unless the electromechanical coupling of the gage is relatively small[14] and the effects of ionized impurities are absent.[15]

O'Brien and Wasley[16] have described methods for packaging the short circuited transducer. The completed transducer was described as having its greatest value in determining the times of arrival of various waves in a disturbance with multiple wave structure. However, no information was given on gage repeatability or on gage failure conditions such as maximum pressure and minimum stress pulse duration.

The use of quartz gages in gun or rocket applications requiring reasonable repeatability poses a different problem. Blackstock et al[17], who were concerned with gage fracture, have used quartz in an acoustic mismatch gage using two dissimilar metals to reduce the pressure on the quartz. Pressure pulses were measured up to about 5 kbar (72 kpsi) with a pulse rise time of 3μsec. Recording of higher pressures was limited due to quartz fracture which was probably caused by a reflected wave giving rise to a tensional stress. Generally, the tensional stress caused by reflected waves may well be the limiting factor in establishing an upper limit for gage applications for those uses where the duration of the pressure pulse is short compared with the stress pulse transit time.[17] The tensional stress limit for quartz as determined by Blackstock et al [17] was about 1 kbar (14.5 kpsi). Fracture could therefore be expected for those cases involving a 1 kbar or less tensional stress pulse with a duration less than about 1 μsec assuming a 1mm thick crystal and a stress velocity of 3.8×10^5 cm/sec.

The present investigation was concerned with a pressure step, having a relatively long step rise time, and problems with reflected waves probably did not occur. However, due to the static component of the pressure step, the present tests may impart a more severe condition to the gage than tests involving a long duration (to reduce the effect of reflected waves) pressure pulse.[17] Nevertheless, two assembled quartz pressure transducers, one natural and one synthetic, attained pressures of about 4 kbar (52 - 62 kpsi, Table II) before fracturing.

The use of tapered pistons[6] would suggest even higher gage pressures; however, Blackstock et al[17] encountered considerable pulse distortion with tapered pistons. They attributed the pulse distoration to a coupling between the longitudinal and other modes of vibration and to velocity dispersion of the stress pulse (caused when the duration of the pressure pulse is short compared with the time required for the pulse to travel a distance equal to the diameter of the gage).

It is conceivable that higher fracture pressures could be achieved if the present investigation were continued. Reducing the size of the crystal might offer significant improvement which was certainly the case in going from a 4mm diameter sample to a 2mm diameter sample. An additional reduction of diameter by a factor of 2 would not be objectionable with respect to the signal to noise ratio or the sample size.

Additional improvements in the sample preparation and mounting procedures might also yield higher fracture pressures. Polishing the electrodes with a finer grit, etching the crystal[19] to smooth out incipient dislocation fracture lines, and selecting crystals based on etch patterns (which may be used for indicating defect density)[19] might all result in significant improvement. Further improvements in the method of electrode mounting might also result in higher fracture pressure. A better acoustic match between the quartz and the electrodes, such as aluminum electrodes, might also give higher fracture pressures. However, aluminum would yield at pressures greater than 25 kpsi with a probable change in the impedance match to the quartz crystal.

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