

FURTHER STUDIES ON THE
HYDROTHERMAL STABILITY OF QUARTZ

by

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A THESIS SUBMITTED IN PARTIAL FULFILMENT OF
THE REQUIREMENTS FOR THE DEGREE OF
MASTER OF APPLIED SCIENCE
in the Department
of
GEOLOGY AND GEOGRAPHY

We accept this thesis as conforming to the
standard required of candidates for the
degree of MASTER OF APPLIED SCIENCE.

Members of the Department of
GEOLOGY AND GEOGRAPHY

THE UNIVERSITY OF BRITISH COLUMBIA

April, 1954

ABSTRACT

Quartz and quartzite are subjected to moderate confining pressures and compressive stresses in water and alkaline media. Planes of liquid inclusions in quartz are not affected nor is quartz recrystallized under the experimental conditions imposed. Calculations of the pressure unit's limitations are made, and modifications for its future use are suggested. A complete analysis of sodium disilicate, produced in the experiments, is presented.

C O N T E N T S

	Page
ABSTRACT	i
INTRODUCTION	1
ACKNOWLEDGEMENTS	3
APPARATUS	4
PHYSICAL CALCULATIONS	6
Pressure Bomb	6
Vise	9
RECOMMENDATIONS FOR BOMB AND VISE MODIFICATIONS	13
EXPERIMENTAL PROCEDURE	15
EXPERIMENTAL RESULTS	15
Group I Experiments (Nos. 1,2,3, and 9)	17
Group II Experiments (Nos. 4,5,6,7, and 8)	18
Group III Experiments (Nos. 10, 11, and 12)	20
NEW DATA ON SODIUM DISILICATE	23
Summary of Properties	23
Composition Calculations	25
Phase Relations	27
CONCLUSIONS	28
BIBLIOGRAPHY	29
ADDITIONAL REFERENCES	31

ILLUSTRATIONS

	Page
Figure 1. Furnace, Pressure Bomb, and Temperature Recorder	4
2. Vice Assembled for Bomb Run, with Static Loading Bar in Place	5
3. Pressure Bomb	4b
4. Assemblage Diagram of Vice	5b
5. Specific Volumes of Water	7
6. Sodium Disilicate Crystals Formed on Quartz Prism Faces	23
7. Sodium Disilicate: X-ray Powder Photograph with Iron Radiations, Manganese Filter	24
8. The Optic Orientation of Sodium Disilicate	25
Table I	
I Volumes of Parts and Changes of Volumes Caused by Pressure and Temperature Changes	8b
II Specifications, Physical Constants, and Dimensions of Materials used in Experiments	12b
III Summary of Experimental Data	16b & 16c
IV Lattice Spacings of Sodium Disilicate	24b
V Chemical Analysis of Synthetic Silicate and Atomic Proportions of Elements Derived from the Analysis by Approximations	25b

INTRODUCTION

Many data in the fields of ore genesis and metamorphic petrology suggest the possibility that quartz and a few other minerals can become mobile under appropriate conditions, recrystallizing in situ to form new textures in which grains have new optical orientations. Thus the metamorphic change from sandstone to quartzite is expressed by a change in texture and, very commonly, by the development of a lineate fabric.

A well-known feature of quartz, particularly vein quartz, is its clouds of inclusions, both solid and fluid. When distributed at random, these inclusions, — or 'vacuoles' — are considered to be primary material which has been trapped in the quartz during crystallization. Smith (1951) has compiled an exhaustive list of the contributions to this particular branch of geology. A more remarkable feature of quartz, however, is the occurrence of well-defined planes of disconnected inclusions that cross grain boundaries without disruption. This planar distribution of vacuoles, usually accompanied by flambouyant or mottled extinction of the quartz, implies recrystallization under conditions quite different from those under which the quartz originally crystallized.

What might these conditions be? Griggs (1938) has shown that quartz is remarkably stable under conditions of elevated temperature and extreme compressive stress, but becomes mobile when a shearing stress is superimposed. Similar effects are now being produced under more natural

conditions by Handin (1953) and others. Such experimental results support the field evidence that high differential stress was present during dynamic metamorphic changes and during the emplacement of most deep-seated veins. The planes of inclusions described above must in some way be related to such differential stresses, even though little other evidence may be present to indicate that deforming forces once existed.

The present series of bomb experiments was designed to study the effect of elevated temperature, moderate hydrostatic pressure, and moderately strong differential stress on the stability of quartz, and on the distribution of inclusions in quartz and quartzite.

The pressure bomb used was a large-chamber type which accommodated a vise for producing a compressive stress on the test piece. The internal stresses produced in the quartz are 'differential' in nature; that is, a tendency of the test piece to shear in one direction or another exists, according to the strain ellipsoid hypothesis (Fairbairn, 1949, p. 199).

The experiments were of three main types:

- (1) those in which quartz was being treated in pure water;
- (2) those in which quartz was being treated in a 5% solution of sodium carbonate;
- (3) those in which quartz sandstone and quartzite were being treated in pure water.

No positive results were obtained in these experiments. It is felt, nevertheless, that this is a fruitful field for continuing research. This paper is, therefore, in the nature of a progress report treating the following subjects:

- (a) Description of the apparatus,
- (b) Limitations of the apparatus,
- (c) Recommendations for modification of the apparatus;

- (d) Experimental results;
- (e) New data on sodium disilicate.

ACKNOWLEDGEMENTS

This study was made possible by a research grant from the Department of Mines and Technical Surveys. The writer wishes to express his appreciation of this grant and of the efforts made by Dr. W.H. White to obtain it. During the course of experimentation invaluable assistance, criticisms, and suggestions were given by Drs. W.H. White, R.M. Thompson, K.C. McTaggart, and H.C. Gunning, and to them the writer extends due thanks. The interest in the project shown by other members of the Geology and Mechanical Engineering Departments of this University has been highly gratifying.

APPARATUS

The apparatus consists of three units: an electric pot furnace and regulator, a pressure bomb, and a vise for producing a differential pressure on the object being examined (Figures 1 & 2).

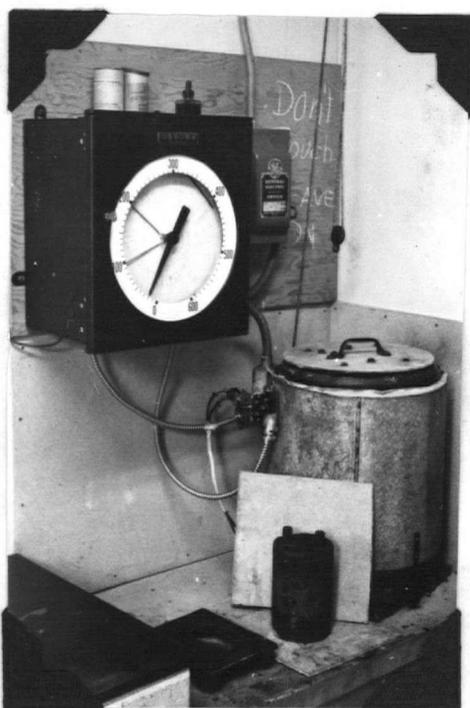
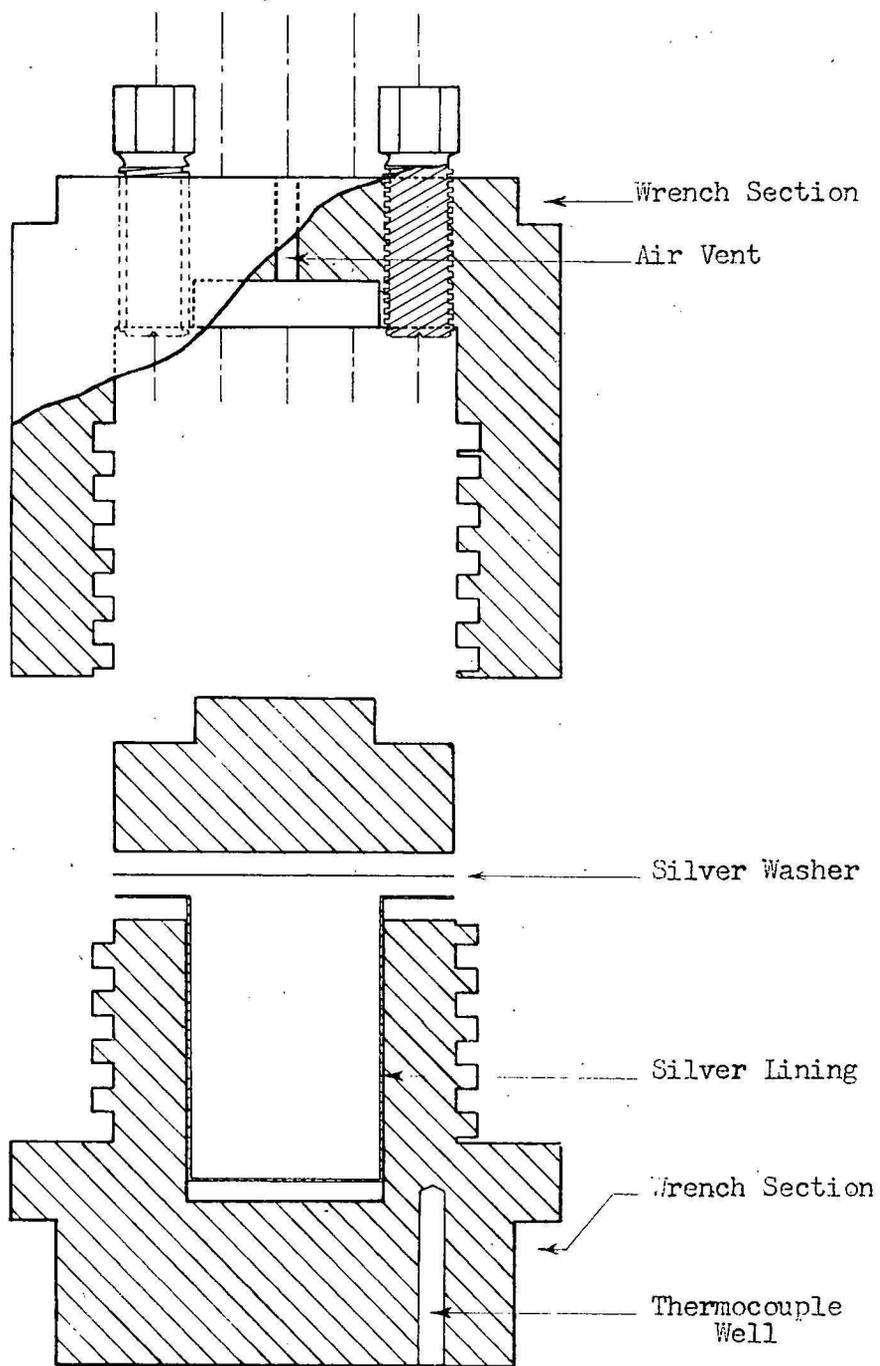


Figure 1. Furnace, Pressure Bomb, and Temperature Recorder

The furnace was designed especially to accommodate the bomb. Six elements are regularly spaced about the well-insulated periphery of the furnace to provide equal heating throughout the interior. Three thermocouple stems protrude into the base of the furnace, one on either side of the bomb space for registering the furnace temperature, and one near the center for obtaining the internal temperature of the bomb. The last mentioned thermocouple fits into a hole drilled in the base of the bomb. The thermocouple leads pass through the base of the furnace to a timing

Figure 3.



PRESSURE BOMB

Scale : X $\frac{1}{2}$

Supplement to Design Given by F.G. Smith

device designed by Dr. W.H. White which permits recording of the furnace temperature constantly, and the internal temperature of the bomb at half-hour intervals. A Brown Electronik recorder controls the furnace temperature and registers it constantly.

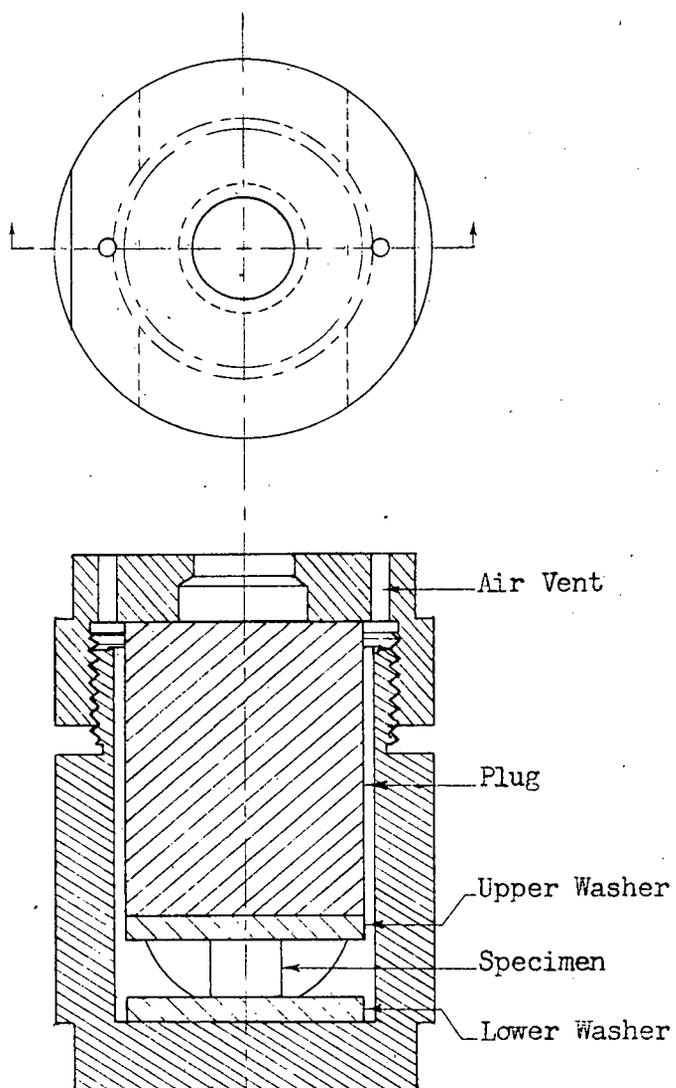


Figure 2. Vise Assembled for Bomb Run, with Static Loading Bar in Place

The pressure bomb is a replica of the type designed by F.G. Smith (1947); a drawing showing its construction is given in Figure 3. The silver lining is used to obtain an effective seal at the joint between the cap and base. The silver lip and washer partly fuse together under pressure. Internal pressures up to a minimum safety limit of 12,000 atmospheres can be maintained by the bomb, although yielding of the wall begins at approximately 4,000 atmospheres. The high confining pressures reduce the tendency of the test object to rupture under the stress produced by the vise.

The vise construction is shown in Figure 4. The case is made of a material having a low coefficient of linear expansion, and the plug

Figure 4.



ASSEMBLAGE DIAGRAM
OF VISE

Scale : Full Size

and washer of material having a high coefficient. The plug and washers thus expand more upon heating than does the case, and a compressive stress is applied to the object between the washers. The washers are used because they can be readily replaced should the elastic limit of the material be exceeded at any time. A slightly convex surface on the lower face of the base washer permits automatic adjustment to the faces of the confined test object should they not be parallel. When the object is mounted, a loading bar (Figure 2) is inserted through a hole in the vise cap, and an external force is applied to it. This static load increases the compressive stress and ensures a tight fit.

PHYSICAL CALCULATIONS

Pressure Bomb

The pressure bomb is designed to produce a hydrostatic, or confining, pressure by preventing water from expanding when heated. Water is only slightly compressible, hence a small change in temperature effects a large change in pressure. Smith (1953) has plotted the P-V-T relations of water graphically from data obtained by Kennedy (1950) and Bridgeman (1949), and all values used here were taken from this source.

The specific volume of water is the ratio of the volume at any given temperature and pressure to the volume at standard conditions, or

$$\text{Sp. Vol.} = \frac{V_{t,p}}{V_{stp}} \quad (1)$$

Values are obtained empirically. An example of the use of the graph (Figure 5) is as follows.

At 160°C, water has a vapor pressure of approximately

6 atmospheres. If allowed to expand against the vapor pressure alone, its specific volume would be 1.10; therefore, at 160°C, an inexpandible container 1.10 times larger than the volume of water used would be completely filled by water.

32

F. G. SMITH.

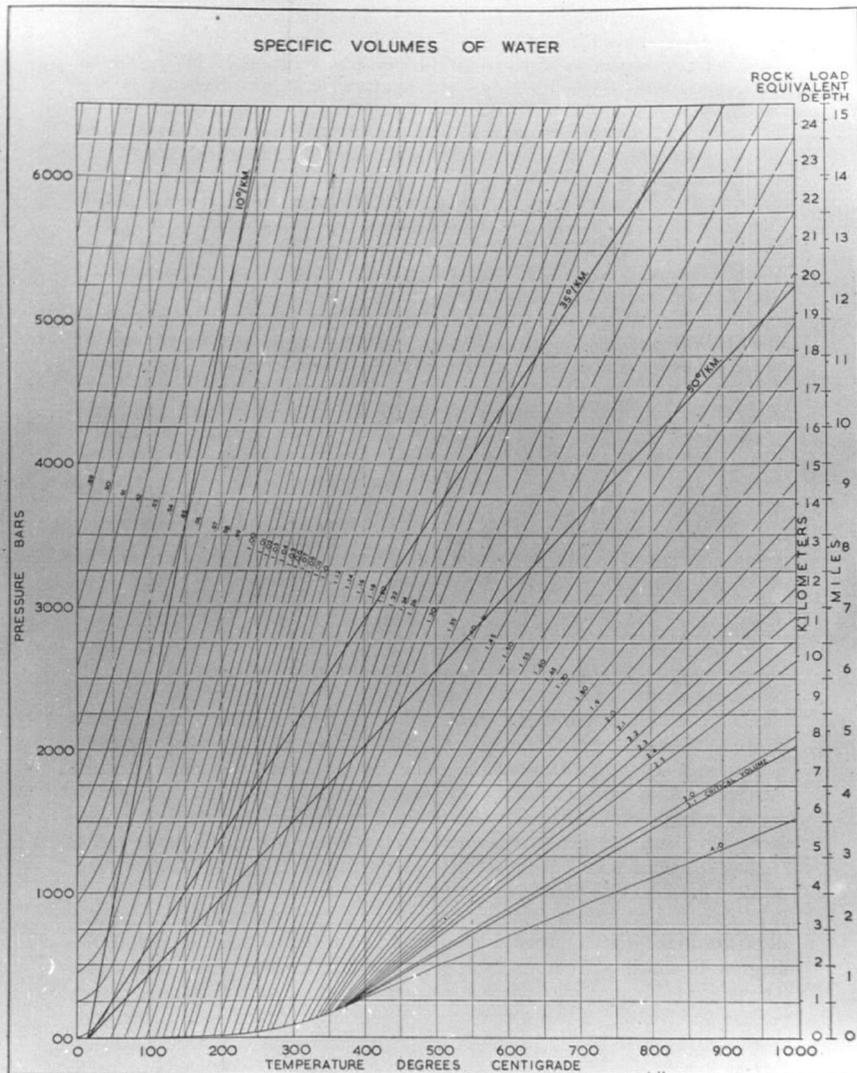


FIG. 3. P-V-T relations of water, with approximate maximum, minimum, and mean geothermobar.

Figure 5. Specific Volumes of Water
(after F.G. Smith, (1953) p. 32)

At temperature greater than 160°C, however, the water must expand against the container walls. If they are rigid, the water will be compressed, and the pressure exerted on the container walls by the water will be that pressure required to prevent the water from increasing its volume. The pressure-temperature relations above 160°C for a specific volume of 1.10 are shown by the appropriate water compressibility curve; if the container is filled to $\frac{1}{1.1}$ of its volume at room temperature, the pressure at any temperature above 160°C can be obtained directly from this curve. Conversely, the specific volume can be obtained for a desired pressure at a given temperature, and from this the volume of water required to produce the pressure can be determined.

Volume changes to be considered are:

- (a) increase of bomb volume with increasing temperature and hydrostatic pressure;
- (b) decrease of volume of the vise assembly caused by the pressure;
- (c) increase of volume of the vise assembly with increasing temperature.

The results of the calculations are given in Table II.

The volume changes calculated for (a) and (b) are added to the initial (room temperature) volume of the bomb, while that of (c) is subtracted, as is the initial volume of the vise assembly. The resulting (free) volume is divided by the specific volume to give the amount of water required for obtaining the desired pressure.

The probability of error is high in determining pressure by this method. The compressibility curves are largely extrapolated; air cannot be removed from the bomb chamber when filled to the desired level; the chamber volume cannot be obtained with an accuracy greater than

TABLE I.

Volumes of Parts and Changes of Volumes Caused by
Pressure and Temperature Changes

Unit	Volume (cc.)			ΔV from T = 320 C (cc.)		ΔV from T = 580 C (cc.)		ΔV from Pressure (cc.)	
								3500 At.	3000 At.
Bomb Chamber	150 (ave.)			2.3		4.2		2.1	1.5
Vise Assemblage	Case & Washers	ss Plug	Cu Plug	ms Case ss Plug	ss Case Cu Plug	ms Case ss Plug	Both Assemblages	Both Assemblages	
	78.0 (ave.)	30.2	27.8	1.5	1.8	2.3			0.5

Total Free Volume Change for Experimental Conditions	Δ Temp. (°C)	P = 3500 At.		P = 3000 At.	
		ms Case, ss Plug	ss Case Cu Plug	ms Case, ss Plug	ss Case Cu Plug
		320	+ 3.4 cc.	+ 3.1 cc.	+ 2.8 cc.
580	+ 4.5 cc.	-	-	-	

\pm 0.2 ml.; and leakage of the seal may occur.

Air trapped in the bomb would increase the total pressure according to the partial pressure it exerts. The effect is to decrease the specific volume, and to flatten the compressibility curve at temperatures near the filling temperature, but the actual and assumed curves converge with increasing temperature and therefore, at higher temperatures, the relative error is reduced. If the air is considered to be insoluble in water, a calculation using the ideal gas equation shows that at 3,500 atmospheres and 600°C, with an initial air space of 12.2 cc. (Experiment No. 12), the volume occupied by the air is 0.09 cc. The value is a maximum for all experiments, and is within the limits of error given for the volume of the bomb chamber; hence, the presence of an air phase can be ignored.

Errors arising from approximations in the compressibility curves and from the measurement of the chamber volume cannot be avoided; however, an error of \pm 100 atmospheres would probably affect the results of the experiments little, and this limitation was accepted in these experiments. Leakage of the bomb seal is intolerable, of course, for the pressure is immediately reduced an unknown amount below the estimated value.

Vise

The case of the vise used for the purpose of illustration was made of machine steel, and the plug and washers were of stainless steel; the effective difference in the expansion coefficients is 11.0 : 16.1. The test material was either quartz or quartzite.

Since the vise is subjected to the confining pressure produced in the bomb, compression of the various parts is expected. The vise

construction permits equalization of pressure across the case, but the plug, washers, and object will be compressed longitudinally, tending to reduce the effective pressure produced by expansion. Assuming that the maximum area of the plug is affected, calculations using the stress equation

$$\Delta L = \frac{PL}{AE} \quad (2)$$

and the expansion equation

$$\Delta L = L_0 \alpha t \quad (3)$$

show that the ratio expansion : contraction = 20:1; hence the hydrostatic factor can be neglected for the accuracy demanded here.

Failure of the vise can occur in two ways:

- (1) by shearing of the vise threads, which are the weakest part of the case;
- (2) by yielding of the washers under a stress exceeding that prescribed by the elastic limit of the material used.

Calculations are as follows:

- (1) Cross-sectional area of threads (A) = 1.1 ins.²

Shear factor for machine steel (f_s) = 50,000 lbs./in.²

$$F = Af_s \quad (4)$$

The force required to shear the threads is thus 55,000 pounds.

- (2) Minimum cross-sectional area of object under stress (A) = 0.25 in.²

Yield point for stainless steel under compression (Y)
= 21 (10⁴) lbs./in.²

$$F = YA \quad (5)$$

the force required to cause failure of the washer under compression is therefore 52,000 pounds.

Failure by compression will occur first, at a maximum pressure of 3,500 atmospheres.

The pressure on the object below the yield point is a function of the temperature change, lengths of the component parts of the vise assembly, the coefficients of expansion of the parts, and their moduli of elasticity. The fundamental equation, expressed verbally, is:

Lengthening of plug, washers, and object due to the increase in temperature minus shortening of plug, washers, and object due to compression by the vise

=

Lengthening of the case due to the increase in temperature plus lengthening of the case due to the expansion of the plug, washers, and object.

The washers are made of the same material as either the plug or the case, hence are incorporated with the measurements. Expressed symbolically with the appropriate coefficients and stress formulae, the equation is:

$$\begin{aligned} (L_{ss} \alpha_{ss} + L_Q \alpha_Q)t - P \left[\frac{L_{ss}}{E_{ss} A_{ss}} + \frac{L_Q}{E_Q A_Q} \right] \\ = \\ L_{ms} \alpha_{ms} t + P \left[\frac{L_{ms}}{E_{ms} A_{ms}} \right] \end{aligned} \quad (6)$$

The effective area for conditions of maximum stress will be that of the quartz faces. The equation now reduces to

$$P = A_Q T \frac{\left[L_{ss} \alpha_{ss} + L_Q \alpha_Q - L_{ms} \alpha_{ms} \right]}{\left[\frac{L_{ss}}{E_{ss}} + \frac{L_{ms}}{E_{ms}} + \frac{L_Q}{E_Q} \right]} \quad (7)$$

where

P = force on the quartz

A = area of compression face of object

t = temperature change in °C

L = length

α = coefficient of linear expansion per °C

E = Young's modulus of elasticity

ss = stainless steel

ms = machine steel

Q = quartz

The equation can be applied to any materials used in the vise. Young's modulus for metals decreases with increasing temperature according to the equation

$$E_t = E_0 \left\{ 1 - \left(\frac{T-32}{1700} \right)^2 \right\} ; \quad (8)$$

the moduli corresponding to the temperature reached should be used in equation (7). The constants used here were obtained either by experiment or by selection from dependable handbooks. Constants, variables, and measurements applicable to these experiments are listed in Table II.

The length of the object varies the effective length of the case. For these experiments the prism lengths were cut to 8.0 ± 1.5 mm.; this limitation gives an acceptable error of approximately 3% if a prism length of 8.0 mm. is used to reduce the equation to a convenient form. The pressure in atmospheres is required, and this is given by

$$S = \frac{P}{14.7A_Q} \quad (9)$$

Insertion of the various constants and lengths reduces the equation to the final form:

$$S = 3.83 t \text{ atmospheres} \quad (10)$$

TABLE II

Specifications, Physical Constants, & Dimensions
of Materials used in Experiments

Material	Young's Mod. (E) at 340 °C $\times 10^6$ lbs/in ²	Young's Mod. (E) at 600 °C $\times 10^6$ lbs/in ²	Coeff. of Lin. Expn. $\times 10^6$ /°C	Yield Point $\times 10^4$ lbs/in ²	Total Axial Lengths (ins.)	
					ms Case ss Plug (23°C)	ss Case Cu Plug
Tool Steel (Base of bomb) Atlas SFC 245	28.9	26.6	16.1	21 (340°C) 13 (600°C)	-	-
Machine Steel (Vise Case) Atlas 'Superior)	28.0	25.8	11.0	6.5	2.097	-
Stainless Steel (Washers, Case, Plug) Atlas 316	26.6	24.5	16.1	8 (ult.)	(Plug & Wash.) 1.782	(Case) 1.705
Copper (Plug) 99.5% Cu	17.4	16.0	17.7	21	-	1.390
Quartz	14.0	3.0	10.0	375	.315	.315

for the stainless steel plug and machine steel case and

$$S = 0.105 \text{ t atmospheres} \quad (11)$$

for the copper plug and stainless steel case. The total axial pressure on the prism is given by the sum of the static, differential, and hydrostatic pressures.

RECOMMENDATIONS FOR BOMB AND VISE MODIFICATIONS

High-pressure experimentation is being carried out in several places at the present time. The devices used to attain the desired pressures are numerous; but they are also expensive. The bomb used in the present experiments is not elaborate, and is inexpensive in comparison with other types; but it is also ineffective. Leakage invariably occurs at high pressures,^{and} confining pressures are not known.

This serious fault can be largely eliminated by using a wedge seal, which makes use of increasing pressure to produce a tighter joint. Niemeier (1953) has discussed several types now being employed. Wedge seals have a distinct advantage over contact seals: only a small initial bolt load is required, the parts are readily separated, and the seals automatically adjust themselves to any elastic displacement of the vessel. Such a seal should be used on this bomb.

Although calculation of confining pressures by the method described appears to be sufficiently accurate, the question always remains whether or not the pressures were actually obtained. A direct measurement of pressures produced in the bomb is therefore desirable; this may be obtained by passing a direct-transmission tube with a fine bore through

the bomb cap and attaching it to a high-pressure gauge or constant-recording unit. The protruding tube would be only slightly inconvenient during assembly of the bomb.

The compressive stresses obtained by the vise are much too low if resistant materials are to be deformed. Griggs (1938) found that quartz would not flow under a compression of 140,000 atmospheres, but such extreme pressures are not necessary in favorable chemical and stress environments, or when time is allowed for adjustment of the material to the deforming stress. The vise may be effective if materials having a large difference in expansion coefficients are employed, such as molybdenum for the vise case and a highly expansible steel for the plug and washers. The maximum stress obtainable, of course, is governed by the yield point of the washers or the tensile strength of the case.

A rotational shear may possibly be produced by seating two pairs of small secondary plugs in the wall of the vise case so that they exert opposing tangential forces on the upper and lower washers. If the specimen is seated in grooves cut in the washers, a torque would be applied to it when the plugs lengthened upon heating. The effective rotational force would be the difference between the applied torque and the opposing frictional force caused by axial compression; the latter is reduced by reducing the contact areas of the washers to a minimum. Calculations to determine the possible shear force obtainable should be made before such a modification is incorporated.

EXPERIMENTAL PROCEDURE

The specimens to be examined were cubical prisms, approximately 1 cm. on an edge, cut from quartz and quartzite. Two opposing faces were ground parallel; these faces were cut approximately perpendicular to the c-axis of quartz crystals, or were oriented at some chosen angle to an S-plane or lineation of quartzite. The prism or its respective thin section was studied closely and the pre-treatment features were noted for later comparison with post-treatment features. The volume and axial thickness were measured, and the prism was mounted in the vise as shown in Figure 2.

The free volume of the bomb chamber at room temperature was obtained by measurement of the volumes of the various parts, and this was corrected for pressure and temperature changes to be encountered during the experiment. From the corrected volume the amount of water required to produce the desired pressure at the chosen temperature was determined from the appropriate specific volume. The vise was then inserted in the bomb chamber, the water was added, and the bomb was assembled and placed in the furnace. After heating, the bomb was removed and quenched in oil, the prism was extracted, and any physical or chemical changes caused by the treatment were studied.

EXPERIMENTAL RESULTS

Leakage of the bomb seal occurred in all experiments but one. The confining pressure in the bomb was therefore not known, although it probably exceeded the vapor pressure of water at the temperature applied because some water usually remained in the chamber. Shattering of the quartz

crystals during the bomb runs presented a second difficulty, in that any secondary inclusions formed could not be differentiated from pre-treatment inclusions. This difficulty was mostly eliminated by ensuring that the compression faces of the test piece were ground parallel and by making the base of the lower washer slightly convex so that it would automatically adjust itself parallel to the crystal face.

The procedure followed in determining stresses obtained in the bomb runs is shown by the following example (Experiment No. 10):

1. Final Temperature	345	°C
2. Desired Confining Pressure	3000	Atmos.
3. Volume of Bomb Chamber	149.6	cc.
4. Volume of Vise Assemblage	105.8	cc.
5. Volume of Specimen	0.71	cc.
6. Free Volume at $t = 23^{\circ}\text{C}$ (3-4-5)	43.1	cc.
7. Change of Volume with Temp. and Press.	+ 3.1	cc.
8. Final Volume	45.2	cc.
9. Specific Volume of H_2O (from chart)	1.10	
10. Volume of Water added ($V_f/\text{Sp.Vol.}$)	41.1	cc.
11. Calculated Stress ($S = 3.83 \text{ t}$)	1200	Atmos.
12. Initial Static Pressure	300	Atmos.
13. Compressive Stress on Specimen	1500	Atmos.
14. Total Axial Stress on Specimen	1500 +	Atmos.
15. Time of Run	40	hrs.

Table III summarizes the conditions met in the twelve experiments and the results obtained from each bomb run.

TABLE III

Summary of Experimental Data

Expt. No.	Temp. °C	Compressive Stress (At.)	Confining Press. (At.)	Sp. Vol.	Total Axial Stress (At.)	RESULTS
1	330	830	3000	1.1	3830	1. Quartz prism shattered under static load, collapsed during bomb run. No dependable information obtained on inclusions in fragments.
2	325	550	?	1.09	550 +	2. Bomb seal leaked; quartz prism shattered into splinters.
3	325	430	?	1.09	430 +	3. Bomb leaked; quartz prism fractured parallel to load; no inclusions observed in fragments.
4	325	460	?	1.09	460 +	4. Bomb leaked totally; quartz crystal digested. Hydrous glass and aomite formed.
5	325	330	?	1.09	330 +	5. Bomb leaked, little water remained; specimen partially digested, remainder cracked. Crystalline silicate, aomite, and hydrous glass produced.
6	325	400	?	1.09	400 +	6. Bomb leaked, much water remained; specimen partially digested. Hydrous glasses and aomite produced. Etch lamellae parallel to base.
7	325	370	?	1.09	370 +	7. Bomb leaked, little water remained; crystalline silicate reproduced with aomite and glasses.
8	335	430	?	1.10	430 +	8. Bomb leaked, little water remained. Silicate reproduced; silver lip corroded.
9	335	390	?	1.10	390 +	9. Bomb leaked partly; specimen intact. No inclusions formed, no change in position of optic axis effected. Etch lamellae parallel to base on two sides, randomly oriented on others.

TABLE III

Summary of Experimental Data

Expt. No.	Temp. °C	Compressive Stress (At.)	Confining Press. (At.)	Sp. Vol.	Total Axial Stress (At.)	RESULTS
10	345	1500	?	1.10	1500 +	10. Bomb leaked partly. Sandstone prism partially disintegrated.
11	345	1560	?	1.10	1560 +	11. Bomb leaked partly. Sandstone prism partly disintegrated. Thin section showed no change of texture; cementing material leached.
12	1000?	3500	?	1.35	3500 ±	12. Furnace control failed, temperature elevated. Quartzite specimen fractured axially into prisms. No Changes in texture observed in thin section.

The experiments were of three main types and are grouped below accordingly to facilitate discussion of the results.

Group I Experiments (Nos. 1,2,3, and 9)

Quartz prisms were subjected to pressure and stress in the presence of water in an attempt to determine whether or not the shear forces set up in a crystal under compressive stress according to the strain ellipsoid hypothesis (Fairbairn, 1949, p. 199) produced planes of liquid inclusions or caused migration of or changes in the shapes of inclusions already present.

Specimens 1 and 2 contained many anhedral liquid inclusions, either with or without air bubbles. The crystals were shattered into splinters and fragments during the static loading or the bomb run, and a study of the effects of temperature and pressure on planes of inclusions could not be made. No development of negative crystals (Tuttle, 1949) was noted to have taken place in those inclusions observed in the fragments. Purdie (1950) found that negative crystals were formed under conditions which were assumed to have been imposed in these experiments. His bomb held its pressure, however, which suggests that hydrostatic pressure is an important factor in experiments of this nature.

Specimens 3 and 9 contained few liquid inclusions; the experiments were made to determine whether or not tension or shear planes might serve as planes of weakness along which fluids introduced under pressure could move. Specimen 3 was partly shattered during the bomb run, but the fragments were examined; no inclusions had formed under the P-T conditions used. Specimen 9 remained intact, but no new inclusions had formed, and the pre-treatment inclusions remained unchanged.

In experiments 3 and 9 smaller crystals from the same source material were also placed in the bomb beside the vise, so that differences in etch patterns between the stressed and unstressed prisms could be studied.

The rate of solution of quartz is greater in one crystallographic direction than another, hence the adjacent twins of a multiple-twinned crystal will stand in contrast with one another upon solution, and a lamellate or etched pattern will result. Shear planes are weak points in a structure, and solution would proceed more rapidly along them than along adjacent unstressed parts. A second set of strong, irregular lamellae would therefore be superimposed on any twin pattern.

No recognizable difference in etch pattern was observed; both stressed and unstressed specimens showed that similar etch lamellae had developed perpendicularly or obliquely to the 'c' - axis, which suggested that the crystallographic structure influenced the rate of solution more than a possible impressed shear. Specimen 9 was more strongly corroded than specimen 3. The former was subjected to a longer period of treatment, and it is possible that the hydrostatic pressure was greater since more water remained; both effects would increase the total amount of silica dissolved.

Group II Experiments (Nos. 4,5,6,7, and 8)

In these experiments, it was proposed to examine the effect of an alkaline solution on the stability of quartz, and to determine whether or not the solution facilitated the development of inclusions in stressed quartz under a confining pressure. The specimens selected had few individual inclusions and rarely a plane of inclusions.

The bomb leaked in all experiments. It is doubtful if further

information would have been obtained had the pressure held, however, for corrosion of the quartz proceeded rapidly and the prisms were mostly digested. A study of the interior of remnants left in four of the experiments showed that shallow conchoidal tension(?) fractures, which had developed sub-parallel to the direction of stress, were crowded with inclusions of minute size. The fractures all terminated at the upper compressive face or at a free face, hence they were accessible to the solutions. The exact nature of the inclusions could not be determined because of their size (0.1 microns), but their transparency in reflected light and opacity in polarized light suggest that the globules were either liquid or gaseous. It is concluded that the hot fluid (aqueous or gaseous) migrated or was forced into the cracks produced by the stress and, upon cooling, contracted to form the innumerable globules observed. The inclusions could be gas bubbles in an aqueous host, or water globules in a gaseous host, depending on the density of the medium introduced and the relative proportion of liquid to gas upon cooling.

The primary stage of dissolution of the quartz appears to have been the formation of a hydrous, plastic glass, for the solution faces were thinly coated with this material upon removal of the specimen. Dehydration proceeded rapidly upon exposure, the final product being an amorphous glass (confirmed by X-ray analysis) only slightly softer than silica itself. Morey (1914) considered such glasses to be 'fused mixtures', the melting point of the alkaline silicate having been reduced by addition of water. Infinite dilution is apparently not obtainable under the present condition of formation, however, for the two phases water and hydrous glass were found to coexist at room temperature. In the light of more recent work by Morey and Hesselgesser (1952) it would appear that both a gas and a liquid phase were present at the temperature of formation; later cooling producing a

'supercooled' liquid from the liquid phase and water from the gaseous phase.

Too little is known of the conditions within the bomb to draw definite conclusions from these experiments.

A second product formed during the treatment appeared as radiating or dendritic groups of crystals on the washers and vise walls. X-ray analysis revealed that the material was a variety of acmite found in boiler scale; apparently a sufficient amount of iron had been extracted from the walls of the bomb or the vise assemblage to form the insoluble pyroxenic compound.

A third well-crystallized compound produced in experiments 5, 7, and 8 was studied in detail (see pages 23 - 27). This compound, determined as the disilicate of sodium, was found as aggregates of prismatic to tabular crystals adhering closely to the quartz prism or to the basal washer. Hydrous silica glass and acmite were also present. The former was in less abundance than in experiments 4 and 6 and was deposited on and about the crystals; the latter was again found spread thinly over parts of the iron surfaces.

Group III Experiments (Nos. 10, 11, and 12)

Experiments 10, 11, and 12 were carried out in a pure water medium to avoid the strongly corrosive action of the carbonate solution. The object was to determine whether or not it was possible to change the optic orientation or cause recrystallization of grains of quartz in sandstone or quartzite by subjecting prisms of these materials to a compressive stress. A unidirectional stress on an aggregate produces high point

stresses between the component grains; if these local stresses are sufficiently high, solution would take place, followed by deposition in areas of lower pressure. Since planes of inclusions in aggregates appear to be related to processes of reorientation and recrystallization, particular attention was paid to their distribution in the untreated specimens so that any changes induced during treatment could be recognized.

Potsdam sandstone was used in experiments 10 and 11. The quartz grains were well rounded and unstressed, and were loosely cemented together with optically oriented rims of quartz. Hematite and micaceous impurities were concentrated along grain and rim boundaries. Vacuoles observed within grains were dispersed at random. A fabric analysis showed a more or less random crystallographic orientation of the grains.

Partial dissolution of the impurities along grain and rim boundaries took place during the treatment, causing loosening of the grains and disintegration of much of the prisms. A fabric study could not be made because the thin sections yielded during their preparation, but superficially the grains did not appear deformed, nor was the fabric notably altered. Intragranular inclusions remained unchanged. Recrystallization may have occurred, but a decrease rather than an increase in the cementing strength suggested that it did not.

Griggs (1938) found that no preferred orientation of quartz grains was produced under extreme static pressures, hence the lack of deformation of the grains is not surprising; an increase in the cementation might be anticipated, however. Fairbairn (1950) produced synthetic quartzite by subjecting loose quartz sand to compressive stresses of 3,000 to 6,000 atmospheres in a 5% Na_2CO_3 solution under confining pressures of 400-2,000 atmospheres. His results were negative when pure water was used at these pressures, however, despite a longer time of run. A comparison

of the conditions imposed here with those obtained in his experiments shows that positive results could not have been obtained under these conditions.

Specimen 12 was cut from a quartzite found immediately below the Moine thrust fault in the Assynt area, Scotland. The quartz grains were extremely elongate and somewhat contorted, most of them consisting of a mosaic of minute grains having a variable optic orientation — the result of microgranulation under high differential stress. Thin sections were cut parallel and perpendicular to a micaceous S-plane which reflected the shear surfaces. The object of the experiment was to produce a coarser texture by crystallization so that inclusions could be studied. To give optimum conditions a temperature of 573°C (the inversion temperature of quartz) was applied.

Failure of the temperature control unit resulted in far higher temperatures ($+1000^{\circ}\text{C}$) being reached than was expected. The silver seal melted and the bomb leaked completely, but the prism was little damaged. Thin sections cut parallel to the pre-treatment sections showed that no visible changes had been caused by the treatment. The microgranular texture, elongation of grains, and undulose extinction of individual grains had survived the imposed conditions apparently unchanged.

Griggs (1936-1941) conducted experiments on deformation of rocks under high compressive and shearing forces. He succeeded first in producing quartzite, as Fairbairn had later, by imposing a high compressive stress on a charge of loose sand in a water environment held at 1,000 atmospheres pressure and $\pm 500^{\circ}\text{C}$. Next he superimposed a shearing stress on the charge, and found that the grains were deformed, their mobility increasing with the stress applied; granulation and the development of

preferred orientation also became apparent at high degrees of stress, the 'c'-axes tending to parallel the motion. More recent experiments by Handin (1953) on marble are confirming Griggs' results, but are being carried out in environments more closely approximating natural conditions.

It is evident that the pressure conditions imposed in the present experiments were not suitable for producing the changes of texture which occur when rock bodies are subjected to deforming stresses.

NEW DATA ON SODIUM DISILICATE

Summary of Properties

The crystals of sodium disilicate produced in experiments 5, 7, and 8 were well-developed plates, without modifying forms, which had aggregated about the base and on the faces of the quartz grains (Figure 6).

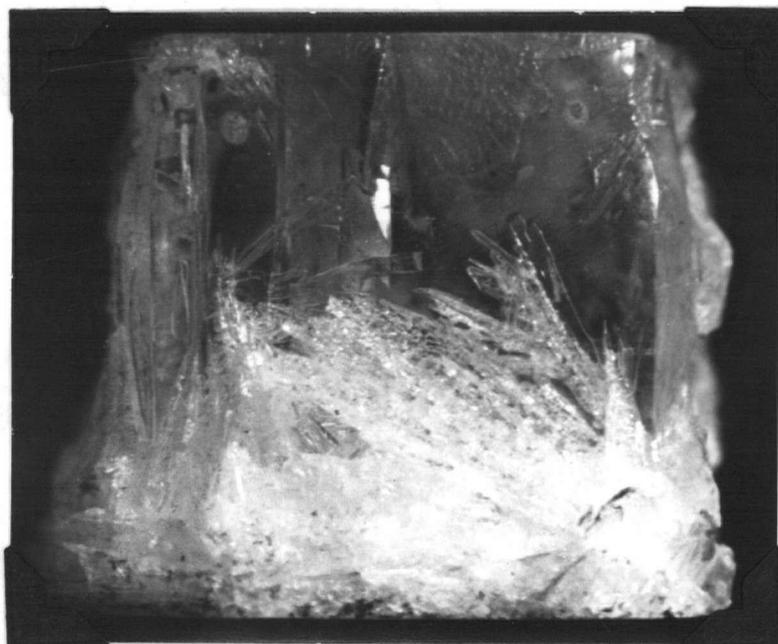


Figure 6. Sodium Disilicate Crystals Formed on Quartz Prism Faces

Most of the physical, optical, and structural properties of the compound could be obtained. Rotation and Weissenberg photographs were taken about

the b- and c-axes, and from them the zero-, first-, and second-layer reciprocal lattices were projected to determine the cell dimensions. The cell content was calculated in terms of the elements present. A rather poor agreement between the calculated and measured specific gravity was found because the crystals were crowded with fluid and gaseous inclusions. Measured lattice spacings given by the X-ray powder diffraction pattern (Figure 7) show good agreement with the calculated spacings; the results

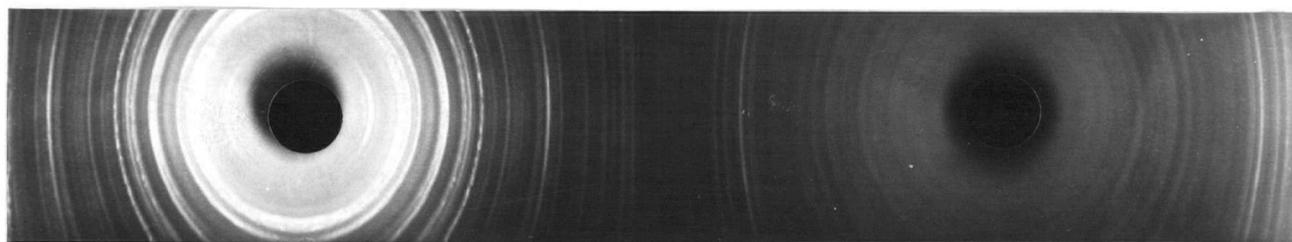


Figure 7. Sodium Disilicate: X-ray Powder Photograph with Iron Radiation, Manganese Filter

are shown in Table IV. Indices of refraction were determined accurately by the double variation method, using a constant-temperature stage designed by Dr. K.C. McTaggart.

The properties are summarized as follows:

Composition : $\text{Na}_2\text{Si}_2\text{O}_5$

Structure Cell :

Monoclinic;
Space group $P 2_1/a$

$a = 12.32$; $b = 4.88$; $c = 8.15 \text{ \AA}$; $\beta = 104^\circ 30'$

Physical Characteristics

Colorless. Vitreous lustre, splendid on good cleavage. Crystals tabular to prismatic.

TABLE IV

Lattice Spacings of Sodium Disilicate

Sodium Disilicate - $\text{Na}_2\text{Si}_2\text{O}_5$										
Monoclinic, $P 2_1/a$; $a = 12.32$, $b = 4.88$, $c = 8.15\text{\AA}$, $\beta = 104^\circ 30'$; $Z = 4$										
II	$\Theta(\text{Fe})$	d(obs.)	hkl	d(calc.)	I	$\Theta(\text{Fe})$	d(obs.)	hkl	d(calc.)	
1	7.08	7.88	001	7.891				411	2.282	
10b	9.30	5.99	200	5.945	2	25.25	2.27	403	2.274	
2	10.15	5.80	$\bar{2}01$	5.458				220	2.258	
4	12.95	4.32	201	4.269	$\frac{1}{2}$	26.00	2.21	203	2.210	
3	13.55	4.13	011	4.151	3	26.85	2.14	510	2.142	
6b	14.20	3.95	002	3.945				402	2.135	
			210	3.776				122	2.092	
1	14.90	3.77	111	3.768	$\frac{1}{2}^B$	27.80	2.08	512	2.090	
5	15.55	3.62	$\bar{2}02$	3.750				320	2.079	
			$\bar{2}11$	3.638				321	2.079	
			310	3.081				022	2.075	
5	18.45	3.06	$\bar{3}11$	3.080	1	28.65	2.02	$\bar{2}04$	2.031	
			012	3.066	$\frac{1}{2}^B$	29.43	1.972	004	1.973	
			401	3.051				611	1.891	
			400	2.979				420	1.888	
9	19.00	2.98	$\bar{2}11$	2.874	2B	30.95	1.884	222	1.884	
			202	2.966				513	1.881	
$\frac{1}{2}^B$	19.85	2.85	112	2.841				404	1.875	
4	21.30	2.67	$\bar{2}03$	2.665				$\bar{2}14$	1.875	
			411	2.587				610	1.840	
4	21.95	2.59	401	2.582				314	1.831	
			410	2.543	8	31.95	1.831	612	1.839	
1	22.45	2.54	212	2.534				313	1.832	
6	23.40	2.44	020	2.440				014	1.829	
$\frac{1}{2}$	24.50	2.34	$\bar{2}13$	2.339				601	1.821	
$\frac{1}{2}$	32.75	1.791			3B	42.82	1.426	$\frac{1}{2}$	51.33	1.241
$\frac{1}{2}$	33.05	1.776			1	43.50	1.407	$\frac{1}{2}$	51.90	1.231
3	33.65	1.745			$\frac{1}{2}^B$	44.54	1.382	3	53.02	1.213
$\frac{1}{2}$	35.80	1.656			2B	46.16	1.344	3	54.41	1.191
3	36.90	1.613			1B	46.90	1.327	$\frac{1}{2}$	55.01	1.183
$\frac{1}{2}$	37.91	1.577			$\frac{1}{2}^B$	47.81	1.308	$\frac{1}{2}$	55.91	1.170
1	39.02	1.539			1	48.55	1.293	5	56.52	1.162
2	39.30	1.529			$\frac{1}{2}^B$	49.52	1.274	1	57.51	1.149
3B	41.53	1.462			$\frac{1}{2}$	50.61	1.255	1	58.03	1.142

Cell Dimensions by J.A. Soles and R.M. Thompson, February, 1954

Cleavage : (100) eminent, yielding thin sub-elastic folia; (001) poor.

H = 4; Sp. Gr. = 2.31 (meas.), 2.55 (calc.)

Fuses at 3.5 to translucent glass.

Optical Properties

$$2V = (-)64^{\circ}$$

The optic plane and acute bisectrix (Figure 8) are

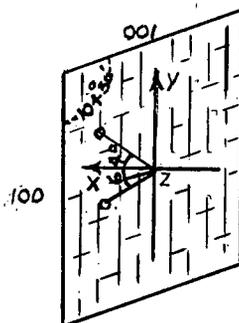


Figure 8. The Optic Orientation of Sodium Disilicate

normal to (100). Elongation is positive parallel to the (001) cleavage traces. Extinction is parallel to both cleavages.

$$N_x = 1.5054$$

$$N_y = 1.5135$$

$$N_z = 1.5167$$

$$N_z - N_x = 0.0113$$

Composition Calculations

A chemical analysis of the mineral was made by G.S. Eldridge and Co. Ltd., Vancouver, B.C.; the analysis is given in Table V.

Possible compounds obtained by considering the atomic proportions of all oxides do not give molecular weights corresponding to that calculated from the unit cell dimensions and measured specific gravity. As a first approximation the $(Fe, Al, Ca)_x O_y$ fraction can

TABLE V

Chemical Analysis of Synthetic Silicate
and Atomic Proportions of Elements
Derived from the Analysis by Approximations

ANALYSIS		APPROXIMATIONS %			ATOMIC PROPORTIONS					
Oxide	%	1 Corrected %	2 Less Fe Oxides	3 Less H ₂ O	Element	1	2	3	3a Less Excess Na ₂ O	3a Reduced
Na ₂ O	32.9	33.9	35.2	36.5	Na	1.10	1.14	1.18	1.057	1
SiO ₂	57.2	58.9	61.1	63.5	Si	0.98	1.02	1.06	1.057	1
(Fe,Ca,Al)O	3.5	3.6	-	-	Fe	0.05	-	-	-	-
H ₂ O	3.5	3.6	3.7	-	H	0.20	0.21	-	-	-
Remainder	2.9	-	-	-	O	2.76	2.82	2.71	2.64	2.50
TOTAL	100.0	100.0	100.0	100.0						

be eliminated (Table V, column (2)); for the oxide's atomic proportion (determined by assuming Fe to be the major cation constituent since it alone is present in the system in abundance) is too low to incorporate it in the composition as a major phase. A recalculation of the proportions of remaining oxides still does not give a satisfactory composition, however, and a second approximation is made by assuming that the water was trapped in vacuoles during crystallization. The resulting atomic proportions are Na:Si:O = 1.18 : 1.06 : 2.71 (Table V, Column(3)). If the Na is considered high because some Na_2CO_3 was also trapped during crystallization, the ratios reduce to Na:Si:O = 1:1 : 2.50, which would give a composition of $\text{Na}_2\text{Si}_2\text{O}_5$. The results of the various calculations are compiled in Table V.

The gram molecular weight calculated from the equation

$$M = \frac{abc \sin \beta}{N} \times \text{Sp. Gr.}$$

(Glasstone, p. 177) is 680, whereas the value obtained by using 4 molecules of $\text{Na}_2\text{Si}_2\text{O}_5$ is 728. The discrepancy in molecular weight appears to be caused by a low measured specific gravity. The specific gravity calculated by using a molecular weight of 728 is 2.55, which gives an error of 6.7%; considering the porous nature of the compound, such a large error is likely.

The above approximations have been made by considering both the conditions under which the compound was formed and the general appearance of the material itself. The evidently anhydrous nature of the compound is of interest, however, for water was a major constituent. Apparently the cell structure prohibits incorporation of the OH ion.

Morey (1914) examined synthetic silicates of sodium produced by heating mixtures of Na_2O and SiO_2 in the ratio 1:1 with various amounts of water; both the metasilicate (Na_2SiO_4) and disilicate ($\text{Na}_2\text{Si}_2\text{O}_5$) were

formed, the latter being produced only with a marked deficiency of water. The optical properties of the disilicate, determined by C.N. Fenner, compare exactly with those observed for the compound produced here, supporting the writer's view that the compound is the disilicate as the computations suggest. Fenner, probably working on the optical properties alone, considered sodium disilicate to be orthorhombic, but X-ray data obtained by the writer show that it is monoclinic. His error is to be expected if his data were obtained from poorly formed crystals, for the position of the optic plane gives parallel extinction in all oriented sections.

Phase Relations

Niggli (1913), Morey and Bowen (1924), and Kracek (1930) respectively examined dry melts of the systems $\text{Na}_2\text{O} - \text{SiO}_2 - \text{CO}_2$, $\text{Na}_2\text{O} \cdot \text{SiO}_2 - \text{SiO}_2$, and $\text{Na}_2\text{O} - \text{SiO}_2$, and reported on the stability ranges of the silicates formed. Examination of water-silicate systems by Morey and Fenner (1917) and Friedman (1950) were carried out successfully, and recently the $\text{Na}_2\text{O} - \text{H}_2\text{O} - \text{SiO}_2$ system was thoroughly examined by Morey and Hesselgesser (1952) at 400°C and at pressures up to 2500 bars.

Observations made in the present study add little to the phase relations outlined in this last excellent paper. A fourth constituent, CO_2 , was introduced in these experiments, but it does not appear to have been an active phase in the system under these experimental conditions, as Niggli had found previously. The disilicate probably was precipitated in equilibrium with gas-liquid phases when the $\text{SiO}_2 : \text{Na}_2\text{O} + \text{SiO}_2$ ratio reached the point where the solubility curve Di - L for the prevailing temperature was intersected; later cooling would consolidate the liquid (probably a viscous fluid at higher temperature) and condense the water vapor still remaining.

CONCLUSIONS

Planes of fluid inclusions in mineral and rock bodies could provide valuable information concerning the physical environments of tectonic movements if the processes of formation of the planes were understood. The planes appear to be related to differential stress applied to the bodies and to the processes of recrystallization resulting from it and other pressure, temperature, and chemical effects. High-pressure experimentation is therefore required to gather information regarding their formation.

The present study yielded no positive information. It is believed, nevertheless, that a step in the right direction has been made, and that further work will eventually give concrete results. The pressure bomb used in these experiments, however, may not give the conditions necessary to produce the desired changes in quartz and quartz aggregates within reasonable experimental time limits of one to two weeks. If further experimental work shows this to be the case, an effort should be made to acquire superior equipment, rather than to relegate the problem to already over-taxed laboratories in which superior equipment is available. The problem of planes of fluid inclusions is worthy of intensive study, and therefore of expenditures to facilitate it.^o

Sodium disilicate is of no commercial value, but its place in the $\text{Na}_2\text{O} - \text{SiO}_2 - \text{H}_2\text{O}$ system is an important one. It is to be hoped that the new structural data presented here may be of use to investigators who are examining this system.

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