SYNTHETIC QUARTZITE*

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ABSTRACT

Quartz sand (grain size 125–250 microns) immersed in weak aqueous Na_2CO_3 solution is converted to sheared quartzite (grain size 100–200 microns) after a few hours' exposure to temperatures in the range 230°–435° C., confining pressures between 5000 and 30,000 psi, and compressive loads between 32,000 and 103,000 psi. The principal feature of the experimental procedure is the use of a piston of rectangular cross-section and of a sand receptacle designed so that a biaxial strain is imposed on the quartz aggregate. The deformed prisms show shortening up to 57%, elongation up to 20%, and net decrease of volume up to 49%. Individual quartz grains have a maximum elongation of 2:1. Undulatory extinction bands are abundant, but no trace of deformation lamellae was found. There is little evidence of recrystallization. Both compaction and dimensional orientation are best developed in the upper range of temperatures and pressures used. Investigation of the lattice orientation (axis diagrams) of the deformed prisms shows random orientation for the most part. Calculation of the coefficient of correlation for each scatter diagram has also been carried out to test statistically this apparent randomness, but with inconclusive results.

Several similar experiments, using H_2O instead of Na_2CO_3, show a much lower degree of compaction and no trace of dimensional orientation; even, for example, after 7 days' exposure to 355° C., 30,000 psi confining pressure and 70,000 psi compressive load.

INTRODUCTION

Rock synthesis, for obvious reasons, has lagged far behind rock analysis as a field of geological investigation. Since successful synthesis depends in large part on knowledge obtained from analytical studies, this is not surprising and indeed is the logical order of events. As in analytical work, the approach may be either structural or compositional. Since the investigation reported here is concerned entirely with quartz, only structural considerations apply. The special theme is the behavior of quartz aggregates within a selected range of temperature and pressure. The following pages relate some details of the transition from uncemented sand aggregates to synthetic quartzite.

BACKGROUND OF THE PROBLEM

The facts regarding quartz orientation in tectonites are now well established as a result of work done by many petrologists from widely scattered parts of the world. I have recently summarized these investiga-

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* It is a pleasure to acknowledge Professor Larsen's part in the development of the petrofabric background which has led to this paper. It was his scholarly interest in this field, then (1930) virtually unknown outside Austria and Germany, which first made me aware of its possibilities. Although not his own special interest, his encouragement at that time of this new work has in no small measure been responsible for my own continued interest in it.

tions (Fairbairn, 1949) and attempted a synthesis of the conflicting hypotheses used to explain the data. Omitting details, it may be fairly stated that the factual side of the matter far outstrips the interpretative side. Experimental data are sorely needed if real progress is to be made. It was with this in mind that the present study was commenced.

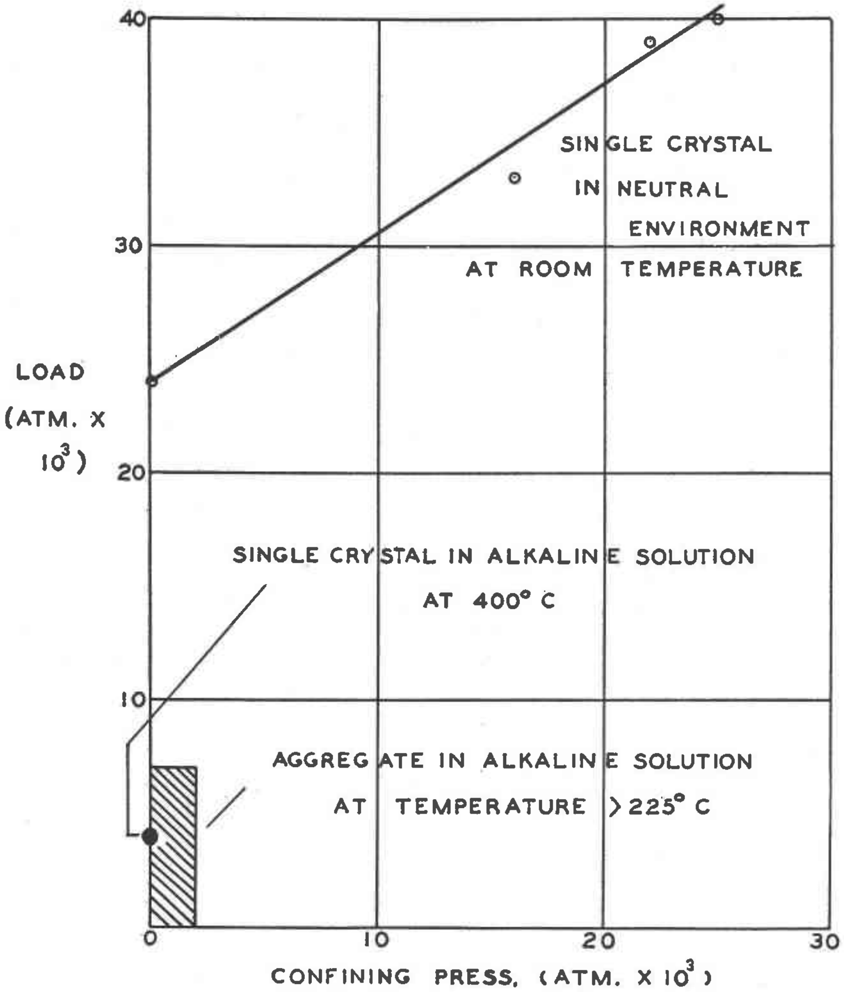

FIG. 1. Diagram showing strength properties of single-crystal quartz relative to experimental range used for cementation of quartz aggregates.

Enough work has been done on the deformation of single-crystal quartz to aid materially in the experimental procedure for deformation of quartz aggregates. Bridgman (1946) has made the most recent determination of the strength of quartz for confining pressures up to 25,000 atmospheres. Griggs and Bell (1938) showed that this high level of breaking strength could be drastically reduced by carrying out the experiment at elevated temperature (400°C.) in an alkaline solution environment. It was found that a crystal would fracture with one-sixth the compressive load required in Bridgman's experiment (Fig. 1). An additional contrast was the tendency toward crystallographic control of the shapes of the fragments in the Griggs-Bell experiment and lack of this feature inBridgman's deformed material.

TABLE 1. EXPERIMENTS WITH QUARTZITE CYLINDERS

Expt. No.	Temp. (° C.)	Fluid Environment	Fluid Press. (psi$\times 10^3$)	Load (psi$\times 10^3$)	Time (hrs.)	Remarks
1	315	1% Na_2CO_3	15	13.5	1	Rupture after 1 hr.
2	315	1% Na CO_3	15	4.7	3	Rupture after 3 hrs.
3	315	1% Na_2CO_3	15	3.5	115	No rupture
4	315	2% Na_2CO_3	9	12.8	140	No rupture. Noticeable transfer of SiO_2 from upper to lower parts of cylinder.
5	150	10% Na_2CO_3	12	5.7	0	Ruptured immediately when load was applied.
6	Room	Air	0.0147±	30 (to produce rupture)	0	Value for load is average of 8 (Handbook Physical Constants—*G.S.A. Special Paper No.* **36,** 1942)

Table 1 summarizes the results of experiments with cylinders cut from quartzite[1] which confirm the contrast in the Bridgman and Griggs experiments. Experiment 6 is an average of 8 normal rupture tests. Experiments 1 to 5 show the effect of varying alkaline fluid concentrations, fluid pressures, and loads.

All of this work provides very definite evidence that the results of room temperature-dry deformation of quartz and quartzite give no indication of the actual confining pressures and compressive loads required to deform the mineral in the earth's crust.

Continuing this work, Griggs later constructed new apparatus and made a preliminary survey (Griggs, 1941) of the deformation of a number of minerals (single and in aggregate) in this temperature-pressure range. These investigations, discontinued because of the war, were taken up by

[1] Massive, fine-grained material, locality unknown. All cylinders have the same orientation.

me in 1946 through the generosity of Griggs in providing details of the equipment and a manuscript of his work as far as it had gone. Support has been provided in the succeeding years by a grant from the Geological Society of America, (Project Grant 466–55), to which society grateful acknowledgment is hereby made.[2]

EXPERIMENTAL PROCEDURE

Full details of the equipment used are in process of publication elsewhere. It is necessary to include here only a description of the receptacle in which the deformation of the quartz sand is accomplished. This consists of a slot with vertical, parallel sides cut in one end of a piece of drill-rod steel (Figs. 2, 3) into which fits a Carboloy piston of similar shape. The piston does not occupy the full length of the slot, the ends being blocked with shaped copper pieces as shown. The space between the copper end pieces is filled almost to the top with the quartz aggregate. A close-fitting copper cylinder is inserted around the steel piece, project-

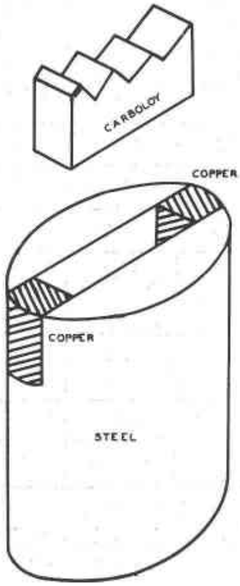

FIG. 2. Receptacle used for quartz aggregate experiments. See also Fig. 3 (*a*) and (*b*).

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[2] Invaluable mechanical assistance by John Solo of the Department of Geology Shop solved many problems which plagued the investigations. Laboratory assistance at various times by J. B. Thompson, Jr., M. C. Wittels, D. L. Kendall, and R. H. Stebbins is also acknowledged.

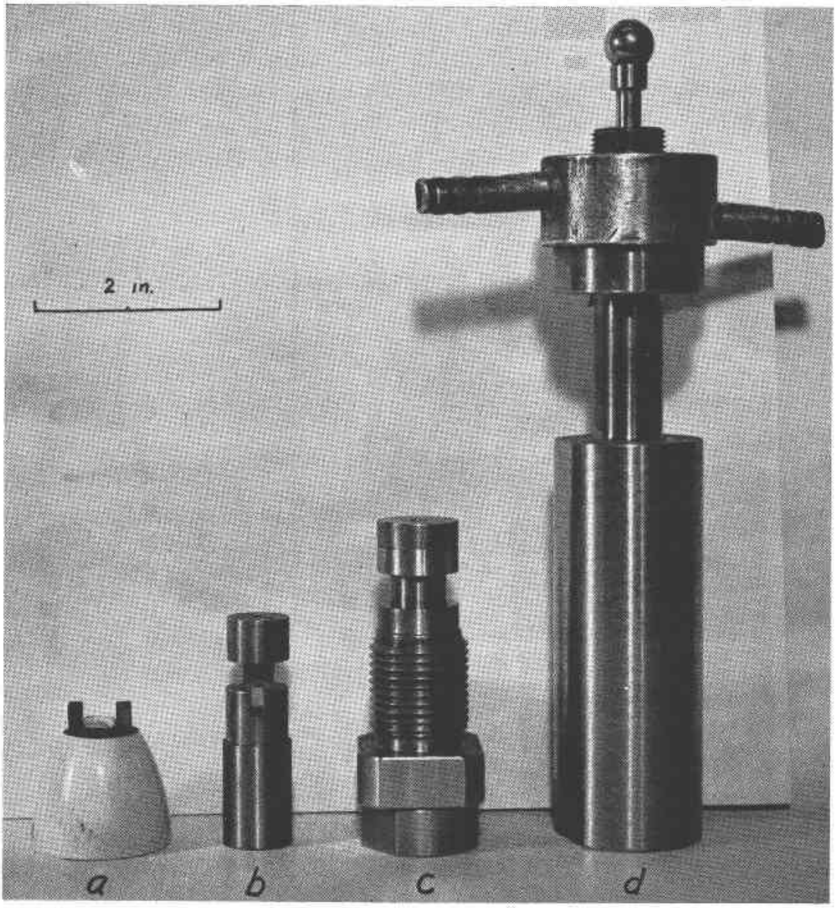

FIG. 3. Pressure chamber and receptacle assembly. (*a*) Copper end pieces enclosing a prism of synthetic quartzite. (*b*) Copper sheath at base; steel receptacle, center; carboloy piston, top. (*c*) Assembly for sealing base of pressure chamber. (*d*) Pressure chamber showing outer piston (top) and water-cooling jacket.

ing above the top. The space above the slot is filled with Na_2CO_3 solution. The piston is then inserted in the top of the slot, excess solution being expelled at the contact with the copper cylinder. By this means air is displaced from the receptacle. The cylinder serves further (1) to hold in place the copper pieces in the slot, and (2) to protect the receptacle from convection currents in the main body of the bomb. The loaded receptacle is now placed in the bomb and the experiment may proceed.

Temperature is first raised to the desired level and the fluid pressure adjusted to the required setting. Finally a load is applied through the piston assembly shown in Fig. 3 (*d*). This may be done in one stage, or by increments at set time intervals. Most of the experiments were run until the dial gage indicated that shortening of the specimen had ceased. Many were run beyond this time.

The grain size of the quartz aggregate was held between 125 and 250 microns so that optical study of the deformed material would not be unnecessarily difficult. No attempt was made to select rounded grains. The quartz sand was obtained by disaggregation and sizing of selected specimens of the Nepean sandstone from Ottawa, Canada (Cambrian). The grains are mostly angular and include both equant and inequant shapes in approximately equal amounts (Fig. 4).

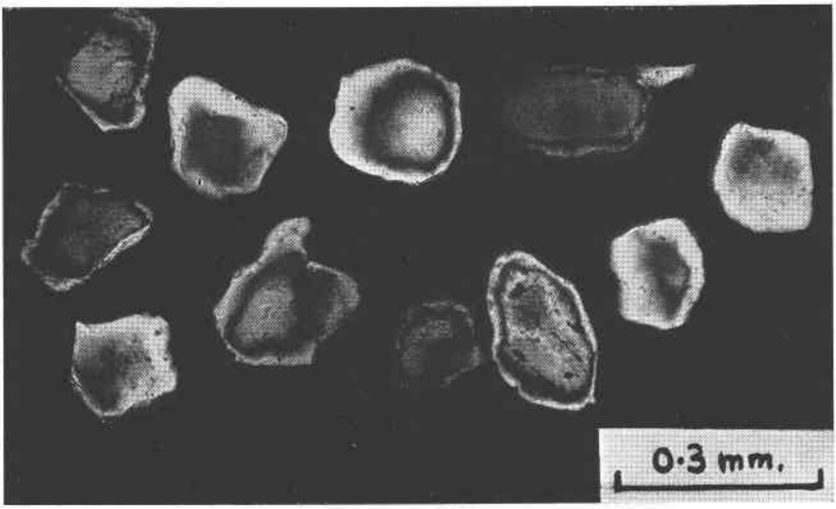

FIG. 4. Photomicrograph of typical disaggregated grains of the Nepean sandstone used in the experiments.

Upon completion of the experiment the receptacle is taken from the bomb and the Carboloy piston and cylindrical copper sheath are removed. The walls of the slot are then carefully sawed off at the base, thus exposing the deformed specimen, which is easily lifted out. Its height and length are then recorded and compared with the original dimensions. Width remains constant because of the design of the receptacle (Figs. 2, 3). A thin section is prepared of the side of the specimen parallel with the vertical wall of the slot. This slide is then examined for visible fabric details and the orientation of the quartz axes is determined with a universal stage.

The equipment used gave temperatures up to about 450°C. in the space occupied by the receptacle. As the temperature in the receptacle is not

TABLE 2. EXPERIMENTS USING 5% Na_2CO_3 SOLUTION (pH=8)

Expt. No.	Temp. ° C.	Fluid press. psi×10^3	Load† psi×10^3	Time of Expt. (hrs.)	Est. time to produce 75% of shortening	% Shortening	% Elongation	Cementation	Dimensional orientation	% Volume decrease	Statistical classification of axes orientation		Remarks
											Random	Significant	
1	150	30	70	19	4.5	43	nd	None	None				Aggregate finely granulated
2	190	30	70	63	40	36	nd	None	None				Temperature threshold for cementation is at 200° C. approximately
3	230	0	70	15	3	11	nd	None	None		X		No fluid pressure used in excess of that generated at the temperature of the experiment
4	230	5	70	9		nd	nd	Moderate	None		X		Time-shortening* curve erratic
5	230	15	70	22	18	36	nd	Moderate	None			X	Temperature threshold for dimensional orientation is at 230° C. approximately
6	230	27	70	66	46	41	nd	Excellent	Moderate		X		
7	230	30	70	9		43	10	Moderate	Moderate	37	X		Time-shortening* curve erratic
8	300	25	14	23 days									Quartz grains transferred to non-stressed parts of receptacle
9	315	7	32	10	10	nd	nd	Excellent	Moderate		X		Shortening continues for longer period than in Expt. 11 because of relatively low fluid pressure
10	315	13	32	19	11	nd	nd	Excellent	Moderate				Thin section missing
11	315	26	32	71	5	nd	nd	Excellent	Moderate			X	Prolonged experiment (71 hrs.) does not visibly improve the dimensional orientation
12	315	30	103	24	6	nd	nd	Excellent	Moderate			X	Load applied by stages. Specimen indistinguishable from preceding experiments at same temperature
13	355	5	70	27	3	36	nd	Slight	Slight		X		Short time (3 hrs.) for 75% of shortening related to poor cementation, resulting from low fluid press.
14	355	15	70	24	9	27	nd	Excellent	Moderate		X		
15	355	25	70	64	22	nd	nd	Excellent	Excellent			X	
16	355	28	70	35		45	nd	Excellent	Excellent			X	Time-shortening* curve erratic
17	435	10	70	9	10	nd	nd	Excellent	Moderate		X		
18	435	20	70	8.5	6	57	20	Excellent	Moderate	48		X	
19	435	20	70	31	5	48	13	Excellent	Moderate	41		X	Load applied gradually. Specimen indistinguishable from Expt. 18, even though run was prolonged almost fourfold
20	435	10	103	9	8	56	15	Excellent	Slight	44	X		Temp. control poor
21	435	20	103	24	8	53	15	Excellent	Excellent	43	X		Expts. 20–22 (103,000 psi load) tend to develop more fracturing of the specimens than in those under smaller load
22	435	20	103	11		55	20	Excellent	Moderate	49		X	No improvement of dimensional orientation is seen
23	435	25	70	3.25	.4	56	15	Excellent	Excellent	49			Saturated solution used (pH=11)

* This is probably due to non-uniform friction characteristics of the outer piston.

† Total load applied at beginning of experiment, except where otherwise noted under "Remarks."

measured directly an error of perhaps ±5° is unavoidable. Therefore control of the temperature is unwarranted to limits closer than ±5° and was not attempted.

Fluid pressures between 5,000 and 30,000 psi were maintained. The sensitivity of the Bourdon gages used is ±1%. Automatic control was not necessary as variations of several thousand psi had no observable effect on the course of the experiments.

Compression loads were used which by calculation should transmit between 35,000 and 115,000 psi to the receptacle. The fraction actually transmitted probably does not exceed 90% of these values on the average. The limits might thus be set at 30,000–100,000 psi.

For most of the experiments aqueous Na_2CO_3 solutions of various concentrations were used. Na_2SiO_3 was used successfully in some runs. H_2O was also tried in a number of experiments.

The time for individual experiments varied between 117 days and $2\frac{1}{4}$ hours. For the most part the runs were of 24 hours' duration or less.

Results of Experiments

Sodium carbonate is one of a number of non-corrosive salts which in aqueous solution provide the alkalinity necessary for these experiments. A 5% solution (pH = 8) was used for most of the runs, since very weak solutions prolong the experiments unnecessarily. On the other hand, under the conditions of the experiments, saturated solutions (Expt. 23, Table 2) react somewhat too rapidly with the quartz for accurate appraisal of the results.[3] Table 2 therefore is largely concerned with data using a 5% solution. The experiments are arranged in order of increasing temperature; within each temperature group the order is that of increasing fluid pressure.

Application of the load in increments (Experiments 12, 19, 22) rather than in one stage makes no observable difference in the deformed specimen, other than increasing the time required for compaction.

A minimum temperature of about 200°C. is necessary for appreciable cementation of the aggregate. In combination with this an appreciable excess fluid pressure[4] is needed. (Experiments 1–4), as well as a compressive load. Cementation was not obtained with less than about 5,000 psi fluid pressure. The smallest compressive load used (32,000 psi, Expts.

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[3] This results in transfer of the grains from the stressed space beneath the piston and redeposition on the walls of the receptacle. Experiment 8 (Table 2) shows that, even with a 5% solution, a prolonged run results in loss of the material. The rounded edges of the deformed specimen shown in Fig. 3(a) are evidence of the beginings of this transfer.

[4] That is, in excess of the relatively small pressure generated at the given temperature level.

9–11) does not represent the minimum possible load. An investigation of the minimum compressive load was not feasible, as the error arising from frictional forces in the external piston sleeve would become disproportionately large. It was not possible either to test effectively the factor of increasing time, since there there is continuous solution of the quartz grains under the piston and re-deposition in unstressed areas of the receptable (e.g. Experiment 8). The time required for 75% of the compaction can be estimated[5] and is a somewhat better basis for comparison of experiments than the actual time of a run (compare time columns in the table).

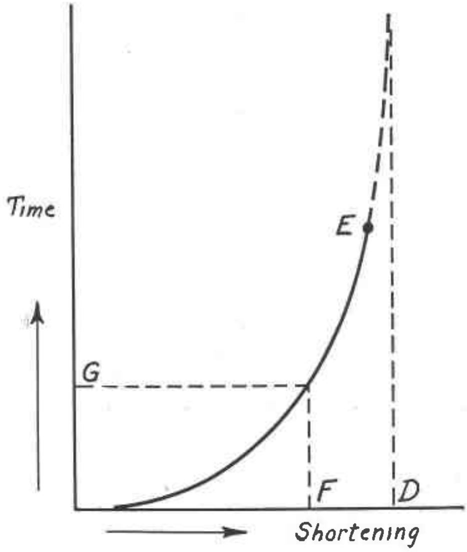

FIG. 5. Diagram showing method of approximating the time required for 75% of shortening to take place. (See footnote in text)

Dimensional orientation of the deformed aggregates first becomes noticeable at about 230°C. There is elongation of the grains normal to the load and parallel with the length of the slot in the receptacle (Fig. 6). Maximum ratio of the long and short axes is about 2:1. Although some grains in the undeformed sample were inequant to almost this extent, it is certain that rotation of these into parallelism is an inadequate explanation of the observed dimensional orientation. There is a net decrease

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[5] The time vs. shortening curve for any experiment is approximately exponential and the time required for essential completion of shortening can be estimated even where an experiment is not run to completion. For example, in Fig. 5, *E*—experiment stopped here; *D*—estimated total time for experiment if completed; *F*—75% of shortening represented by *D; G*—time required for 75% of estimated total shortening.

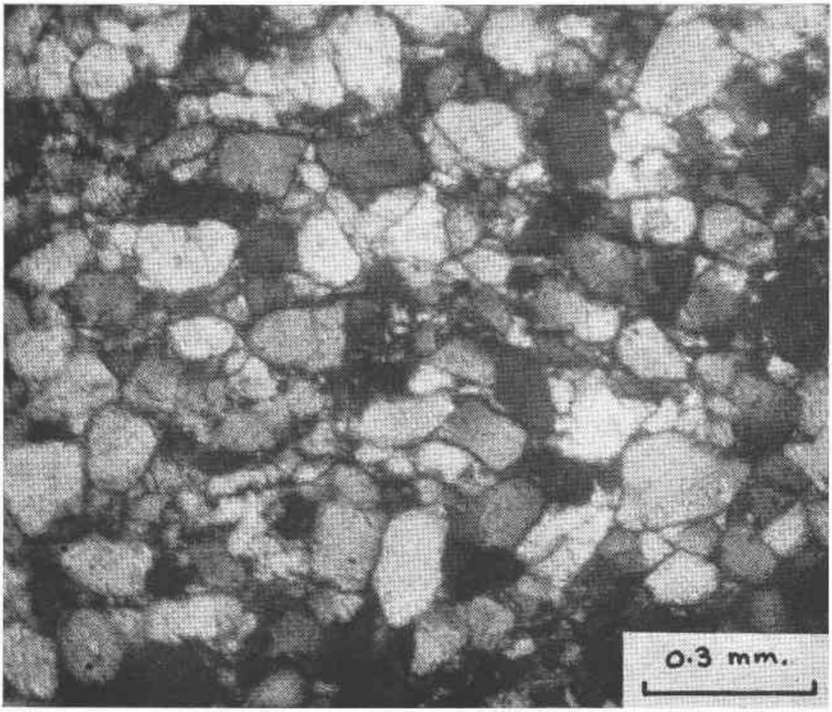

FIG. 6. Photomicrograph of synthetic quartzite showing elongation trend parallel to long axis of slot in the receptacle (east-west). Note non-uniform grain size and brecciation.

in range of grain size from 125–250 (undeformed) to 100–200 microns (deformed) and considerable less angularity in individual grains after deformation than before (Fig. 7).

Examination of thin sections shows the consolidated, deformed material to be a sheared quartzite (Fig. 6). There is considerable brecciation around some of the grains and all show undulatory extinction. No trace of deformation lamellae has been found. Axes diagrams have been made by means of the universal stage for all deformed aggregates from which a thin section could be made. No definite orientation pattern could be determined by inspection alone, although, as Table 2 shows, a wide range of variables was tried out. To test statistically the degree of randomness the coefficient of correlation was computed for each point diagram, as outlined by Chayes (1949). This study indicates that about one-half the diagrams are probably random (isotropic), the other half probably significant (anisotropic). As this statistical classification does not, however, show any rational relation with the corresponding experi-

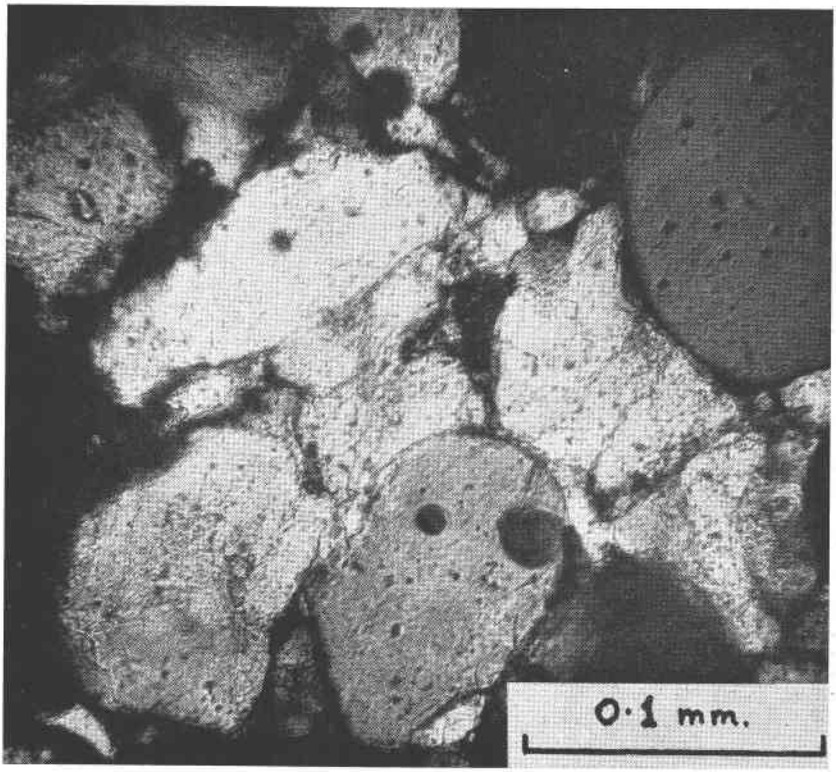

FIG. 7. Photomicrograph of synthetic quartzite at high magnification showing rounded grain outlines and interstices filled with small grains.

mental conditions (see Table 2) the analysis is indecisive as matters stand at present. It is possible, however, that the lattice orientation, as determined with the universal stage, does not represent the entire deformation story. Many grains in these sheared quartzites escape measurement because of their minute size. Until their part in the stress-strain picture is known the final word regarding the overall character of the axes orientation can not be said. It is hoped that this additional study can be successfully carried out using an improved type *x*-ray spectrometer.

With respect to the optically measurable, larger grains in the deformed quartz aggregates it would appear that changes in the fabric involve transfer of material from points of high stress to points of lower stress, resulting in elongate grains which tend to be arranged perpendicular to the direction of shortening. No important grain rotation or change in the axes orientation seems to take place. The process is essentially Riecke's principle applied to a polycrystalline aggregate. The conditions

TABLE 3. EXPERIMENTS USING H_2O (1–3) AND Na_2SiO_3 (4–5)

Expt. No.	Temp. ° C.	Fluid press psi×10^3	Load† psi×10^3	Time of Expt.	Est. time to produce 75% of shortening	% Shortening	% Elongation	Cementation	Dimensional orientation	% Volume decrease	Remarks
1	300	20	14	117 da.				None	None		Time-shortening* curve erratic. See footnote to Table 2
2	355	28	70	3½ da.	20 hr.	36	nd	Slight	None		
3	355	30	70	7 da.	22 hr.	36	nd	Slight	None		
4	275	20	70	4 hr.	1 hr.	45	12	Excellent	None	40	Load added by stages
5	435	25	70	2¼ hr.	4 min.	51	23	Excellent	None	40	

* This is probably due to non-uniform friction characteristics of the outer piston.

† Total load applied at beginning of experiment except where otherwise noted under "Remarks."

of the experiments have not produced, therefore, any of the lattice orientation patterns commonly associated with quartz tectonites.

A few experiments with water glass (Na_2SiO_3) indicate behavior like that observed with saturated Na_2CO_3 (Table 3, Table 2, Expt. 23). No extended series of experiments was carried out, however, as it is believed to be $(OH)^-$ concentration, not the compound itself, which accounts for the effects observed.

A number of experiments were run using H_2O as a fluid environment. No satisfactory cementation was obtained by this means (Table 3) and no dimensional orientation was observed. Only with difficulty could thin sections of this deformed material be made. Possibly the slight cementation observed in two of the experiments arises from traces of OH^- in the bomb as a result of inadequate cleaning. It is concluded that absence of $(OH)^-$ is the key to the matter.

DISCUSSION OF RESULTS

It has already been stated that the failure to produce evidence of lattice orientation in these synthetic quartz fabrics may be total, or only partial, depending on what additional evidence may be obtainable by *x*-ray methods. At any rate the large, optically measurable grains lack evidence of lattice orientation. The development of the parallel dimensional orientation goes on during the stage of cementation and elimination of voids. After this stage is completed, the fabric of the synthetic quartzite is frozen into a permanent pattern which remains unchanged as far as the present group of experiments is concerned. To test this from another angle, thin, parallel-sided plates of natural quartzite[6] were subjected to the same range and combination of variables as listed in Tables 2 and 3. In the half-dozen experiments run there was found to be, without exception, no observable change in lattice or dimensional orientation of the plates, thus confirming the conclusions reached for the synthetic material. The conditions of the experiments are therefore inadequate for reproduction of the girdle orientation patterns of tectonite quartz.

Higher temperature might produce lattice orientation, but as the present equipment is not designed for much higher temperatures and pressures than those already used, this aspect of the investigation is problematical. Lengthening the time of an experiment carries with it two serious restrictions, (1) the short active life of the external piston, and (2) the continuous solubility of quartz in alkaline carbonates (enhanced by the pressure differential of the experiments).

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[6] The Lorrain quartzite (Huronian), Sudbury District, Ontario, was used for this purpose.

Natural quartzites are known which have random lattice orientation (Phillips, 1937), but without more detailed information as to their tectonic history it would be unwise at present to attempt correlation with the lattice orientation of the synthetic product described in this paper. Fairbairn (1949, pp. 167–169) has outlined some hypothetical stages of recrystallization which, if applied here, would involve paratectonic recrystallization and an intermediate state of dimensional orientation, with no change in lattice orientation. The deformed material, however, is poorly recrystallized, indicating possibly that granulation has outstripped the recrystallization process.

The conditions of the experiments impose a nonrotational strain, or restricted type of transport on the material (Fairbairn, 1949 Chap. 17). As already seen, this does not reproduce the common girdle orientation of tectonite quartz. If rotational strain is necessary for production of these girdles, the only feasible way to impose it on the experimental material is to apply a torque in addition to the normal compressive load. Some work has been commenced in this direction, but since *x*-ray analysis of the deformed material appears to be necessary, no thorough study has yet been made.

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