[AMERICAN JOURNAL OF SCIENCE, VOL. 276, NOVEMBER, 1976, P. 1120-1130]

AN INDEX OF CRYSTALLINITY FOR QUARTZ

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ABSTRACT. A crystallinity index (CI) for quartz minerals is based on the degree of resolution of the $d(212)$ X-ray reflection at 1.3820A. Clear euhedral quartz shows the maximum resolution of this reflection, and measurements are scaled to give a maximum CI of 10 for such quartz samples. Examples of poorly crystallized quartz (CI of <1.0-3.0) are chrysoprase from Australia, cherts of the Monterey Shale of California, and Caballos Novaculite of Texas. Well-crystallized quartz (CI 8.0-10.0) occurs in Franciscan cherts of California and in the Arkansas Novaculite at places where these rocks seem to be strongly metamorphosed. The crystallinity index seems to be largely a function of crystalline size (up to about 1 μ diameter) but may also be affected by lattice distortions induced by mechanical stress. That time alone will not improve crystallization is shown by a silicified wood of *Callixylon* with a CI of 1.2 from the Devonian of Ontario, Canada.

INTRODUCTION

The wide range in crystallinity of quartzose cherts recovered from deep-sea sediments has been discussed qualitatively by Hathaway (1972) in terms of profiles of certain X-ray diffraction peaks, particularly a set of five peaks at 2θ of 67° to 69°, CuK_α radiation, which is fully developed only in diffractograms of well-crystallized quartz. For convenience of discussion, we refer to this set of peaks as the 68° quintuplet. All quartzose cherts, regardless of degree of crystallinity, yield a strong diffraction pattern of quartz in the lower range of 2θ (20°-50°) which is usually examined. Deterioration in quality of the diffraction peaks of quartz at high angles of 2θ has been shown by Klug and Alexander (1954, p. 529) to be characteristic of crystallites smaller than about 1 to 5 μ in diameter.

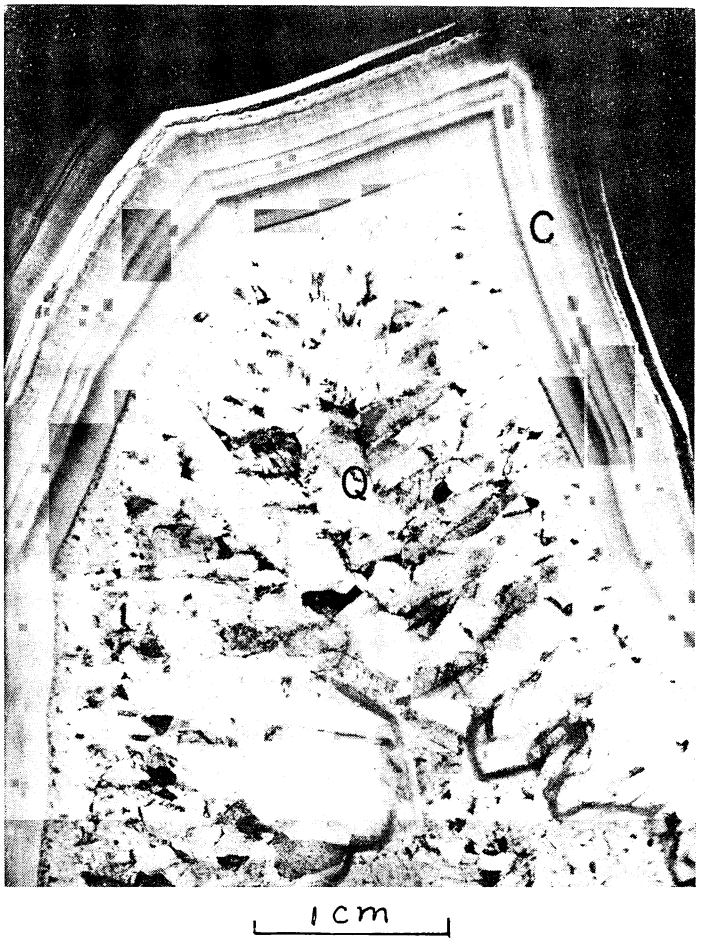
We have found that the 68° quintuplet is always poorly developed in diffractograms of the recently described (Murata and Larson, 1975) quartzose cherts from the Miocene Monterey Shale of California. The same is true for chalcedonic silica of agates and petrified wood of even Paleozoic age. Fournier (1973) has shown that chalcedony is appreciably more soluble in water than macroquartz at temperatures of 50° to 200°C. Chalcedony with its poorer crystallinity is thus metastable relative to macroquartz but can persist in a metastable state for geologically long periods. The factors that determine the degree of crystallinity of quartz and a semiquantitative X-ray method of measuring the crystallinity are described here.

X-RAY PROCEDURE FOR DETERMINING CRYSTALLINITY

In characterizing the bulk crystallinity of chert, silicified wood, or other forms of microquartz, care is taken to exclude materials of veins and drusy patches with possibly different crystallinity. Likewise in studying different parts of a texturally zoned sample, like the agate of plate 1, each part is separated as cleanly as possible from others. The fine grinding and the mounting of the sample in a conventional aluminum holder are done according to the procedure of Tatlock (1966).

PLATE 1

A thick section of a simply zoned Brazilian agate (sample 22 of table 1) showing a margin of chalcedony C and a core of coarsely granular quartz Q.



The 2θ interval of 66° to 69° is scanned at ¼°/min with nickel-filtered copper radiation. A sample of clear euhedral quartz, such as that from Herkimer County, N.Y., is used originally to fix the counting parameters, so that the strongest peak (2θ of 68.14°) of the quintuplet will stay on the diffractometer chart. We use a counting rate of 320 counts/sec and a chart speed of ½ in./min.

Reproducibility of the results seems to depend mainly on the consistency with which the finely ground powder is mounted in the aluminum holder, firmness of packing and flatness of the sample surface being especially critical factors (Klug and Alexander, 1954, p. 299). Deter-

minations are made in triplicate, either with three separate splits of the sample or, if supply is limited, three separate mounts of the same sample.

CHARACTERISTICS OF THE 68° QUINTUPLET

Among the quartz minerals studied by us, the profile of 68° quintuplet ranges from a simple broad hump through a series of profiles of increasingly better resolution to a sharply defined quintuplet (fig. 1). We derive an index of crystallinity from the intensity of the (212) peak at 2θ of 67.74°, which is marked with an asterisk in figure 1.

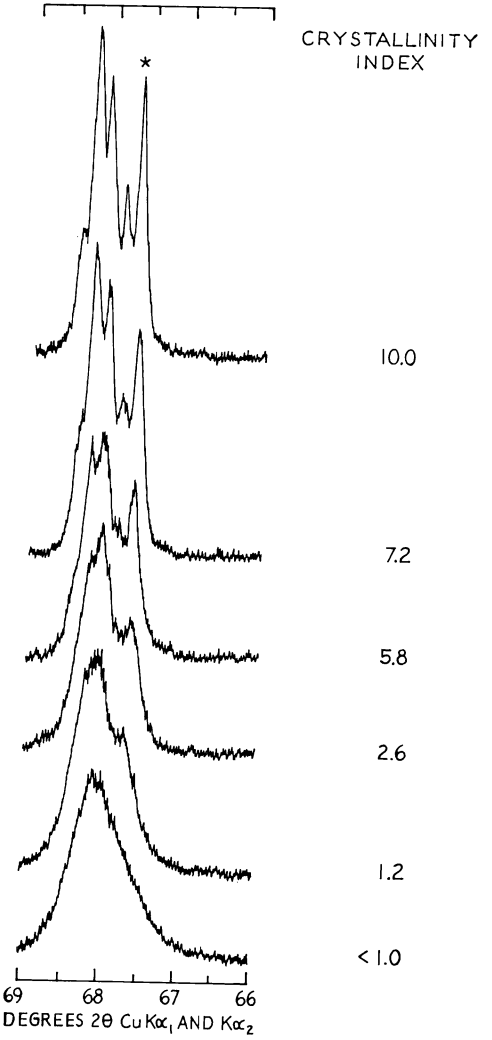


Fig. 1. Spectrometer tracings of the 68° quintuplet arranged (in the upward direction) in order of increasing crystallinity of quartz. Asterisk marks the (212) peak at 2θ of 67.74° which is measured to obtain the crystallinity index given to the right of the tracings.

The relative intensities of the peaks of the quintuplet are generally as shown in the figure, but they are rather sensitive to the orientation of the grains within the mounted sample. For example, among the better crystallized samples, an occasional pattern is obtained in which the (212) peak is stronger than all others. Such a pattern is rejected, and another prepared after remounting the sample in the holder.

The method of computing the crystallinity index from the intensity of the (212) peak at 67.74° is shown by means of a pattern of a moderately well crystallized sample (fig. 2). Height a of this peak is first divided by its total height b above background in order to compensate for minor variations in the overall intensity of the pattern. The quotient a/b is further multiplied by 10 in order to convert fractions into numbers generally greater than 1.0. The highest value (6.0) of quantity $10a/b$ was obtained most consistently with clear euhedral crystals of quartz, like those from Herkimer County, N.Y.

In order to express crystallization indexes on the familiar scale of 10, our maximum determined value is raised from 6.0 to 10.0 by using a "scaling" factor F of 1.67. The complete equation used for all samples is

$$\text{crystallization index} = 10a\text{F}/b, \quad \text{where F is 1.67.} \tag{1}$$

Scaling factor F would probably vary from one diffractometer to another, so its separate determination on a sample of clear euhedral quartz could help to standardize results obtained with different instruments. When the 67.74° peak is barely seen or does not appear at all (bottom pattern in fig. 1), the sample is assigned an index of <1.0; such materials cannot be said to have zero crystallinity, because they always give a strong pattern of quartz at lower angles of 2θ. The precision of the method in terms of 2σ is 0.50.

Iron-rich materials, such as hematitic chert, pose a special problem for diffractometers equipped with copper tubes, because iron alters the absorptive properties of silica and also fluoresces strongly under copper radiation. The fluorescence enhances the background (quantity c in fig. 1), which causes the determined crystallinity index to be too high. With some samples, the error can be reduced by extracting the iron with a hot acid. A roughly correct result can be obtained for iron-bearing samples by arbitrarily assigning a normal background to their diffractograms, thereby correcting for the main effect induced by iron (Tatlock, 1966).

ANALYTICAL RESULTS

The materials studied are grouped in table 1 into five broad categories: (A) cherts and novaculites, (B) silicified plant remains, (C) zoned agate nodules, (D) granitic and pegmatitic quartz, and (E) miscellaneous quartz. Within each category, samples are generally listed in order of increasing geologic age of the host formation. The general trend of increasing crystallinity of microquartz with geologic age has been discussed by Laurent and Scheere (1971).

TABLE 1

Crystallinity index of quartzose materials (scale of 10; precision of determination = 2σ of 0.50)

Sample	Mineral or rock	Host formation	Age of host formation	Locality	Crystallinity index	Reference and notes
			A. Cherts and novaculites			
1	Chert from magadiite	Rome beds of informal usage	Pliocene	Malheur County, Oreg.	1.3	Sheppard and Gude, 1974
2	Bedded chert	Monterey Shale	Miocene	Temblor Range, Calif.	2.0-3.2 (8)*	Murata and Larson, 1975
3	Chert (black flint)	Chalk	Cretaceous	England	<1.0	U.S. Natl. Museum 36012
4	Chert	Franciscan Formation	Jurassic and Cretaceous	Cerro Colorado quad., San Benito Co., Calif.	8.0-9.0 (2)	M. B. Norman II, collector
5	Bedded chert	"	"	Penitencia Creek, Santa Clara County, Calif.	8.5	Crittenden, 1951
6	Replacement bedded chert	Calera Limestone Member of Franciscan Formation	"	Permanente quarry, Santa Clara County, Calif.	6.2 (2)	Wachs and Hein, 1974
7	Novaculite	Caballos Novaculite	Devonian and Mississippian	Marathon Basin, Tex.	2.2 (2)	King, 1937
8	Novaculite	Arkansas Novaculite	"	Hot Springs region, Ark.	5.7-9.8 (7)	Miser, 1943
9	Replacement bedded chert	Dolomite in Allamoore Limestone	Precambrian	Van Horn, Tex.	4.8	Rohrbacher, 1973
10	Bedded chert	Ironwood Iron-Fm.	Huronian	Gogebic Range, Mich.	6.5-7.0 (2)	Huber, 1959
			B. Silicified plant remains			
11	Wood	Neroly Sandstone	Miocene	Tesla, Alameda County, Calif.	1.1	Huey, 1948
12	Wood	Lamar River Formation	Eocene	Yellowstone Natl. Park, Wyo.	1.8	Smedes and Prostka, 1972
13	*Tempskya* trunk	— — — —	Cretaceous	Greenhorn, Oreg.	<1.0	Read and Brown, 1937
14	Wood	Chinle Formation	Triassic	Holbrook, Ariz.	2.2-3.5 (2)	Daugherty, 1941
15	*Osmundites* root sheath	— — — —	"	Mt. Augusta and Fremouw Peak, East Buckley Island Sheet, Antarctica	1.4-1.7 (2)	J. M. Schopf, collector
16	Silicified peat	— — — —	Permian	West Coalsack Bluff, central Antarctica	4.1	Schopf, 1970
17	Wood	Tradewater Formation	Pennsylvanian	Christian Co., Ky.	4.6	R. A. Peppers, collector
18	*Psaronius* with quartz druses	Monongahela Formation	"	Shade, Ohio	8.9	R. M. Kosanke, collector
19	*Callixylon*	— — — —	Devonian	Kettle Point, Ont., Canada	1.2	F. M. Hueber, collector

			C. Zoned agate nodules			
20	Chalcedony rim	John Day Formation	Oligocene and Miocene	Madras, Oreg.	1.0	Peck, 1964
	Macroquartz core	— — — — — — — — — — — — — — — —			9.3	— — — —
21	Chalcedony rim	Volcanic rocks	Tertiary	Chihuahua, Mexico	2.4	Sinkankas, 1959
	Macroquartz core	— — — — — — — — — — — — — — — —			9.2	— — — —
22	Chalcedony rim	Basalt	Triassic	Rio Grande do Sul, Brazil	<1.0	Sobrinho, 1958
	Macroquartz core	— — — — — — — — — — — — — — — —			9.0	— — — —
23	Chalcedony rim	Basalt	Keweenawan	Hibbard Bay, Ont., Canada	3.3	M. B. Norman II, collector
	Macroquartz core	— — — — — — — — — — — — — — — —			9.0	— — — —
24	Chalcedony rim	Amygdaloid Island Flow	Keweenawan	Isle Royal, Mich.	4.7	Huber, 1973
	Macroquartz core	— — — — — — — — — — — — — — — —			9.2	— — — —
			D. Granitic and pegmatitic quartz			
25	Granitic quartz	Half Dome Quartz monzonite	Cretaceous	Yosemite Natl. Park, Calif.	8.4	Bateman and others, 1963
26	"	Biotite granodiorite	"	Pt. Reyes, Marin County, Calif.	8.6	Ross, 1972, sample 531A
27	"	Quartz monzonite	"	La Panza Range, Calif.	8.9	Ross, 1972, sample P-14
28	"	Hornblende quartz monzonite	Jurassic	Gold Hill, Monterey County, Calif.	8.9	Ross, 1972, sample 133A
29	Massive quartz	Granite pegmatite	Precambrian	Hugo mine, Keystone, S.D.	9.1	Page and others, 1953
30	Clear euhedral quartz	"	Cretaceous	Kyburz, Calif.	9.9	E. F. Murata, collector
			E. Miscellaneous quartz			
31	Silicified coral	Tampa Formation	Miocene	Hillsborough County, Fla.	<1.0	Cooke, 1945
32	Chrysoprase	Laterite on serpentine	Paleozoic	Marlborough, Queensland, Australia	<1.0	Crowe, 1973; Brooks, 1964
33	Milky white gold-quartz vein	Serpentine	Mesozoic	Coulterville, Mariposa County, Calif.	7.0	Knopf, 1929
34	Tiger-eye, quartz replacement of crocidolite	Transvaal Supergroup	Precambrian X	Cape Province, Union of South Africa	8.4	Cilliers and Genis, 1964
35	Clear euhedral quartz	Little Falls Dolomite	Cambrian	Herkimer County, N.Y.	9.8	Tuttle, 1973
36	Clear euhedral quartz in veins	Several Paleozoic formations	Cambrian to Carboniferous	Ouachita Mtns., Ark.	10.2	Miser, 1943
37	Clear euhedral quartz	— — — — — — — — —		Synthetic quartz, manufacturer unknown	10.1	— — — —

* The number in parenthesis denotes the total number of subsamples analyzed from different parts of the formation. When no such number is given, only a single sample was analyzed.

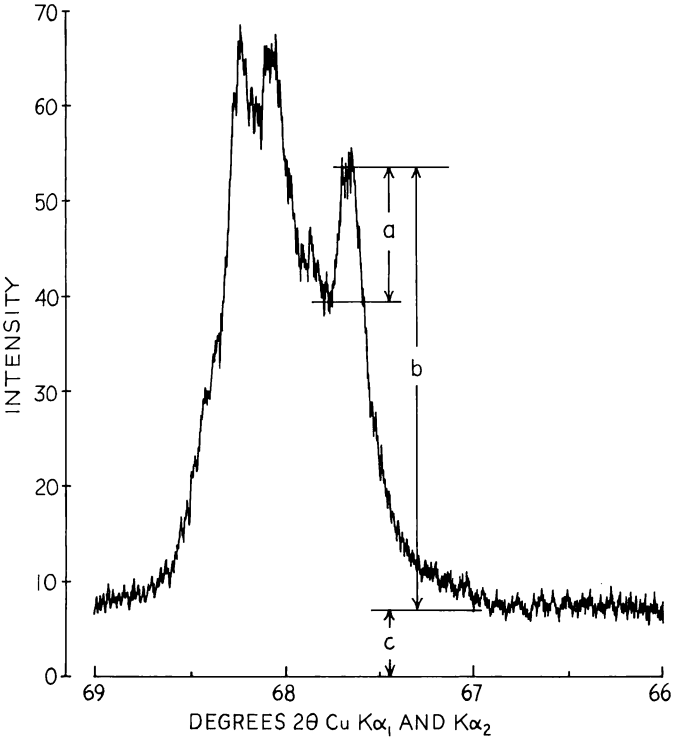


Fig. 2. The 68° quintuplet in the diffractogram of a moderately well crystallized quartz. Quantities *a*, *b*, and *c* are measured to obtain the crystallization index of the sample, as explained in the text.

Several important varieties of quartz minerals had to be ignored in this preliminary survey, and our conclusions regarding factors controlling the crystallinity of the minerals studied must be highly tentative because of the small number of samples examined. Nevertheless, the data suggest interesting relations between crystallinity and mode of origin of some quartz minerals and indicate possible use of the crystallinity index in studying metamorphism of quartzose rocks.

A. *Cherts and novaculites.*—The crystallinity of cherts and novaculites ranges widely from the surprisingly low value of <1.0 for chert from the Chalk of England (sample 3 of table 1) to 9.8 for one of the novaculites from Arkansas (sample 8). The chert of sample 1 represents an unusual type derived through alteration of sodium silicate minerals in an alkaline playa (Eugster, 1967; Hay, 1968; Sheppard and Gude, 1974). Its poor crystallinity of 1.3 probably reflects its mode of formation—rapid precipitation from a silica-rich solution at near-surface temperatures.

Many of the other cherts of category A were probably derived from biogenic opal of diatoms, radiolaria, and other siliceous organisms. We

are most familiar with this kind of bedded and nodular chert from the Miocene Monterey Shale of California (sample 2). Through progressive burial, biogenic opal of diatomaceous Monterey Shale is converted diagenetically into cryptocrystalline cristobalite that, at greater depth, is converted into microquartz (Murata and Larson, 1975). Quartzose chert occurs in the bottom 800 m of a 2300-m-thick Monterey Shale in the Temblor Range, and its crystallinity does not change systematically with depth, varying randomly between 2.0 to 3.2. Poor crystallinity may be characteristic of such diagenetic microquartz, which would be metastable relative to better crystallized macroquartz.

Cherts from the late Mesozoic Franciscan Formation of California (samples 4-6) are better crystallized (6.2-9.0) than those of Miocene Monterey Shale. Bailey, Irwin, and Jones (1964) suggest that these cherts did not form from biogenic opal but rather from silica that was liberated when hot submarine lavas reacted with seawater. However, the higher crystallinity of these cherts probably resulted from the more intense metamorphism to which they were later subjected (Bailey, Irwin, and Jones, 1964; Ernst, 1965) than from their different mode of origin. E. H. Bailey (oral commun. May, 1975) adds the caveat that conditions of metamorphism of some Franciscan cherts were zeolite facies, so that more poorly crystallized examples might be found when samples from more localities are tested.

Such a relation between intensity of metamorphism and crystallinity of quartz is suggested by a comparison of the Devonian and Mississippian Caballos Novaculite of Texas (sample 7) with the contemporaneous Arkansas Novaculite (sample 8). A white and a green variety of the Caballos Novaculite both show the same low crystallinity of 2.2, which is comparable to that of the Miocene Monterey Shale of California (sample 2). The Arkansas Novaculite is much better crystallized (5.7-9.8), the best crystallinity being found in a rock said to be of "abrasive honestone grade." The low crystallinity of the Caballos Novaculite seems to correlate with its mild diagenetic history (McBride and Thomson, 1970), and the higher crystallinity of the Arkansas Novaculite with the more intense metamorphism imposed by the Ouachita orogeny (Miser, 1943; Goldstein and Hendricks, 1953).

Under the electron microscope, novaculite of the Arkansas Novaculite appears as a tightly packed aggregate of polyhedral grains, mostly 1 to 10 μ in diameter (Folk and Weaver, 1952). That of the Caballos Novaculite typically has a bimodal texture consisting of coarser grains with 3 to 8 μ diameter and finer grains with 0.1 to 0.5 μ diameter (McBride and Thomson, 1970; W. D. Keller, written commun., May 1975). Apparently the difference in the X-ray index of crystallinity of the novaculites from the two regions is mostly a reflection of the difference in average size of their constituent crystallites.

The moderate crystallinity (6.5-7.0) of the Huronian cherts of Michigan (sample 10) is unexpectedly low in view of the great age of

these cherts but seems to be in keeping with the chlorite grade of metamorphism sustained by the host formation (James, 1955).

B. *Silicified plant remains.*—Silicified wood is a familiar kind of fine-grained petrification (Murata, 1940; Ransom, 1955) which occurs in sedimentary rocks of all ages since the Silurian. Our limited tests on quartzose woods (sample 11-17, 19) suggest that they are generally poorly crystallized (< 5.0), except when extensively recrystallized and permeated with drusy quartz (sample 18). The Devonian *Callixylon* (sample 19) is a remarkable example of the persistence of poorly crystallized (1.2) microquartz. Besides poor crystallinity, such quartz may retain some other original or early characteristics such as oxygen-isotopic composition.

C. *Zoned agate nodules.*—Zoned agate nodules that formed as vesicle fillings in lava flows are another familiar form of quartz. We selected agates that were zoned most simply, consisting of a rim of chalcedony around a solid core of coarsely granular quartz (fig. 1). Among the five samples (nos. 20-24) that range in age from Tertiary to Keweenawan, the crystallinity of the coarse quartz of the core is high and virtually constant (9.0-9.3), whereas that of the chalcedonic rim ranges from < 1.0 to 4.7. The somewhat higher crystallinity (3.3-4.7) of the rim in samples 23 and 24 from Keweenawan flows may reflect the greenschist (quartz-epidote-pumpellyite) facies of metamorphism of these flows (Stoiber and Davidson, 1959; N. K. Huber, oral commun., May, 1975) compared to the zeolite facies of the younger flows (Hay, 1963, for sample 20; Franco, 1952, for sample 22).

D. *Granitic and pegmatitic quartz.*—The crystallinity index of quartz separated from four granites (samples 25-28) and of massive quartz from a pegmatite (sample 29) falls in the range of 8.4 to 9.1, which is significantly lower than the index 9.9 of clear euhedral crystals from a cavity in pegmatite (sample 30). That granitic quartz should fall substantially short of perfect crystallinity is surprising because the anhedral crystals have a size in the range of millimeters rather than microns and because they crystallized out of a magma under presumably favorable conditions of high temperature and slow cooling. But granitic quartz usually shows wavy extinction in thin sections, which indicates strain or possibly a fine-grained mosaic structure (Bailey, Bell, and Peng, 1958; Blatt and Christie, 1963). The imperfect crystallization may be due to random distortions of the quartz lattice, induced by various mechanical and thermal stresses inherent in crystallizing granitic magma or brought to bear on a solidified pluton.

E. *Miscellaneous quartz.*—Miscellaneous quartz includes extremes of both poorly and well-crystallized quartz. The poorly crystallized samples, 31 and 32, are believed to have formed under near-surface conditions, probably from silica solutions that were excessively supersaturated relative to macroquartz. Samples 35 to 37 are further examples of well-crystallized clear euhedral quartz. Synthetic quartz (sample 37)

is of special interest, because it is grown from alkaline solutions very different from those commonly found in nature (Frondel, 1962, p. 156), which suggests that many different combinations of pressure, temperature, time, and composition might yield crystals of high perfection.

ACKNOWLEDGMENTS

Professor W. D. Keller of the University of Missouri kindly provided unpublished SEM data on the Caballos Novaculite of Texas. We are also indebted to many other people for their generous donation of samples. R. G. Coleman, B. R. Haley, and R. M. Kosanke made special collections of chert and silicified wood for this study.

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