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SCANNING ELECTRON MICROSCOPY OF CHERTS IN RELATION TO THE OXYGEN ISOTOPIC VARIATION OF SOIL QUARTZ

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Abstract—Quartz isolated from soils by the pyrosulfate-H_2SiF_6 method and chert samples of various origins were examined with the scanning electron microscope. Quartz isolates of the 20–50 μm from the A2 and B2lt horizons of the Baxter soil (AR), with quartz $\delta^{18}O$ of 18·2 and 19·0‰, respectively, showed a mixture of detrital quartz particles and chert clusters of aggregates of fine euhedral quartz crystals. The 2–5 μm fractions of both horizons consisted mainly of euhedral quartz particles. The 20–50 μm fraction from the underlying chert, with a $\delta^{18}O = 29·6‰$, consisted of aggregates of euhedral quartz particles 1–10 μm dia. and of subhedral particles 0·1–0·5 μm dia. In the soil fractions, the size and shape of the quartz particles as well as oxygen isotope data indicated that the aggregates were from the underlying chert but that irregular, unaggregated grains were detritally admixed loess, particularly in the medium and coarse silt fractions. This mixing of chert (of low temperature origin and heavy oxygen) with detrital quartz (of high temperature origin and light oxygen) gave rise to the intermediate $\delta^{18}O$ values in the soil quartz. The SEM of cherts of different geological ages showed different morphologies. Prismatic, polyhedral microcrystalline quartz of 1–10 μm size as well as submicron, euhedral particles were observed in cavities. Submicron, subhedral particles and interlocking quartz grains were characteristic of Precambrian chert. Quartz grains more than 100 μm in size isolated by HCl from Ordovician dolomite (WI) had large (many microns) subhedral overgrowths and attached clusters of 0·1–0·5 μm microcrystalline quartz. A Danish flint formed in chalk had calcite-lined cavities (x-ray emission determined) in which spheroidal fibrous chalcedony occurred.

INTRODUCTION

The objective of this paper is to show that the micromorphology of quartz in soil silt, as seen by scanning electron microscopy (SEM), can be used to identify particles of chert as distinct from particles of detrital origin. This relates to the evidence from oxygen isotopic composition that soil quartz is a sediment of mixed provenance from eolian, fluvial and glacial transport of silts of low temperature and high temperature origin (Jackson *et al.*, 1968, 1971; Clayton *et al.*, 1968, 1972; Syers *et al.*, 1969).

The dictionary of geological terms (American Geological Institute, 1960) defines chert as "a compact, siliceous rock formed of chalcedonic or opaline silica, one or both, and of organic or precipitated origin". Chert may form by inorganic processes (Eugster, 1967) as well as by hydrothermal and replacement processes (Pettijohn, 1957). This paper concerns mainly the quartz of chert. Through the replica technique of transmission electron microscopy, chert was shown as microcrystalline aggregates, either as well-defined polyhedral blocks with somewhat curved surfaces a few microns in size or as optically fibrous, spongy chalcedony (Folk and Weaver, 1952). With the same technique, Namurian bedded cherts of North Wales, U.K., were shown to have two dominant surface textures, granular and spongy, with intermediate textural gradations (Oldershaw, 1968). The observed spongy texture of chert in both replica studies may correspond to the aggregates of subhedral quartz crystals resolved much more clearly by this SEM study.

MATERIALS AND METHODS

Samples consisted of quartz isolates from the horizons of the Baxter soil on Mississippian cherty dolomite, Rogers, Benton Co., AR (Syers *et al.*, 1969); a chert nodule, chips and fragments from Wisconsinan Cary dolomitic glacial till and soil and a red bedded chert from lower Ordovician (Oneota) dolomite, Madison, WI; Prairie du Chien (Ordovician) dolomite from Richland Co., WI; a black chert (flint) developed in chalk from glacial till 15 km west of Copenhagen, Denmark; an Eocene chert from 15 km east of Gainesville, FL (accompaniment in the field collection by W. K. Robertson); a Pennsylvanian chert from 2 km east of Tuscola, IL; a Precambrian chert from the basal portion of the Biwabik iron formation of the Mesabi range, MN (courtesy G. LaBerge).

The cherts were lightly crushed and freshly fractured chips were placed in 6N HCl (except for control samples, see text) and heated to boiling for 2 hr to remove carbonates and iron oxide coatings. The chert chips were then cemented to 9 mm dia. Al stubs

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with wet silver cement. Soil quartz isolates were attached to the stubs with double-stick tape. The mounted samples were then coated with a film of Au–Pd alloy of several hundred Å thickness evaporated at multiple angles of incidence to form an electrically conductive surface. SEM observations were done with a JEOL JSM50A Scanning Electron Microscope with x-ray emission detector.

RESULTS AND DISCUSSION

Quartz from soils and subsoil cherts

Scanning electron micrographs of the quartz isolate from a chert chip from Baxter C horizon show aggregates of about 10–50 μm at low magnification (Fig. 1a). At successively higher magnifications (Fig. 1b,c, d), the aggregates are revealed to be composed of very fine euhedral and subhedral grains as well as irregular particles, mainly 1–10 μm in size. Still higher magnification (Fig. 1e,f) shows that the smaller particles are subhedral crystals about 0·1–0·5 μm in size. Aggregates or clusters similar to those of the Baxter soil were found in weathered chert bodies (discussed in a following section). Silica, presumably amorphous initially, has crystallized into quartz (shown by X-ray diffraction identification) grains of varying size. Submicron particles (Fig. 1f) are generally believed to contain some water and, hence are closer to X-ray amorphous silica, i.e. opal. The size of these submicron particles are comparable to 0·15–0·35 μm spheroidal opal (Greer, 1969); however, the SEM of crystal faces on these submicron particles suggests a gradation to crystalline silica. Further crystal growth has produced the common prismatic shape of quartz crystals (Sayin and Jackson, 1974).

Soil quartz isolates from Baxter soil A2 horizon (AR), developed in silty material, show in the 2–5 μm fraction mainly euhedral quartz particles (Fig. 2a) which appear to have come from cherty limestone parent material. Two rhombohedral terminations of prismatic quartz particles are clearly seen at higher magnification (Fig. 2b). SEM of the 20–50 μm fraction shows a mixture of irregular detrital quartz particles and chert aggregates (Fig. 2c). The detrital quartz, absent in underlying chert (Fig. 1a, 1b, 1c), may have been added to soil as loess. Higher magnification again shows the euhedral quartz grains as well as smaller particles (Fig. 2d and 2e) in the chert clusters. Granular particles are revealed to be subhedral under higher magnification (Fig. 2f). Figure 2c, d, e and f are at the same magnification as Fig. 1a, c, d and f and can be compared to shape and size.

Some authigenic quartz particles are seen in the 2–5 μm fraction of the B21t horizon of the same soil profile (Fig. 3a). The 20–50 μm size fraction revealed the same morphology (Fig. 3b, c, d) as the quartz isolate from the overlying horizon. The characteristic aggregation of microcrystalline quartz in the soil coarse silt is believed to occur during crystallization (discussed below) from much finer gel or skeletal particles of silica.

Relation of $\delta^{18}O$ values of quartz with the SEM

The lower oxygen isotope ratios in quartz of different size fractions of loessial Baxter soil horizons $\delta^{18}O$ values of 18·2–25·9‰) than that of underlying chert (29·6‰; Table 1) indicate that the silty Baxter soil is not derived exclusively from chert. The SEM pictures of cherty quartz mixed with detrital quartz (Figs. 1, 2 and 3) further support the mixed provenance of sedimentary silt inferred from the $\delta^{18}O$ data (Jackson *et al.*, 1968, 1971; Clayton *et al.*, 1968, 1972). The $\delta^{18}O$ value of quartz in the 2–5 μm fraction is much higher (more chert, less detrital quartz) than that of the 20–50 μm fraction, since loessial silt is dominantly in the 10–50 μm size range.

Study of chert controls

The surface in the weathered portion of the freshly fractured chert nodule (Fig. 4a) reveals many subangular grains, on the order of 1 μm (Fig. 4b,c). They tend to occur as aggregates or clusters a few tens of microns in diameter. The cement between the clusters is evidently less resistant chemically than the grains themselves. With time, these aggregates evidently separate from the main body to form soil chert aggregates (noted above). The smooth, banded agate portion (B) displays large plate-like, smooth fracture surfaces (Fig. 4d), which appear to be results of splitting through the clusters of quartz grains, i.e. the cementation is physically as strong as the grains in the unweathered chert. Some of the subangular quartz particles, in the plane of cleavage (arrows) and below the plane, are similar to those in the weathered portion. More toward the chert nodule center (C), a microcrystalline quartz portion shows many homogenous euhedral and prismatic microcrystalline quartz crystals (Fig. 4e), much like those of the Baxter 2–5 μm fractions (Fig. 2a, 3a). The crystals are somewhat larger than those in the weathered part of the chert body (Fig. 5b). The margin of the central geode of the chert nodule has macrocrystalline, euhedral quartz grains, visible to the naked eye (Fig. 4f). They have large, well-developed, smooth crystal faces.

Table 1. Oxygen isotopic composition and content of soil quartz in loessial Baxter soil over calcareous substrata*

Horizon	Depth (cm)	Fraction (μm)	Quartz: Weight in residue (%)	Quartz: $\delta^{18}O$ (‰)
A2	5–25	20–50	86·4	18·2
		2–5	82·2	24·6
B21t	25–43	20–50	88·8	19·0
		2–5	87·7	25·9
Powdered chert	76–183	20–50	87·8	29·6

* Data from Syers *et al.* (1969).

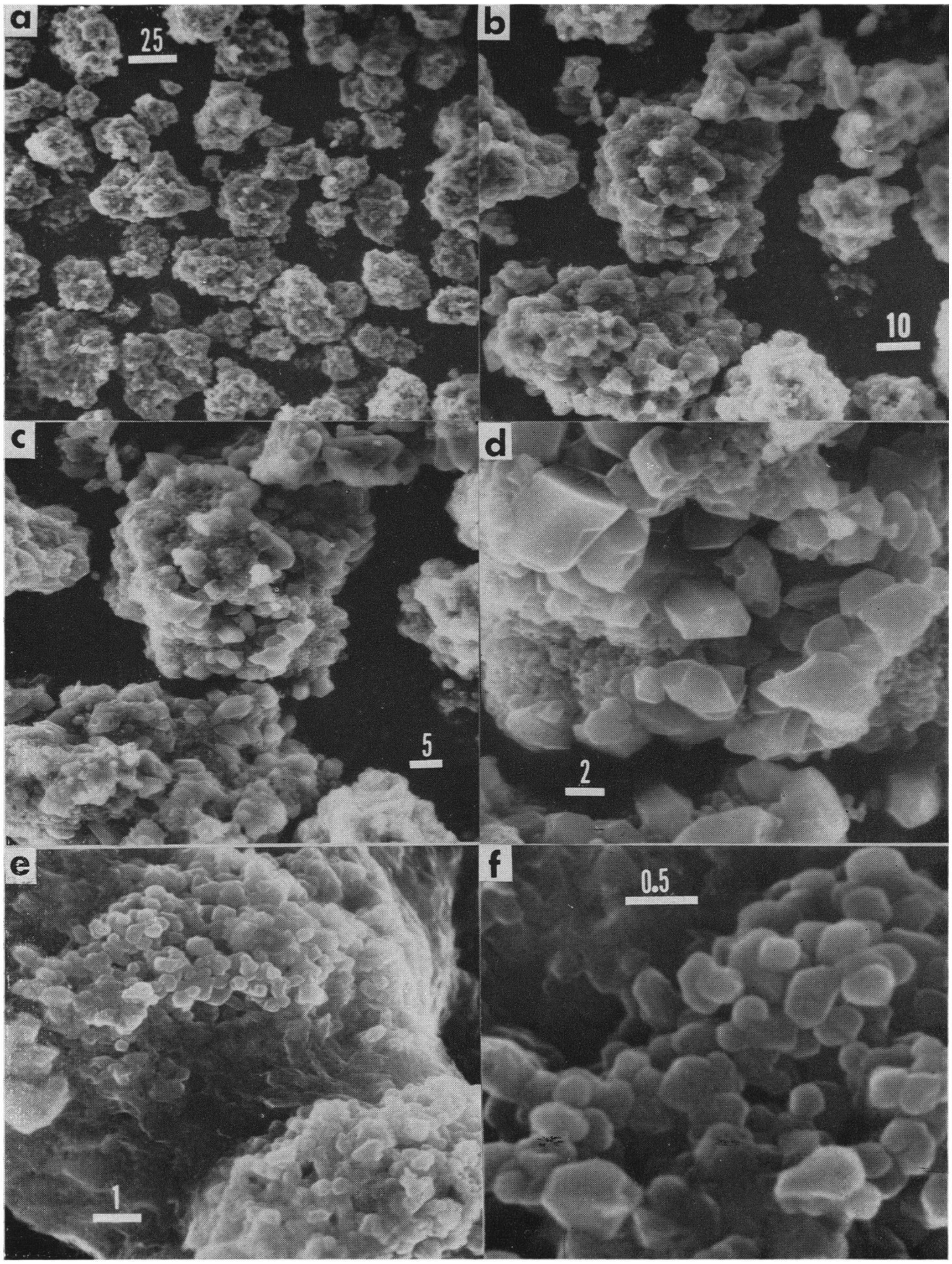

Fig. 1. Scanning electron micrographs of the 20–50 μm quartz isolate from chert under Baxter soil at progressively increasing magnifications. Number on bar indicates microns.

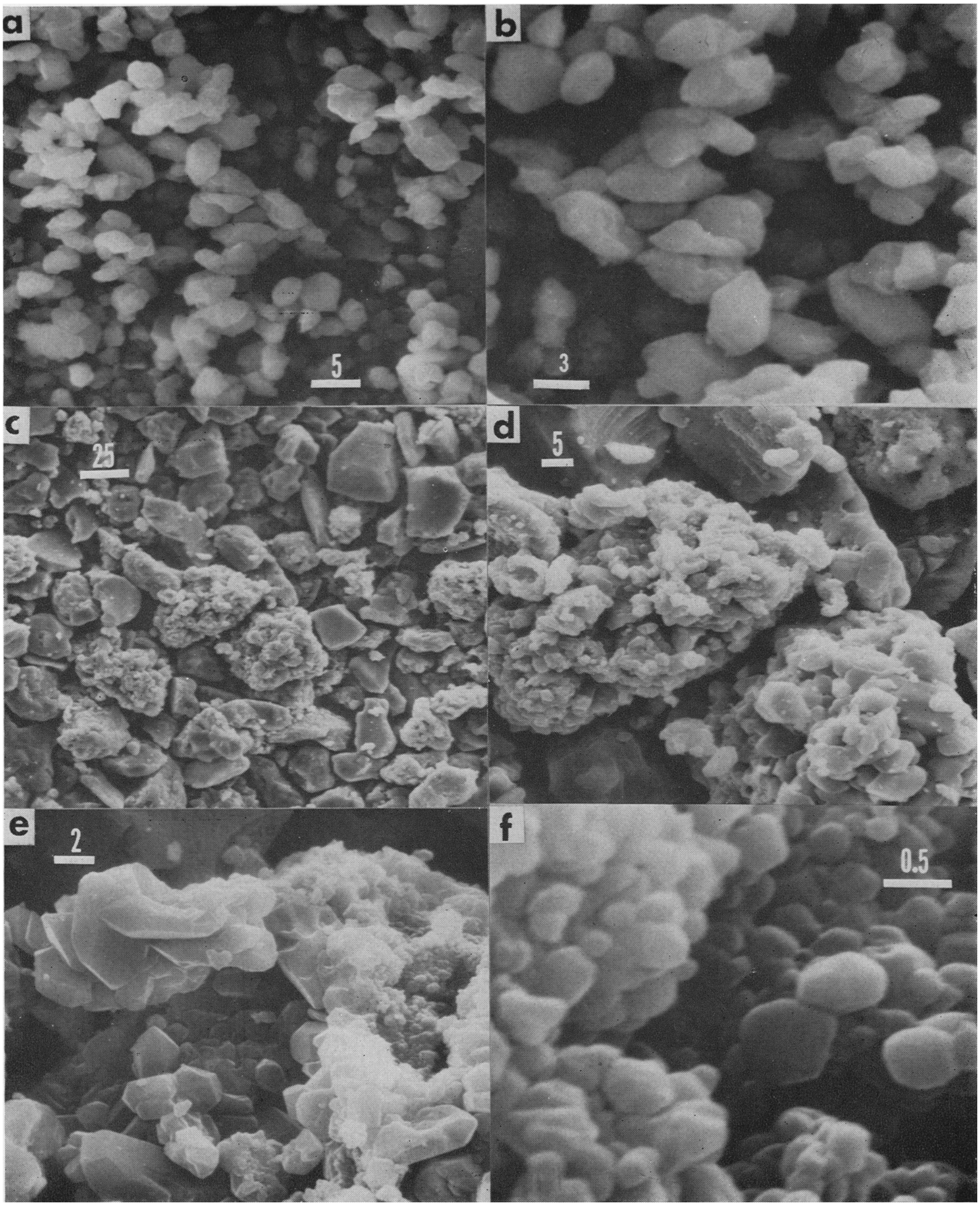

Fig. 2. Scanning electron micrographs of the quartz isolates from the Baxter soil, A2 horizon: of the 2–5 μm fraction (a) and (b); of the 20–50 μm fraction at progressively increasing magnifications, (c), (d), (e), (f). Number on bar indicates microns.

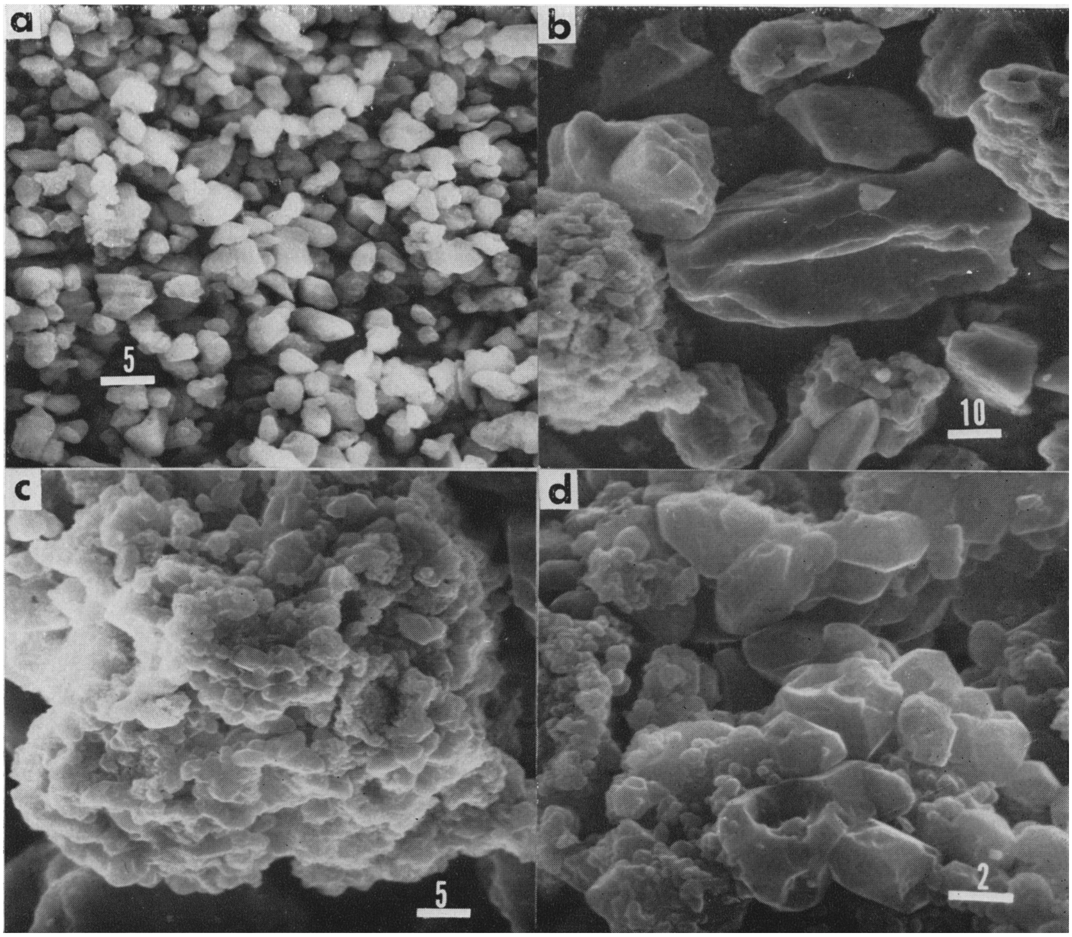

Fig. 3. Scanning electron micrographs of the quartz isolates from the Baxter soil, B21t horizon: of the 2–5 μm fraction (a); of the 20–50 μm fraction at progressively increasing magnification: detrital grains and chert aggregates, (b), chert aggregate, (c), and further enlargement of the latter, (d). Number on bar indicates microns.

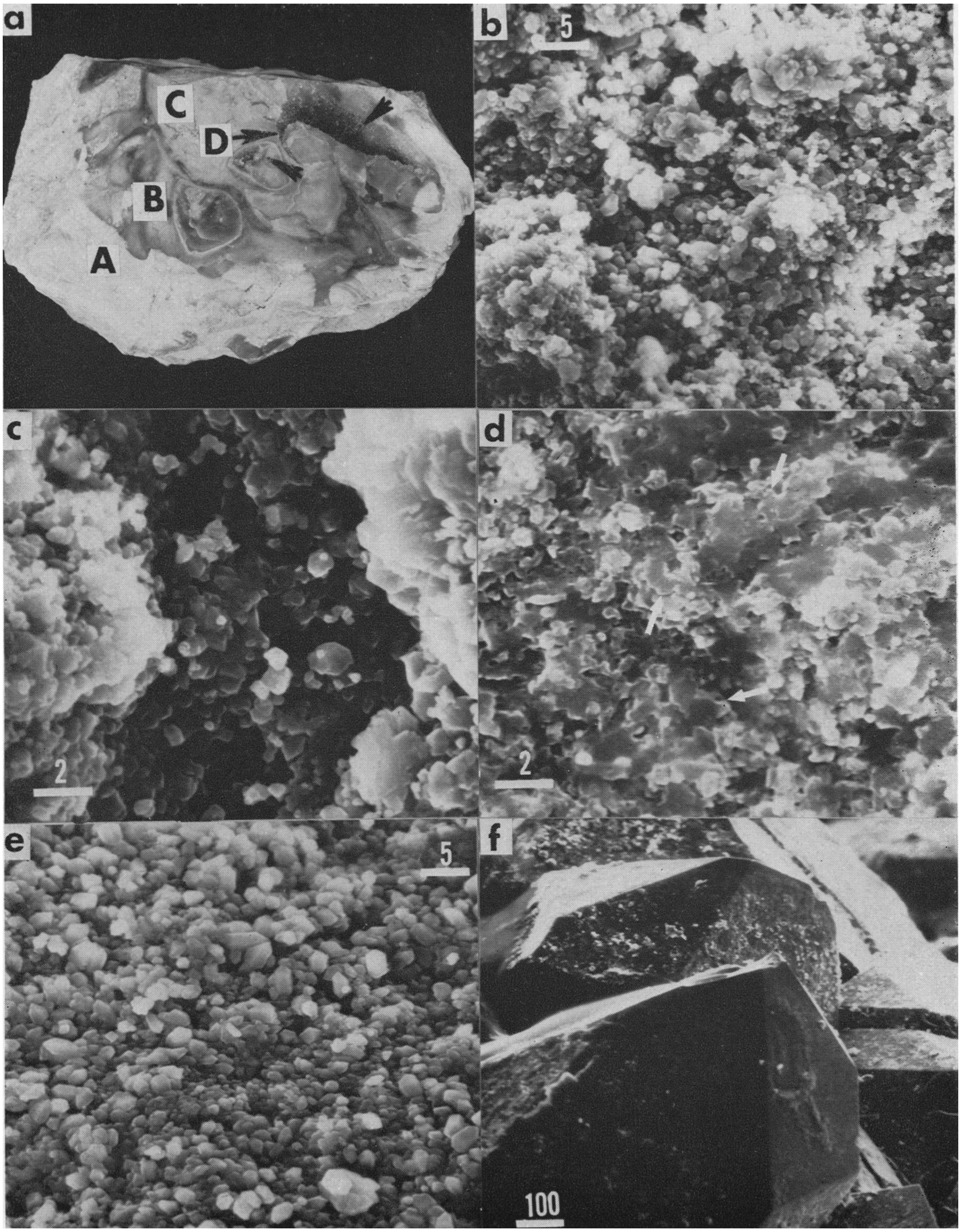

Fig. 4. Picture of a chert nodule and scanning electron micrographs of different portions of it: (a) picture of chert nodule, 8 cm long A (weathered), B (agate), C (microcrystalline), and D (macrocrystalline). SEM views: of the outermost weathered portion composed of clusters or aggregates at different magnification, (b) and (c); of unweathered agate fracture through clusters (arrows), (d); of microcrystalline euhedral and prismatic quartz grains, (e); and of macrocrystalline quartz, (f). Number on bar indicates microns.

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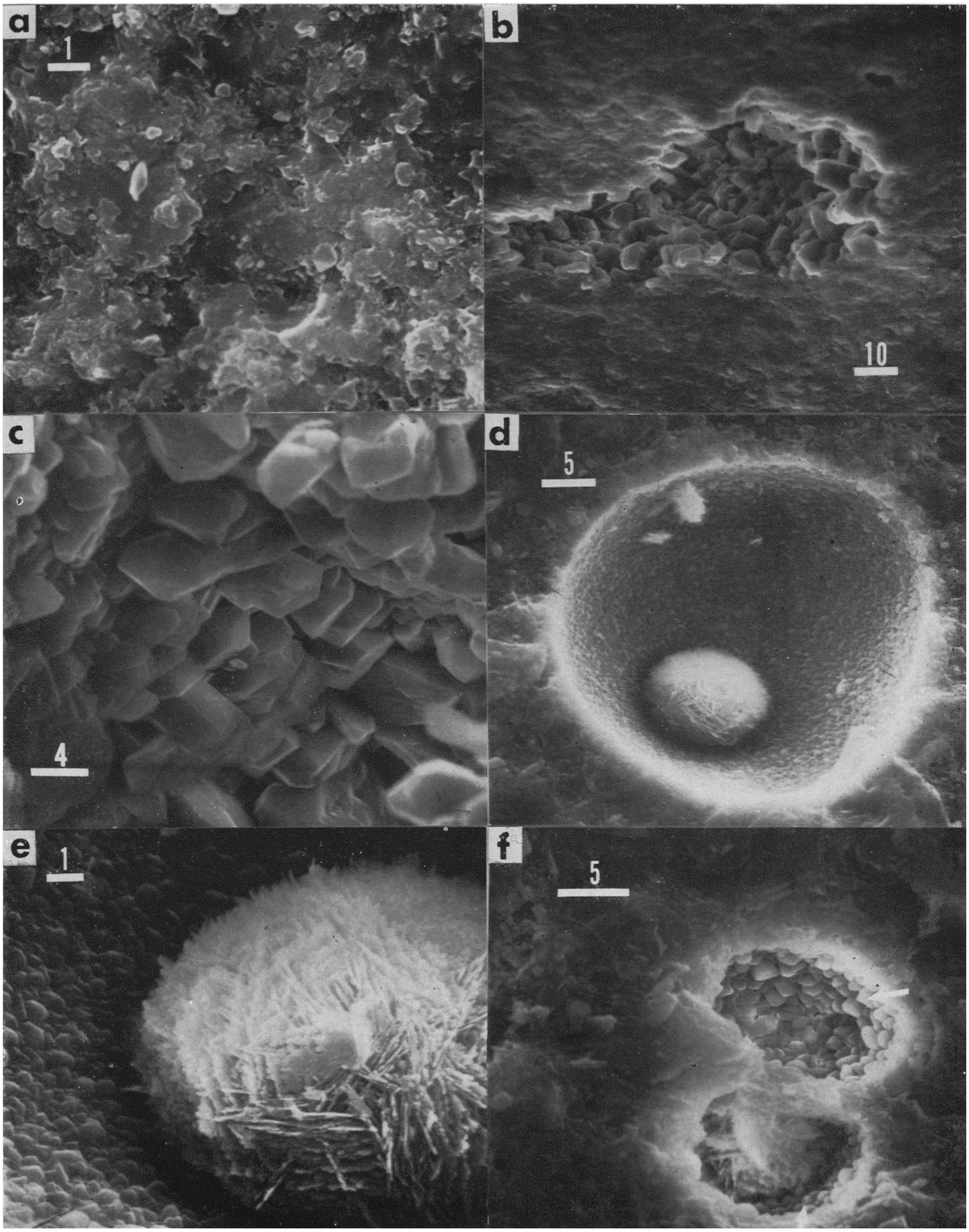

Fig. 5. Scanning electron micrographs of Danish chert: (a) fracture surface; (b) microcrystalline quartz in a cavity; (c) closer look at the microcrystalline quartz; (d) calcite hole and chalcedony spheroid; (e) closer look to chalcedony spheroid; (f) two cavities formed of calcite crystals (arrows) and chalcedony fibers in one cavity. Number on bar indicates microns.

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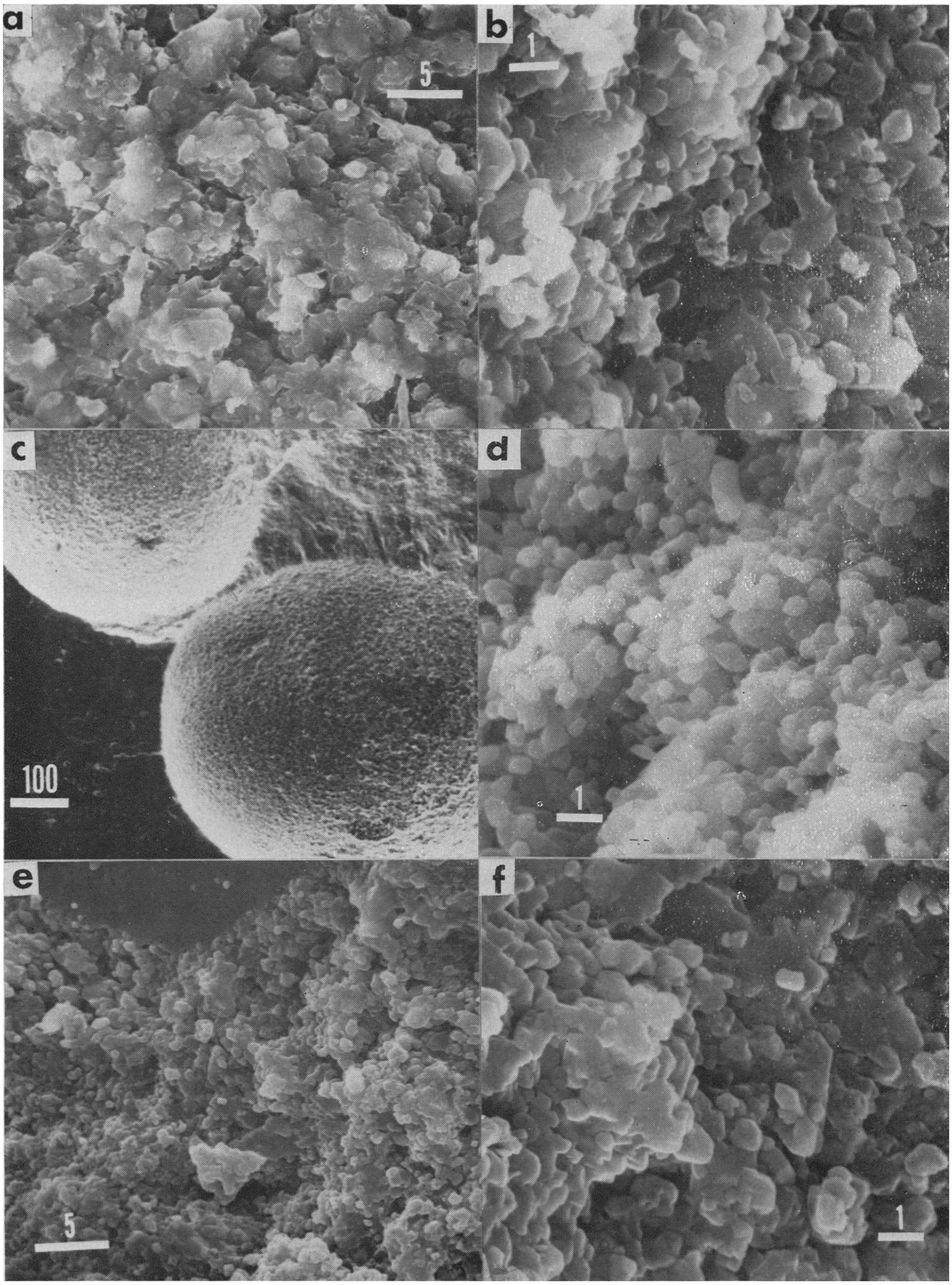

Fig. 6. Scanning electron micrographs of red bedded and nodular (from southern Wisconsin till) cherts: (a) fracture surface of red bedded chert; (b) microcrystalline quartz in a cavity of red bedded chert; (c) two spheroidal cavities in red bedded chert; (d) submicron quartz inside of one of the cavities. (e) Surface of nodular chert; (f) closer look to the surface of the nodular chert. Number on bar indicates microns.

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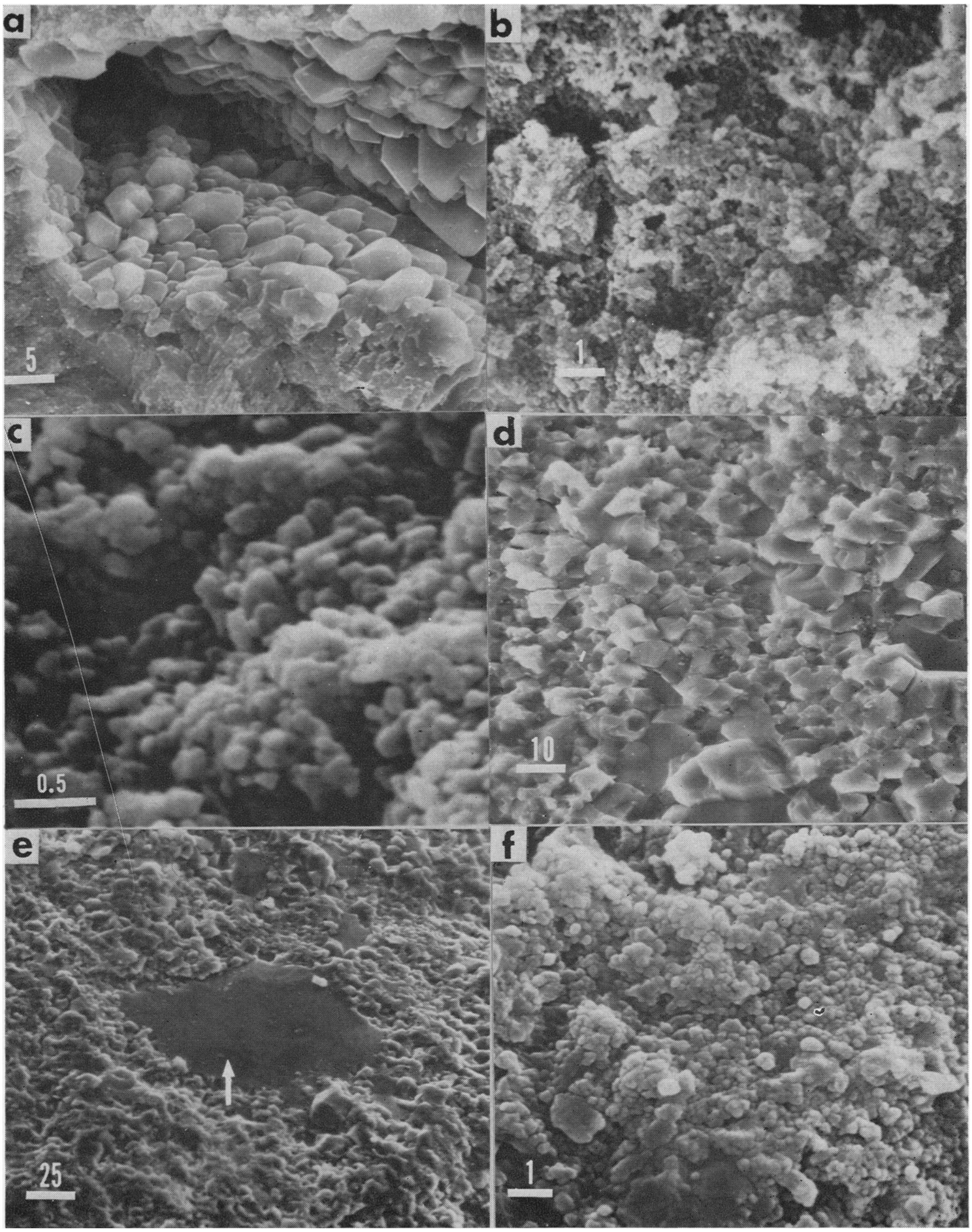

Fig. 7. Scanning electron micrographs of cherts: (a) fracture surface and cavity in unweathered FL chert. (15 km east of Gainesville; Eocene); (b) fracture surface of weathered part; (c) closer view of weathered part; (d) surface of IL chert (quarry, 2 km E. of Tuscola, IL; Pennsylvanian); (e) smooth fracture (arrow) on the IL chert; (f) surface of a chert fragment from a southern WI soil. Number on bar indicates microns.

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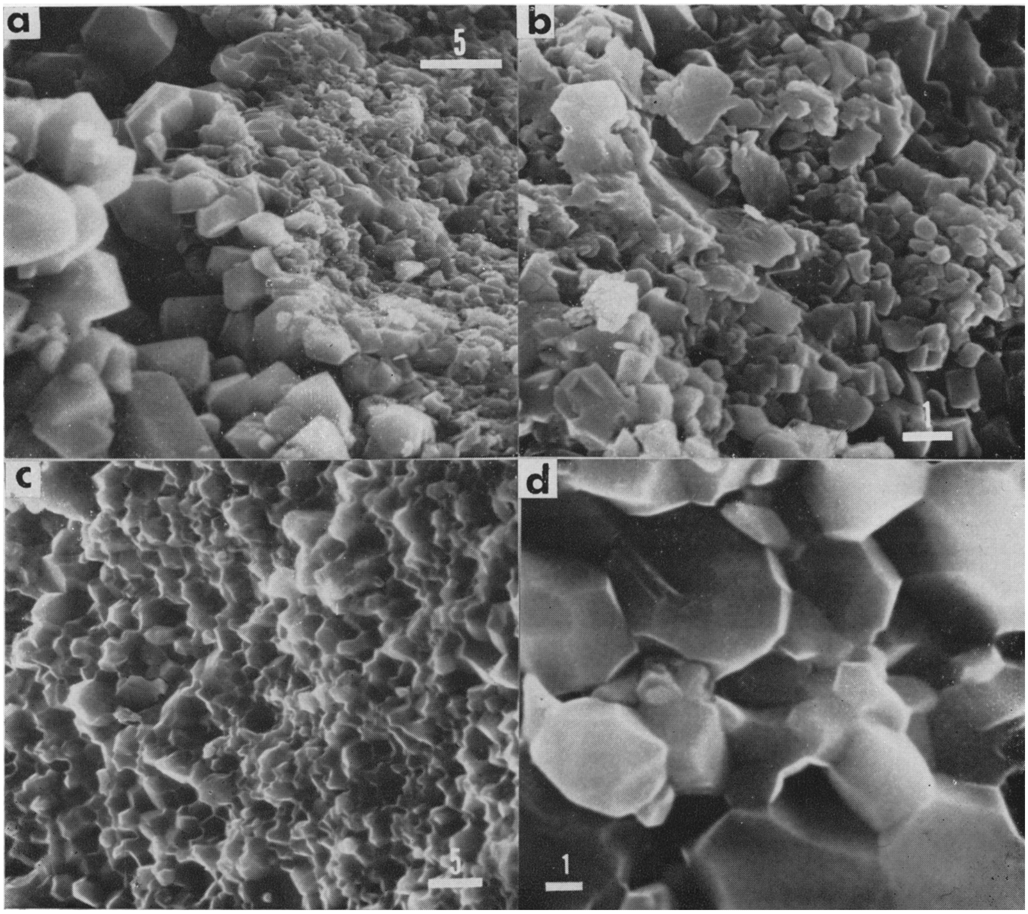

Fig. 8. Scanning electron micrographs of cherts: (a) a microcrystalline quartz cavity on the chert associated with Cary till, WI; (b) closer view of the surface of above chert; (c) surface of Precambrian chert, Mesabi Range, MN; (d) closer look to the surface of the Precambrian chert, showing detail of interlocking grains. Number on bar indicates microns.

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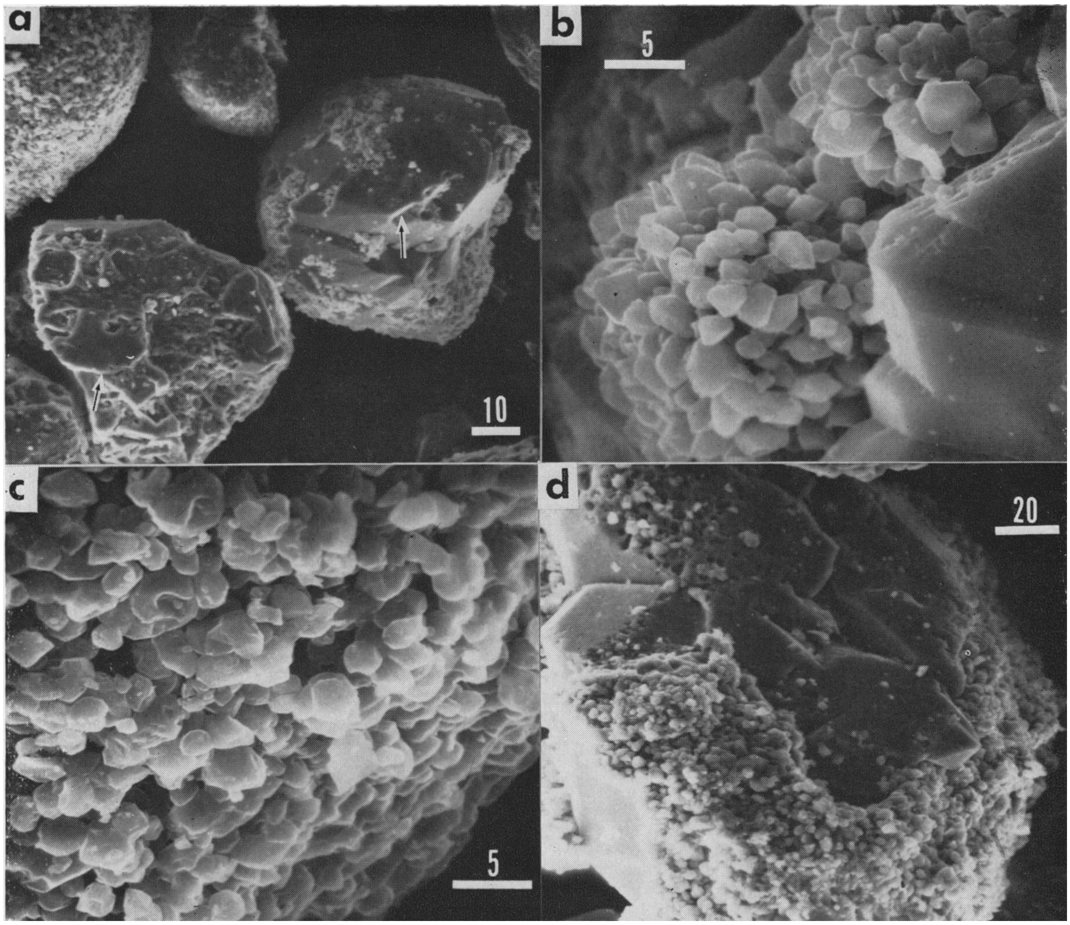

Fig. 9. Scanning electron micrographs of quartz isolate from Prairie du Chien (Ordovician) dolomite: (a) macrocrystalline quartz particles with secondary enlargements (overgrowths, arrows); (b) microcrystalline quartz on the surface of macrocrystalline quartz, the one at center in (a); (c) microcrystalline quartz on the surface of the particle at upper left in (a); (d) larger quartz particle with microcrystalline quartz. Number on bar indicates microns.

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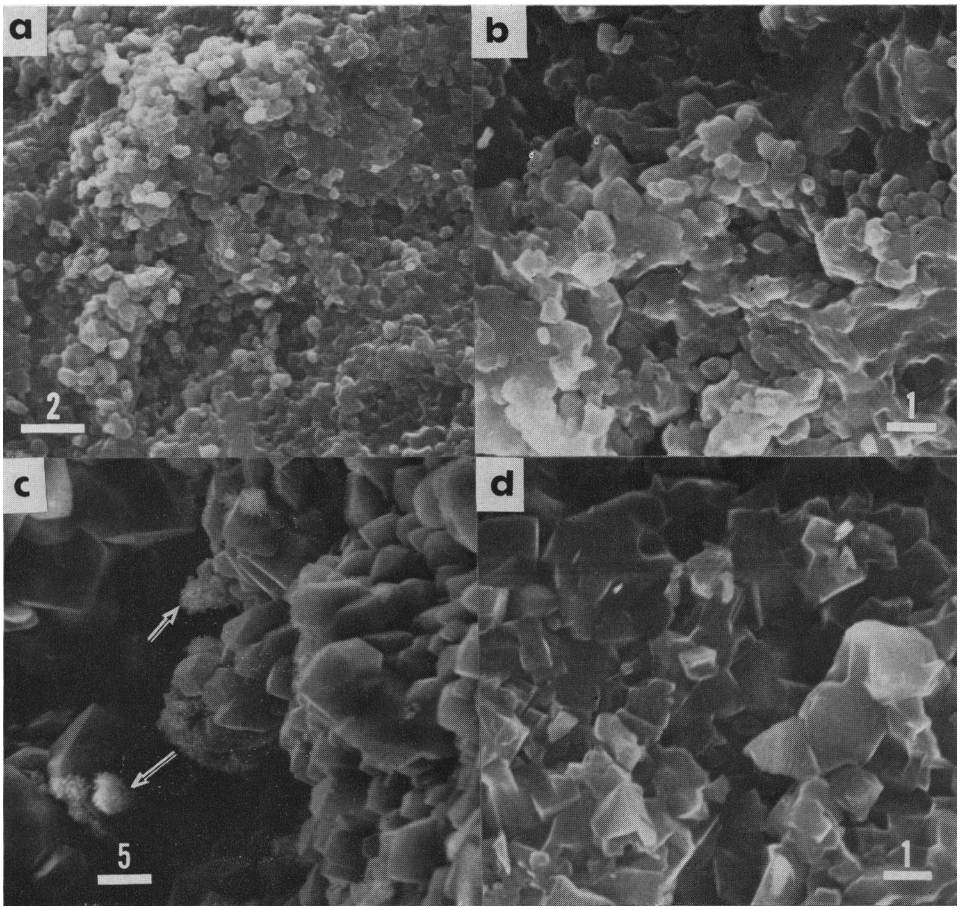

Fig. 10. Scanning electron micrographs of control samples, showing similarity with HCl treated samples (figure reference in parantheses): (a) chert nodule, weathered section (Fig. 4c); (b) nodular chert (Fig. 6f); (c) FL chert (Fig. 7a) with Fe bearing substances (arrows); (d) chert associated with Cary till, WI (Fig. 8b). Number on bar indicates microns.

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Chert of glacial till west of Copenhagen, Denmark (Fig. 5a) has a smooth platy fracture surface, similar to that of agate (Fig. 4d). A cavity in this chert (Fig. 5b) is filled with microcrystalline quartz. Higher magnification reveals euhedral quartz crystals about 5 μm dia. (Fig. 5c). A fibrous spheroid of about 10 μm dia. was observed in a hole in the white colored section of chert not treated with 6 N HCl (Fig. 5d). X-ray emission showed the presence of abundant Ca in the walls of the hole, suggesting that the rhombohedral crystals are calcite. Closer examination of the spheroid (Fig. 5e) revealed subparallel fibers making up the whole particle. X-ray emission showed Si and Ca indicating an intimate mixture of quartz and calcite crystallites. However, the fibers probably belong to chalcedony, a microcrystalline variety of quartz which exhibits fibrosity under a light microscope (Pelto, 1956), although the same compound looked spongy in the electron microscope replica pictures (Folk and Weaver, 1952; Monroe, 1964). In another area, one hole was filled with calcite (Fig. 5f, upper) and the other one (lower) with calcite and fibrous silica or chalcedony. The X-ray spectrum of the surface showed both Ca and Si, reflecting the replacement of $CaCO_3$ by SiO_2 in the formation of chert (or flint) in the chalk.

The surface of red-bedded chert (Fig. 6a) has a platy morphology similar to the agate and Danish chert (compare with Fig. 4d and Fig. 5a), a result of splitting the grains of the clusters or aggregates. A cavity in the same sample (Fig. 6b) showed microcrystalline, euhedral and prismatic quartz crystals, 1 μm or less in size. In another area on the surface (Fig. 6c, upper hole), similar but smaller grains were observed (Fig. 6d). The euhedral grains grown in the voids are mostly less than 1 μm in size. These euhedral grains are presumably not present in the chert rock because of the lack of space for free growing. Chert associated with Ordovician dolomite showed the same unresolved grain boundaries (Fig. 6e) as agate (compare to Fig. 4d). The presence of large quartz plates (left) may suggest formation of crystals from them. A higher magnification of the surface (Fig. 6f) shows that many of the particles are 1 μm or less in size.

An Eocene chert from Florida had an unweathered inner part and a weathered outer part. The SEM picture from unweathered section shows a platy surface with microcrystalline quartz crystals on the order of a few microns, precipitating into or forming freely in the cavity (Fig. 7a). The platy surface is a result of the splitting of the quartz grains (compare Fig. 5a; Fig. 6a), although the truncated particles may not be necessarily as euhedral and large as those growing in the cavity. The fracture of the weathered outer portion shows very fine granules (Fig. 7b). Higher magnification (Fig. 7c) reveals that the particles are about 0·1 μm or less, hence in the size range of colloidal silica. Close examination reveals that these submicron particles show some face development, although less clear than that of the subhedral particles of the Baxter chert (compare Fig. 1f). X-ray powder diffraction patterns of the samples from both sections gave sharp peaks indicating 3·34 Å spacing of $(10\bar{1}1)$ structural planes and 4·25 Å spacing of $(10\bar{1}0)$ structural planes of quartz without any difference in intensities. Although crystallization of amorphous silica through an intermediate mineral phase such as opal or cristobalite to quartz takes a long time (Siever, 1972), in an alkaline environment with frequent pH changes such as Na_2CO_3 lakes (Eugster, 1967) and lagoons (Peterson and von der Borch, 1965) direct precipitation of silica from the solution and subsequent crystallization is quite fast. Furthermore, laboratory experiments proved that quartz can be precipitated directly from sea water with a constant pH of 8·1 and reaches equilibrium with 4·4 ppm silica in the short time of 3 yr (Mackenzie and Geer, 1971). This evidence can help explain the formation of quartz in lagoonal areas of Florida in a relatively short geologic time span since the Eocene Epoch.

The chert from Illinois (Fig. 7d) shows very dense, broken quartz particles of about 5 μm on the surface; platy formations of more than 50 μm in size were seen (Fig. 7e). Closer examination of the edges indicates that smaller particles are forming by the weathering of this plate. The X-ray emission spectrum indicates only Si on the plate and perhaps this was a face of a much larger quartz grain. The surface of a Wisconsin soil chert fragment (Fig. 7f) shows that it is composed of subhedral particles of few tenths of a micron in size.

A cavity in a chert body developed in Ordovician dolomite in Wisconsin Cary till is lined by prismatic and polyhedral quartz particles (Fig. 8a) 1–10 μm in size, similar to those found in the Danish chert (Fig. 5c), red bedded chert (Fig. 6b) and Florida chert (Fig. 7a). Several rhombohedral terminations of prismatic quartz particles are visible (Fig. 8b) among the many fractured grains about 1 μm in size.

As a representative of earlier geological time, Precambrian black chert (Fig. 8c) has a conchoidal fracture surface, being homogenous through the whole sample. Higher magnification reveals that the surface of particles is somewhat curved (Fig. 8d), similar to the novaculite-type surface (compare Folk and Weaver, 1952, their Plate 1, Fig. 2). Extreme compactness apparently prohibits the formation of euhedral quartz particles.

Quartz isolated from the Ordovician Prairie du Chien dolomite blocks occurs in extraordinarily large particles (Fig. 9a) mostly in the size range of 100–250 μm. Ninety five per cent of the acid residue was recovered in the quartz isolate indicating that is composed almost entirely of quartz. The particles at the center (Fig. 9a) show secondary enlargements or overgrowths (compare, Krinsley and Doornkamp, 1973, their Fig. 28) and have fine particles on them. The fine particles (Fig. 9b) are also microcrystalline quartz a few microns in size. The same micromorphology

(Fig. 9c), as well as small holes, occurs in clusters on the macrocrystalline quartz in the larger particle (upper left, Fig. 9a). The particle on the lower right (Fig. 9d) also shows both overgrowths and fine crystal clusters probably on precipitated silica. Authigenic overgrowths of quartz are common in sandstones, siltstones and shales as well as all types of carbonate rocks (Smithson, 1956; Siever, 1972).

The microcrystalline quartz on the macrocrystalline quartz with secondary enlargements may be considered as chert. Authigenic enlargements on quartz were said to increase the $\delta^{18}O$ value of the quartz as well as that of the whole sample (Silverman, 1951). We believe that overgrowths and chert clusters are the main cause of the higher $\delta^{18}O$ values of quartz in carbonate rocks and in silts of sediments into which the finer cherts are mixed.

Control chert chips (not acid treated) from the weathered section of the chert nodule (Fig. 10a) compare well with Fig. 4b or c, from the platy nodular chert (Fig. 10b) compare well with Fig. 6f, from a FL chert cavity (Fig. 10c) compare well with Fig. 7a, and from the Cary till, WI (Fig. 10d) compare well with Fig. 8b. These controls show that the quartz morphology of non-treated samples has been retained through the 6 N HCl preparation; the Fe-bearing (X-ray emission determined) spongy material in FL chert (Fig. 10c, arrows) was removed by the acid treatment.

CONCLUSIONS

(1) Chert occurs as silt-sized clusters or aggregates of microcrystalline quartz, euhedral or prismatic grains from 1 to 10 μm cemented together by subhedral grains of 0·1 to a few tenths of a micron dia.

(2) Such chert aggregates also occur in the coarse and fine silt fraction of soils. The silt fractions of soils also contain irregular, single-grained quartz particles which are presumed to be detrital from igneous and metamorphic sources, since intermediate $\delta^{18}O$ values of quartz are noted in silty sediments such as shales, alluvium, loess, and aerosols.

(3) Chert nodules from the Ordovician dolomite (WI) weather to a loose fabric of silt-size clusters or aggregates composed of microcrystalline subhedral quartz grains. Unweathered agate-like fractures show platy discontinuous surfaces resulting from splitting of the aggregates. Some areas of unweathered microcrystalline quartz are euhedral and prismatic, a few microns dia. Subhedral overgrowths occur on large (over 100 μm) quartz grains.

(4) Bedded chert has prismatic, polyhedral, interlocking quartz particles too densely packed to form euhedral quartz. In cavities there are euhedral quartz grains a few microns in size. Flints from chalk have spheriodal clusters of chalcedony fibers in calcite-lined cavities.

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