1) $a=7.0$ Å; $b=14.0$ Å. No conditions limit the presence of $hk0$ reflections, which implies the absence of an n glide plane normal to the c axis, and of screw axes parallel to a or b.
2) $a=14.0$ Å; $b=14.0$ Å. The condition limiting the presence of possible $hk0$ reflections, $h=2n$, implies an a glide plane normal to c.

If the weak spots belong to the $hk1$ reciprocal net, the cell has $a=7.0$ Å; $b=14.0$ Å. The limiting condition for the presence of possible $hk0$ reflections, $k=2n$, implies a b glide plane. For every set of conditions, several orthorhombic space groups are possible.

These few results point to the convenience of a more complete investigation of the minerals of this group by electron diffraction methods.

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LOW-ANGLE X-RAY DIFFRACTION MAXIMUM DUE TO TEXTURAL PERIODICITY IN HEATED GIBBSITE

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Oriented specimens of powdered gibbsite (from Minas Gerais, Brazil), <1 micron, were heated for two hours at 300°, 400° and 500°C and scanned on a conventional Philips X-ray diffractometer with CuKα radiation. At each temperature only one X-ray diffraction maximum occurred. The diffraction angle decreased slightly with increasing temperature. The maxima were reproducible. At 500°C the maximum was the most pronounced and was situated between 1.5 and 2°2θ, corresponding to about 50 Å. See Figure 1.

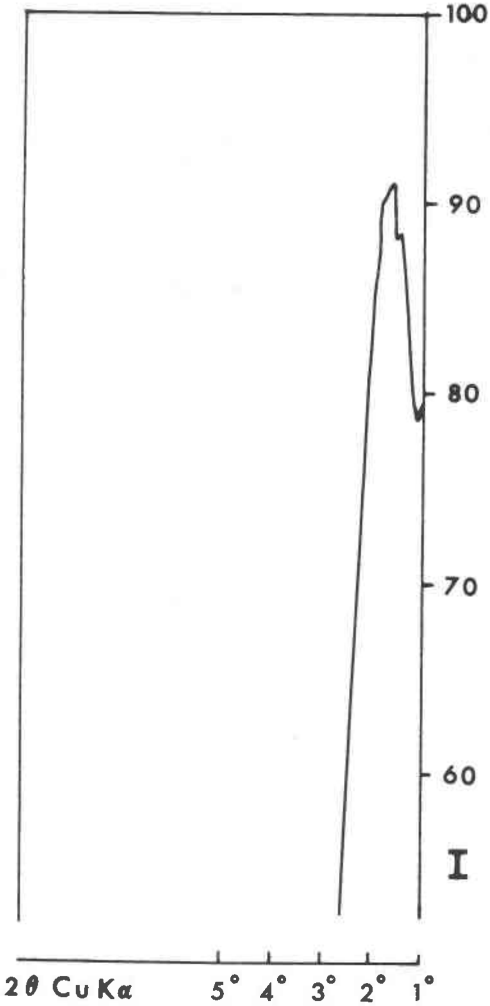

FIG. 1. X-ray diffraction maximum of oriented gibbsite, after heating for two hours at 500°C.

A similar and presumably the same "long spacing of the order of 30–90 Å" from progressively dehydrated gibbsite was found by Brindley and Nakahira (1959). They considered this spacing to be "additional evidence for structural order in nearly anhydrous alumina." One of us however developed an alternative hypothesis, that is, to assume the existence of an approximately regular pattern of small particles in the dehydration product which would give rise to the diffraction maximum.

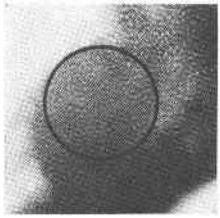

FIG. 2. Electron-micrograph of the heated gibbsite. Original magnification of this negative 52,000 ×, photographical enlargement 2×.

The second author investigated the heated gibbsite with a Philips E.M. 200 electron microscope. The electron-micrograph (Fig. 2) shows indeed rather regularly spaced electron-dense areas on an average mutual distance corresponding to about 50 Å in the heated material. A fragment is shown that lies over a fissure in the supporting carbon membrane. Confusion with the granular appearance of the carbon film caused by refraction contrast (see Van Dorsten and Premsela, 1966) is therefore excluded.

The average shortest distance of denser areas, determined from the micrograph is of the order of magnitude of the particle size observed by Weitbrecht and Fricke (1945) in γ -Al_2O_3 and of the pore-width in heated gibbsite as found by De Boer *et al.* (1954). We wished to test whether the regularity of the pattern observed in the electron-micrograph could give something similar to the low-angle maximum in the X-ray dif-

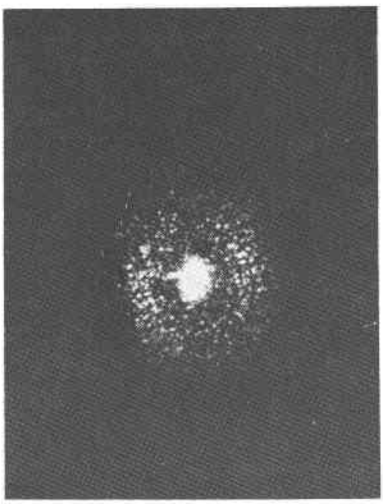

FIG. 3. Optical transform from the encircled part of the negative of Figure 2.

fraction pattern. In electron diffraction such a low-angle maximum cannot be resolved from the primary beam. Therefore Prof. C. H. MacGillavry suggested to use the negative of the electron-micrograph as a mask for the preparation of an optical transform. An optical diffractometer as described by Taylor and Lipson (1964, p. 30), was used. In fact, the pattern in the negative produced a broad optical diffraction maximum around the primary beam, Figure 3. The centre of this maximum corresponds to a distance of 0.3 mm in the mask or to a pseudoperiod of about 50 Å in the heated gibbsite material. Therefore it is concluded that the low-angle X-ray diffraction maximum is caused by the same textural periodicity. This explanation is expected to hold true for the maxima observed after heating at lower temperatures. The periodicity is probably brought about by concentration of mass after expulsion of structural water by heating.

Analogous X-ray diffraction maxima, caused by similar textural ordering might occur in diffraction patterns of heated minerals with structures similar to gibbsite, and also in patterns of heated natural clays, containing such minerals.

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SYNTHESIS OF THE GROSSULARITE-SPESSARTITE SERIES[1]

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Although a solubility gap has been thought to exist under natural conditions between the pyralspite and ugrandite groups of garnets, an interpretation stemming from the surveys of garnet analyses by Boeke (1914) and by A. N. and H. Winchell (1951), analyses have recently been sum-

[1] Mineralogical Contribution No. 450.