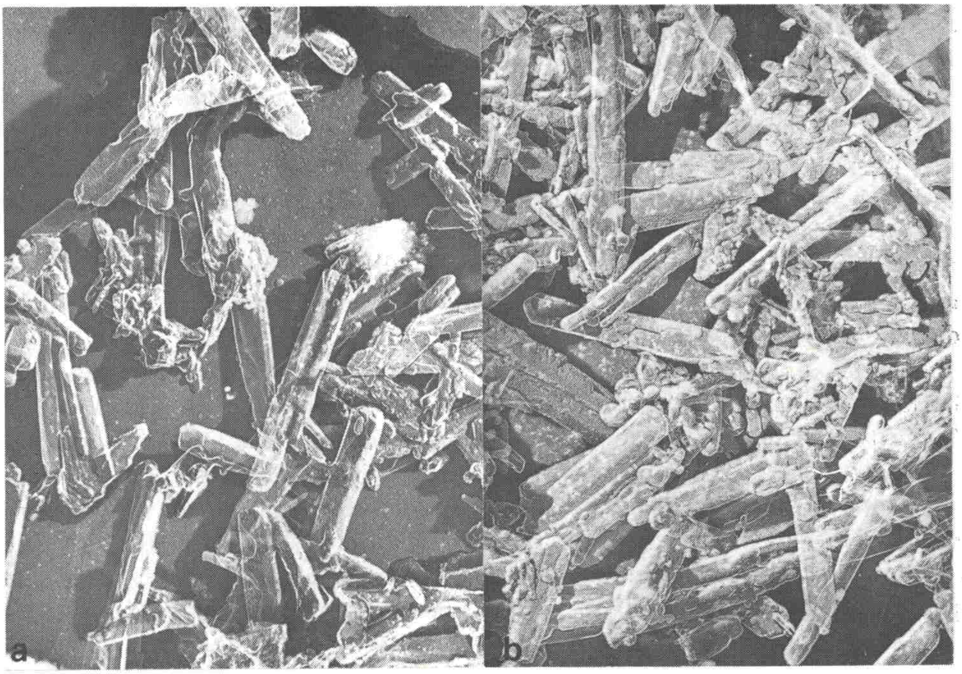

Fig. 14. Electron micrographs of products from γ-FeOOH (preparation 2). (a) In vacuo, 400 °C, 1 h: γ-Fe_2O_3 (30′000 ×). (b) Atmospheric pressure, 380 °C, 1 h: α-Fe_2O_3. Mind the beginning crystallite growth (30′000 ×).

ments must remain highly dubious. Decomposition of hydroxides, etc. yielding reaction water must be studied in vacuo to produce meaningful kinetics.

9.2 Texture of γ-Fe_2O_3. As mentioned before, the initial Bragg fringe contrast of the γ-FeOOH crystal vanishes with proceeding reaction. For interpretation of this phenomenon it is necessary to recall what the presence or absence of Bragg contours actually means.

In very thin crystals, the reciprocal lattice points will be elongated to spikes vertically to the platelet zone. As the Ewald sphere is, due to the very short wavelength of the electron beam, so large that a tangent plane may replace it, there will virtually always be a cut of this tangent plane through some reciprocal lattice spikes. In other words, the Bragg condition of diffraction is always fulfilled and a diffraction pattern will be produced by the entire crystal. The real crystal differs from this situation only in that it is, as a rule, slightly bent, which means that the Bragg condition is not equally well fulfilled throughout the crystal. Where it is fulfilled, the diffracted beam will be deflected at an angle prohibiting this beam to pass through the objective diaphragm. That is to say this part of the initial beam does not contribute to the final image and there will, thus, be a dark region in this image showing where the missing part of the beam (i.e., the diffracted beam of one particular Bragg condition, defined by the hkl of the according lattice plane family) originally came from. By shifting the objective diaphragm from the primary beam to the said reflection, a dark field picture in the light of this particular beam, or reflection, will be obtained. In this dark field picture the region which before, in the normal position of the objective diaphragm, has been dark against bright background, will show bright against dark background. By switching the image conditions to diffraction, the reflection can be identified. Hence, it is possible to attribute each dark zone in a picture of γ-FeOOH to a particular family of planes named hkl which originates there. Such regions are called Bragg fringes, or Bragg contours, or extinction contours, etc. In many cases it is possible, with the present state of the art, to make directly visible these lattice planes.

A closer investigation of partly decomposed γ-FeOOH crystals, now, reveals at higher magnification some surprising and highly characteristic features (Fig. 15). Some parts of the crystal exhibit the said Bragg fringes, while other parts do not. Along the needle axis another contrast phenomenon is visible, in the form of strings of aligned "particles" of the size of about 70 Å. These can only be the crystallites of γ-Fe_2O_3 which are forming in the γ-FeOOH matrix crystal. Where Bragg fringes occur, the γ-FeOOH is still coherently scattering, and where these γ-Fe_2O_3 crystallite strings intrude, a sharp contrast indicates the interface. By depicting a γ-FeOOH

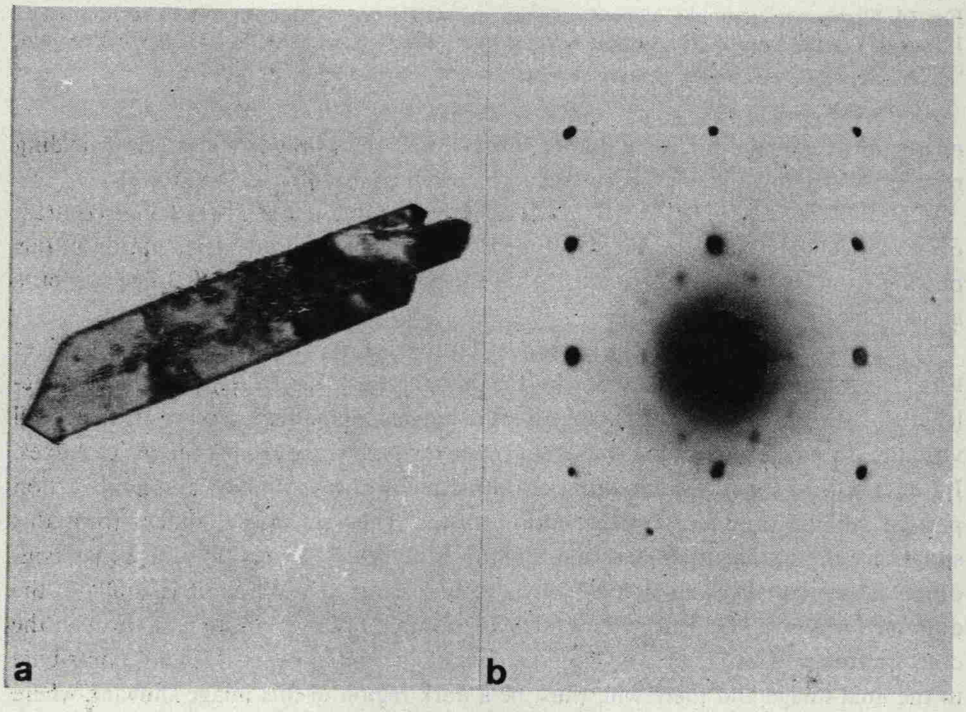

Fig. 15. (a) Partly decomposed crystal of γ-FeOOH (115 °C, 10^{-5} torr, $\alpha \approx 25\%$) (75′000 ×). (b) Selected area electron diffraction of the above crystal. This is a superposition of the γ-FeOOH pattern and the (weak) γ-Fe_2O_3 reflections.