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Crystalline Arrangement of Organic Molecules in Ammonium Urates as Determined by Electron Microscopy

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Uric acid is a heterocylic organic compound. Normally, health concerns are associated to uric acid precipitation i.e., crystals. Here a transmission electron microscopy (TEM) work is presented for a rather beam sensitive material. Additionally there is a need to completely characterize the molecular distribution in synthesized ammonium urate crystals (NH4U). Thus the main goals of this work are such a characterization together with the corresponding image analysis. Synthesis is described elsewhere, briefly the NH_4U crystals are prepared in an alkaline ammonium urate growth solution (pH 11), outside the range of physiological pH for the purpose of NH_4U structure determination. In basic media, NH_4U crystallizes as rods as shown in Fig 1a. Two types of crystals are characterized by TEM at the highest possible resolution, bent and straight crystals. The crystals are too sensitive to the beam and image exposure to the beam needs to be limited to around 10-15 s before clear structural variations can be appreciated. The TEAM 1.0 electron microscopy at the Molecular Foundry (LBNL) [1] is used at 80 KeV in low dose conditions i.e., the samples are observed with a dose of 10 $e^-/Å^2s$. Image series are used, they are taken at a fixed defocus condition. The stability of the samples can be preserved only for a low number of images, normally 10 and taken for 1 s. A visible contrast can be developed with atomic and molecular contrast after overlapping the aligned images. In all cases, the software MacTempass X is used. After an exposure longer than approximately 10 s, there is a noticeable sample damage. Thus beam sample interaction had to controlled in order to achieve the genuine structure of these organic crystalline arrangements.

The molecular structure of Amonium Urate crystals is here identified with transmission electron microscopy. Figure 1 shows typical results of these crystals. Fig. 1a shows a representative low magnification image with examples of straight and curved crystals. The magnification for different parts of the crystals is increased in the images shown in Figs. 1b to 1d. Clearly the molecular and atomic distribution is preserved and visible. Figs 1c and 1d can be further used to analyze the molecular arrangement in these organic crystals. All images are produced by overlapping 10 experimental images and adjusting contrast by means of the software MacTempass. Figure 2 shows several areas at different magnification of bent Amonium Urate crystals. Both types of crystals contain defects such as dislocations but they more easily seen in these bent crystals. Figures 1c and 1d are a direct magnification of Fig 1b. The circled area In Fig 1b has a dislocation (see arrow in Fig. 1c) that becomes more easily seen after a Wiener filter is applied (Fig. 1d). Figures 1e and 1f give further examples of the atomic distribution in these crystals with a variety of structures and defects. Analysis of the results will be proposed and discussed. The molecular arrangement of these Amonium Urate Crystals can be clearly determined from the achieved images for both straight and bent crystals. The use of low dose conditions is especially important for these samples.

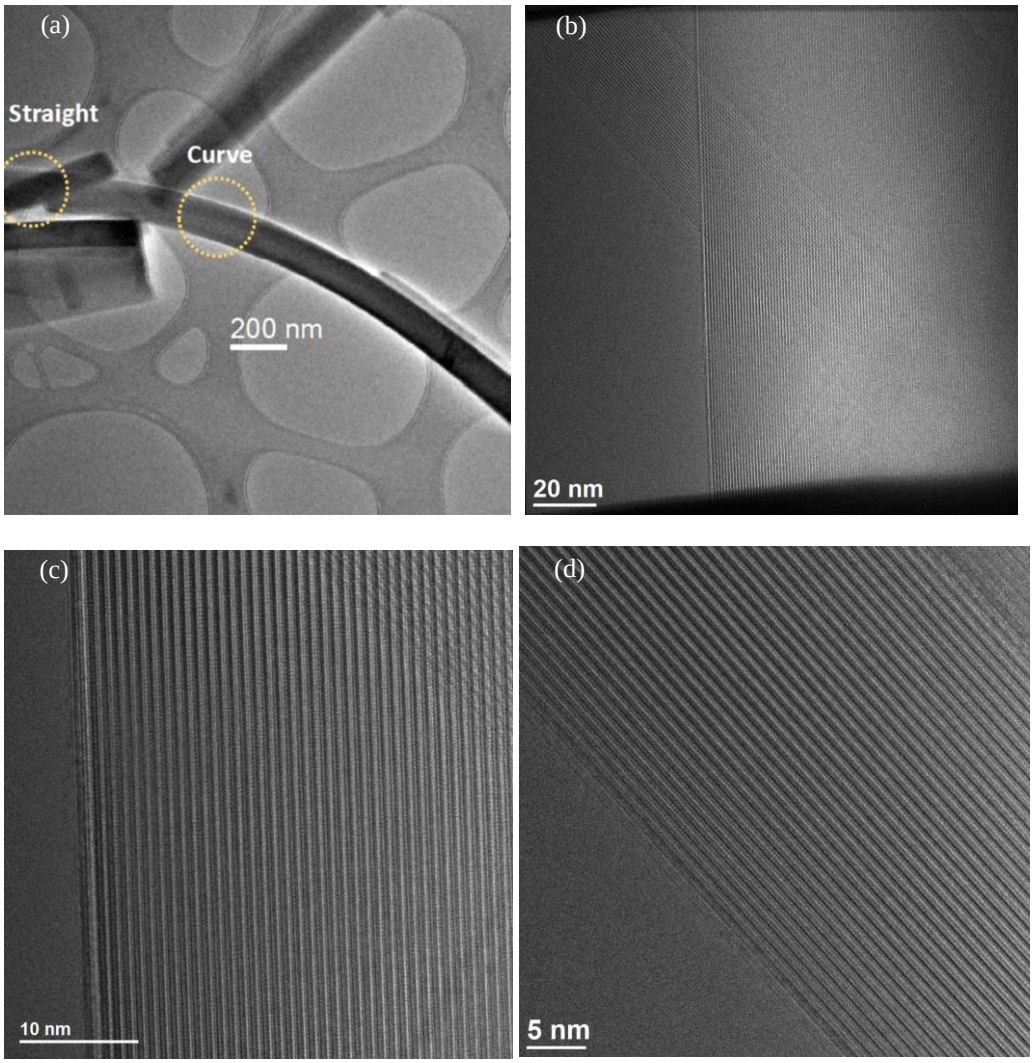

Figure 1. Amonium Urate crystals. (a) Typical low magnification bend and straight crystal on grid.(b) – (d) typical images of straight crystals at different levels of magnification. The ordered structure of the crystals can be seen.

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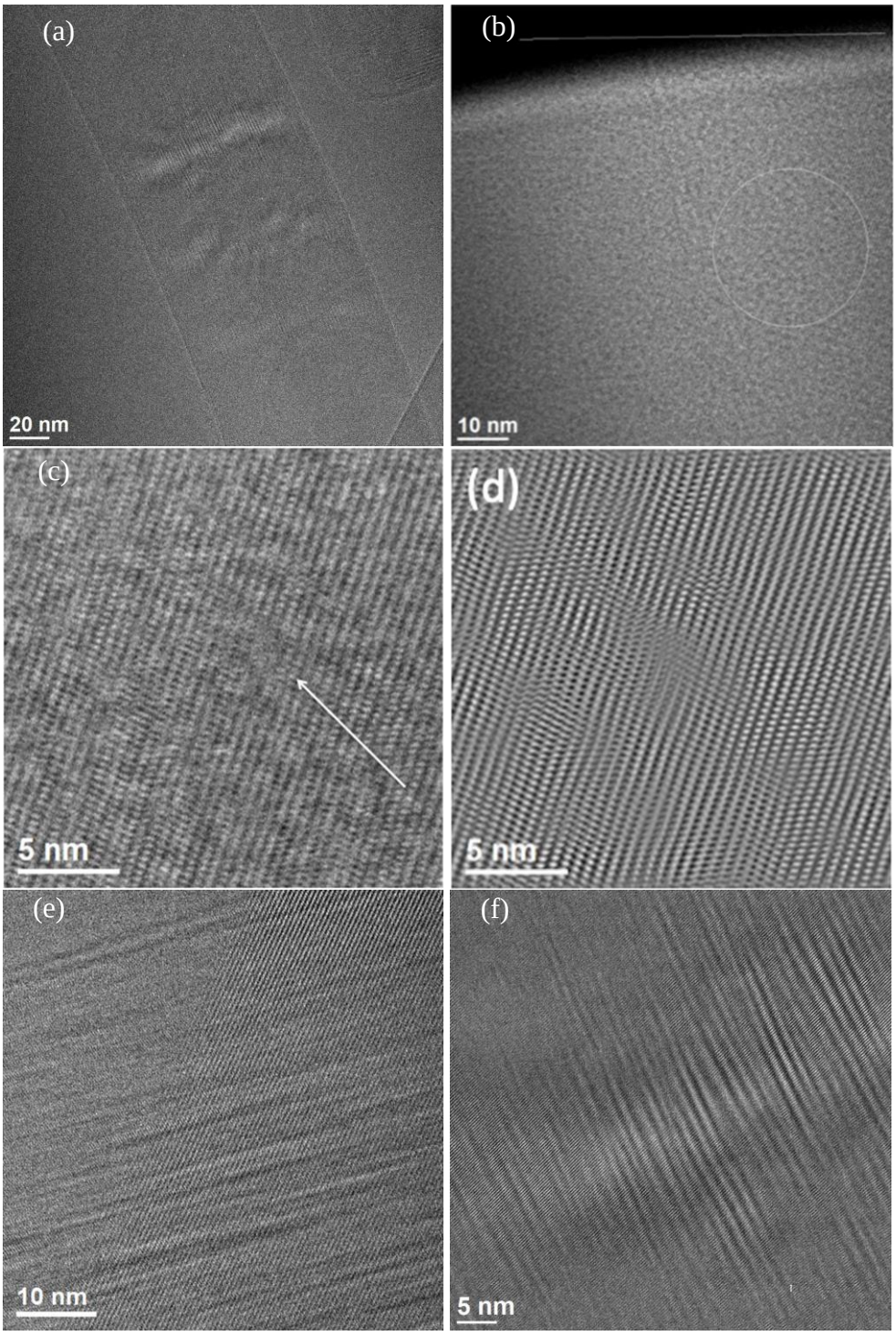

Figure 2. Images of bent Amonium Urate crystals in low dose conditions. (a) Crystal in low magnification but retaining resolution. (b) Area in a circle containing a dislocation that is better seen in (c) and the Wien filtered image in (d). (e-f) selected areas of a bent crystal.

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