

## Decoration of Cleavage Steps on $\text{CaF}_2$ Crystal Surfaces

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(Z. Naturforsch. 30 a, 394 [1975];  
received January 11, 1975)

Cleaved crystal surfaces of  $\text{CaF}_2$  were decorated by means of gold deposited on the crystal surface by vacuum evaporation. The influence of the substrate temperature on the decoration behaviour was studied.

Experiments were carried out in order to decorate cleavage surfaces of  $\text{CaF}_2$  for a study of crystal imperfection. The technique used was introduced by Basset<sup>1</sup>.

A small quantity of gold, chromium or platinum, which would give a film of mean thickness of less than  $10 \text{ \AA}$  if spread uniformly over the surface, is deposited on the crystal surface by vacuum evaporation ( $10^{-6}$  Torr). A film of carbon is then deposited on top of this layer. The carbon film is removed from the substrate carrying with it the deposit of the decoration material. With the amount of deposition material used no continuous film is formed but large numbers of small nuclei form on the crystal surface. Nuclei form in greater numbers along the edges of steps on the crystal surface which

are thus made visible in the electron microscope as chains of nuclei. This technique was proved to show very informative results when investigating NaCl-surfaces<sup>2</sup>.

Now it is – as far as we know – the first time that this technique was applied successfully to study other types of structures ( $\text{CaF}_2$ : face centered cubic, cleavage in 111 direction).

One of the main problems was the removing of the carbon film including the decoration material. Using chromium as decoration material – chromium is very mobile – it was impossible to remove the film, while, when using gold, the film could be removed from the substrate by means of a dilute (8%) solution of HF.

The influence of the substrate temperature on the decoration behaviour was also studied. Temperatures between  $300^\circ\text{C}$  and  $900^\circ\text{C}$  were applied. Substrate temperatures in the range of  $350^\circ\text{C}$  up to  $500^\circ\text{C}$  proved to be best suited for decoration purpose (Figure 1 \*\*). Also, in this temperature range epitaxial growth of the gold particles onto the surface could be seen (Fig. 2), while increasing the substrate temperature results in the disappearance of the epitaxial growth of the nuclei (Fig. 3) and in a certain ridge and valley surface macrostructure (see also Bezerianos et al.<sup>4</sup>) which makes it very complicated to detect the gold nuclei.

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\*\* Figures 1–3 on page 394 a.

<sup>1</sup> G. A. Basset, Phil. Mag. 8, 1042 [1958].

<sup>2</sup> V. Schmidt, H. Bethge, R. Schulz, and K. W. Keller, Third Europ. Reg. Conf. Electr. Micr. 1964, 263

<sup>3</sup> Y. Fukano and C. M. Wayman, J. Cryst. Growth 15, 32 [1972].

<sup>4</sup> N. Bezerianos and R. W. Vook, J. Appl. Phys. 43, 1416 [1972].

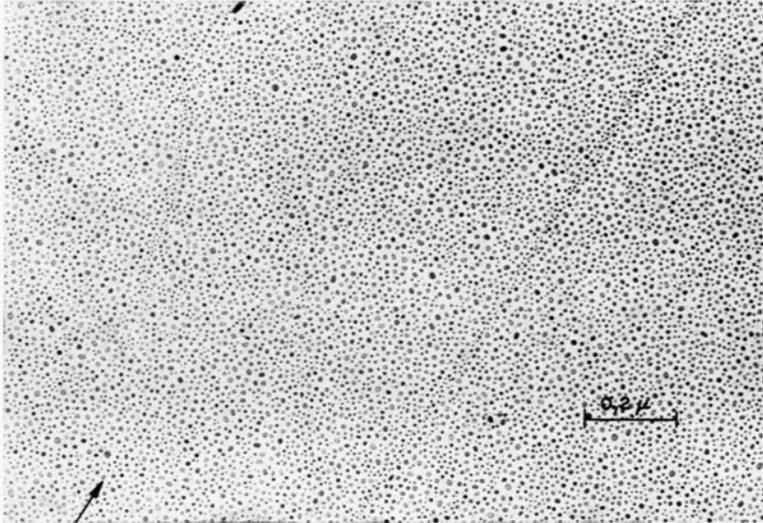


Fig. 1. Replica of cleaved CaF<sub>2</sub> surface decorated with gold. The cleavage direction (arrow) is along the (111) direction.

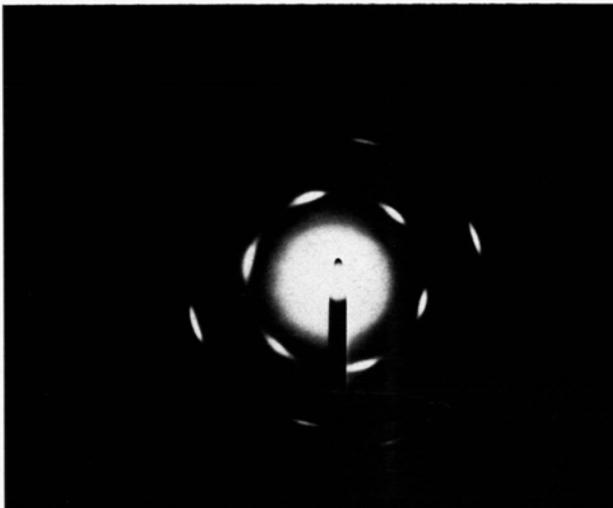


Fig. 2. Electron diffraction pattern taken from the specimen of Figure 1.



Fig. 3. Electron diffraction pattern taken from a specimen decorated at a substrate temperature of 900 °C.