that competition from recovery has reduced the driving force for boundary migration to the point where it is insufficient to overcome the pinning force exerted by the particles. Surface tension will therefore try to reduce the interfacial area generated by the different migration rates of the pinned and unpinned boundary segments. As the boundary straightens up it may be impeded by particles in its path and this in principle will explain the cusps at "T" being convex to the direction of general migration.

Fig. 3 shows part of a recrystallisation nucleus consuming neighbouring subgrains. This material was processed slightly differently in that after hot-rolling at 340° C it was cold-rolled 75% and then annealed for 1 h at 480°C. The component of misorientation across the boundaries between the growing nucleus and the recovered matrix is 15° in the plane of the foil.

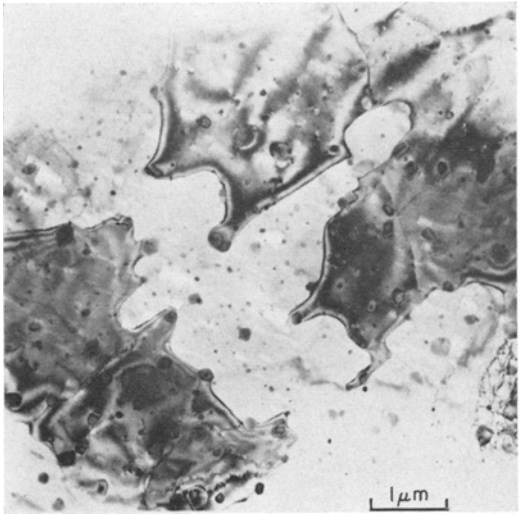

Figure 3 Recrystallisation nucleus consuming neighbouring subgrains and so becoming a new recrystallised grain. Al-Mn-Si hot-rolled 340°C, cold-rolled 75% and annealed 1 h at 480°C.

With the evidence of the previous micrographs it is postulated that the recrystallisation nucleus in fig. 3 originated by subgrain boundary-migration rather than by the subgrain coalescence mechanism of Hu [3] and Li [4].

Thin-foil annealing in the conventional 100 kV electron microscope is not representative of bulk annealing because only a section of a subgrain can be enclosed within the foil thickness and the free surfaces play a very important part in reducing boundary movement [5, 6]. However, high-voltage electron microscopy will enable annealing experiments to be performed in the microscope on aluminium foils [7] containing two or three subgrains within the foil thickness.

It is hoped that until such experiments are performed the observations reported here will help furnish a fuller description of the process of recrystallisation.

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Received 12 January
and accepted 15 March 1971

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A SEM Study of the Conversion of Clay to Type A Molecular Sieve

When the morphologies of a solid reagent and the final product differ markedly, changes can be followed qualitatively by scanning electron microscopy – for example, when metakaolin clay, a calcined amorphous alumino-silicate with a silica alumina ratio of about 2:1, is converted [1] to a crystalline alumino-silicate, type A molecular sieve [2], with the same approximate silica-alumina ratio.

The starting material (fig. 1) is composed of stacks of irregularly shaped platelets up to 7 μm across. One hour after crystallisation begins the mixture contains 0.2 μm particles and 0.5 to 3.0 μm cubes (fig. 2); at this stage the material is about 55% crystalline. After 4 h the material consists of agglomerated cubes (fig. 3) protruding from one another; the cube size has not

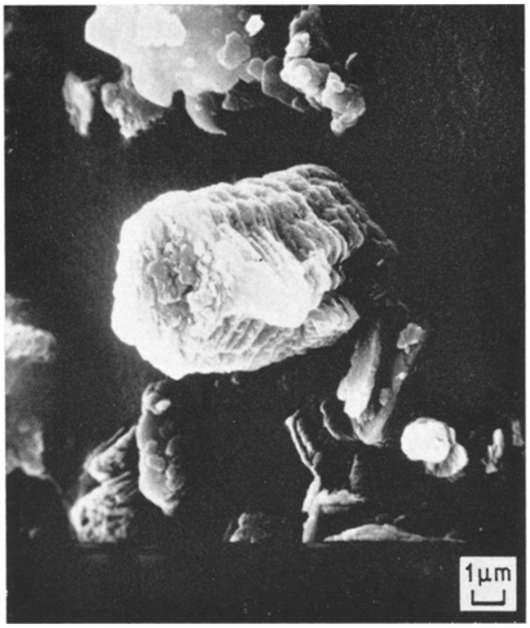

Figure 1 Metakaolin (× 5000).

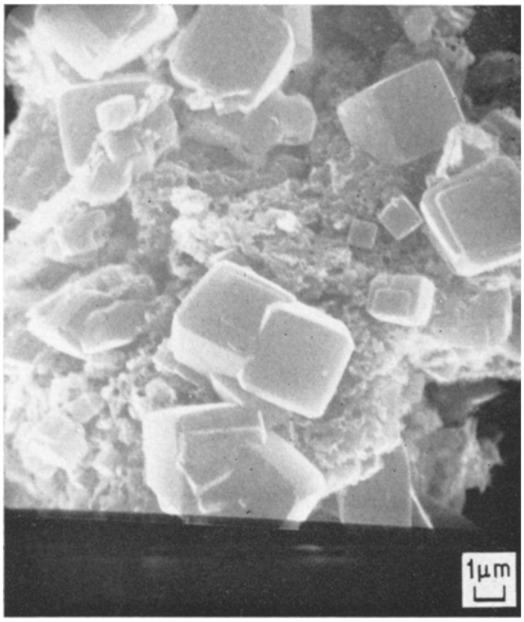

Figure 2 Solids – 1 h crystallisation (× 5000).

changed significantly. This material is only 80% crystalline, although no amorphous material can be seen (fig. 3). Table I summarises X-ray diffraction data. Extending the time past 4 h increases crystallinity without perceptibly changing particle morphology. Fig. 4 illustrates a preparation where metakaolin was not converted as completely. The crystallisation time was also 4 h, but a considerable amount of the granular intermediate remains. No improvement in appearance or crystallinity was noted for longer crystallisation periods.

TABLE I

Crystallisation time	Peak intensity index	%crystallinity
0	0	0
1 h	181	55.4
2 h	241	73.6
3 h	248	75.9
4 h	265	81.0
Standard	327	100

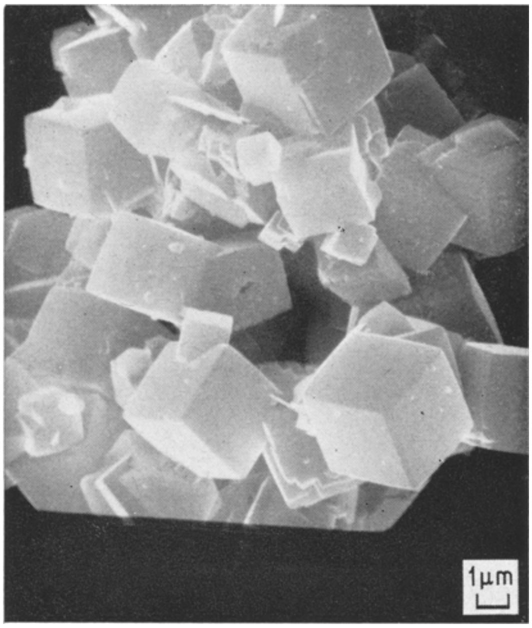

Figure 3 Solids – 4 h crystallisation (× 5000).

To prepare the sieves a Georgia (USA) kaolin was calcined at 700°C for 3 h to form metakaolin, the necessary intermediate. This was added to a 10% solution of NaOH at room temperature. The slurry was aged at 50°C for 18 h and then the temperature was raised to 100°C to begin crystallisation. Samples were removed from the slurry at 1 h intervals and washed, dried and activated. One portion of the samples was coated with 40 to 60 Pd-Au alloy to eliminate charging of the surface and scanning

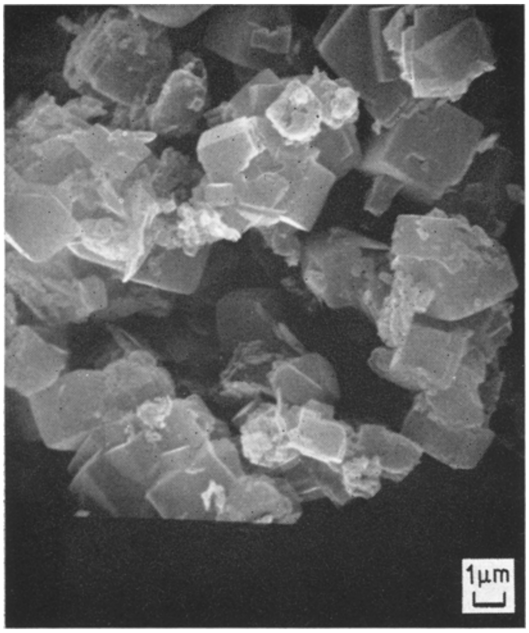

Figure 4 Solids – unsatisfactory conversion (× 5000).

electron micrographs were made, using a Cambridge Mk 2A Stereoscan. Another portion was analysed by X-ray diffraction to determine crystallinity.

Acknowledgements

The samples were prepared by Mr Ray Lambert and the micrographs were made by Mr Richard Roberts.

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Received 16 March
and accepted 26 March 1971

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The Annealing of ZnS Films on Silicon

Although epitaxial films of ZnS have been grown successfully by electron beam sublimation of the powder in UHV [1, 2] there are circumstances when it is not feasible to adjust growth conditions such that good quality single crystal films are produced. Consequently the possibility of using annealing to produce epitaxy or substantially to improve the crystal structure of films deposited under various conditions was examined.

Previous heat treatment work with films of II-VI compounds has mainly been concerned with recrystallisation and activation for luminescence purposes using various impurities [3]. However, it has been shown possible to produce epitaxy by periodic annealing of thin GaAs films deposited on GaAs [4]. In the work [4] on GaAs, deposition and examination were carried out in the same system, the films (up to 30 nm in thickness) were examined by glancing angle electron diffraction.

In this work films were deposited on to silicon substrates by electron beam sublimation in UHV [1], and by RF triode sputtering under ordinary vacuum conditions in a special "clean" sputtering system. The material used for sublimation was ZnS powder of the highest purity available commercially from various sources, mass spectrographic analysis showed that the purity was about 100 ppm. The sputtering target was an Eastman Kodak Irtran 2 disc and analysis of deposited films showed that the target must have a similar purity to the powders. Samples were examined after deposition using the high resolution diffraction stage of an AEI EM6G electron microscope by glancing angle electron diffraction. Annealing of the films was done in the microscope (a vacuum of 1 to 10 mNm^{-2}, 10^{-4} to 10^{-5} torr) by means of electron bombardment of the silicon substrate on the opposite face to that of the film. Thermocouple readings were calibrated so that surface temperature could be quoted to an accuracy of ± 10%. Diffraction photographs of the hot sample were taken after equilibrium had been reached, which took between 1 and 3 min, and the sample was then heated up to the next temperature. Longer annealing periods similar to those which Cho [4] has found to be necessary to produce complete structural transformations were tried. Samples were annealed for periods up to 15 min (after reaching equilibrium); generally no observable effects occurred provided the