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.

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.

References


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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
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 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>
<thead>
<tr>
<th>Crystallisation time</th>
<th>Peak intensity index</th>
<th>%crystallinity</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>1 h</td>
<td>181</td>
<td>55.4</td>
</tr>
<tr>
<td>2 h</td>
<td>241</td>
<td>73.6</td>
</tr>
<tr>
<td>3 h</td>
<td>248</td>
<td>75.9</td>
</tr>
<tr>
<td>4 h</td>
<td>265</td>
<td>81.0</td>
</tr>
<tr>
<td>Standard</td>
<td>327</td>
<td>100</td>
</tr>
</tbody>
</table>

Figure 1 Metakaolin (× 5000).

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

Figure 3 Solids – 4 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