A Note on the Formation of a Recrystallisation Nucleus

This note is intended to present evidence to show how an individual subgrain may grow during annealing by the migration of its boundaries, to become a viable recrystallisation nucleus. Cahn [1] and Cottrell [2] proposed such a mechanism many years ago and whilst the theory has never been called into question, neither has it gained wide acceptance because convincing experimental evidence is lacking.

Brief mention will be made only of the necessary experimental details since a more complete account of this work will be published later. An alloy of composition Al–0.5 wt % Mn–0.4 wt % Si–0.07 wt % Fe was chosen for an investigation into the inhibition of recrystallisation by particles. From measurements of electrical conductivity the ingot was judged to be strongly supersaturated and during hot-rolling at 340° C fine particles of αAl(Mn, Fe, Si) were nucleated. Recrystallisation did not occur during hot-rolling, but on subsequent annealing at 430° C the material rapidly polygonised. Further matrix decomposition took place by the growth of the particles, especially those associated with subgrain boundaries. The fine dispersion generated consisted of particles, approximately 500 Å in diameter spaced 1600 to 2000 Å apart, which were stable at temperatures well above the annealing temperature. Even after isothermal annealing at 430° C for 8 h, recrystallisation was only about 75% complete.

Fig. 1 is a transmission electron micrograph from a foil prepared from bulk material which was hot-rolled at 340° C, then annealed for 8 h at 430° C. The micrograph shows part of the low angle boundary between two subgrains which overlap within the thickness of the foil. Some segments of the boundary are in the process of growing in the direction of the arrows marked "F" but migration is being retarded by particles. Since boundaries move away from their centres of curvature during recrystallisation, the growth direction can be judged from the cusps in the boundary between the particles. Fig. 2 shows a more advanced stage of subgrain growth in a different area of the same material. Since most subgrains observed measured between 1.5 and 2.5 μm in diameter, it is fairly certain that the subgrain shown is an individual subgrain and not a coalesced group. Some segments of the boundary have migrated in the direction of the arrows marked "F" and where the boundary is pinned by particles, "P", it is bulging out and growing between them. From the splitting of the matrix spots on the selected area diffraction pattern, the component of misorientation across this boundary in the plane of the foil is only $3\frac{1}{2}$°. It is probable, however, that most of the misorientation is in a plane perpendicular to the foil.

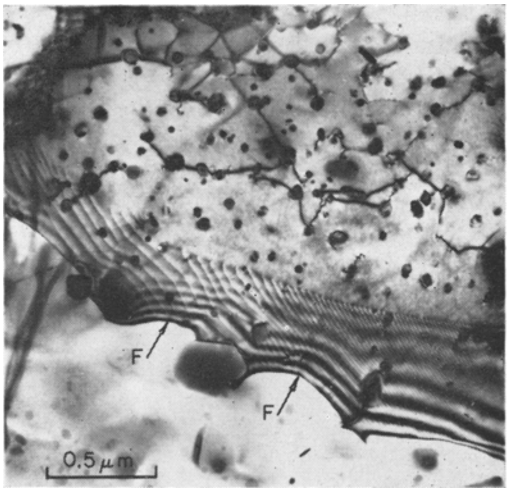

Figure 1 Initial stage in the growth of a low angle subgrain boundary. Al-Mn-Si hot-rolled 340° C and annealed 8 h at 430° C.

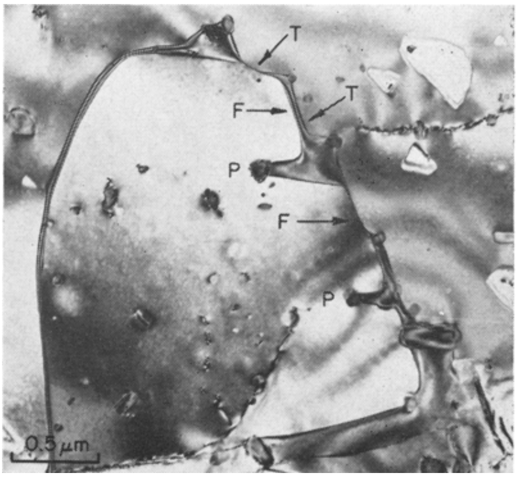

Figure 2 Subgrain in the process of becoming a recrystallisation nucleus. Its mobile boundary is pinned by particles. Al-Mn-Si hot-rolled 340° C and annealed 8 h at 430° C.

The cusps in the boundary marked "T", suggest movement contrary to the direction of boundary migration. One possible explanation is 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.

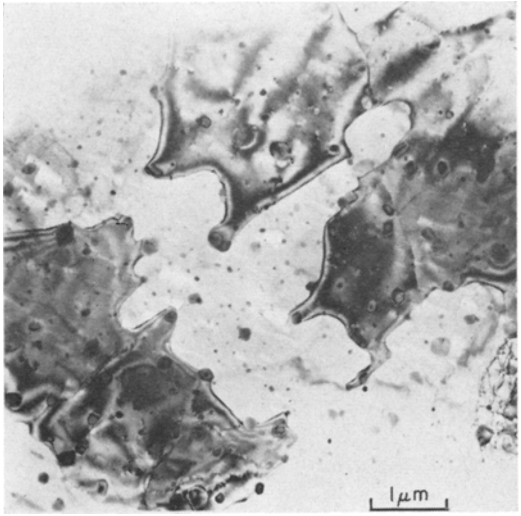

Figure 3 Recrystallisation nucleus consuming neighbouring subgrains and so becoming a new recrystallised grain. Al-Mn-Si hot-rolled 340°C, cold-rolled 75% and annealed 1 h at 480°C.

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.

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