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Chemical and Deformational Controls on Recrystallization of Mica

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Abstract. Chemical and microstructural data from one experimentally and two naturally deformed and recrystallized micas are discussed in terms of established metallurgical mechanisms for strain-induced nucleation and growth during recrystallization. It is suggested that none of these nucleation mechanisms is applicable, and that nucleation is driven by energy from both chemical and permanent strain sources. Subsequent growth of the nuclei is influenced by the deformation or by coincidence lattice relationships.

Introduction

Recrystallization—the nucleation and growth of grains of one phase within grains of different or like phases—is of great interest to both structural and metamorphic petrologists. The former tend to think of recrystallization being controlled by local strains and stresses or defects in the crystalline structure, without recourse to controls exerted by changes in chemical composition. The latter have traditionally adopted another extreme view and tend to think of recrystallization processes being controlled solely by mineralogical changes which move the system towards equilibrium under the prescribed conditions of temperature, pressure and chemical activities. Both views involve the formation of a discrete nucleus with minimum free energy, but neither includes all the possible energy sources for a deforming multi-component system. It has also been shown (Gibbs, 1961; Cahn, 1961) that nucleation may involve either a large rearrangement of material at a localised site or small rearrangements distributed over broad regions in the host phase. The first of these nucleation processes produces a discrete nucleus with minimum free energy, and is the type of event traditionally envisaged by geologists. The second process, however, normally requires more work than the first, but is favoured kinetically because it involves easier diffusion paths (Cahn, 1961). This second process is typically associated with small fluctuations in chemical composition, and although the products are metastable they may persist simply for kinetic reasons. The quasiperiodic fluctuations in composition with wavelengths from 400 to 1000 Å now recognised in the intermediate plagioclases (Nissen and Korekawa, 1969; Nissen *et al.*, 1967; McLaren, 1973; Lorimer and Champness, 1973) may be examples of this second kind of nucleation process.

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It is thus apparent that the simple situations envisaged by both structural and metamorphic geologists may not be applicable to the complexities of silicate systems in geological environments. In particular, a metastable phase may nucleate and grow for kinetic reasons, especially if it is favoured by strain energy considerations. Similarly, nucleation controlled by chemical energy requirements in a deformed crystal may produce preferred orientations unrelated to accepted nucleation mechanisms, especially if reaction rates are increased by the presence of easy diffusion paths.

This paper presents chemical, microstructural and fabric data from experimentally and naturally deformed and recrystallized mica. The initial aim of the project was to examine the role of strain-induced recrystallization in the development of mica preferred orientation. However, the accepted deformation controlled nucleation models are unsatisfactory, and slight differences in chemical fluctuations during the nucleation stage help promote the recrystallization process.

2. Experimentally Recrystallized Mica

a) Experimental Techniques. Cores 6.35 mm in diameter and 15 mm long were drilled parallel to (001) from large single crystals of phlogopite from the Strangways Range, Central Australia (Hudson and Wilson, 1966). The phlogopite is very faintly pleochroic (colourless to pale brown) in thin sections, and has a 2V of about 5°. The chemical composition is given in Table 1.

The specimen described in detail below was shortened 35% at 300°C[1] and 10 Kbars, and annealed for 10 min at 1150°C and 10 Kbars in a solid pressure medium apparatus of the type described by Griggs (1967). Ten other specimens were deformed and annealed under the following range of conditions: deforming pressures of 3 Kbars to 10 Kbars, deforming temperatures of 300°C and 800°C, annealing temperatures of 800°C to 1150°C, and annealing times of 5 min to 4 hrs. Despite this variation, all of the specimens showing any recrystalliza-

Table 1. Microprobe analyses of experimentally deformed and recrystallized phlogopite RH1

Element	Area 1	Area 2	Area 3	Area 4	Area 5
	Deformed		Recrystallized		
SiO_2	39.15	39.17	41.39	39.21	39.15
TiO_2	0.43	0.56	0.43	0.61	0.63
Al_2O_3	14.66	15.06	14.23	15.60	15.16
FeO	2.66	2.83	1.40	1.63	1.42
Fe_2O_3					
MnO	0.00	0.00	0.00	0.00	0.00
MgO	27.34	27.17	27.72	27.13	27.13
CaO	0.00	0.00	0.00	0.00	0.00
Na_2O	0.16	0.12	0.04	0.04	0.04
K_2O	10.62	10.86	11.06	11.24	10.96
H_2O[a]	4.31	4.31	4.31	4.31	4.31
Total	99.33	100.08	100.58	99.77	98.80

[a] H_2O was not determined, and the figures quoted are the theoretical weight percentages, assuming no fluorine is present.

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[1] All temperatures quoted from this apparatus are thermocouple temperatures corrected in a manner described by Carter and Ave-Lallement (1970).

tion contained the same features. No recrystallization was detected below 1050° C after four hours of annealing. Similarly prepared specimens of biotite and muscovite were also deformed and annealed but no recrystallization of either phase took place after several hours annealing just below their breakdown temperatures.

b) Microstructures. Because of the temperature gradient within each specimen (Carter and Ave'Lallement, 1970), it is possible to examine a range of microstructures in a single specimen varying from deformed mica at the ends to wholly recrystallized material in the centre. The microstructure of deformed phlogopite which has not been annealed is identical to that described for biotite by Etheridge, Hobbs and Paterson (1973).

There appear to be three stages in the recrystallization process in these experiments:

(i) Migration of kink boundaries to produce serrated shapes.
(ii) Nucleation and rapid growth of large new grains, often with (001) similarly oriented to that of the host.
(iii) Nucleation and growth of a further generation of new grains within these larger grains.

In addition, a number of less common features are attributable to annealing: the growth of small new grains within the old deformed material but with a different orientation, the optical "clearing" of small regions of the deformed crystal away from the hottest regions, and the growth of new grains from one kink band, having the approximate orientation of that band, into the adjacent kink band.

Serration of kink boundaries is the first visible evidence of annealing with increasing temperature. It is present in the hottest regions of specimens which display no other sign of annealing, and in the slightly cooler regions in specimens with extensive new recrystallization. Fig. 1a illustrates the early visible stages of this boundary migration, with small, isolated bulges occurring in the original plane. With increasing temperature and/or time these displacements or serrations increase in amplitude and spread along the boundaries. Eventually, a kink band may disappear completely in places along its length, being replaced by bleb-like regions (Fig. 1b). The migrated segments of a kink boundary are always bounded by the (001) plane of one of the two orientations on each side of the kink boundary and commonly are so bounded on both sides (Fig. 1a).

Evidence of new grain growth, from the bleb-like regions mentioned above, is occasionally seen. A fairly large grain may grow from a shrinking band into the adjacent one with an (001) orientation close to that of the former (Fig. 1c).

In the early stages of kink boundary migration, some boundaries become highly serrated while others remain quite straight. The angle of bending (ω) was measured across a selection of boundaries in such a region. These were translated into misorientations across the boundary by replacing ω by $180°—\omega$ if ω was greater than 90°. The misorientations were then plotted as frequency diagrams (Fig. 2), with separate diagrams for serrated, straight and total boundaries. Comparison of Fig. 2a, b and c indicates that boundaries with a misorientation of 70° to 80° are more mobile than others.

The central area of the specimen is extensively recrystallized (Fig. 3a). It consists of an interlocking mosaic of grains commonly bounded by (001) and up

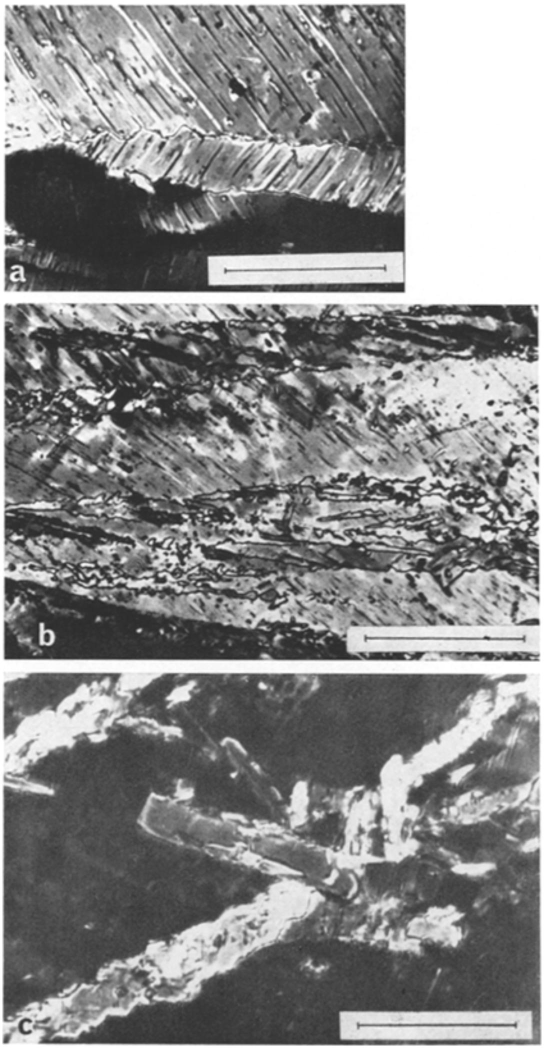

Fig. 1. a Small to large amplitude serrations in a pair of kink boundaries near the hottest part of RH14. Note planar boundaries of serrations parallel to (001). Crossed nicols parallel to plate edges. Scale bar is 80 μm. b Isolated remains of once continuous kink bands formed by exaggerated KBB migration. Some KBB pairs must have coalesced and cancelled each other out during this process. Crossed nicols parallel to plate edges. Scale bar is 200 μm. c Isolated patch of recrystallisation in region of KBB migration. The largest new grains have an (001) orientation close to that of the host on either side of the boundary. Crossed nicols at 45° to plate edges. Scale bar is 80 μm

to 0.4 × 0.08 mm in size. These grains are optically "clearer" than the deformed material, and always enclose smaller grains (Fig. 3a). Examination of the gradation from the deformed material towards the central recrystallized region reveals that many of the new grains originate with (001) parallel or almost parallel to that of the adjacent deformed material (Fig. 3b). In fact, residual kink boundaries can be traced through parts of the recrystallized material as serrated boundaries between two groups of mica grains, each having a dominant (001) orientation. However, some grains cut across these grain groups at moderate to high angles, without any apparent relationship to the deformed material. The interlocking mosaic of grains in the very hottest parts of the specimen includes narrow, wedge-shaped pockets of glass (Fig. 3a).

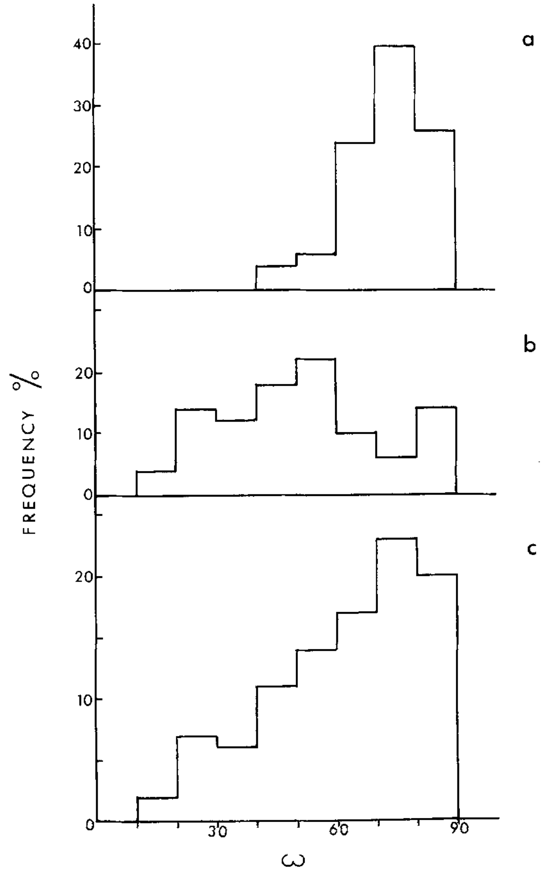

Fig. 2. Acute angle between (001) on either side of a selection of KBB's. (a) 100 serrated (mobile) boundaries; (b) 100 straight (immobile) boundaries; (c) 200 total boundaries showing overall distribution

Along the margins of some specimens adjacent to the furnace, and just away from the areas of extensive recrystallization, narrow (about 0.04 mm wide) strips of optically clearer material are common (Fig. 3c.) These strips are characterised by lower density of visible cleavage planes, slightly lower birefringence, than the adjacent material, and the presence of coarse new grains. In fact, these zones are identical in appearance to the large grains described above, except that they appear to be crystallographically continuous with the original grain.

Smaller new grains occur (1) within the larger new grains described above; (2) in the "clear" bands; (3) along some low angle kink boundaries; and (4) scattered throughout deformed crystal in areas adjacent to the hottest zones. There is an overall decrease in their average size in the order listed above, but the grains never exceed 0.04 mm in length. The larger ones tend to be rectangular in section and to have one well-developed pair of planar sides. Where the orientation of these small grains can be measured with certainty, they show no apparent relationship either to the host in which they grow or the overall deformation.

The small new grains along kink boundaries are generally restricted to boundaries with a low misorientation across them. They are commonly elongate parallel

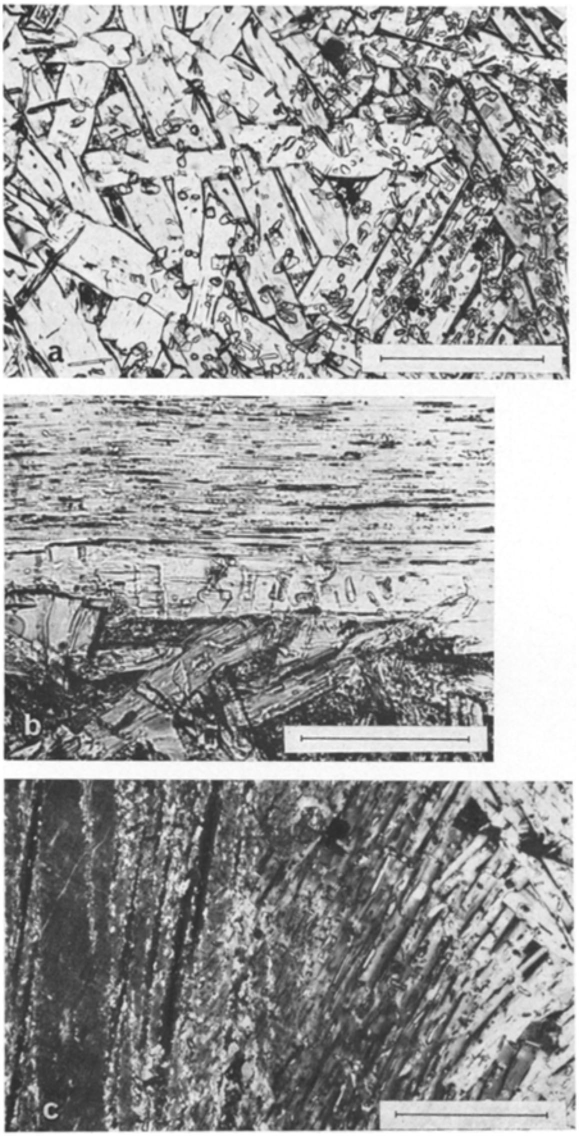

Fig. 3. Central, wholly recrystallised region near the centre of the specimen RH1. Most of the coarse grains are oriented NE-SW or NW-SE. The small equidimensional grains are also phlogopite, and the prismatic needles on the right hand side are forsterite. Plane polarised light. Scale bar is 200 μm. b Photomicrograph showing the gradation from deformed crystal displaying only KBB migration (on left) to completely recrystallised material (on extreme right). Note that many of the coarse new grains are subparallel to the host orientation. The specimen axis is horizontal, and the temperature increases from left to right. Crossed nicols parallel to plate edges. Scale bar is 500 μm. c Optically "clearer" strip along the margin of a specimen is outlined. It contains new grains up to 40 μm long, whereas the material above it is unrecrystallised. The coarsely recrystallised material below the cleared strip is the reaction rim between the specimen and the talc jacket. Plane polarised light. Scale bar is 80 μm

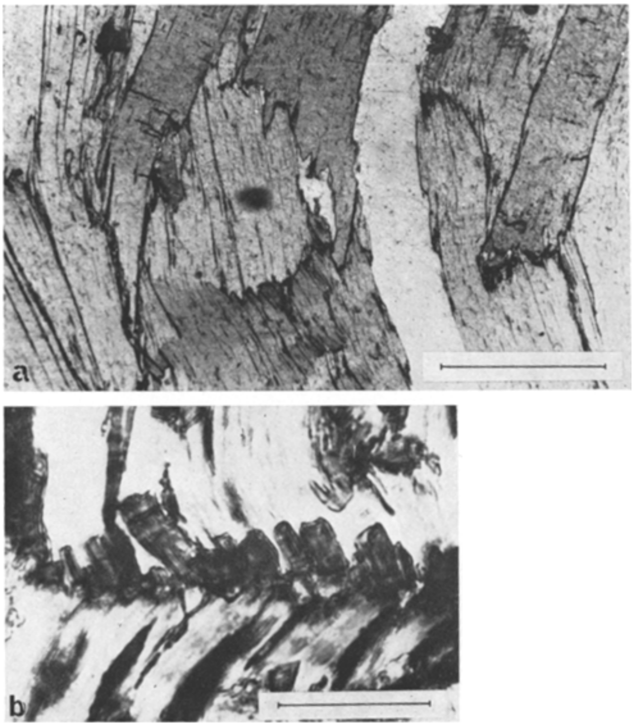

Fig. 4. a Extensive migration of a low angle KBB in the natural single crystal to produce new grains with (001) orientation between those of the host on either side. Plane polarised light. Scale bar is 500 μm. b New grains nucleated along a kink boundary in the recrystallised dacite. They all have (001) at 70° to 80° to that of the host into which they have grown. Plane polarised light. Scale bar is 60 μm

to the kink boundaries, but are too small for their crystallographic orientation to be determined optically. Very rarely, descrete small grains can be seen adjacent to high angle kink boundaries. These have an orientation intermediate between those of the matrix on either side of the boundary.

c) Chemical Composition of Phases. Electron microprobe major element analyses of five areas within a single specimen are presented in Table 1. Three of these areas are within the deformed host crystal (old grain), and three within new grains. Small but consistent changes in iron, sodium and potassium are associated with recrystallization.

3. Naturally Recrystallized Mica

a) Location and Geological Setting of Specimens. One specimen is a large single crystal of greenish-brown biotite which contains variously appressed kinks; extensive recrystallization has taken place along these kink boundaries. It was found in a private collection at the Australian National University, and its origin is unknown.

Other specimens were collected from a deformed dacite within the Goobragandra Formation, south of Tumut, New South Wales. The deformed rock contains

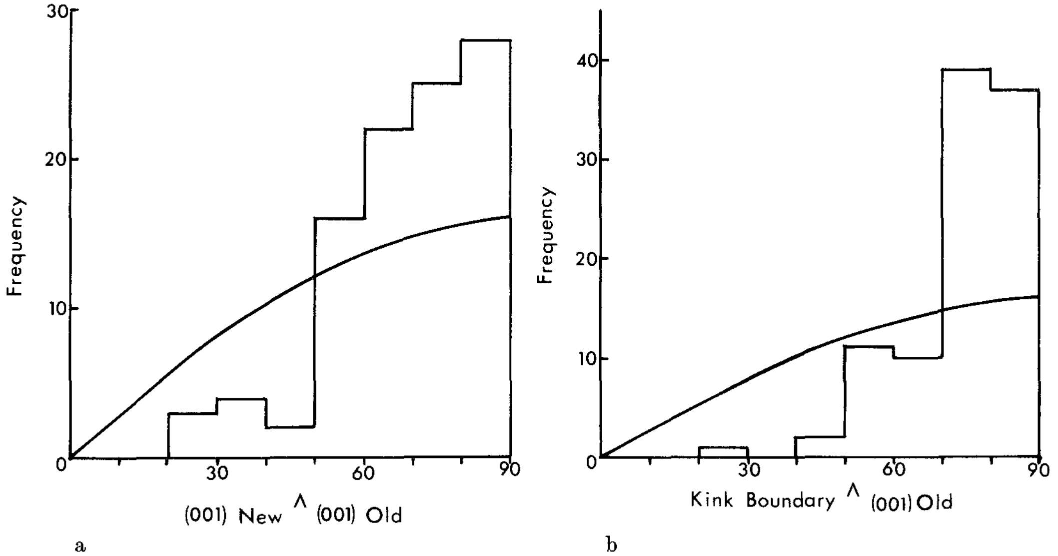

Fig. 5. a The distribution of the angle between (001) of 100 new grains and (001) of their host in the recrystallised dacite. b The distribution of the angle between 100 KBB's and the average (001) in each grain

kinked biotite phenocrysts which recrystallized within the aureole of the Bogong Granite (Ashley *et al.*, 1971), so that recrystallization has taken place by essentially hydrostatic annealing after deformation.

b) Microstructures. Three separate thin sections of the deformed single crystal were examined. Recrystallization is almost wholly restricted to the immediate area bounding high angle kink boundaries. Almost the whole area of sharp boundaries of this type is recrystallized, but where the kinks are broader regions of bending, recrystallization is much less common. Migration of the lower angle boundaries results eventually in the formation of a distinct euhedral grain with one end in common with the host and one end at a low angle to it (Fig. 4a). Along the high-angle, sharp kink boundaries, the recrystallized grains tend to be parallel to the boundaries. The shapes of the new grains vary somewhat with orientation, being longer and thinner (length in section parallel to (001)) where at a low angle to the kink boundary (*cf.* Jones and Galwey, 1972; Etheridge, 1973).

In the field, the transition from kinked mica phenocrysts in the dacite to completely recrystallized fine-grained mica takes place over 100 m of poor outcrop. Few specimens contain both old and new mica grains, and these contain the whole range from almost unchanged single crystals to completely recrystallized patches. The grain size of the new grains ranges from 0.003 × 0.006 mm to 0.05 × 0.1 mm. The distribution of 100 angles between (001) of old grains and new grains within them is shown in Fig. 5a, and their mean is 69°. For comparison, the distribution of the angles between 100 kink planes and undeformed (001) of their grains is given in Fig. 5b, and the mean of these is 75°. Very few kink planes remain visible in the recrystallized specimens,

Table 2. Microprobe analyses of naturally deformed and recrystallized biotite single crystal

Element	Area 1	Area 2	Area 3	Area 4	Area 5	Area 6
	Deformed			Recrystallized		
SiO_2	36.97	36.69	36.83	36.76	36.41	36.64
TiO_2	2.49	2.52	2.57	3.45	3.20	3.51
Al_2O_3	16.20	16.58	16.50	15.82	15.96	15.76
FeO	20.49	20.45	20.85	21.21	21.11	21.67
Fe_2O_3						
MgO	10.45	10.11	10.09	9.65	9.97	9.84
MnO	0.00	0.00	0.00	0.00	0.00	0.00
CaO	0.00	0.00	0.00	0.00	0.00	0.00
Na_2O	0.10	0.14	0.14	0.14	0.13	0.13
K_2O	10.07	10.09	9.92	9.97	10.01	9.71
H_2O[a]	3.75	3.75	3.75	3.75	3.75	3.75
Total	100.52	100.33	100.65	100.75	100.54	100.01

[a] H_2O was not determined, and the figures quoted are the theoretical weight percentages, assuming no fluorine is present.

but where they do, recrystallization is closely associated with them (Fig. 4b); however, they are not serrated. Recrystallization is uniformly distributed in most grains, without being controlled by major deformation features.

c) Chemical Composition of Phases. Electron microprobe analyses of new and old phases in the single crystal (Table 2) are internally consistent within each phase, and the differences in Fe, Ti, Al and Mg between new and old phases are small but consistent. The new grains on kink boundaries in the dacite were too small for reliable analysis.

4. Discussion

Most of the recrystallization microstructures described above are related to visible intracrystalline deformation, and others may be associated with lattice defects not now visible on this scale. These microstructures will thus be discussed first in terms of the recognised strain induced recrystallization mechanisms, mostly derived from metallurgical studies. The limited application of these single component studies to complex geological situation is evident, so that the effects of chemical energy contributions and kinetic factors will also be considered.

a) Nucleation. Two stages are recognised in the recrystallization process, nucleation and growth. The boundary between these stages is ill-defined; but nucleation may be considered as the formation of lower energy embryos, whereas growth is their expansion and mutual adjustment. It is still argued whether preferred orientations in recrystallized metals are due to a selection of the orientation of original nuclei, or random nucleation followed by selective growth (Beck and Hu, 1966). It has become apparent that both may play some part, and they are discussed separately here.

Three main nucleation processes have been proposed:

1. Random or homogeneous nucleation (Becker, 1938; Christian, 1965).

2. Preformed or subgrain nucleation (Hu, 1962; Li, 1962; Walter and Koch, 1962; Cahn, 1966).

3. Bulge nucleation (Bailey and Hirsch, 1962).

The first of these has been discounted as a viable mechanism for metals because the minimum size of stable nuclei (approx. 0.001 mm radius) and the activation energy for nucleation (Cahn, 1966) are too large to be the result of random thermal fluctuations. Assuming a cleavage surface energy for mica of 10^3 ergs/cm^2 (Gutshall, Bryant and Cole, 1970), a stored deformation energy of 1 cal/gm (*cf.*, Gross' (1965) value for calcite), and a flat, rectangular solid with sides in the ratio 10:10:1, the critical size for mica is about $0.01 \times 0.01 \times 0.001$ mm, again too large.

In the preformed nucleus models, subgrains form along high-angle deformation boundaries, and selected ones grow by absorbing their neighbours, increasing their misorientation with respect to the host as they proceed. Eventually they form nuclei surrounded by high-angle boundaries suitable for rapid growth. The kink boundaries in deformed mica are thought to approximate these deformation boundaries (Etheridge *et al.*, 1973) so this process may be applicable here. However, the dislocation substructures so far observed in micas (Amelynckx and Delavignette, 1962) favour neither the formation of subgrains, nor large rotations of subgrains during growth. Firstly, the dominance in mica of a single slip plane and only one or two slip directions appears to be unfavourable for the formation of subgrains. This follows from the need for dislocations of a number of orientations for the complete enclosure of a small, stable sub-grain (Jaoul *et al.*, 1957). Secondly, dislocations in layer silicates are generally split into partials (Silk and Barnes, 1960), which restricts dislocation climb, an important process in subgrain formation. Even if subgrains do form, rotation of them during coalescence and growth will be minimised by this restricted dislocation structure (Hu, 1962; Walter and Koch, 1962), and grains will tend to form with (001) at a moderate to high angle to kink boundaries. This is not the normal orientation observed in recrystallized mica.

Bulge nucleation (Bailey and Hirsch, 1962) is not dependent on the presence of preformed subgrains, but only of a preformed high-angle boundary. Bailey and Hirsch established that, with a difference in dislocation density on either side of a high-angle boundary, annealing causes the boundary to move towards the side with the higher dislocation density, absorbing dislocations as it goes. Local fluctuations in dislocation density causes bulges in the boundary leaving dislocation-free regions behind them. Eventually these regions reach a critical size and become nuclei for recrystallization. This type of nucleation is common in moderately deformed metals annealed at a temperature below that required for dislocation climb and subgrain formation (*cf.* Bailey, 1964, for an example), and is thus applicable to layer silicates, where such processes are apparently difficult. However, the recrystallizing nuclei must have an orientation close to a part of the deformed material, which is rarely observed in these examples. The serrated kink boundaries could be the result of this type of process, as could the rare new grains which appear to nucleate on these boundaries in the experimental specimens and the dacite.

Thus, few of the new grain orientations described in this paper can be explained by the established strain-induced nucleation mechanisms. It is suggested that stored plastic strain energy is generally insufficient to overcome the barriers to nucleation in layer-silicates. However, nucleation does take place along sites of deformation, which indicate that the strain energy contributes to the process, but another energy source is necessary. This energy source can be found in the changes in chemical composition which accompany recrystallization. It is possible to write an energy equation for nucleation as follows (*cf.* Christian, 1965, p. 418):

$$\Delta G = (g_\beta - g_\alpha + g_s) + \xi A - \sigma V$$

where ΔG is the overall free energy difference between host phase α and the new phase β; g_β and g_α are the chemical free energies of β and α respectively; g_s is the increase in elastic strain energy involved in the change from α to β; σ and ξ are the stored plastic strain energy and surface energy respectively; and A and V are the area and volume respectively of the nucleus of β. If ΔG is negative, the phase change is accompanied by a lowering of the overall free energy of the system and is thus enhanced. From a similar equation (neglecting stored plastic strain energies), Becker (1938) calculated the critical size for growth of a particular phase from a solid solution to be only about 100 atoms. The activation energy for the process is a very reasonable 500 cals/mole. It thus seems that if a phase becomes chemically unstable under certain conditions, the chemical driving forces for nucleation of a new stable phase add significantly to those resulting from stored strain energy. The inclusion of both in the equation above increases the tendency for nucleation. To carry out such a calculation for mica, one would need to know the activation energy for diffusion, to have some way of assessing the entropy changes, and to be able to consider the effects of elastically accommodating a nucleus of a particular volume, shape and orientation in the matrix. Without suitable data, such a calculation is impossible, but the work on metal alloys (Christian, 1965, p. 606ff.) shows that the mechanism is a thermodynamically viable one.

Spinodal decomposition (Burke, 1965, p. 118; Yund and McCallister, 1970) provides other possible loci for nucleation of discrete grains. It is apparently less thermodynamically favourable than the direct formation of a lower energy nucleus with a sharp boundary, but kinetic factors related to diffusion may make it feasible. Kink boundaries may be paths of easier diffusion related to such nucleation, especially in the natural single crystal.

b) Growth. The growth stage of the recrystallization process depends on the rate of movement of the high-angle boundaries of nuclei formed by any of the above processes. Boundaries with certain misorientations across them will migrate fastest (Aust and Rutter, 1959), although the exact reason for this is debated (Gordon and Vandemeer, 1966; Aust and Chalmers, 1970). The most mobile boundaries are those between two lattices which are so oriented as to share a common superlattice, especially when the boundary coincides with a high density plane of this superlattice. Such boundaries are called coincidence boundaries and various types are illustrated by Spry (1968, p. 21). Not all coincidence boundaries are mobile (e.g., twin boundaries), and there is some evidence that *near* as opposed to *strict* coincidence boundaries in fact move fastest (Gordon and Vande-

Table 3. Lattice coincidence relationships in phlogopite

Rotation axis	Polymorph type	Angle of rotation	Unit cell coincidence ratio
[010]	1M	42°	1:18
[010]	1M	73°	1:16
[100]	1M or 2M	40°	1:9
[100]	1M or 2M	70°	1:6
[010]	2M	$75^1/_2$°	1:8

meer, 1966). Nuclei which are in a coincidence relationship with the surrounding host grain will thus grow most rapidly, having a marked influence on the resulting preferred orientation.

An example of boundary mobility varying with lattice misorientation has been described above from the experimentally recrystallized mica (Fig. 2). To test this example against the coincidence model, unit cell networks projected normal to [010] and [310] (the axes of kinking in mica, *cf.* Etheridge *et al.*, 1973) were overlaid and rotated with respect to one another to identify common superlattices. Two relatively high coincidences were found at rotations of 42° and 73° and, of these, that at 73° has the highest density (see Table 3). The second of these is close to the mean of misorientation angles across the serrated (mobile) kink boundaries, suggesting that they behave in a similar fashion to high-angle grain boundaries.

There is also evidence of a growth control in both natural examples. In the dacite, much of the recrystallization is apparently unrelated to intracrystalline deformation, but there is a specific angular relationship between new and old grains (Fig. 5). The misorientation is again about 70°, and may relate to the [010] rotation above, or to a similar coincidence rotation of 70° about [100] which was found in the same manner. However, it is also consistent with new grains growing at low angles to kink boundaries, as is observed in the single crystal. The paucity of observed kinks in the dacite biotite may favour the former mechanism, but the extensive recrystallization may have obscured pre-existing low angle kinks.

5. Conclusions

(i) Nucleation and growth in deformed mica of mica grains with a composition different to that of the host is apparently promoted by free energy differences resulting from differences in both chemical composition and stored strain energy. In the biotite single crystal especially, nucleation is wholly restricted to zones of high strain, but cannot be explained by solely strain-induced processes.

(ii) If this combination of strain energy and the chemical energy derived from *small* chemical fluctuations is kinetically favoured in inducing recrystallization, the chemical composition of the new phase need not be that for equilibrium at the existing temperature, pressure and chemical activities. Such a situation is pertinent to interpretation of mineral chemistry in some deformed metamorphic rocks.

(iii) There is some evidence that coincidence lattice relationships between new and old grains in these specimens may determine grain growth rates and orientations. However, it is difficult to distinguish such effects from those causing mica to grow with (001) (the fastest growing plane) parallel to kink boundaries (regions of likely high diffusion and mismatch).

(iv) The role of recrystallisation in producing a preferred orientation of mica (001) is still unclear. However, the parallelism of new grain (001) to kink boundaries in the naturally deformed single crystal may be relevant. Such kink boundaries, and therefore new grain (001), form at high angles to the principal finite shortening direction, in agreement with the majority of data from foliated rocks. The reluctance of mica to recrystallise during the experiments, and the paucity of descriptions of partially recrystallised mica indicate, however, that it may not be a widespread mechanism.

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