

## CHEMISTRY

# ON THE NATURE OF RECRYSTALLIZATION NUCLEI AND THE ORIGIN OF RECRYSTALLIZATION TEXTURES

BY

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### *On the nature of recrystallization nuclei:*

1. Considerable thought has been given to the question of the nature of the nuclei of crystals growing in deformed metals by the process of recrystallization (see for a discussion of the literature up to 1940 BURGERS, 1941). Generally speaking, several authors, though differing perhaps in details of their conception, favour the view that nuclei are small lattice regions already present in the deformed matrix, which, for some reason or other, obtain the faculty to grow during heat treatment. The experimental fact that, in several cases, the lattice orientations of the crystals formed after recrystallization can also be found in the deformed test-piece, has been considered to support this conception (DEHLINGER, 1929; BURGERS, 1942, 1949). Based on the assumption that a lattice region could grow only at the expense of surrounding regions if it is more stable (less strained), it was assumed that those lattice blocks could serve as "potential nuclei" which, being originally in a strained state, on heat treatment suffered some stress-releasing process, which transformed them from "potential" into "actual" growth nuclei (VAN ARKEL, 1930; VAN LIEMPT, 1931). The necessity of such an activation may account for the occurrence of an incubation period before visible growth starts, as observed in recrystallization experiments. Considerations regarding the atomic character of the activation process have been given, as early as 1929 by DEHLINGER (1929, 1933) and, more recently, by BURGERS (1947; 1949). The latter, starting from the assumption that recrystallization was essentially a process of elimination of dislocations, advanced the idea that by a proper elimination of dislocations of "opposite sign" somewhere in the deformed matrix stresses could be reduced locally and so create a "remaining" stress, capable of displacing a dislocation layer between two adjoining domains, initiating crystal growth. It was thought that a lattice block such as *b* in the schematic figure 1, lying in the inflexion point of a S-curved region, was particularly favorably placed for such a local neutralization process, as it is separated from the neighbouring blocks *a* and *c* by dislocations of opposite sign. This conception of the activation process

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and of the most probable nuclear spot is much akin to DEHLINGER's conception of 1929. The "S"-curved regions were considered to be a direct consequence of the occurrence of local disturbances of the glide-process, which produced local rotations (so-called "local curvatures") of the active glide-lamellae about the normal to the glide-direction (see BURGERS, 1934).

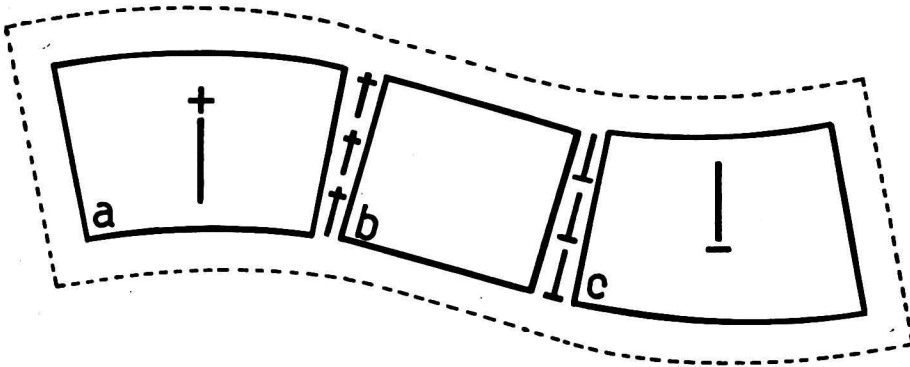


Fig. 1. Three adjoining lattice blocks, which, taken together, can be considered to form a "S-curved" lattice region. Block *b* in the "inflexion point" can presumably function as a nucleus for recrystallization.

2. The conception that an elimination of dislocations, involving a release of strain energy, may lie at the root of the process required to "activate" a potential nucleus to growth, was considered to be supported by the phenomenon of "stimulation" of crystal growth. As set forth in various papers (ref. in BURGERS, 1947), in recrystallized aluminium plates crystals may be found of a special "pointed" shape, the occurrence of which can be understood on the assumption that growth of such a "stimulated" crystal starts at the moment that an already growing "stimulating" crystal comes into contact with its nucleus. As it was found that such crystal pairs were mutually oriented as spinel twins (with a precision of less than a minute of arc: GUINIER and TENNEVIN, 1949; May, 1950), a practically perfect fit was possible between the growing crystal and the potential nucleus of the stimulated crystal. This led to the suggestion that the sudden elimination of dislocations produced when contact was established was the actual cause of the stimulating process and thus constituted a direct example of the growth-activation of a potential nucleus (that of the stimulated crystal), this time not brought about by thermal agitation as such, as in spontaneous nucleation, but in a kind of "artificial" way.

In the light of this phenomenon, a conception of spontaneous nucleation might be conceived, which is somewhat different from that advanced in paragraph 1. From a recent paper by SHOCKLEY and READ (1950) on dislocation models of crystal boundaries (supported by the experimental

work of DUNN and co-workers, 1949; 1950), it follows that the energy content of the boundary layer between two adjoining lattice regions with special mutual orientations is extremely sensitive for slight variations in the orientation of the boundary layer, in this sense that it increases at an infinite rate with deviations from a special position. If then, in a deformed test-piece, adjoining lattice regions happen to be present in such mutual positions, it seems reasonable to assume that on heating a release of strain energy by such slight displacements may occur at their boundary and so transform a potential nucleus into a growth nucleus.

There may be some relation between this conception and that brought forward by KRONBERG and WILSON (1949) in connection with their investigation of the growth of large crystals on prolonged annealing of fine-grained copper with cube-texture by "secondary recrystallization" (abnormal grain growth). The fact that, according to their experiments, this occurs only in twin-bearing material, leads KRONBERG and WILSON to the assumption that "nucleation" occurs preferentially at twin boundaries and is connected with stacking faults existing at such boundaries. Moreover, they point out that the orientation relationship existing between the new crystals and the primary texture (they are related by a rotation about either a [111]- or a [100]-axis over approximately definite angles) is such that the atoms in the (111)-resp. (100)-planes show definite coincidences or near-coincidences in both orientations, so that the atoms of one net can be brought into the sites of the new net by simple movements. If, reasoning along the lines set forth above, two such lattice regions were adjacent in the deformed state, it might be envisaged that such movements, bringing about better fit, were apt to give a stress release and to initiate growth.

3. A more defined conception of the nucleation process has been given in a paper by CAHN (1950). This paper, which starts from the assumption given above that the growth nuclei are actually formed in the most distorted parts of the lattice, i.e. in the "local curvatures", postulates (as is also done in a note by BECK, 1949) that the process which transforms the potential nuclei in growth nuclei, is essentially the recently much discussed process of "polygonization" (OROWAN, 1947; CAHN, 1949). This process, taking place in curved lattice regions, is considered to consist of a diffusion of dislocations parallel to the slip planes, thus producing a redistribution of dislocations causing a change of a continuously bent lattice into a number of polygon elements, each keeping the orientation of that part of the bent lattice from which it is formed but free of elastic strain <sup>1</sup>). (Cf. CAHN, 1950 p. 326). This is illustrated in a schematic way in

<sup>1</sup>) It was pointed out to us by Dr W. SHOCKLEY that also local redistributions of dislocations in a somewhat different way may involve a release of strain energy. For example under special conditions a displacement of two sets of dislocations along intersecting glide-planes can built up a boundary between lattice elements including a definite angle under release of strain.

figure 2, which is taken from CAHN's paper (1950). Figure 3, also taken from this paper, illustrates the effect of polygonization of a local curvature, representing a potential nucleus (PP in figure 3a) and suggests (figure 3b) that a strain-free element formed in this way is able to grow at the expense of the surrounding lattice. For details of CAHN's conception, in particular his assumption that the "incubation period" for

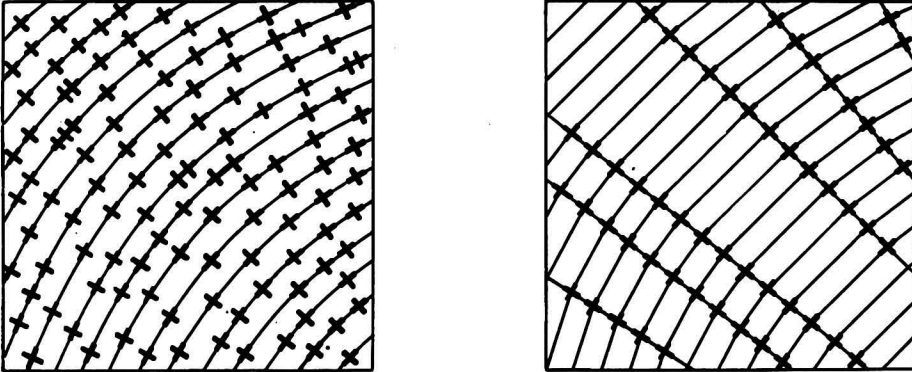


Fig. 2. Nature of polygonization in a bent crystal lattice: *a*) as bent; *b*) annealed. The crosses represent excess positive dislocations remaining on the glide-planes after bending. [After CAHN (1950)].

activation is inversely proportional to the radius of a local curvature, and the possibilities of his theory to explain a.o. ANDERSON and MEHL's observations on the kinetics of nucleation we must refer to his paper.

4. The conceptions of the process of nucleation, discussed in the foregoing paragraphs, are in our view supported by X-ray diffraction results recently obtained by TIEDEMA (1950). These results show that LAUE photographs of the "center" (the nuclear region) of aluminium crystals formed by recrystallization have a peculiar striated appearance, as if they are accompanied by satellite spots, these peculiarities being absent on photographs of parts of the crystal *outside* the nuclear spot region. This fact points to the presence, in the nuclear region, of lattice elements differing in orientation of the order of a degree of arc from the main body of the crystal, which have been left unconsumed by the growing nucleus.

As it is well established that a growing crystal cannot, or in any case only very reluctantly, consume lattice regions of approximately parallel orientation (TIEDEMA, MAY and BURGERS, 1949; LACOMBE and BERGHEZAN, 1949), the above result is in excellent agreement with the idea that a crystal grows from a lattice element, which forms part of a local curvature, as schematically shown in CAHN's paper (see figure 3), leaving unabsorbed some neighbouring elements of approximately the same orientation and growing at the expense of the deformed matrix outside the local curvature, which differs from it far more in lattice orientation.

Moreover, the non-focussed LAUE photographs taken by TIEDEMA

according to GUINIER and TENNEVIN's method (1949) show that *not one but a few elements* of the local curvature, with *slightly* deviating orientation (of the order of minutes of arc), may function simultaneously as actual growth nuclei, growing as it were side by side and producing a crystal consisting of as many parts with the same slight orientation differences <sup>2)</sup>.

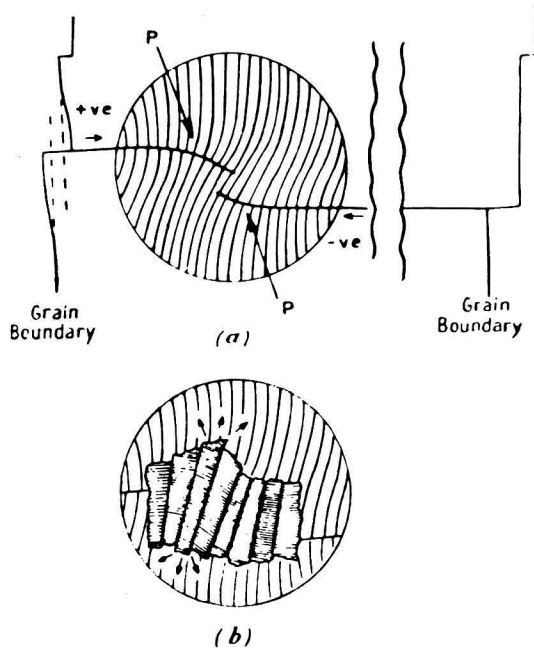


Fig. 3. Local curvature in a deformed crystal: *a*) as deformed; *b*) after annealing. [After CAHN (1950)].

Such a behaviour seems compatible with either of the two conceptions of nucleation discussed above, (1) by elimination or (2) by redistribution of dislocations. In fact, it seems a priori very well possible that in both cases more than one element of the original local curvature becomes sufficiently free of strain to obtain the faculty to grow. These, due to their approximately coinciding orientations, can grow at equal rate at the expense of the surrounding matrix, thus forming a straight "boundary" between them as observed in TIEDEMA's photographs, while leaving unabsorbed those elements of the original local curvature, which differ from them in orientation to a larger degree.

In this connection it seems of interest to remark that in rapidly heated samples KRONBERG and WILSON (see paragraph 2) often observed roughly

<sup>2)</sup> These regions of *macroscopic* size, dividing the final crystal in a few parts only, must not be confused with the very much smaller lattice regions (dimensions  $\sim 0.1$  mm) with still smaller (less than one minute of arc) orientation differences existing over the whole extension of the crystal and therefore inside each of the larger blocks, as discussed by LACOMBE and GUINIER and co-workers (see f.e. references in CHAUDRON, 1949).

elliptical grains containing a twin boundary near the center and along the major axis, the occurrence of which they take as an indication that in that case growth develops a twinned crystal as the first unit of growth, which continues to grow most rapidly parallel to the twin boundary. This may perhaps be compared with the occurrence of two or three side-by-side growing parts of slightly different orientation observed by TIEDEMA in aluminium crystals. We are, however, aware that this comparison is highly speculative. For instance Dr SHOCKLEY raised the question whether in our case such a process will give a sufficient release in strain energy (angle between two lattice elements about  $1/200$  radians corresponding to about 1 dislocation per 200 interatomic distances), and if not the origin of the crystal parts must be explained in quite a different way.

*On the origin of recrystallization textures:*

5. The nucleation theories discussed in the foregoing paragraphs are denoted by BECK and co-workers (1949, 1950) as "oriented nucleation", in so far they imply the supposition that the occurrence of potential nuclei in definite orientations, as refound later after completed recrystallization in the orientation of the new crystals (recrystallization texture), is a direct consequence of the foregoing deformation process. To state an extreme case: BURGERS and LOUWERSE (1931), when recrystallizing aluminium single crystal discs, which had been subjected to homogeneous compression between flat discs, thus causing almost pure shear parallel to definite glide-combinations (glide-plane (111), glide-direction in this plane [110]), found crystal orientations, which could be deduced from those of the deformed crystals by a rotation about the normal to the glide-direction (a [112]-direction).

The conception of "oriented nucleation" has been disputed by BECK c.s. in favour of a theory of "oriented growth". These authors, in an interesting series of papers (1949, 1950) found that in aluminium a pronounced orientation relationship exists between grains growing in a matrix with a strong single orientation texture and the matrix itself, namely a rotation of  $30-40^\circ$  around a [111]-axis. This relationship exists as well when the matrix is a cold worked single crystal as in coarsening when the matrix is an annealed primarily recrystallized fine-grained material with a pronounced preferential orientation. Similar orientation relationships have been found (in some cases together with other ones) in other cubic face-centred metals: in copper by BOWLES and BOAS (1948) and by KRONBERG and WILSON (1949) (see paragraph 2), and in nickel-iron alloys by RATHENAU and CUSTERS (1949).

The occurrence of the same relationship in all these cases is considered by BECK c.s. as an indication that grains with definite orientations with respect to the matrix grow much faster than others. Taking this as starting point, and assuming that in a deformed matrix, and also in a recrystallized material, even when a strong texture is present, there are always some

lattice elements in practically any orientation <sup>3)</sup>, it is supposed that only those domains can serve as actual growth nuclei, which are favorably oriented with regard to the matrix for their growth. The resulting texture would thus be caused by "selective growth" and not by "selective nucleation" of domains in special positions only. A similar view was tentatively advanced by BARRETT (1940), and discussed by DUNN (1948).

6. When comparing these two conceivable theories (see also DUNN, 1948), it must be said first of all that also in the opinion of the present authors it is certain that a selectivity of the growth process exists, in that sense that the ease with which a crystal can grow in a matrix depends very much on the mutual orientation of growing and disappearing lattice domain. This was already evident from early researches by VAN ARKEL (1932) on "secondary recrystallization" of aluminium, in which it was observed that a large crystal, growing at the expense of a fine-grained matrix, often stops its growth on attaining a part of the matrix, which had a texture different from that present on the part where the crystal was growing. Also experiments on crystal growth in locally deformed single crystals of aluminium (by scratching: BECK, SPERRY and HSUN HU, 1950) or in pseudo-unicrystalline nickel-iron foil (by pinprick: RATHENAU and CUSTERS, 1949) show that the largest crystals developed occupy special positions with regard to the matrix texture. Finally the non-consumability by a growing crystal of lattice domains in approximately identical or twin position (TIEDEMA, MAY and BURGERS, 1949; BURGERS and DALITZ, 1949; LACOMBE and BERGHEZAN, 1949) is an exponent of this phenomenon (cf. also BOWLES and BOAS, 1948).

A quantitative measure of the variation of rate of growth with orientation of large crystals growing in a given texture can be deduced from experiments by DUNN (1948) with silicon iron. A variation up to about 30 % was observed. From these investigations it may be concluded that a high rate of growth is connected with a considerable difference in orientation between growing and disappearing lattice domains. The question remains, however, whether the selectivity is itself sufficiently pronounced to explain the observed orientations after recrystallization. In considering this question it seems to us important to take into account the following experimental facts:

*a.* A consideration of the results obtained with aluminium by BECK c.s., according to which a 30–40° rotation about an [111]-axis between growing grain and matrix is particularly favorable for growth, shows that deviations from such positions up to at least 15° in some direction are present. This is a considerable amount in a lattice with cubic symmetry

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<sup>3)</sup> There is no doubt that this is certainly true in most cases (cf., however, paragraph 6, under (b)), as X-ray photographs apart from the interference spots due to the preferred orientation, practically always show at least some intensity along the Debye-Scherrer rings outside the intense reflection region due to the texture.

and means that crystals with a quite different crystallographic orientation with regard to the matrix can also grow with a comparable rate <sup>4)</sup>.

That this is actually true can be demonstrated in a direct way by forcing a crystal with a prechosen orientation to grow in a matrix with a pronounced texture, using the method of "growing round the corner", as realized by TIEDEMA (1949) with aluminium and by DUNN (1949) with silicon-iron. For aluminium, such experiments (TIEDEMA, unpublished results) show that in a matrix, with a sharp texture, obtained by stretching a single crystal, apart from crystals deviating from this texture by a rotation about [111] as considered by BECK, also crystals related to the matrix by a rotation about for example an [110]-axis can grow over large distances. This agrees to some extent with the fact that in some cases (for example in RATHENAU and CUSTERS' work on nickel-iron and in that of

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<sup>4)</sup> The above can be illustrated in another way. As set forth in paragraph 5, the recrystallization texture of homogeneously compressed aluminium single crystals was interpreted by BURGERS and LOUWERSE on the basis of oriented nucleation in lattice regions rotated about an axis [112], perpendicular to the glide-direction [110]. In the original paper it is shown that the observed orientations could approximately be ascribed to rotations around the perpendiculars to the various active glide-directions. BECK and HSUN HU (1949), in an effort to fit these results into the theory of selective growth, show decidedly that the same orientations, at least the prominent groups, can also be described, perhaps even somewhat better, by a rotation around [111]-direction. \*) As far as this statement goes, the argument may be taken as an example that also in this case the scattering of the observed orientations is so considerable that they can approximately be described by rotations about different sets of axes. One may say also that a description of a scattered texture by a rotation about a definite axis or set of axes has in itself not much value. It derives this value from the interpretation: either (BURGERS and LOUWERSE) on the basis of "oriented nucleation" in local curvatures, or (BECK and HSUN HU) in terms of growth selectivity.

\*) In this connection it seems of interest to remark that, as set forth by KOCHENDORFER (1950) (cf. also BILBY, 1950), shear along a [111]-plane may be produced as well by propagation in a direction parallel to the glide-direction of a "Taylor" or "line" dislocation, as by a sideways displacement of a screw-dislocation. Whereas the holding-up of line-dislocations gives rise to local curvatures about the normal to the glide-direction [112], considered in paragraph 5, the holding-up of screw-dislocations will presumably cause local lattice rotations about the normal to the glide-plane [111] (cf. J. M. BURGERS, 1940). Only the first type of curvatures was considered at the time of the BURGERS and LOUWERSE 1931-paper. If gliding in these experiments was also produced by the second mechanism, rotations about the [111]-axis normal to the active glide-planes might be expected. They were apparently observed by HEIDENREICH and SHOCKLEY (1948) (cf. also FRANK, 1948). If such local rotations actually existed, the presence of [111]-related deformation and recrystallization textures could even be expected on the ground of an oriented nucleation theory.

It must be mentioned, however, that the [111]-axes applied by BECK and HSUN HU (1950) (cf. also BARRETT, 1940) to explain the recrystallization textures in compressed aluminium single crystals, were *not* the [111]-axes perpendicular to the prominent glide-planes.



KRONBERG and WILSON on copper), besides the "[111]"-rotated crystals, occasionally also large crystals with other orientations with respect to the matrix, grew by "secondary recrystallization".

b. A second point to be considered is the following: a deformation process apparently not always produces growth nuclei in every possible orientation, from which the recrystallizing matrix may "chose" those best fitted to grow. This is clear from the following (unpublished) experiments: a drawn and annealed aluminium wire, on prolonged heating, often shows the formation of large crystals with a [210]-direction parallel to the wire axis (BURGERS and SANDEE, 1942). This fact itself appears to fit in BECK's et al. selective growth theory, as the [210]-orientation can be deduced from the [111]-texture by a  $40^\circ$ -rotation about one of the [111]-axes, which is not parallel to the wire axis.

If, however, a *single* crystal wire, with a [111]-direction parallel to the wire axis, is extended circa 8 %, and then subjected to prolonged annealing, then among the large crystals developed by "secondary recrystallization", the formation of a [210]-crystal was in no case observed. Yet, by the method of "growth round the corner", it was found that such a crystal, if presented to the matrix, could consume the deformed [111]-crystal readily. This can be interpreted that in this case 8 % extension of the single crystal did not produce potential nuclei in the [210]-orientation.

An analogous conclusion can be drawn from the experiments of RATHE-NAU and CUSTERS with nickel-iron, mentioned in paragraph 5. Although in their experiments with locally deformed sharp-texture matrixes, the largest crystals developed possessed the same orientation with respect to the matrix as those formed "spontaneously" on prolonged annealing, a fact apparently pointing to a dominant influence of growth selectivity in choosing from the available nuclei, yet it must be mentioned that the local deformation after subsequent annealing not always gave rise to the growth of large crystals (loc. cit. fig. 5): this again might be interpreted in the sense that the local deformation (pin-prick) had not produced growth nuclei in all possible orientations, so that the matrix could not "chose" the right one.

c. Thirdly, there is the phenomenon of "stimulated crystal growth", discussed in paragraph 2. Here we find an example of two crystals, growing both at the cost of the same fine-grained matrix, of which the one with the faster rate of growth, viz. the "stimulated" crystal, starts to grow at a *later* moment than the one with the slower rate of growth, viz. the "stimulating" crystal. Therefore, notwithstanding its faster rate, which undoubtedly means that its orientation with respect to the matrix texture is more favorable for consuming this texture, the "stimulated" crystal would perhaps not have developed at all, if not, according to our view, the *establishing of contact* with the approaching "stimulating" crystal had "activated" its "potential" growth nucleus to a centre actually capable to grow.

7. The points raised in the foregoing section make us ask whether the theory of "selective growth" is capable of explaining the observed recrystallization textures without taking into account the part played by the preceding deformation process in producing specially oriented lattice elements, which we may call "potential nuclei", which have to undergo some "activation process" before they can start to grow, their growth then being subjected to the laws of "growth selectivity".

To state our point more precisely, we think that for growth of a crystal a combination of factors is required:

- 1) there must be, in the deformed matrix, a lattice region (lattice element) in the orientation, later found again in the resulting crystal;
- 2) in order to be able to grow, this element must be essentially "strain-free" and in contact with strained (c.q. higher strained) lattice regions;
- 3) the "strain-free" state is brought about by some activation process, which probably consists of a proper redistribution of dislocations (as in "polygonization") or perhaps of a mutual dissolution of dislocations at the boundary of two adjacent lattice elements, which transforms a "potential" growth nucleus into an "actual" growth nucleus;
- 4) the capacity of actual growth nuclei to grow at the expense of the surrounding matrix depends on their lattice orientation with respect to the matrix texture ("growth selectivity factor").

Of these four points, 1) and 2) are probably widely accepted and considered inherent to both the "oriented nucleation" and the "selective growth" theories. With regard to 3), however, the conceptions differ, apparently not so much as to the idea that some "activation" process in the nuclear region has to precede actual growth (cf. Бекк, 1949), but in the assumption in the former theory, that the orientations of the lattice-elements, where such an activation can take place, are in first instance determined by the deformation process, whereas the latter theory supposes that in general activation may take place in elements with all kinds of orientation, leaving it to the selective character of the growth process to choose those properly oriented with regard to the matrix for growth.

We think that, generally speaking, neither of the two factors 3) or 4) can be considered to be the exclusively active one in determining the orientation of the crystals observed after recrystallization is complete; but that the final texture has to be considered to be the combined effect of both, it depending on the experimental conditions of deformation and annealing, which one has the dominating influence.

If actually the deformation process produces potential nuclei in all possible orientations and with nearly equal "incubation periods" (cf. the end of paragraph 3), then growth selectivity may be expected to be the dominant factor, as all nuclei start their growth at the same moment. Therefore the faster growing ones may impede to a large extent the growth of the slower ones, and thus determine the final texture.

If, however, the incubation periods are widely varying, then the

potential nuclei with the shortest periods become actual growth nuclei and start their growth before nuclei with longer periods. If the "short period nuclei" happen to possess an orientation different from that most favourable for growth, then, due to the fact (cf. point *a* of paragraph 6) that growth selectivity is not so very pronounced, such nuclei may consume a considerable part of the matrix before nuclei with a longer incubation period (and perhaps more favourable orientation with respect to the matrix) may start their growth, or such nuclei may even be consumed by the already growing crystals. In such a case the final texture is not that to be expected according to the growth selectivity conception. The occurrence of a large scattering in the observed textures as well as that of large crystals with orientations different from that of the main group (cf. point *a* of paragraph 6) can be understood in this way.

In other cases, however, the deformation process may have a less all-round character (cf. point *b* of paragraph 6) and produce "local curvatures" or adjacent lattice elements with "unstable" boundaries only in special positions with regard to the matrix, either because the "curvatures" are directly correlated to the orientation of the glide-planes, or because unstable boundaries, corresponding to "cusp positions" in SHOCKLEY and READ's picture, require quite definite orientation relationships between two adjacent lattice elements, one of which may belong to the main orientation of the deformed matrix. In such cases the orientation of the new crystals, according to our view, is determined by the orientation of the potential nuclei, produced by the deformation process, i.e. by oriented nucleation. If these orientations do not conform to that which is most favourably oriented with respect to the matrix from the point of view of growth selectivity, then such "favorable" orientations cannot develop and (to quote CAHN, 1950, footnote p. 333) growth must necessarily occur from the available nuclei, whatever their orientations. Again the selectivity of the growth process seems to us to be sufficiently weak to allow such growth, if only the orientation of the available nuclei is sufficiently different from that of the matrix.

In conclusion, the considerations given above do not pretend in any way to give a definite solution to the problem of the origin of recrystallization textures. We think it impossible at the present state of our knowledge to decide with certainty between the various possibilities. However, we thought it opportune to draw attention to some experimental facts, which, at least in our opinion, are not readily explained on the basis of the selective growth theory *alone*.

*Summary:*

The first part of this paper intends to show that X-ray results obtained by TIEDEMA (1950) on the structural state of the nuclear region of aluminium crystals grown by recrystallization, support the idea that new crystals originate in "local curvatures" of the deformed matrix.

The second part discusses the question to what extent the orientations of crystals grown by recrystallization of a given matrix can be understood on the assumption of what may be called "oriented nucleation" or "selective growth".

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