

OBSERVATIONS ON THE NATURE OF UNMIXING IN PERISTERITE PLAGIOCLASES

By

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Abstract

The exsolved phase of the structurally inhomogeneous peristerite plagioclases (An_2 - An_{17}) has been observed to take the form of minute (-0.5μ) domains scattered in apparently random array throughout the host feldspar (Ribbe and Van Cott (1962)). It is shown, however, that there must be mutual crystallographic orientation of the two phases, $An_{2.5}$ and An_{25} , to explain the schiller phenomenon. A detailed comparison of the atomic structures of albite and oligoclase (An_{22}), while failing to indicate any geometric relationships that would explain the directional schiller (Bøggild (1924)) or the related (08 $\bar{1}$) lamellae (Brown (1960b)), indicates a close similarity of the structural frameworks in the two end-members.

The peristerite subsolidus is discussed in detail from two viewpoints: that of heat-treated natural specimens and that of natural (equilibrium) plagioclase. It is postulated that the unmixing solvus is asymmetric, rising sharply in the vicinity of $An_{2.5}$ and sloping off to An_{17} where a steep gradient marks the limit of peristerite exsolution. From An_{17} the solvus is flared out at very low temperatures to the An_{20-30} compositions, and evidence is given that a sodic peristerite will tend to unmix to $An_{2.5}$ and An_{30} and a calcic peristerite will unmix to $An_{2.5}$ and An_{20} . Unmixing is postulated to be contiguous with the Al/Si ordering process in the peristerite range.

Introduction

Structural inhomogeneity of the soda-rich low-temperature plagioclases was first reported by Laves (1951). His later work (Laves (1954)) indicated that in the range An_5 to An_{17} this inhomogeneity is expressed by the co-existence of two geometrically distinct plagioclases corresponding roughly to low albite and low An_{30} . Gay and Smith (1955) gave these compositions more precisely as $An_{3\pm 2}$ and $An_{23\pm 2}$. Further detailed examination of this composition range showed that unmixing occurs in plagioclases containing as little as 2 wt. % An (Brown (1960b)). It was also found by measuring the reciprocal lattice angle γ^* that the unmixed phases were not always of the same composition from grain to grain. Variations ranging from An_1 to An_5 and An_{20} to An_{35} were determined for the Ab- and An-rich components (Ribbe (1960)), using Smith's (1956) plot of γ^* versus wt. % An content. The mean compositions of these based on 43 measurements compiled from the literature are $An_{2.5}$ and An_{25} .

Heating experiments were performed on a peristerite by Schneider (1957). It was discovered that at high temperatures the albite phase requires 3 to 7 times as long to attain its disordered geometry as does the An_{25} phase. Because the lattice parameters of the high-temperature forms of the two phases are indistinguishable by X-rays, it was tempting to assume homogeneity at this point in the heated specimen's history. However, the persistence of schiller in this form belied true homogeneity. It was demonstrated by the present author (1960) that optical heterogeneity between high albite and the high An-rich phase caused this schiller in the same way that it did in the low-temperature peristerites. True homogeneity could only be achieved by destroying the exsolved domains, thereby making a single-phase disordered solid-solution crystal. Difficulty of diffusion of tetrahedral Al/Si over any appreciable distance (Goldsmith (1952)) was proposed as the reason for extreme sluggishness of the homogenization process.

Bøggild (1924) observed that peristerite schiller was visible in an orientation inclined 13–14° to [010] which roughly corresponds to [08 $\bar{1}$]. He suggested the possibility of lamellar unmixing in the (08 $\bar{1}$) plane causing schiller by reflection. Brown (1960b) observed just such lamellae in schillered peristerites, noting that their thicknesses were 1 to 3 microns and their occurrence and separation sporadic.

Recently Ribbe and Van Cott (1962) have extended the peristerite studies, employing special microscope techniques to observe the exsolved phases, thereby confirming the cause of peristerite schiller previously predicted (Ribbe (1960)). The results of this work are reviewed below as a preliminary to discussions of the structural causes of unmixing and the phase changes related to it.

Optical studies of unmixing [1]

Phase-contrast and dark-field microscope techniques reveal small differences in refractive index or optic path in an optically heterogeneous material by enhancing the brightness contrast of one phase against the other. At high magnifications they can be used to study the relationships of exsolved and host feldspars in peristerites, because albite and An_{25} differ in refractive index by $\sim .012$ both in their low- and their high-temperature forms.

It is thereby observed that the exsolved phase takes the form of spherical or ellipsoidal domains ranging downward in size from ~ 0.5 micron (5000 \AA). See Figure 1. It is presumed that there is a lower limit of domain size in the vicinity of 0.02 micron because diffracted spots on X-ray diffraction photographs are generally sharp and clear, except in the case of incomplete unmixing as discussed by Brown (1960b). These exsolved domains are distributed linearly or on planes—often the composition face (010) of albite twins—but are far more frequently observed in apparently random disarray. However, because the schiller produced by the optical heterogeneity of peristerites can only be seen in a direction normal to $(08\bar{1})$ (Bøggild (1924)) and because Brown (1960b) has reported the occurrence of $(0\bar{8}1)$ ($\equiv (08\bar{1})$) lamellae in several schillered specimens, it must be assumed that there is a unique crystallographic relationship between all the exsolved domains and the host feldspar that produces these effects. This will be discussed later, but for the moment it is important to emphasize the necessity of mutual orientation of the domains to one another and to demonstrate that although fairly extensive lamellae of the exsolved phase may exist, it is not these that cause peristerite schiller [2].

[1] This is a condensed review of the work of Ribbe and Van Cott (1962).

[2] Jenkins and White (1957) provide the theoretical approach to this schiller model. Raman, Jayaraman and Srinivasan (1950) develop analogous ideas for the two-phase potash-soda feldspars.

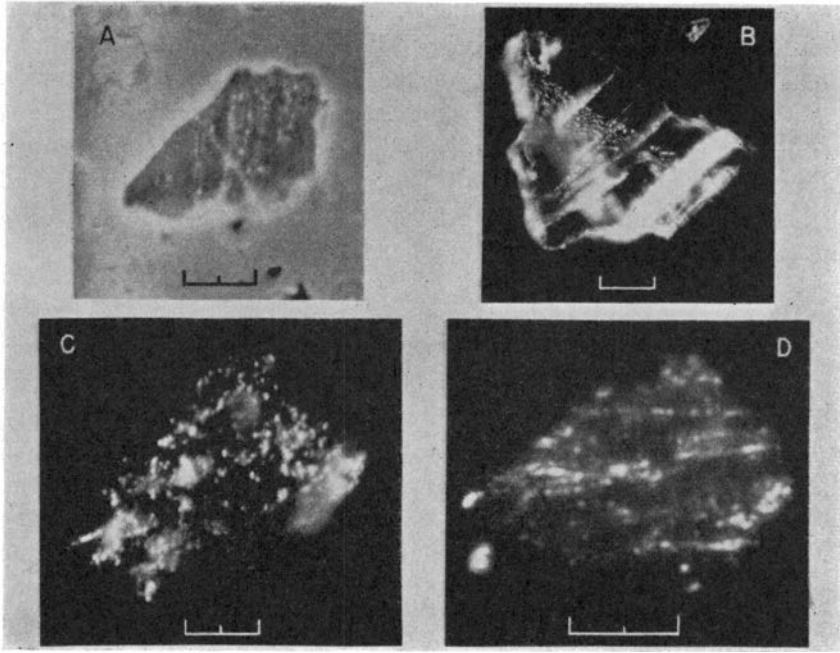


Figure 1. Views of peristerite fragments using special microscope techniques. The spherical or ellipsoidal domains are exsolved plagioclase shown in contrast to the host material.

A. Phase-contrast lighting.

B, C, D. Dark-field lighting.

All scales are 10 microns from end to end.

(After Ribbe and Van Cott (1962).)

The white and blue schiller can consistently be explained only in terms of diffuse reflections and scattering from optically distinct, mutually oriented domains such as those shown in Figure 1. When domains of a size near or somewhat larger than that of a wavelength of light predominate, diffuse reflections from these cause a white schiller. When most of the domains are very much smaller than this, light scattering results, and the schiller is blue. It follows that the intensity of blue schiller increases with increasing proportion of scattering domains to reflecting domains. It is presumed that the absence of schiller indicates that the exsolved feldspar is in domains so small that scattering is unobservedly weak.

Should it happen then that the observed (08 $\bar{1}$) lamellae are continuous, i. e. not made up of closely spaced, minute domains, they can be said neither to contribute to nor detract from the schiller phenomenon as described for this model. Their existence is evidence that there is cause on an atomic scale for a preferred geometric relationship between the exsolving phases. In search of this relationship the low albite and oligoclase (An₂₂) structures have been the subjects of careful scrutiny and comparison.

Structural comparisons of albite and oligoclase

A point-by-point comparison of the crystal structure of albite (Ribbe, Ferguson and Taylor (this volume)) and An₂₂ (Waring (1961)) was undertaken to provide the answer to the riddle of the directional nature of schiller and to the cause of unmixing itself. Differences in T-O (T for Tetrahedral) and O-O bondlengths and the splitting of the large cation in the two structures were of course expected. But although the degree of randomness of Al/Si in the T-sites of oligoclase had been predicted by Schneider (1957) (discussed below), there were at that time certain difficulties in accepting this argument, notably because it had been presumed by this author (1960) that some sort of unusually stable Al/Si ordering would exist near the An₂₅ composition which would dynamically necessitate its formation as an end-member in the unmixing process. Megaw (1959) had ventured that local electrostatic charge balance might dictate such an arrangement. But within the

Table 1. The distribution of tetrahedral Al/Si in albite and oligoclase.

Site	Low albite (Ab) Mean T-O distance	% Al*	Low oligoclase (An ₂₂) Mean T-O distance	% Al*
T ₁ (0)	1.74 ₄ Å	95	1.68 ₂ Å	45
T ₁ (<i>m</i>)	1.61 ₀ Å	0	1.63 ₄ Å	15
T ₂ (0)	1.61 ₆ Å	3	1.63 ₂ Å	14
T ₂ (<i>m</i>)	1.61 ₃ Å	2	1.65 ₂ Å	26

* These values are based on the assumption that the Al/Si bond lengths in feldspars vary linearly from 1.75 Å for 100 % Al to 1.61 Å for 100 % Si. (Smith and Bailey (forthcoming)).

accuracy of a two-dimensional analysis, oligoclase was found to have an irregular, but not totally random distribution of Al/Si on the T-sites (Table 1) and a rather poor local charge balance ($\sum|A| = .31$ to $.51$).

Explanations failed on these accounts, so a detailed geometric comparison was made. Table 2 indicates that angular distortions of the tetrahedral framework of low albite to account for the compositional differences of oligoclase are non-significant. The only difference is a uniform, multidirectional expansion of the fourfold tetrahedral ring in oligoclase to accommodate the larger Al cation. This amounts to 0.01 \AA —very nearly the difference between the radii of the average tetrahedral cations in the two structures: $Al_{\frac{1}{4}}Si_{\frac{3}{4}} = 0.325 \text{ \AA}$; $Al_{\frac{5}{16}}Si_{\frac{11}{16}} = 0.334 \text{ \AA}$.

This striking geometric similarity of frameworks is perhaps the most important discovery of this study. It may be that An_{25} as a structurally stable peristerite end-member is best considered from an engineering viewpoint as simply an expanded model of the least-strain, low-energy albite structure. This would provide an explanation for its appearance as a peristerite phase but leaves the problem of directional schiller and unmixed lamellae unanswered.

It should be re-emphasized that the Al/Si distributions on the T-sites of Ab and An_{22} are not notably similar, nor are the splittings of the large cations. The sodium half-atom "splitting" is 0.35 \AA in low albite; it is 0.66 \AA in oligoclase. The directions of the splittings are nearly parallel in the [100] projection and are inclined by $\sim 9^\circ$.

Table 2. Comparison of interatomic angles in albite and oligoclase.

Angles	Differences between Ab and An_{22}		R.M.S. deviation of angles from a mean value within the refined albite structure
	$ \bar{\Delta} $	$\Delta_{\text{max.}}$	
T-O-T	$1^\circ 01'$	$3^\circ 01'$	$10^\circ 08'$
O-T-O	$1^\circ 11'$	$3^\circ 21'$	$3^\circ 19'$
T-T-T*	$50'$	$2^\circ 50'$	—

* Although these are not interbond angles, they are useful in demonstrating that the expansion of the framework discussed in the text is not compensated for by distortion of the basic feldspar unit, the fourfold tetrahedral ring.

Even if one were to assume that these cations were disordered in their respective cavities and could be optically activated, thereby causing schiller, one would find that this inclination is in no ordinary way related to the schiller direction in peristerites. Nor apparently are any other geometric parameters.

The peristerite subsolidus phase relations

Although there are the aforementioned difficulties in explaining the structural reasons why peristerites unmix, there have emerged from this study two reasonable diagrammatic representations of what is known to occur in this the soda-rich portion of the plagioclase system. It is advisable to treat these separately. The first is constructed from experiments involving the heat-treatment of natural low-temperature peristerites. The second is based on observations from numerous sources of natural specimens and is somewhat more tentative than the first for reasons discussed below.

Before proceeding to specific details, some clarification of terminology is necessary. The word "ordering" is used herein to indicate the positioning of Al and Si on specific tetrahedral sites in a totally regular array. "Disordering" means that this regular array is being destroyed (partial disorder = partial order) and ultimately total randomness of occupation of tetrahedral sites by Al/Si is introduced, and the structure becomes a "disordered" or "high-temperature" structure. The "low-temperature" structure, however, is not necessarily a fully ordered structure in the classic geometric sense. It is the structure that exists in a geologic low-temperature environment and is stable or perhaps even metastable under those conditions.

The peristerite subsolidus has generally been regarded as a simple solvus with Ab and An₂₅ as the end-members (Christie (1959; 1962); Noble (1962)). This representation is oversimplified, as indeed are most attempts to explain plagioclase dynamics. Because it is fairly well established that only natural specimens of less than An₁₇ content unmix to form peristerites (Laves (1954); Brown (1960b); Ribbe (1960)), it must be supposed that there is a thermodynamic reason that unmixing is only initiated in the An₂-An₁₇ range and is unable to start at higher anorthite compositions. This is shown in Figures 2a and 2b as a skewed solvus which has a very steep slope in the An₁₇ region and a

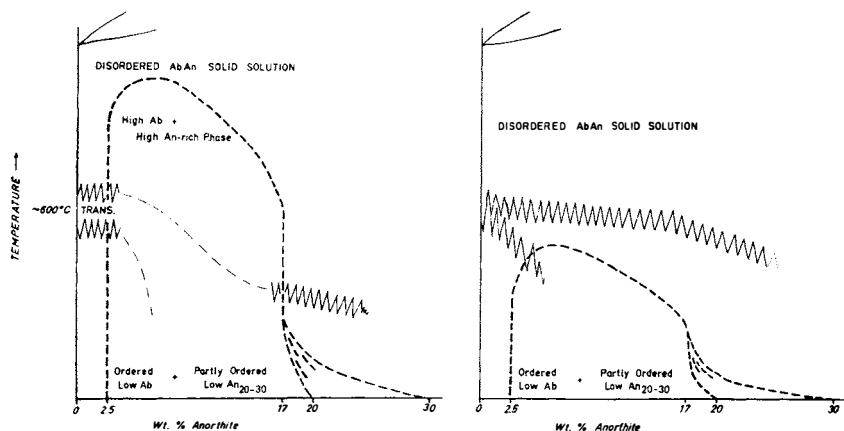


Figure 2. Tentative diagrams of the peristerite plagioclase subsolidus phase relationships. The jagged lines represent the "smeared transformation" of McConnell and McKie (1960).

- a.* The subsolidus derived from laboratory heat treatment of natural peristerites. Probably a non-equilibrium diagram.
- b.* A proposed phase diagram for peristerites which form from a high temperature under natural geologic conditions.

flaring out at low temperatures to the An_{20-30} compositions. It is thus assumed that a material of composition greater than An_{17} remains a solid-solution crystal even at low temperatures when its isopleth intersects the flared solvus, because at these temperatures there is too little thermal energy to impel the exsolution process.

This interpretation finds support from various sources. First, it must be recalled that Waring's (1961) structural analysis of An_{22} shows it to be a homogeneous single crystal whose local electrostatic charge balance is rather poor. That this structure would have to be more nearly disordered than albite had previously been predicted by Schneider (1957), who reported that the An_{25} phase of his peristerite attained its high-temperature geometry 3 to 7 times as rapidly as the fully ordered albite phase. Secondly there are indications that the less anorthite in a potential peristerite, the greater will be the tendency to unmix to Ab and An_{30} (Figure 3) [3]. On the other hand an initial

[3] This tendency toward An_{30} may be confirmation that DeVore's (1956) An_{33-3} is the ultimately stable end-member, but the appearance of intermediate plagioclase forms near An_{25} leaves this open to question. There may, of course, be a range of

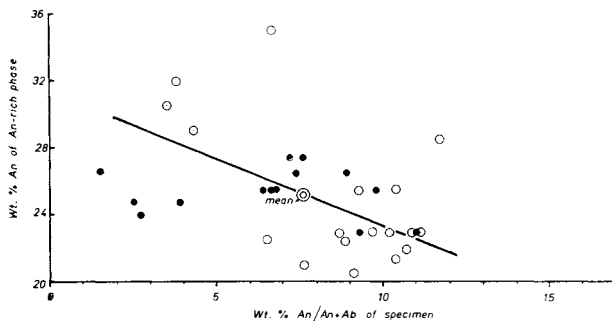


Figure 3. A plot of the wt. % An of the An-rich phase against the total wt. % An/An + Ab of peristerite specimens. Black dots represent values from Brown (1960b), open circles those from (Ribbe (1960). A line of best fit indicates the tendency of An-rich peristerites to unmix to Ab and An₂₀. Ab-rich specimens tend to unmix to Ab and An₃₀.

composition not far below An₁₇ will only have energy enough to unmix to low Ab and An₂₀.

Further confirmation of this type of solvus is found in Brown (1960b), pp. 341–342: “To date no plagioclase with more than 18% anorthite . . . has been found to be unmixed. Many plagioclases just below this composition have diffuse tails to strong reflections [4]. This is interpreted as meaning that the unmixing temperature in the compositional range above approximately An₁₇ is below that at which such oligoclases are stable in rocks”. He goes on to state that these crystals would form reaction products in certain metamorphic facies. In plutonic rocks, supposedly, the crystal would retain its metastable form.

Figures 2a and 2b, therefore, have similarly shaped solvi, but in that Figure 2a represents the subsolidus as deduced from laboratory heating experiments, it differs from that proposed in Figure 2b for natural occurrences where geologic ages are involved in cooling, heating, and reaction rates.

The subsolidus diagram in Figure 2a demonstrates the following things that can only be observed in the heating of low-temperature natural peristerites. (1) The “smeared transformation” from low to structures near An₂₂ which have the geometric stability mentioned in the previous section.

[4] Discussed at length below.

high albites has been drawn in the vicinity of 600 °C, following McConnell and McKie (1960). (2) The upper limit of this transformation intersects the opposite boundary of the field at a considerably lower point, indicating the fact that the more An-rich of the peristerite phases achieves random Al/Si distribution with far greater ease than the Ab phase (Schneider (1957)). The lower limit of the transformation is not shown, because it is assumed that the structures An₂₀₋₃₀ are partially disordered with respect to Al/Si (as An₂₂ is known to be) and thus fall between the transformation boundaries. (3) The inclusion of a two-phase region above the transformation is based on evidence that although the initial peristerite phases have attained their indistinguishable high-temperature geometries, they can still be seen microscopically in specimens heated between 1045 and 1070 °C for as long as 65 days (Ribbe and Van Cott (1962)). Schiller retention when geometrical homogeneity has been reached further demonstrates that the optical heterogeneity seen by phase-contrast and dark-field techniques results from the coexistence of high albite and a high An-rich phase in these specimens. The statements made under (2) and (3) above are analogous to Laves' (1952) explanation of the heat-treatment of cryptoperthites which consist of exsolved, highly ordered albite and K-rich areas of "common orthoclase" in which a considerable degree of disorder is still prevalent." It is to be emphasized that the phase changes shown here are non-reversible and that this diagram does not purport to represent the equilibrium that is gained by natural specimens in the course of geologic ages.

The subsolidus in Figure 2*b* differs from that in 2*a* in that the solvus is placed lower on the diagram; therefore, no two-phase field is shown at high temperatures. This diagram, in fact, only shows what is presumed to have happened during the formation *from some high temperature* of natural albite and oligoclases, because specimens of intermediate or high thermal state are rare or absent in nature with the possible exception of those mentioned below.

Brown (1960*b*) has assumed that ordering within crystals in the peristerite range occurs *before* unmixing; but to the present author this is somewhat contradictory, because unmixing begins during and occurs simultaneously with the ordering process, the unmixed state representing a much lower energy state than the proposed "ordered" solid solution crystal of the same average composition. Such an as-

sumption eliminates the only apparent driving force for unmixing [5].

Brown states (p. 342) that "the lattice constants of oligoclases in the range An_{10-18} which show only the beginnings of unmixing fit on to the curves for ordered plagioclases (Brown (1960a), Figures 1-5)." However, if one fits reported γ^* measurements from his specimens — An_{11} , An_{13} , An_{14} , An_{16} — to a curve γ^* vs. An content drawn up independently of these specimens (Smith (1956)), one discovers that these γ^* angles represent compositions of An_{13} , An_{17} , $An_{18.5}$, and An_{18} , or else they indicate that the structure of each is partially disordered. If the former assumption is correct, then three of the four specimens fall on the flared portion of the solvus and confirm the idea that only the slightest indication of unmixing can be expected at or above An_{17} . If, on the other hand, one assumes the structures to be partly disordered forms, it then follows that unmixing begins before Al/Si ordering is complete and is in fact contiguous with the ordering process.

Notwithstanding the interpretation of these somewhat unusual specimens, the argument for unmixing being initiated during the ordering process is tenable in view of (1) the evidence given by McConnell and McKie (1960) that on heating albite some disordering occurs at rather low temperatures, (2) the expectation that full ordering in the classical sense will never be attained by the An-rich phase (Waring (1961)), and (3) the lack of a motive force for exsolution if viewed differently. There is the additional evidence of the observations of heat-treated peristerites which, of course, must be treated cautiously in view of its non-equilibrium nature.

The fact that a nearly pure, fully ordered albite phase is always found in unixed peristerites leads to the conclusion that the driving energy for its formation greatly exceeds that which dictates the final structure and composition of the other phase. The gross geometric similarity of the frameworks of the Ab and An_{25} structure may or may not be significant in furthering unmixing. In any case, there yet remains for future understanding the directional nature of the peristerite phenomenon.

[5] Intentionally ignored in this paper is the growth of sodic plagioclase in metamorphic facies where reaction with other minerals (and often water) is essential (Christie (1962); Rutland (1962)). Such a study would involve ternary and quaternary systems that are beyond the scope of this paper. According to Rutland (1962), Brown's (1960b) argument is valid, but other phases or degrees of freedom would be needed to achieve this state.

Acknowledgements

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Discussion

Olav H. J. Christie (Oslo)

Paul Ribbe claims that my peristerite solvus (1959, 1962) is oversimplified. This may or may not be so. Quite generally I think one should use simple models as long as they do not conflict with our general experience with the matter dealt with.

Among the few plagioclases in the compositional range An_{20-26} which have been examined so far, none have shown peristeritic exsolution. In the compositional range An_{2-14} most plagioclases are exsolved, and the exsolution products have the composition An_2 and An_{26} respectively.

I believe that a model, slightly different from that one presented by Ribbe, may explain this, using a solvus shape that is well known in

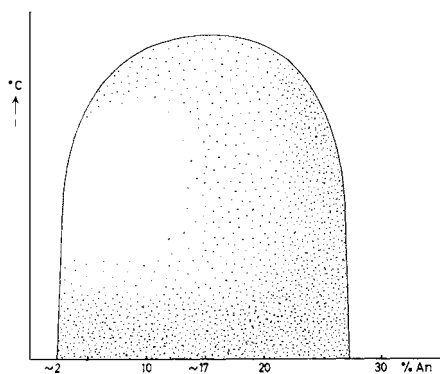


Figure 1. Tentative exsolution diagram of peristerites. The area of difficult nucleation of exsolved phases is dotted. (The matter dealt with is nucleation, not growth and atomic rearrangements in the exsolved phases.) Within the undotted area the nuclei have the compositions An_2 and An_{25} .

a large number of silicate systems (Figure 1). The dotted area enclosed by the solvus represents an "area of difficult nucleation" of the exsolved phases. From this diagram one may explain why plagioclases in the compositional range An_{17-20} to An_{26} do not exsolve, or do so under very special conditions only: it is so simply because the nucleation energy barrier is too high in this compositional (structural) range to allow the formation of the phases An_2 and An_{26} .

The peristerite solvus by Ribbe, however, shows nicely what is found in nature: that plagioclases in the compositional range An_2 and An_{17} exsolve and that the exsolution products are An_2 and $An_{25}-An_{30}$. If the solvus by Ribbe is correct, there must be a *dominant* structural control over the peristerite formation. I think Dr. Ribbe and I agree that such a control exists. Neither of us, however, know how strictly or to what extent this control acts.

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