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Intensity differences of subsidiary reflections in calcic plagioclase

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Abstract

The intensity distribution of *e* reflections in intermediate plagioclase is different from that of corresponding *b* reflections in anorthite. While *e*-reflection pairs with strong intensity asymmetry occur at positions corresponding to strong *b* reflections in anorthite, symmetrical *e*-reflection pairs occur at positions typical of weak *b* reflections. The differences in intensity between *e*-pair sums and corresponding *b* reflections (*e.g.* 071, 073, $09\bar{3}$, $09\bar{5}$) can be used to distinguish between the intermediate plagioclase and anorthite structural types, particularly in cases with highly diffuse reflections for which geometric criteria are ambiguous. Application of this method has documented very diffuse *e* reflections in volcanic labradorites and bytownites previously thought to only have *b* reflections. These plagioclases also have diffuse *c* reflections with the intensity distribution of *c* reflections in anorthite, and we designate them as *transitional plagioclase* to indicate presence of diffraction features characteristic of both structural types.

Introduction

Subsidiary reflections in plagioclase characterize superstructures with either compositional or positional order or both. The structures of albite and anorthite are reasonably well understood, while structures of plagioclase with intermediate composition are still controversial. Of particular interest is a superstructure which is evidenced by satellite reflections in the diffraction pattern. It has been described as a modulated superstructure with antiphase relationships and may have regions which resemble albite and anorthite. In this paper we investigate the intensity distribution of subsidiary reflections in intermediate plagioclase and compare it with that of corresponding reflections in anorthite.

Weak reflections in addition to those observed in albite were first noticed by Taylor *et al.* (1934) and Chao and Taylor (1940), but only later were these reflections classified and studied systematically. Cole *et al.* (1951) noted that "split *b*" reflections (later called *e* reflections) occurred in pairs, of generally unequal intensity, and were satellitic about *b* positions in anorthite ($h + k$ = odd, l = odd). Bown and Gay (1958) classified and introduced the current nomenclature for reflections in plagioclase (*a, b, c, d, e, f*), and reviewed observations of Gay (1956) that the geometry of *e* reflections varied as a function of An content. Wenk (1977) recently found a similar variation with changing metamorphic grade.

While geometrical variations have been clearly documented, there have only been speculations about changes in intensity. Gay (1956, p. 29) suggested a general relationship between *b* and *e* reflections. He noticed that "a strong (*b*) reflection in body-centred anorthite is usually replaced by a relatively strong pair of split reflections in the intermediate pattern."

Bown and Gay (1958, p. 6) further advanced such a relationship. They reported that the average intensity of a pair of *e* reflections is comparable to the intensity of the corresponding *b* reflection. This has generally been accepted by feldspar researchers (*e.g.* Smith and Ribbe, 1969, p. 175; Smith, 1974, p. 199). In the course of a survey of calcic plagioclase we noticed that the intensity relationship between *b* and *e* reflections was not so simple, and decided to study differences and variations in some detail. Some of these and other aspects of calcic plagioclase are described in more detail in an M.S. thesis (Rainey, 1977).

Techniques and material

X-ray experiments were done with the precession technique using Mo radiation, long exposure time (one week to two months) and a graphite mono-

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chromator (Huber type) in order to optimize intensity, peak-to-background ratio, and resolution of weak and often diffuse subsidiary reflections. We are aware of the drawbacks of the precession technique when photographing reflections slightly out of the plane of the photograph. Oscillation photographs may have been easier to interpret quantitatively, and also exposure times would have been reduced. On the other hand, the precession technique provides for fast orientation (of often small fragments without morphology which we picked out of thin sections), ease of indexing, and cameras were available.

A graphite monochromator yields, in addition to $K\alpha$-radiation, weak radiation resulting from a second-order reflection on the monochromator crystal with a wavelength of $K\alpha/2$ from the continuous spectrum. Some weak but sharp reflections at low diffraction angles at positions of *d* and *c* reflections in Figure 1c can be attributed to such radiation. They did not cause any problems.

The intensities of *b, e,* and *c* reflections ($h + k$ even, l odd, some strong ones marked with arrows in Fig. 1) were first visually estimated with the aid of a calibrated intensity scale. We observed that some reflections such as 071, 073, $09\bar{3}$, $09\bar{5}$ (indices of *e* reflections are given as those of the corresponding *b* reflection between the pair) varied in intensity more than others, and we investigated the pairs 071/073 and $09\bar{3}/09\bar{5}$ more closely. These pairs were chosen, apart from the large relative variation, because they are close together in reciprocal space (similar *Lp* factor) and easy to observe on 0*kl* precession photographs. Intensity profiles were then scanned with a Jarrell Ash 21-000 microphotometer, in case of *e* reflections in the direction of the *t* vector (Smith, 1974, p. 152). In the range of observed intensities there was a practically linear relationship between exposure and measured intensity. Peak profiles (Fig. 2) were integrated with a cartographic planimeter, for *e* reflections summing both reflections of a pair. Since absolute values of intensities from crystals of different size and non-standardized exposures were difficult to compare we only considered intensity ratios. Ratios for two pairs of reflections are plotted in Figure 3. Values obtained with the microphotometer

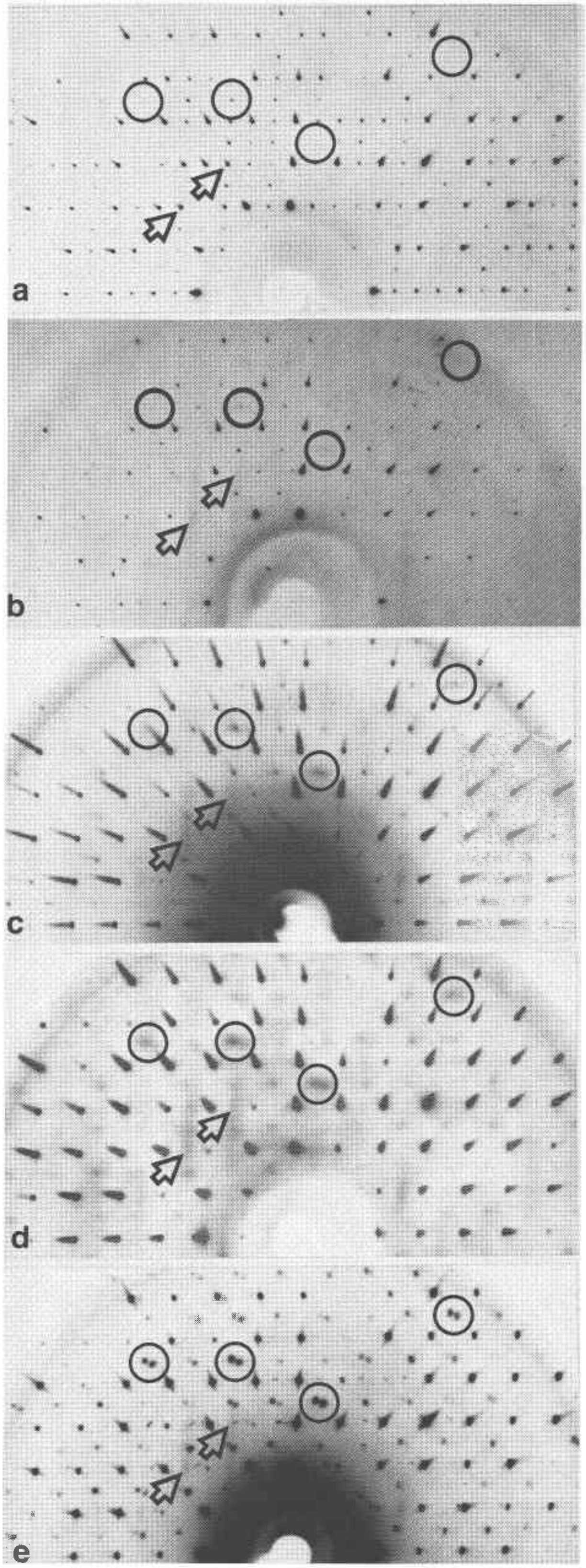

Fig. 1. Monochromatic precession 0*kl* photographs of calcic plagioclase. Characteristic satellite reflections are circled. Strong *c* reflections are indicated by arrows. Notice the different intensity distribution of subsidiary reflections in anorthite (a) and intermediate plagioclase (e). a: anorthite, Grass Valley (14); b: bytownite, Crystal Bay (8); c: labradorite, Lake County (7); d: labradorite, Surtsey (5); e: labradorite, Central Alps (4).

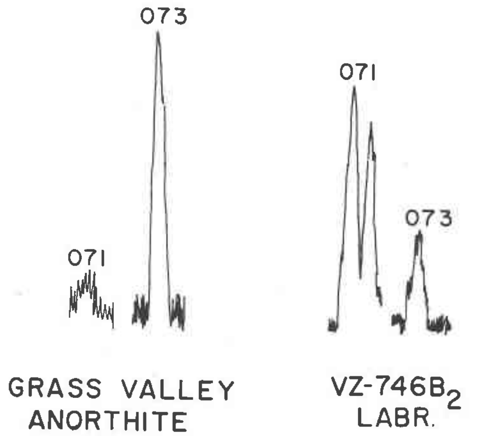

Fig. 2. Densitometer profiles across the reflections 071 and 073 of Grass Valley anorthite (a) and intermediate plagioclase from the Central Alps (b). Notice the switch in intensity.

compare well with diffractometer measurements which were available for selected specimens.

Information about the plagioclases studied is summarized in Table 1.

Intensity variation

Comparing a precession $0kl$ photograph of anorthite (Fig. 1a) with intermediate plagioclase (Fig. 1e) we recognize considerable differences. For instance, circled *e* reflections in intermediate plagioclase such as 071 and $09\bar{3}$ are strong while corresponding *b* reflections in anorthite are weak. The reversal in intensity between reflection pairs such as 071 and 073 is also demonstrated with microphotometer profiles in Figure 2. In Figure 3 the intensity ratios for two pairs of characteristic reflections of 15 calcic plagioclase samples are plotted. There are two main groups, one typical of intermediate plagioclase with 071 (resp. $09\bar{3}$) strong and 073 (resp. $09\bar{5}$) weak, and one typical of anorthite with 073 and $09\bar{5}$ strong and 071 and $09\bar{3}$ almost missing.

There are a few transitional samples (6, 7, 8), mainly volcanic labradorites and igneous bytownites with highly diffuse *e* reflections either together with or without *b* reflections. Lake County labradorite (7) has been described as "body-centered" with only sharp *b* reflections (Stewart *et al.*, 1966) but examination of $0kl$ photographs shows, in addition to sharp *b* reflections, diffuse streaking in positions typical of strong *e* reflections (Fig. 4*b*, compare also Tagai *et al.*, 1977). The same is true for Crystal Bay bytownite (Fig. 4*a*), which has never been associated with intermediate plagioclase (Grundy and Brown, 1967). In Surtsey labradorite diffuse *e* reflections have been described (Wenk, 1966) but only by checking positions typical of strong *b* reflections (e.g. $0\bar{9}5$) could we document presence of diffuse *b* reflections (Fig. 1d). Both *e* and *b* reflections become sharper after heat treatment at 870°C (Wenk, 1978, compare his Fig. 2), and as is shown in Figure 4, the intensity ratios are lower and more similar to anorthite.

Although we have documented a change in intensity of $h + k$ = odd, l = odd (*b* and *e*) reflections as we proceed from anorthite towards more sodic plagioclase, the intensity distribution of *c* reflections in the same samples remained the same. Even though *c* reflections decrease in intensity with increasing sodium content, and become diffuse and streaked roughly parallel to b^* (compare Ribbe and Colville, 1968), the strongest intensities are always observed at positions of strong *c* reflections in anorthite (Fig. 1).

Intensity distribution

Inspection of films has shown that the intensity pattern of *b* and *e* subsidiary reflections is different. This can be further advanced by investigating correlation of structure amplitudes $|F_{obs}|$ for many reflections. In Figure 5 we compare three data sets: labradorite An 52 from Labrador (measured and published by Toman and Frueh, 1976), labradorite Vz433 An 66 from the Central Alps (H.R.W., unpublished),

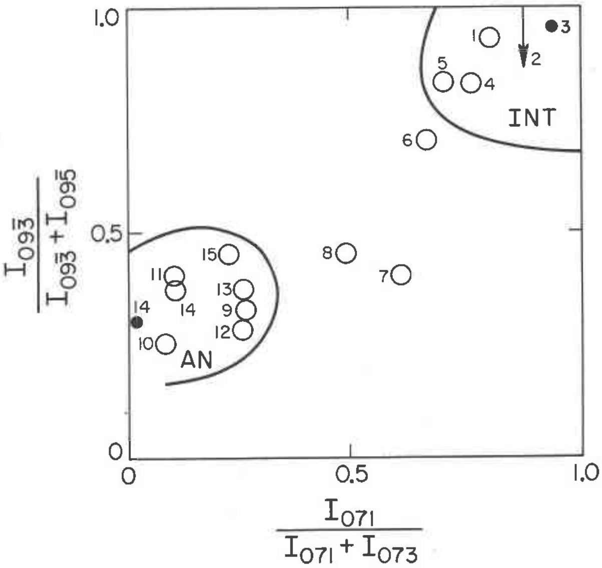

Fig. 3. Plot of intensity ratios 071/073 versus $09\bar{3}/09\bar{5}$ for calcic plagioclases. Circles represent densitometer measurements, dots diffractometer determinations. Numbers are in order of increasing An content and correspond to Table 1. Plagioclase #2 (Toman and Frueh, 1976) is represented by a line because the intensities for $09\bar{3}$ and $09\bar{5}$ were not available.

Table 1. Plagioclases examined in this study (for more information see Rainey, 1977)

	Specimen	Formation Conditions	Selected References
1	An 44, Sci 1014 Val Codera, Central Alps	Metamorphic, feldspathized ultramafic breccia	H. R. Wenk et al. (1977)
2	An 52 Labrador, Canada	Plutonic, anorthosite	Toman and Frueh (1976)
3	An 66, Vz433 Gordemo, Central Alps	Metamorphic, amphibolite facies	E. Wenk et al. (1975)
4	An 66, Vz 746b$_2$ V. Carecchio, Central Alps	Metamorphic, amphibolite facies intergrown with 11	E. Wenk and H. R. Wenk (1977)
5	An 66, Surtsey, Iceland	Volcanic, phenocrysts in vitric tuff, ejected 1964	H. R. Wenk (1966)
6	same crystal as 5	same as 5, heated at 870°C for 50 days	H. R. Wenk (1978)
7	An 66 Plush, Lake County, Oregon	Volcanic, phenocrysts in basalt	Stewart et al. (1966) McLaren and Marshal (1974) Tagai et al. (1977)
8	An 77 Crystal Bay, Minnesota	Plutonic, coarse grained gabbro	Grundy and Brown (1967) McLaren and Marshal (1974)
9	An 84 Cape Parry, E. Greenland	Volcanic, porphyritic basalt dike	E. Wenk et al. (1968)
10	An 89 #332 Stillwater, Montana	Igneous, gabbro from basal zone of Stillwater complex	H. R. Wenk and Nord (1971)
11	An 90, Vz 746b$_2$ same sample as 4	Metamorphic, amphibolite facies, intergrown with 4	E. Wenk and H. R. Wenk (1977)
12	An 93, #12118C Moon, Apollo 12 site	Volcanic, lunar basalt	H. R. Wenk et al. (1972)
13	An 94 Serra de Magé meteorite, Brasil	eucritic achondrite, fall 1923	Müller et al. (1972)
14	An 94.5 Grass Valley, Calif.	Plutonic, anorthosite	McLaren and Marshal (1974) Muller and Wenk (1973)
15	same as 14	same as 14, heated at 1200°C for 7 days	Müller and Wenk (1973)

and anorthite An 94.5 from Grass Valley (H.R.W., unpublished).

There is a good linear relationship between the sum of *e* satellite structure amplitudes in the two labradorites (2) and (3) (Fig. 5a). This indicates that the intensity measurements of these weak reflections at irrational reciprocal lattice positions are reliable, and also demonstrates that the intensity variation within these intermediate plagioclase structures of different composition is minor.

In Figures 5b and c the sum of structure amplitude of pairs of *e* reflections in intermediate plagioclase is plotted versus the structure amplitude of corresponding *b* reflections in anorthite. Structure amplitudes $|F|$ are expressed in electron units. These graphs confirm the observations on films of no direct correlation of intensity between *b* and *e* reflections. Figure 5c shows that there are equal numbers of strong *b*'s corresponding to weak *e*'s as there are strong *e*'s corresponding to weak *b*'s. There is possibly an underrepresentation of strong *b*'s corresponding to strong *e*'s, suggesting an inverse relationship. This pattern is similar to Figure 5b. Clusters and gaps are attributed to poor statistics due to fewer data points (only 233 in Fig. 5b, versus 765—all *e* reflections with $\sin\theta/\lambda <$ 0.55 in Fig. 5c). We have outlined in Figure 5c two areas which contain the most characteristic subsidiary reflections to distinguish intermediate plagioclase (+) from anorthite (×).

We noticed on precession photographs that pairs of strong *e* reflections consisted of two satellites of similar intensity and decided to investigate relationships between intensity and asymmetry. In this analysis we define positive satellites ($e+$) as those for which Δl is positive when l is positive and introduce a measure for the asymmetry $a = |F_{e-}|/(|F_{e-}|+|F_{e+}|)$. Those reflections with a between 0.35 and 0.65 are called symmetrical and the remaining ones asymmet-

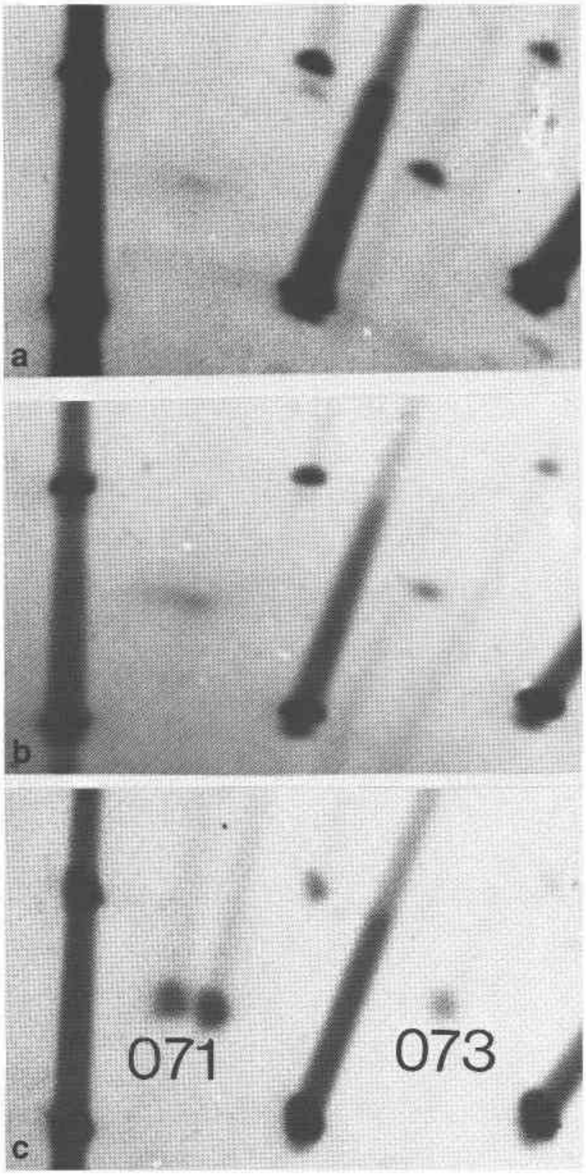

Fig. 4. Enlarged portion of precession photographs to illustrate presence of diffuse *e* reflections in Crystal Bay bytownite (a) and Lake County labradorite (b). (c) is an intermediate plagioclase (4) for comparison.

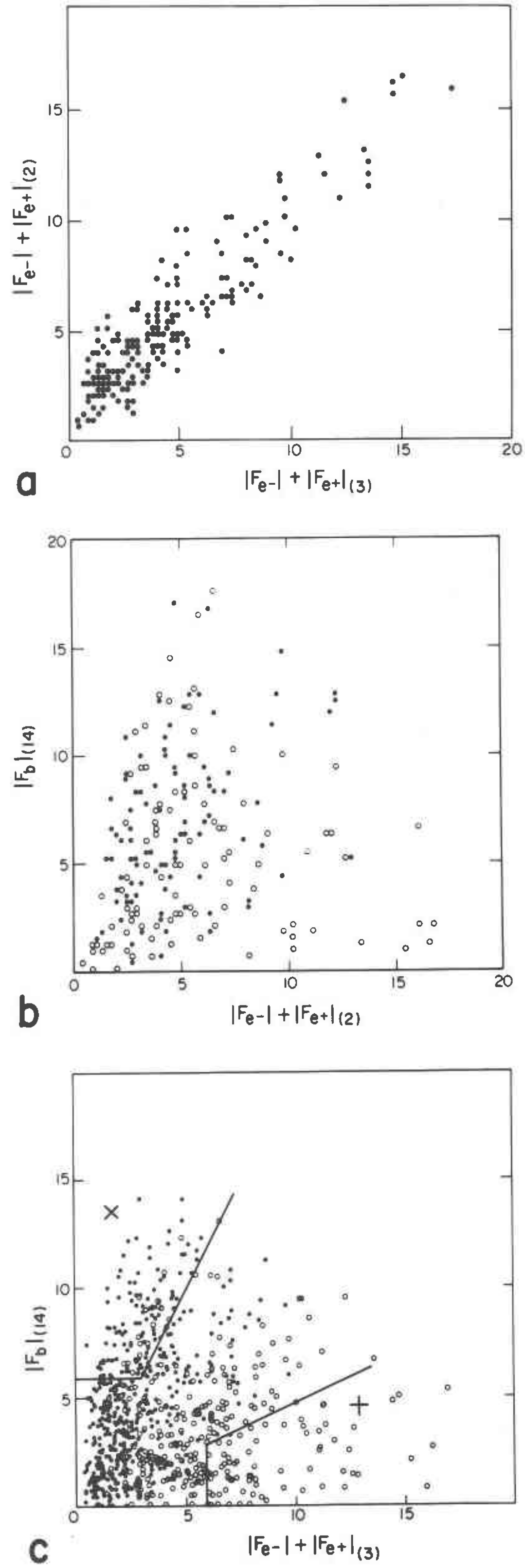

Fig. 5. Plots showing the correlation of structure amplitudes $|F_{obs}|$ for *e* reflections in intermediate plagioclase and *b* reflections in anorthite. a: *e* reflections of An 52 labradorite (Toman and Frueh, 1976, #2) versus *e* reflections of An 66 (3); b: *e* reflections of An 52 (2) versus *b* reflections in anorthite (14); c: *e* reflections of An 66 (3) versus *b* reflections in anorthite. Electron units. Circles represent symmetrical satellites ($0.35 < a < 0.65$), dots asymmetrical ones. Fields with reflections most characteristic of anorthite (+) and intermediate plagioclase (×) are indicated.

rical; both are assigned special symbols in Figures 5b, c. Symmetrical reflections (circles) correspond to the strongest satellites, and there are no symmetrical satellites in positions of strong *b* reflections in anorthite. This is also clear from the representations in Figure 6a, b, which plot the structure amplitudes of *b* and *e* reflections versus the asymmetry of the *e* reflections. Characteristic *e* and *b* reflections (fields outlined in Fig. 5c) are marked with different symbols. Figure 6a shows that all *strong* satellites are *symmetrical*. It also shows that there are equal numbers of pairs in which the negative reflection is stronger as there are pairs in which the positive dominates. Figure 6b illustrates that characteristic *b* reflection intensities (×) are not related to the asymmetry of corresponding *e* reflections.

Discussion

The empirical finding that the intensity of *b* reflections in anorthite bears *no* direct relationship to the intensity of *e* reflections in intermediate plagioclase at first seemed to indicate that structural features giving rise to *b* reflections in anorthite may be different from those causing the superstructure in intermediate plagioclase. However, Böhm (1975), in his treatment of satellite theory, has shown that there is no relationship between the intensity of zero-order (*b*) and first-order (*e*) satellites. Intensity observations neither exclude models for intermediate plagioclase which assume albite- and anorthite-like domains (Smith and Ribbe, 1969) or a stacking of subcells with the atomic arrangement of primitive anorthite (Megaw, 1960), nor do they favor the semiquantitative models based on modulated superstructures with antiphase relationships (Kitamura and Morimoto, 1977; Korekawa and Horst, 1974; Toman and Frueh, 1976). At this point we do not venture an explanation for the difference in intensity distributions, and the most striking pattern of symmetrical versus asymmetrical satellites (Fig. 5b). It may be useful to consider the different intensity distributions and particularly asymmetry relationships in structure determinations of intermediate plagioclase.

The fact that there are subsidiary reflections which are strong in intermediate plagioclase and weak in anorthite and vice versa allows use of the *intensity* of these reflections as a criterion to *identify* superstructures. This is especially important if reflections are highly diffuse or cannot be recognized by their characteristic splitting. As an identification procedure in such cases we recommend the use of strongly exposed $0kl$ photographs so that 071, 073, $09\bar{3}$ and

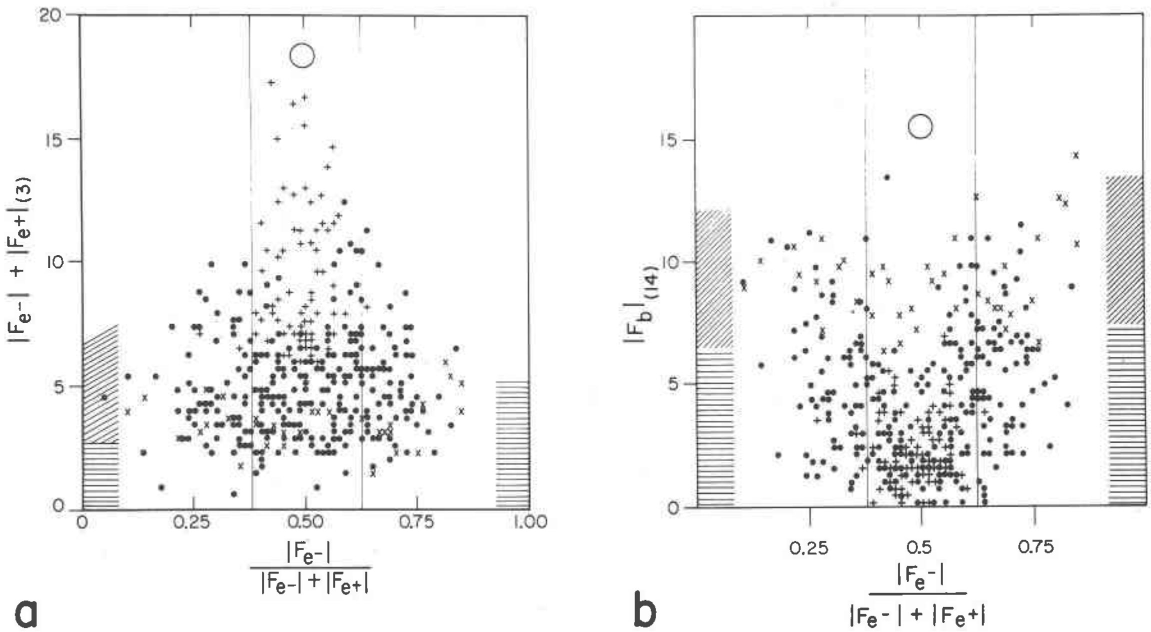

Fig. 6. Asymmetry of satellites in relation to total structure amplitude of *e* reflections in An66 (a) and to corresponding *b* reflections in anorthite (b). Characteristic *e* (×) and *b* (+) reflections are indicated (compare Fig. 5c). Remaining reflections which do not fall into a field for characteristic reflections in Fig. 5c are plotted with dots. Shaded area represents fields for which one satellite had an intensity of less than 1 sigma. Horizontal shading corresponds to dots, diagonal shading to *x* reflections.

$09\bar{5}$ are visible. Through determination of intensity ratios, *e.g.* 071/073 and $09\bar{3}/09\bar{5}$, it is possible to classify the structure of crystals as primitive anorthite, intermediate plagioclase, or a transitional structural state. These transitional phases which have diffuse *c* reflections and both diffuse or sharp *b* and *e* reflections have been called "body-centered anorthite" (Sörum, 1953), "body-centered bytownite" (Smith and Ribbe, 1969), and "transitional anorthite" (Gay, 1956). These names fail to properly describe the space-group symmetry or the chemical composition, and we propose to use the more general term *transitional plagioclase* for calcic feldspars which show features of anorthite *and* intermediate plagioclase.

Many authors have described heterogeneous structures with submicroscopic intergrowths in this composition range. Nissen (1974) and Grove (1976) have shown lamellar intergrowths of intermediate plagioclase and anorthite by transmission electron microscopy, others have postulated them on the basis of X-ray photographs (*e.g.* Jagodzinski and Korekawa, 1976, Fig. 6, and McConnell, 1974, Fig. 1b). These published photographs confirm our observations, *i.e.* strong satellites occur at positions with only weak *b* reflections; interestingly, intensity differences have never been mentioned. If heterogeneities occur on a very fine scale (*e.g.* tweed-structures described by Heuer *et al.*, 1972, and Wenk *et al.*, 1972) corresponding superreflections are very diffuse, and it is difficult to identify the structural states with electron microscopy or to apply conventional darkfield techniques for phase identification; examination of intensity ratios in X-ray photographs provides for a better characterization. While we have seen faint heterogeneities in Surtsey labradorite, and McLaren and Marshall (1974) imaged fuzzy lamellar structures in Lake County labradorite and Crystal Bay bytownite, it has not been possible to identify intermediate plagioclase with the electron microscope. Yet X-ray evidence suggests that such a phase does exist.

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