

Interdiffusion of NaSi – CaAl in Peristerite

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Abstract. The ‘average’ interdiffusion coefficient (\bar{D}) for NaSi – CaAl exchange in plagioclase for the interval from An_0 to An_{26} was estimated from experimentally determined homogenization times for peristerite exsolution lamellae. The average spacing between adjacent (unlike) lamellae is 554 ± 77 Å. Dry heating in air at 1,100 °C for 98 days produced no change in the exsolution microstructure; thus $\bar{D}(\text{dry}) < 10^{-17}$ cm²/s. This limit is consistent with the recently reported ‘average’ $\bar{D}(\text{dry})$ values for the Huttenlocher interval (An_{70-90}) at this temperature. At 1.5 GPa with about 0.2 weight percent water added the ‘average’ diffusion coefficient from 1,100 °C to 900 °C is given by:

$$\bar{D}(\text{wet}) = 18 \frac{+108}{-15} (\text{cm}^2/\text{s}) \exp(-97 \pm 5 (\text{kcal/mol})/RT),$$

where R is the gas constant, and T is °K. This $\bar{D}(\text{wet})$ at 1,100 °C is more than three orders of magnitude greater than $\bar{D}(\text{dry})$ for Na- and Ca-rich plagioclases.

Introduction

Many of the microstructural relations, as well as the chemical zoning, observed in plagioclase crystals owe their origin to the slow rate of the coupled interdiffusion of NaSi and CaAl. This low diffusivity is also responsible for the difficulty of determining many of the phase relations and the kinetics of the transformations which plagioclases undergo in the subsolidus. One of the most promising experimental approaches to these and related problems is to determine the interdiffusion coefficients for NaSi – CaAl at high temperatures and extrapolate these data to the temperature of interest. This method is subject to the usual problems that the extrapolation may not be linear for several reasons which are discussed later. Nevertheless, this approach represents one way to make meaningful progress on understanding the kinetics of transformations and chemical zoning which characterize natural plagioclases.

Although it has been recognized for many years that the interdiffusion of NaSi – CaAl is rate limiting for many of the solid state processes which occur in plagioclase, the diffusion rate was believed to be too low to be measured by conventional methods. Morse (1984) has argued on the basis of the preservation of chemical zoning in plagioclase that the value of the diffusion coefficient in the interval from 1,000 °C to 1,250 °C must be about 10^{-20} cm²/s. Re-

cent experimental diffusion data for Ca-rich plagioclase have been reported by Grove et al. (1984) for the interval from 1,100° to 1,400 °C. Their study provided important limiting, and probably minimum, values for this diffusion because their samples were annealed in air and contained little if any water.

The presence of a trace amount of intracrystalline water increases the kinetics of several solid-state processes in silicates including diffusion of some cations and oxygen in feldspar, the disordering rate of Al,Si and the ease of dislocation creep (hydrolytic weakening) (e.g., Yund 1983; Tullis 1983). It is not known how water alters these kinetics, but hydrolyzation of Si-O bonds has been suggested (Donnay et al. 1959; Griggs 1967). We will continue to refer to this as the effect of the impurity water, without knowing the exact nature of the chemical species or the mechanism involved. Furthermore, the effect is more pronounced at high confining pressure (e.g., Yund and Tullis 1980), presumably because this increases the concentration of water or affects the type of defect which is formed. Thus the rate of a given solid-state process is often a minimum in a dry crystal and a maximum in a crystal containing a small amount of water.

This study reports on the diffusivity of NaSi – CaAl in peristerite at 1.5 GPa with a small but unknown amount of water present in the crystal. The ‘average’ diffusivity can be estimated from the time required to homogenize the lamellar microstructure of the peristerite. This method has been previously used for oxides (Price 1981), pyroxenes (Brady and McCallister 1983), and alkali feldspars (Brady and Yund 1983), in addition to the recent data for NaSi – CaAl diffusion in Ca-rich plagioclase (Grove et al. 1984). A comparison of this method with calculations based on independent tracer diffusion data for the alkali feldspars has demonstrated the potential of this method (Brady and Yund 1983). Preliminary results of the present study were reported earlier (Yund 1984).

Experimental Procedure

A peristerite from Hybla, Ontario (Brown Univ. No. 2583) was selected because of its uniform lamellar spacing. This large crystal (50 × 50 × 10 mm) is clear to white and displays blue iridescence on the (010) cleavage. (See Ribbe 1983, for a review of schiller in feldspars.) An electron probe analysis gave a bulk composition of $Ab_{91.9}An_{6.7}Or_{1.4}$ with no detectable Fe, Mg, Ba, Ti, and Mn. *Transmission electron microscopy* (TEM) examination of the sample shows

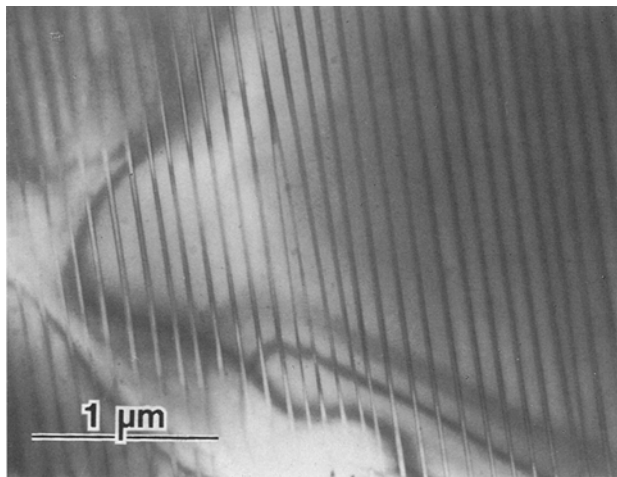


Fig. 1. Bright-field TEM micrograph of a (001) foil of the peristerite used for the homogenization experiments. The coarser lamellae are the Ab-rich phase

that there is only one orientation of exsolution lamellae which is approximately parallel to (041) when indexed using $c = 14 \text{ \AA}$. The only other microstructural feature present is twinning on the Albite law. These twin lamellae are often only several times wider than the exsolution lamellae, although not nearly as abundant or as uniformly spaced. In a (100) foil these twin lamellae make an angle of about 13° with the exsolution lamellae. Occasionally micron size areas of incipient alteration are observed in the crystal.

A typical bright-field (001) micrograph is shown in Figure 1. The lamellar spacing is fairly uniform and measurements of numerous micrographs yield an average spacing from the center to center of adjacent (unlike) lamellae of $554 \pm 77 \text{ \AA}$. This value was used for the calculation of the diffusion coefficients described below. Further evidence that this spacing is uniform throughout the crystal is provided by TEM observation on samples which did not homogenize after heating. These samples had a lamellar spacing consistent with the above value.

The composition of the more An-rich set of lamellae can be estimated from the microstructural observations. The ratio of the width of the albite to the oligoclase lamellae is 2.9. Given the bulk composition and assuming that the Na-rich lamellae are Ab_{100} , the oligoclase lamellae are about An_{26} . This is consistent with previous estimates ($\text{An}_{25 \pm 5}$) for the composition of the oligoclase lamellae in peristerites (e.g., Smith 1974).

The homogenization experiments were done either by annealing fragments of this sample in air, or at a high confining pressure with added water. A core about 3 mm diameter by about 3 mm long (about 50 mg) was placed in a Pt tube along with about 50 mg of powdered peristerite (5–50 μm diameter), some above and some below the core to prevent rupture of the Pt tube by the sharp ends of the core when it was pressurized. About 0.8 mg (0.5–1.2 wt.%) water was added to the capsule before sealing. The samples were annealed in a solid-medium apparatus of a modified Griggs-type using a sample assembly similar to that described by Kronenberg and Tullis (1984). The pressure uncertainty is less than $\pm 100 \text{ MPa}$, and the temperature uncertainty is believed to be less than $\pm 10^\circ \text{ C}$. Samples were first taken to about 300 MPa, then to about 300 $^\circ \text{C}$, then to 1.5 GPa, and finally to the desired tempera-

ture. Many experiments were pretreated at 1.5 GPa and 800 $^\circ \text{C}$ for about 12 h before proceeding to the desired homogenization test temperature. This procedure was used to allow water to penetrate into the sample and is discussed in the next section. The time required to raise the temperature from 800 $^\circ \text{C}$ to the run temperature, or to cool the sample through this interval, was only a few minutes.

At the end of each experiment two polished sections were made of the sample, each normal to the axis of the tube with one passing through the single crystal core, yielding an (001) section, and another through the compacted and sintered powder. These sections were thinned for TEM study by ion bombardment sputtering. Samples were examined on either a JEM-7 or a Philips 420 instrument.

The value for the interdiffusion coefficient at a particular temperature can be obtained from observation of the homogenization of exsolution lamellae. This procedure is discussed by Brady and McCallister (1983), and applications of this method are described by Brady and Yund (1983) and Grove et al. (1984). In order to homogenize a lamellar microstructure, diffusion must occur normal to the boundaries and this involves exchange of the chemical species between the lamellae. If the rate of change of the average compositional difference between the two sets of lamellae is known, from X-ray diffraction data for example, then a curve can be fit to the data assuming a constant value of \bar{D} for the compositional interval represented by the lamellae. However, it is difficult to determine accurately the compositions of the phases in a peristerite from diffraction data. An alternate and almost as accurate estimate of \bar{D} can be obtained from a series of experiments which determines the time (t_h) required to homogenize the lamellar microstructure:

$$\bar{D} t_h / L^2 = 0.5, \quad (1)$$

where L is equal to the spacing from center to center of adjacent (unlike) lamellae.

The value of \bar{D} obtained by either of the above procedures is only an estimate of the 'average' diffusion coefficient for the compositional interval between the lamellae. At high temperature where the solid solution may be nearly ideal, this is a good approximation, but for temperatures near a miscibility gap, a plot of \bar{D} versus composition will have a pronounced minimum. This is illustrated for Na–K interdiffusion coefficients in alkali feldspars at 600 $^\circ \text{C}$ in Brady and Yund (1983). In the aforementioned study it was observed that the 'average' value for \bar{D} determined from homogenization data was approximately equal to the minimum in the predicted value of \bar{D} for a given compositional interval. The uncertainty in \bar{D} will also depend on how close the homogenization time is bracketed and on the constancy of the lamellar spacing in the samples used for the homogenization experiments.

Experimental Results

The experimental results are listed in Table 1 and the data plotted on a graph of $\log \bar{D}$ versus $1/T(\text{K})$ in Figure 2. Also shown on the ordinate of this graph are the homogenization times corresponding to the $\log \bar{D}$ values as determined from equation (1). It should be remembered that these homogenization times are only valid for the given lamellar spacing (554 \AA). The uncertainty in each \bar{D} value due to the estimated uncertainty in the lamellar spacing

Table 1. Experimental Data for Peristerite Homogenization. All experiments at 1.5 GPa, water added and preheated at 800 °C except where noted

T (°C)	t (h)	TEM observation	Comment
1,100	2,352.0	sharp lamellae	1 atm., no water or preheat
1,100	219.0	sharp lamellae	no water, no preheat
1,100	219.0	no lamellae	ran with above sample
1,100	4.0	lamellae visible	no preheat
1,100	2.5	no lamellae	
1,050	12.0	no lamellae	
1,050	5.5	no lamellae	
1,050	2.0	faint lamellae	
1,000	40.0	no lamellae	no preheat
1,000	11.5	no lamellae	no preheat
1,000	1.5	lamellae visible	
950	73.7	no lamellae	
950	48.0	lamellae visible	
950	28.0	lamellae visible	
900	300.0	no lamellae	
900	137.0	faint lamellae	
900	48.0	lamellae visible	
900	264.5	lamellae visible	0.5 GPa

(± 77 Å) is shown by the error bar in the upper right-hand corner of Figure 2.

There was no change in the lamellar microstructure when the peristerite was annealed in air at 1,100 °C for 98 days. Thus $\bar{D}(\text{dry}) < 1.8 \times 10^{-18}$ cm²/s. This result is shown by the large, open arrow on Figure 2. Inasmuch as 1,100 °C is very close to the melting temperature of this sample, higher homogenization temperatures could not be used and a lower limit for the diffusion coefficient under these conditions could not be obtained.

The effect of adding water on the homogenization rate was determined at high confining pressure. Two samples were sealed in Pt tubes, one with 0.8 weight percent water added, and placed side by side in the solid medium assembly. These were annealed at 1.5 GPa and 1,100 °C for 10 days. The peristerite with the added water homogenized completely, whereas the sample without added water showed little if any evidence of lamellar homogenization. Thus this set of experiments demonstrated that when water is available it penetrates the crystal and increases the rate of NaSi–CaAl interdiffusion. The concentration of water, its speciation, or how this impurity alters the mechanism of the interdiffusion is not known.

This set of experiments also provides two other important observations: (1) the effect of a high confining pressure alone is not the reason for the increased homogenization rates, and (2) there is not sufficient ‘water’ present in the initial peristerite in the form of fluid inclusions, etc. to give rise to this faster rate. In addition, an experiment at 0.5 GPa and 900 °C with water added did not produce homogenization of the lamellae, although at 1.5 GPa for the same time with water added the lamellae would have been homogenized. (See Table 1.) Therefore, the remaining experiments were done with water added at 1.5 GPa.

There was no evidence of melting in any of these experiments; homogenization occurred by solid state diffusion and not by a dissolution-precipitation process. The absence of melting also indicates that the small quantity of water was either absorbed by the peristerite, or present only

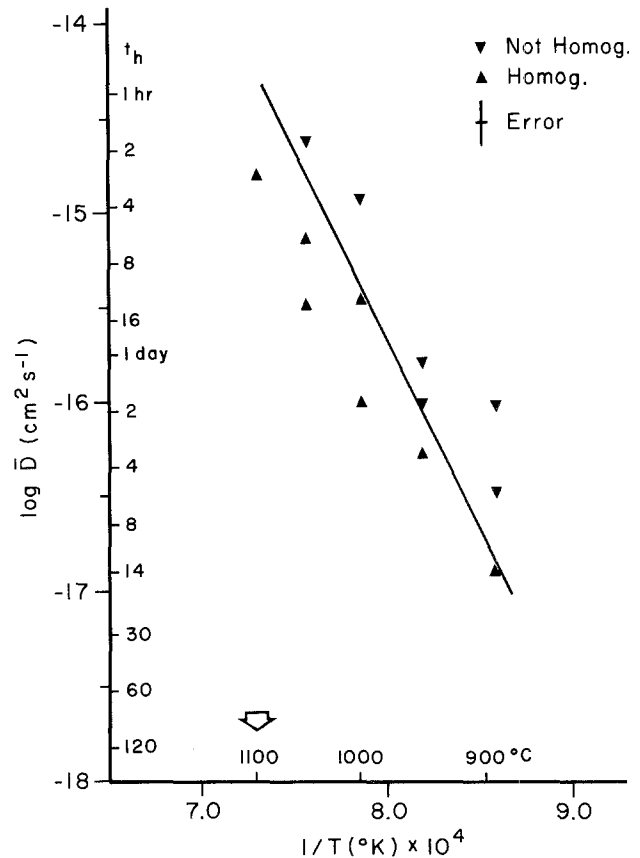


Fig. 2. Arrhenius plot showing the results of annealing experiments at 1.5 GPa with water added (solid arrows), and one experiment at 1,100 °C in air (large, open arrow). The equation for the line is given in the text

as a grain boundary film, and $P(\text{H}_2\text{O})$ was less than the confining pressure. Dislocations were occasionally observed which were not present in the initial sample. These are undoubtedly due to plastic deformation of the peristerite during the experiment. There was no noticeable difference in the degree of homogenization near dislocations, but these areas were not common and were easily avoided when making the TEM observations.

Although the above experiments demonstrated the importance of ‘water’ in peristerite on NaSi–CaAl interdiffusion rates, they do not define the time required for water to penetrate the crystal. Unless this penetration is rapid compared to the homogenization rate, the time observed for homogenization would be at least partially dependent on the rate of ‘water’ diffusion in peristerite. Thus lack of homogenization could be due to slow water penetration but would be interpreted as interdiffusion coefficients for NaSi–CaAl which are too low.

To obtain information about these relative rates, two experiments were done at 1.5 GPa and 1,100 °C. One sample was first preheated at 800 °C and 1.5 GPa for 12 h before being annealed at 1,100 °C for 3 h. This sample was completely homogenized. The experiment which was not preheated at 800 °C was annealed at 1,100 °C for 4 h and did not completely homogenize. In other experiments (such as at 1,000 °C for 7 days), it was observed that this preheat had no effect on the microstructure. It appears that the water has penetrated throughout the crystal during this pre-heat treatment because there was never any observable dif-

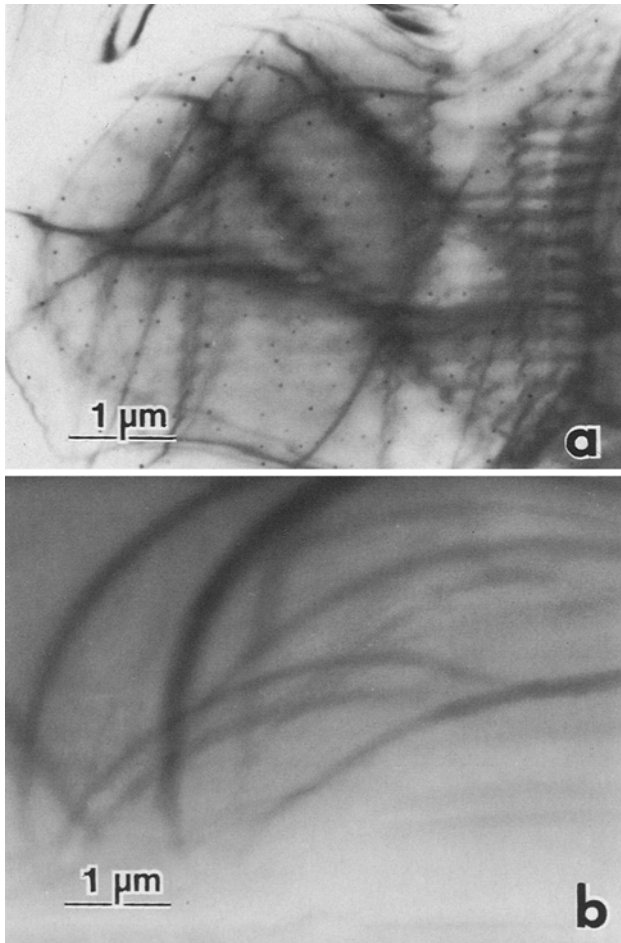


Fig. 3a, b. Comparison of peristerite after being annealed at 1.5 GPa, 950 °C, with water added: (a) for 48.0 h and (b) for 73.7 h. Lamellae (horizontal) are faintly visible in (a) but not in (b). Both micrographs are bright-field TEM images of (001) foils. (The dark curved lines are bend contours)

ference in the degree of homogenization between the edge of a crystal and its center, or between the foils prepared from the single crystal and the powder. (There was some cracking of the single crystal during pressuration, and the effective diffusion distance for water was probably only about 1/10 of the sample radius.)

Thus from these observations it appears that water penetration into peristerite is fast compared to the homogenization rate at 800 °C and 1.5 GPa. This preheat treatment was used for subsequent experiments. Some earlier experiments which had been done with no preheat and which indicated complete homogenization are also valid. (See Table 1.) These experiments also demonstrate that there was a difference between preheated and not preheated samples only for experiments in which the homogenization times were less than about 12 h.

The sharpness of the lamellae in the TEM decreased when samples were annealed above the line shown in Figure 2, but the lamellae were still visible. For example, an electron micrograph of a (001) section taken from a sample annealed at 950 °C for 48.0 h is shown in Figure 3a. The lamellae are faint but clearly visible. The micrograph shown in Figure 3b is from a sample annealed at 950 °C for 73.7 h (below the line in Fig. 2). No lamellae are visible in this

sample for any tilt angle and imaging conditions. Only rarely were lamellae faintly visible in samples annealed at conditions below the line in Figure 2. These corresponded to areas in the crystal in which the lamellar spacing was slightly larger than average, and these were ignored for the purpose of deciding whether the sample had homogenized or not.

The Al,Si distribution in the peristerite was essentially disordered at the end of all experiments including those at the lowest temperatures for the shortest annealing times. This distribution was estimated from the separation of the (131) and (1 $\bar{3}$ 1) X-ray reflections (e.g., Kroll 1983).

The bracketing experiments in Figure 2 are from 900° to 1,050 °C. The homogenization rate at 1,100 °C is so rapid, less than one hour, that an upper limit could not be accurately determined. A straight line can be drawn through these data which separates the homogenized samples from the not completely homogenized samples. This is the best estimate for the ‘average’ NaSi–CaAl interdiffusion for these conditions. The straight line shown in Figure 2 can be expressed by the Arrhenius relation:

$$\bar{D} \text{ (cm}^2\text{/s)} = \bar{D}_0 \exp(-Q/RT) \quad (2)$$

where R is the gas constant and T is °K. The pre-exponential factor (\bar{D}_0) is equal to 18^{+108}_{-15} cm²/s and the activation energy (Q) is equal to 97 ± 5 kcal/mol (405 kJ/mol). The quoted errors are one standard deviation and include the uncertainties in the lamellar spacing (± 77 Å) and temperature (± 10 °C). Although the data appear to define a straight line on the Arrhenius plot shown in Figure 2, the relation may not be strictly linear because the values are an ‘average’ of different \bar{D} ’s as discussed earlier. In addition, even \bar{D} values for a specific composition will not be linear on an Arrhenius plot unless the solid solution is ideal. See, for example, Christoffersen et al. (1983) and below.

Discussion of Results

The \bar{D} value at 1,100 °C for the high pressure, water added experiments is at least three orders of magnitude larger than that for the dry experiment at atmospheric pressure. The effect of a high confining pressure without adding water was not systematically investigated, but the 1.5 GPa experiment with no water added produced no indication of homogenization. An increase in confining pressure, independent of water, is expected to decrease the diffusivity, but only slightly. For comparison, Na–K interdiffusion in alkali feldspar was found to be independent of pressure between 500 MPa and 1.5 GPa within a factor of about $2\bar{D}$ (Christoffersen et al. 1983). In a peristerite annealed at 0.5 GPa and 900 °C for 264.5 h the lamellae were not homogenized. At 1.5 GPa the lamellae would have homogenized in this time; the difference is believed to be due to either a lower concentration of ‘water’ in the peristerite or to a lower rate of water diffusion into the sample at the lower pressure. It will be necessary to carefully evaluate the effect of confining pressure on NaSi–CaAl interdiffusion in plagioclase when water is present, but the data in equation (2) probably represent a maximum value when water is present in rocks at depths less than about 42 km (1.5 GPa).

The 1.5 GPa line (equation 2) and the ‘dry’ upper limit for \bar{D} at 1,100 °C are shown in Figure 4 along with the

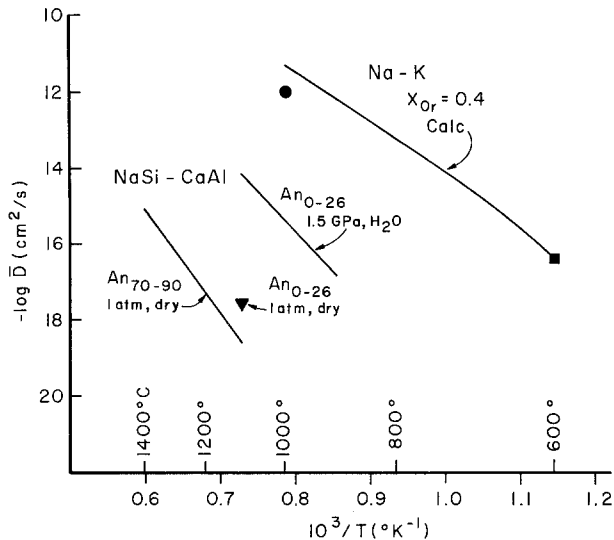


Fig. 4. Comparison of interdiffusion data for plagioclase and alkali feldspars. The Na–K curve for Or_{40} is calculated from tracer diffusion data (Foland 1974 and Kasper 1975) using thermodynamic data for the disordered, coherent solvus (Sipling and Yund 1976). The square is from lamellar homogenization (Brady and Yund 1983) and the circle is from diffusion couple experiments Christoffersen et al. (1983). The CaAl–NaSi data are for lamellar homogenization experiments for the intervals from approximately An_{70} – An_{90} (Grove et al. 1984) and approximately An_0 – An_{26} (this study) at the conditions shown

recent results for NaSi–CaAl interdiffusion from Grove et al. (1984). Their data are for diffusion at atmospheric pressure (dry) in the Huttenlocher interval (approximately An_{70-75} to An_{88-90}) and were also obtained by the lamellar homogenization method.

The ‘dry’ limit for peristerite is consistent with the data for the Huttenlocher interval. Grove et al. (1984) could do experiments at higher temperatures because of the higher melting point of Ca-rich plagioclase; in addition, the smaller spacing of the Huttenlocher lamellae (175 Å compared to 554 Å) facilitated rapid homogenization. Given the available results it is not possible to determine whether there is a difference in the interdiffusion rate of NaSi–CaAl in Na- and Ca-rich plagioclases. The temperatures at the crests of the peristerite and Huttenlocher miscibility gaps are not well known, but the Huttenlocher gap may be hundreds of degrees higher than the peristerite gap (e.g. McConnell 1974); thus Ca-rich plagioclases are expected to show a significant departure from ideality at a higher temperature than Na-rich plagioclases. This would lead to downward curvature of the $\log \bar{D}$ versus composition plot near the miscibility gap, and curvature in an Arrhenius plot (see below).

The activation energy for the dry Huttenlocher data is 123 ± 5 kcal/mol (Grove et al. 1984), which is significantly larger than the value of 97 ± 5 kcal/mol from the present study for peristerite at 1.5 GPa with water added. This difference may reflect the difference in the Q 's for interdiffusion in Ca- and Na-rich plagioclases, but it is more likely that the lower Q for peristerite is due to ‘water’ in the crystal which modifies the diffusion mechanism. The lower value is consistent with the decrease in the activation energy of Al, Si disordering in albite in the presence of water (Yund and Tullis 1980). They found that the activation energy

for dry samples annealed in the atmosphere was about 80 kcal/mol and Q decreased to about 67 kcal/mol when the disordering was done at 1.0 GPa with water added. The larger activation energy for interdiffusion than for disordering, although both involve migration of Al and Si, may be related to the coupled migration of NaSi and CaAl, or it may reflect that the exchange mechanism for disordering is different than the diffusion mechanism.

Interdiffusion data for Na–K in alkali feldspar are shown on Figure 4 for comparison. The line was calculated using the relation between \bar{D} and the tracer diffusion coefficients and thermodynamic data for the solid solution (Brady 1975). The tracer diffusion data were determined from experiments done at 200 MPa water pressure (Foland 1974 and Kasper 1975), and the thermodynamic factor based on Margules parameters (Thompson and Waldbaum 1969) for the disordered, coherent solvus (Sipling and Yund 1976). The curve in Figure 4 was calculated for an intermediate composition (Or_{40}) which shows pronounced curvature at low temperature (coherent solvus crest at 573 °C) due to non-ideality. The experimental point at high temperature (shown by a solid circle) is from a diffusion couple experiment at 1.5 GPa with no water added (Christoffersen et al. 1983). The experimental point at 600 °C (solid square) is from homogenization experiments which were done in air (Brady and Yund 1983). There is fair to good agreement of these points with the calculated curve even though some of the data are from ‘dry’ experiments and others from hydrothermal experiments. Thus the rate of tetrahedral ion migration is affected by the presence of water in a crystal whereas the alkali diffusion rate is not (e.g., Yund 1983). Even so, the rate of NaSi–CaAl interdiffusion is several orders of magnitude slower, and confirms what has been qualitatively recognized for many years from the large difference in the kinetics of exsolution for these two feldspar series.

Caution must be exercised if the interdiffusion data for either the peristerite or Huttenlocher intervals are extrapolated to lower temperature. An Arrhenius relation using ‘average’ \bar{D} values will not be strictly linear. Even \bar{D} values for a specific composition do not yield a linear Arrhenius plot if the solid solution departs from ideality such as near a miscibility gap. See, for example, the curvature in the Na–K interdiffusion data at lower temperature shown in Figure 4. There is also the possibility of a change in the diffusion mechanism which could cause a change in slope of the Arrhenius relation. At the present time we cannot evaluate these possibilities for plagioclase.

The dry and water added experiments probably provide lower and upper bounds for diffusion rates in plagioclase. What is clearly needed for comparison are dry diffusion data for peristerite, which could be obtained by using a sample with a smaller lamellar spacing in order to obtain a bracket for 1,100 °C. This together with the high pressure data for a Huttenlocher sample with water added, preferably the same material used by Grove et al. (1984), would provide useful limits for diffusion in the Na- and Ca-rich plagioclases. The effect of varying the confining pressure when water is present also needs to be investigated. Samples containing Boggild lamellae could be homogenized in order to obtain data for the intermediate plagioclases.

A more fundamental problem is to determine the nature of the ‘water’ impurity and the mechanism of NaSi–CaAl interdiffusion. To help solve this problem we need informa-

tion about the speciation of 'water' and the concentrations of the different species in feldspar as a function of confining pressure and temperature.

The above discussion focuses on 'water' as the principal impurity responsible for extrinsically controlled diffusion in plagioclase. Substitution of other cations or minor departure from stoichiometry may also be important (e.g., Lasaga 1981). One way to assess whether other impurities are important, without attempting to evaluate the specific defect, would be to do homogenization experiments on other peristerites or Huttenlocher samples in order to determine the variability in \bar{D} for different samples at the same experimental conditions.

Progress has been made on providing fundamental diffusion data for plagioclase, and although additional studies are required, the potential applications justify the effort. The available data for the peristerite and Huttenlocher intervals provide a beginning, but extrapolation of these data to lower temperatures and their application to other compositional intervals, or for crystals with intermediate water contents, should be done cautiously until additional questions have been answered.

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