American Mineralogist, Volume 68, pages 1126–1133, 1983

Inter-diffusion of K and Na in alkali feldspars: diffusion couple experiments

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Abstract

Inter-diffusion of K and Na in alkali feldspar was studied at 5 and 15 kbar confining pressure using diffusion couples annealed at 900° and 1000°C. Concentration profiles were measured using an electron microprobe and results were obtained for inter-diffusion in four crystallographic directions at 1000°C. Inter-diffusion coefficients ($\overline{D}$) were calculated as a function of composition from Or_{10} to Or_{80}. Alkali inter-diffusion is about 10 times faster normal to (001) than normal to (010), and the rate is intermediate parallel to [100] and [011]. A trace amount of water appears to have no influence on the inter-diffusion rate, and within experimental error there is no difference in $\overline{D}$ between 5 and 15 kbar. There is agreement between the $\overline{D}$ values determined experimentally and those calculated from published self-diffusion coefficients. The nonideality of the alkali feldspar binary at 1000°C has a significant effect on $\overline{D}$, and this effect becomes increasingly important at lower temperature.

Introduction

The self-diffusion coefficients (D*) for Na and K in alkali feldspar have been reported in previous studies. These results are mostly tabulated by Freer (1981) and have been discussed and evaluated by Yund (1983). Most of the self-diffusion studies used the so-called integrative method in which either Na or K isotopes were exchanged between a collection of grains and a fluid in chemical equilibrium with the feldspar. The bulk isotopic exchange rate (Lin and Yund, 1972; Foland, 1974; Kasper, 1975) or the isotopic profile within a single grain (Giletti *et al.*, 1974) was used to calculate D*. Inter-diffusion coefficients ($\overline{D}$) as a function of the K/Na ratio have not been determined, although Petrović (1974) made estimates of both $\overline{D}$ and D* by measuring K/Na gradients over a limited compositional interval. Average $\overline{D}$ values have been determined at 600° and 650°C from homogenization experiments on crytoperthites (Brady and Yund, 1983).

The purpose of this study was to determine alkali inter-diffusion coefficients for a wide range of K/Na ratios from direct measurements of diffusion profiles, and to use these data to test the theoretical relation between $\overline{D}$ and D* (*e.g.*, Brady, 1975). Because alkali inter-diffusion is relatively slow and the electron microprobe was used to determine the diffusion profiles, accurate values for $\overline{D}$ could only be determined at 900° to 1000°C. In addition to direct determination of $\overline{D}$, we were also concerned with evaluating the pressure dependence of $\overline{D}$ up to 15 kbar, obtaining additional data on the anisotropy of alkali diffusion in feldspar, and further evaluating the effect of a trace amount of water on diffusion of the alkalis. These diffusion data are useful for evaluating the exsolution rates of alkali feldspars, the compositional relations between the phases in a perthite or cryptoperthite, and the preservation or lack of preservation of K/Na zoning in feldspars.

Experimental procedure

Diffusion couples were prepared by placing a K-rich and an Na-rich feldspar crystal in contact after the surfaces had been carefully polished. After annealing the couple at the desired temperature and pressure, electron probe traverses were made to determine the K and Na concentration profiles. Inter-diffusion coefficients were calculated using Wagner's (1969) method which has been applied successfully to oxides (Greskovich and Stubican, 1970), other silicates (Misener, 1974), and sulfides (Boctor and Brady, 1980).

The K-rich half of the diffusion couple was an adularia from Val Kristallina, Switzerland. (It is from the same locality, but not the same crystal, as the material used for oxygen diffusion studies by Yund and Anderson, 1974 and 1978, and Giletti *et al.*, 1978.) Its composition as determined by electron microprobe analysis was $Or_{89.6}Ab_{10.1}An_{0.3}$. The crystals were clear, colorless, and unfractured.

A low albite from Amelia Co., Virginia, was used for the Na-rich crystal and its average composition as determined by electron probe was $Or_{1.1}Ab_{98.6}An_{0.3}$. The An

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0003–004X/83/1112–1126$02.00

content was variable and ranged from 0.1 to 1.0. These Amelia crystals are not perfectly clear and the white color is due to internal cleavage cracks and occasional microscopic inclusions. (This same material was used by Kasper, 1975, for alkali self-diffusion experiments.) Because of the cleavelandite habit and the size of the Amelia crystals, samples of suitable size and quality could not be obtained for all orientations, and for one experiment a sample of polycrystalline low albite from the Hale quarry, Connecticut, was used. This has a grain size of 100–200 μm, a composition of $Or_{1.0}Ab_{96.0}An_{3.0}$, and contains less than 0.1% muscovite and garnet. (This material was used for studying the dislocation-assisted diffusion of oxygen in albite by Yund *et al.*, 1981.)

Cores with 3½ mm diameter were drilled from the crystal, and the ends were ground and polished. The final polishing was done with 1 μm diamond paste followed by 0.05 μm alumina in water. After cleaning, the cores were dried at 60°C before they were sealed in a Pt tube. The diffusion couples were then annealed in a modified Griggs apparatus using $CaCO_3$ as the confining medium. After annealing at pressure and temperature, a longitudinal section was cut and a section prepared for microprobe analysis. The samples usually showed a few extension fractures (normal to the sample core) due to unloading at the end of the experiment, but there was no evidence of any microfracturing normal to the couple interface. Concentration profiles for Na and K were determined on the electron microprobe using a spot size of 1 to 3 μm, with 5 μm steps between each spot. The average error in spot location was 1.5 μm. For each analysis intensities were simultaneously collected for Na, Ca, and K; Si and Al were determined by stoichiometry for use in the Bence and Albee (1968) corrections. Na volatilization was avoided by using a low beam current and relatively short counting times. Two to four traverses were made on each sample and for two experiments traverses were made on different polished sections to further check for reproducibility of the measured profiles.

The inter-diffusion coefficient as a function of composition, $\overline{D}(N^*_{Or})$, was determined from Wagner's (1969) relation, which when written for alkali feldspar is:

$$\overline{D}(N^*_{Or}) = (N^+_{Or} - N^-_{Or})\ Vm^*/2t\ (\partial N_{Or}/\partial x)_{x=x^*} \cdot \left[(1 - Y^*) \int_{-\infty}^{x^*} Y/Vm\ dx + Y^* \int_{X^*}^{+\infty} (1 - Y)/Vm\ dx\right] \tag{1}$$

where N^+_{Or} and N^-_{Or} are the initial concentrations (in mole fraction Or) in the couple at time (t) = 0; N^*_{Or} is the concentration corresponding to the inter-diffusion coefficient; Vm* is the molar volume of the solid-solution for this composition; Y is an auxiliary compositional variable defined by $Y = (N_{Or} - N^-_{Or})/(N^+_{Or} - N^-_{Or})$; and Y* is the Y value corresponding to N^*_{Or}.

Although Wagner's method includes a correction for the change in the molar volume due to the compositional change in the couple, this correction is small and the effect on $\overline{D}$ is less than one percent. Computational errors in evaluating Equation (1) are small and their effect on $\overline{D}$ is less than 1 percent. The error in $\overline{D}$ arises primarily from the uncertainty in the shape of the profile. Even a small error in the profile has a large effect near the ends of a profile, and for this reason $\overline{D}$ was evaluated only between Or_{10} and Or_{80}. The average analytical error of a probe analysis was 0.015 mole fraction Or, and this uncertainty was used to define a maximum and a minimum curve for each profile. The difference between these profiles corresponds to a 15 percent error in $\overline{D}$, and this is a measure of how well the shape of a given profile is known. The absolute error and reproducibility of $\overline{D}$ are larger. $\overline{D}$ values for different profiles from the same experiment, or for experiments that differed only in their annealing times, are reproducible to within a factor of about 2. There are no data with which we can compare our $\overline{D}$'s directly, but in the next section they are compared with $\overline{D}$'s calculated from self-diffusion data.

Experimental results

Four experiments were done to determine the crystallographic anisotropy of the inter-diffusion rate at 1000°C and 15 kbar, for times of 5.9 to 6.8 days. Diffusion was measured normal to (001), normal to (010), parallel to [100], and approximately (±5°) parallel to [011]. An additional experiment was done at these conditions normal to (001) for 18.2 days to demonstate that the concentration profiles were diffusion controlled. The $\overline{D}$ values from this experiment were the same within a factor of two as those for the shorter experiment at the same conditions. From this we conclude that the profiles are due to diffusion and that the transfer of ions across the interface of the bi-crystal is fast compared to the diffusion rate. Typical profiles at 1000°C for different crystallographic directions are shown in Figure 1.

In addition to the above experiments, one was done at 1000°C and 5 kbar to determine the effect of pressure; one at 900°C and 15 kbar to partially establish the temperature dependence of $\overline{D}$; and one at 1000°C and 15 kbar in which the sample was vacuum dried at 600°C before being sealed in the Pt capsule. The latter experiment was done to determine whether $\overline{D}$ is a function of the trace amount of water in the crystal.

The diffusion rate normal to (001) is the same, within the limits of the errors, at 15 and 5 kbar confining pressure. The similarity in these rates can be seen in Figure 1 by comparing the concentration profiles normal to (001) for the different pressures. Because the higher confining pressure ensures good contact between the two halves of the bi-crystal, and the pressure dependence of $\overline{D}$ is so small, the rest of the experiments were done at 15 kbar.

The variation in $\overline{D}$ as a function of composition for

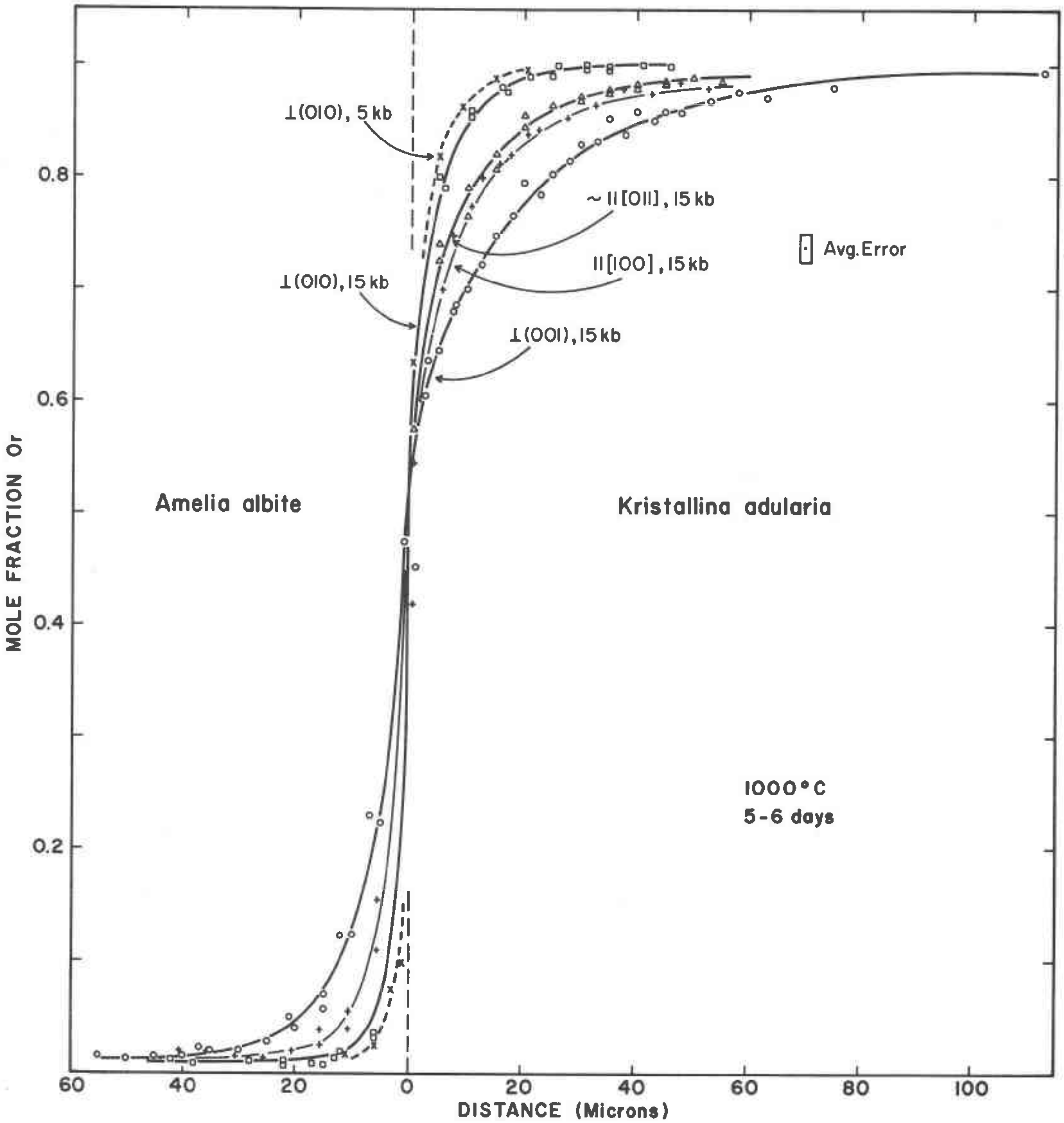

Fig. 1. Concentration *vs.* (mole fraction Or) *vs.* distance (microns) profiles for alkali interdiffusion experiments at 1000°C. All samples were pre-dried at 60°C. The diffusion direction and confining pressure are shown for each curve. The vertical dashed line indicates the inferred position of the couple interface.

diffusion in the different directions at 1000°C and 15 kbar is shown on Figure 2. The microprobe data are tabulated by Christoffersen (1982). (The calculated curves on Figure 2 will be discussed in a later section.) The experiment to measure diffusion parallel to [011] was done with an adularia-Hale albite polycrystal couple because it was not possible to core the Amelia albite crystals with this orientation. Hence the diffusion profiles were only measured in the single crystal adularia, and $\overline{D}$ values for this experiment are shown on Figure 2 between 0.6 and 0.8 mole fraction Or.

As shown on Figure 2, $\overline{D}$ as a function of composition goes through a minimum at N_{Or} equal to about 0.4. There is little if any significant difference in the shapes of the experimental curves of $\overline{D}$ versus composition for any of these four crystallographic directions, but there is a significant difference in the values of $\overline{D}$ for a given composition. Diffusion normal to (001) is approximately 12 times faster than that normal to (010), but only about 6 times faster than that parallel to [100]. There is little if any difference in $\overline{D}$ parallel to [100] and [011].

The difference in $\overline{D}$ as a function of composition for diffusion normal to (001) at 1000°C and 900°C is shown on Figure 3. Again there is little if any difference in the shape

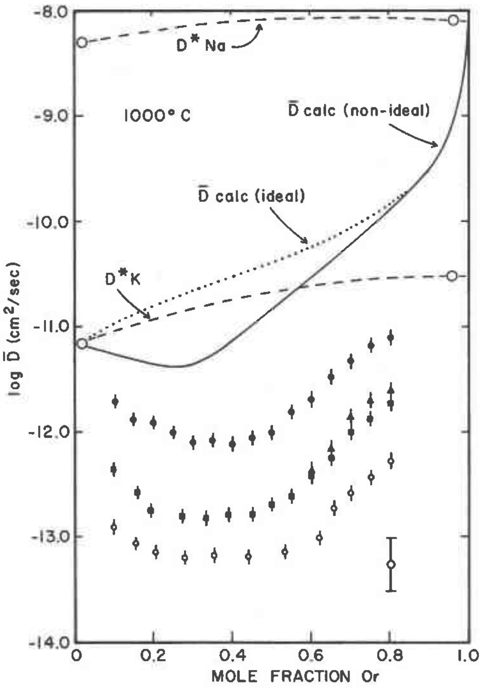

Fig. 2. Log $\bar{D}$ vs. mole fraction Or across the alkali feldspar binary. The experimental data are as follows: solid dots = diffusion normal to (001); solid squares = diffusion parallel to [100]; solid triangles = diffusion approximately parallel to [011]; and open circles = diffusion normal to (010). All for 1000°C and 15 kbar. For these data the absolute (separate error bars) and the relative error (bars on points) are indicated (see text). The solid and dotted curves are for $\bar{D}$ calculated from self-diffusion coefficients that have been extrapolated across the binary (dashed curves) from data for the near end-members (small open circles).

of the two data sets, but $\bar{D}$ at 900°C is about an order of magnitude smaller than that at 1000°C for a given composition.

All but one of the experiments were done with crystals that had been air dried at 60°C before being sealed in Pt capsules. In order to determine whether a trace amount of water affects the alkali inter-diffusion rate in feldspar, as it does oxygen and silicon/aluminum diffusivities (see Yund, 1983), we measured diffusion normal to (001) in a sample that was vacuum dried for about 12 hours at 600°C before being annealed. The concentration profile for this experiment at 1000°C and 15 kbar was the same within experimental error as that for the sample which had been air dried at 60°C. The water concentration in the original sample and after heating at 600°C is not known, but the water content is probably about as low as can be achieved for this material. Even if water was lost only from the outer region of the crystal, this should have produced a change in the profile if the water concentration had any effect on $\bar{D}$.

Interpretation

Effect of confining pressure

The dependence of D ($\bar{D}$ or D*) on temperature and pressure is given by the relation (Lazarus and Nachtrieb, 1963):

$$D = D_0 \exp(-Q/RT) \cdot \exp(-P\Delta V^*/RT) \qquad (2)$$

where D_0 is a constant, Q is the activation energy, R is the gas constant, T is in °K, P is pressure, and ΔV^* is the activation volume. The activation volume is small for oxides; it is approximately 5.5 cm³/mole for Mg–Fe interdiffusion in olivine (Misener, 1975). Thus between atmospheric pressure and several kbar one expects a small decrease in D with increasing confining pressure.

The difference in the self-diffusion rate of K in microcline between 0.5 and 2.0 kbar water pressure was found to be less than the experimental error in the value of D* (Lin and Yund, 1972), and this is apparently true for $\bar{D}$ between 5 and 15 kbar as well. Figure 1 shows that the profile for the 5 kbar experiment was slightly shorter than that for 15 kbar. This difference is almost within the experimental uncertainty. If this difference is real, it may represent slightly better contact of the couple at the higher pressure. The activation volume is apparently very small for alkali interdiffusion and the pressure dependence can be ignored for geological applications in the crust. The temperature dependence of this inter-diffusion is discussed in a later section.

Diffusion anisotropy

The anisotropy of Na self-diffusion in albite at 595°C and 1 atmosphere was investigated by Bailey (1971). He reported that D^*_{Na} normal to (010) was 0.1 to 0.6 as large

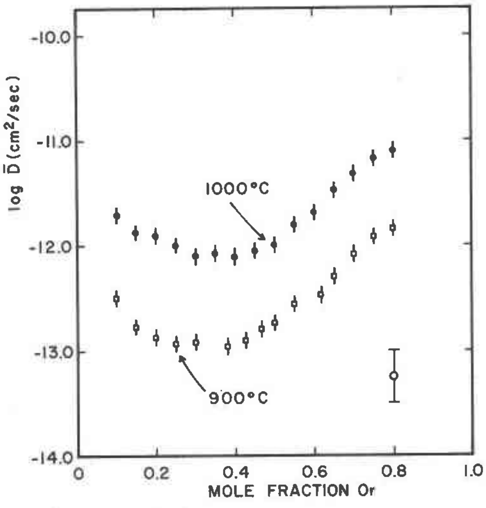

Fig. 3. Log $\bar{D}$ *vs.* mole fraction Or for diffusion normal to (001) at 15 kbar. Solid circles = 1000°C, and open squares = 900°C. Errors indicated as in Fig. 2.

as that for diffusion normal to (001), but within his experimental error these diffusivities could have been equal. Also in albite, Petrović (1972) found inter-diffusion normal to (010) to be slower than that normal to (001), but he believed that this difference was due to fractures and spallation of the crystal. He also reported that the alkali inter-diffusion rates in adularia normal to (001) and (110) were about equal, but diffusion normal to (120) was somewhat slower. In reference to unpublished data, he reported that inter-diffusion normal to (010) was a factor of 0.01 slower than that normal to (110) at 890° and 1000°C (Petrović, 1974). Giletti *et al.* (1974) reported that K diffusion in a low albite was ten times faster normal to (001) than normal to (010).

Our experiments have shown that alkali inter-diffusion normal to (001) is about ten times faster than that normal to (010), but that within the (010) plane diffusion is not isotropic. It is approximately 6 times faster normal to (001) than it is parallel to *a*. Diffusion parallel to *a* is about the same as that approximately parallel to [011].

Regardless of the alkali diffusion mechanism, the fact that diffusion within the (010) plane is faster than that normal to it is consistent with the feldspar structure. The alkali sites are much closer to each other in the (010) plane than they are normal to (010). Petrović (1974) suggested a vacancy mechanism as opposed to an interstitialcy mechanism for alkali diffusion because he believed the latter would only be effective normal to (001). Now that alkali diffusion has been observed to be slower parallel to *a* than approximately parallel to *c*, an interstitialcy mechanism should not be excluded. However, for a complex silicate such as feldspar it is difficult to argue convincingly for an atomic jump mechanism on the basis of the available data.

Effect of water

The rate of oxygen isotope exchange between feldspar and aqueous fluid is much faster than it is between feldspar and dry gas (*e.g.*, Yund and Anderson, 1974; Yund, 1983). This enhancement of the volume diffusion rate is believed to be related to the presence of water or its components in the feldspar structure. The diffusion rate increases at higher water pressures (Yund and Anderson, 1978), presumably because the water content of the crystal increases. Although Si and Al diffusivities are too slow to be measured by standard techniques, the rate of Si/Al disordering is strongly influenced by water pressure (Yund and Tullis, 1980). The above studies have shown that the oxygen exchange and the Al/Si disordering are solid state processes, but that their rates depend on water pressure. A trace amount of water also strongly influences the strength of feldspars in the ductile regime, the so-called hydrolytic weakening effect (Tullis and Yund, 1980; Shelton *et al.*, 1981). Thus the migration rates for these ions and the nature of other solid state processes are dependent on the presence of small amounts of water, OH^-, or H^+ in the feldspar structure.

Alkali diffusion does not appear to be dependent on the water content of feldspar, although the earlier evidence has been somewhat open to debate. Petrović (1974) and Lin and Yund (1972) found no difference in the self-diffusion rates of the alkalis when determined in hydrothermal or molten salt experiments. However, as noted by Foland (1974), the molten salts could have contained trace amounts of water. Indirect support for the idea that alkali diffusivities are independent of water content is provided by the observation that cryptoperthite lamellae coarsen at the same rate when annealed in air and at 1 kbar water pressure (Yund and Davidson, 1978).

Most of our inter-diffusion experiments were done with nominally dry samples, but the crystals contained their natural concentrations of water. There was no difference in the inter-diffusion rate when the couple was vacuum dried at 600°C before the diffusion anneal. The loss of water due to drying at 600°C does have a pronounced effect on the plastic yield strength of polycrystalline feldspar aggregates (Tullis and Yund, 1980; Shelton *et al.*, 1981). Thus it appears that a trace amount of water does not significantly alter the alkali diffusion rate. Furthermore, as discussed in the next section, our inter-diffusion rates are in reasonably good agreement with those calculated from the self-diffusion data that were determined hydrothermally. Thus it appears that alkali diffusion, unlike that of oxygen and probably Si/Al, is not affected by the water content of the feldspar crystal. This makes the geologic application of the alkali diffusion data more straightforward than that for the other major ions in feldspars. However, Mardon and Yund (1981) have suggested that the alkali interdiffusion rates may depend on the anorthite content of the feldspar if more than several percent An are present.

Calculation of $\overline{D}$ from D^*_K and D^*_{Na}

The relation between the self-diffusion and inter-diffusion coefficients for charged cations in binary ionic crystals differs from the standard Darken relation for metallic crystals (Darken, 1948) because of charge balance effects (Manning, 1968; Brady, 1975). For inter-diffusion of monovalent alkali ions in feldspars the relation between $\overline{D}(N_{Or})$ and the self-diffusion coefficients for the same composition, $D^*_K(N_{Or})$ and $D^*_{Na}(N_{Or})$, is given by:

$$\overline{D}(N_{Or}) = \frac{D^*_K(N_{Or})D^*_{Na}(N_{Or})}{N_{Or}D^*_K(N_{Or}) + (1 - N_{Or})D^*_{Na}(N_{Or})} \cdot [1 + (\partial \ln \gamma_{Or}/\partial \ln N_{Or})] \qquad (3)$$

where γ_{Or} is the activity coefficient for the Or component in the solid solution. If the binary is an ideal solution, then the thermodynamic term in brackets equals one and the inter-diffusion coefficient is a function only of composition and the self-diffusion coefficients for that composition.

Although there is no reason to doubt the validity of equation (3), it has not been verified for any binary solid solution. We will use it to calculate inter-diffusion coefficients and compare these with the experimentally determined values. This will provide both a comparison of the agreement between the different experimental data as well as an approximate test of equation (3).

The most accurate and internally consistent sets of self-diffusion coefficients appear to be those of Foland (1974) and Kaspar (1975). Their data are for K and Na self-diffusion in an orthoclase (Or_{94}) and a low-albite (Or_1). (See Yund, 1983, for a discussion and evaluation of the alkali self-diffusion data for feldspar.) In order to use these data we must make several assumptions or approximations which include the following. (1) Foland (1974) and Kasper (1975) measured the bulk or non-directional exchange rate of K and Na isotopes and calculated self-diffusion coefficients assuming either that diffusion is isotropic (spherical diffusion model) or that diffusion in the (010) plane is infinitely fast compared to that parallel to *b* (cylindrical diffusion model). We have calculated interdiffusion coefficients using their data for the cylindrical diffusion model because this most closely approximates the observed diffusion anisotropy, and these calculated coefficients should correspond most closely to our diffusion data normal to (001). (2) The self-diffusion coefficients are only known for the two near end-member compositions, and for the intermediate compositions across the binary we will assume that both the K and Na self-diffusion coefficients are linear functions of composition. Hence the curves for these self-diffusion coefficients appear as slightly curved lines on the log $\overline{D}$ *vs.* composition plot on Figure 2. (3) The self-diffusion data were determined at and below 800°C and must be extrapolated to 1000°C using Foland's (1974) and Kasper's (1975) data. These data were also obtained from hydrothermal experiments at 1 kbar, but as we have shown, neither the presence of water nor a high confining pressure significantly affects this diffusion rate.

For the thermodynamic term we have used activity coefficients for the sanidine–high albite series calculated from the excess Gibbs free energy data published by Waldbaum and Thompson (1969). The Si/Al distribution in the adularia half of the diffusion couple remained essentially unchanged during the time of the diffusion anneal, but the Si/Al distribution in the low-albite became almost completely disordered. This apparently has little if any effect on the diffusivities, but it would change the Gibbs free energy slightly.

Two calculated curves for $\overline{D}$ at 1000°C are shown on Figure 2. The upper curve (dotted) assumes that the solid solution is ideal and the solid curve includes the thermodynamic factor. The difference between these two curves is much larger at lower temperature where the system is more non-ideal. (See the inter-diffusion data obtained from cryptoperthite homogenization experiments, Brady and Yund, 1983.) The calculated curves for $\overline{D}$ can be compared with the experimentally determined data on Figure 2, and the best agreement is for $\overline{D}$ normal to (001), the fast direction.

Although there is up to an order of magnitude difference between the calculated curve which includes the thermodynamic factor and the experimental $\overline{D}$ values normal to (001), this difference is only slightly outside the combined experimental errors for $\overline{D}$ and D*. The shapes of the curves are very similar and the minimum in the experimental curve surely reflects the nonideality of the feldspar binary even at 1000°C. Considering that the diffusion coefficients were determined by totally different methods and at temperatures which differed by 200°C, the agreement between the calculated and experimental curves is good, providing strong support for the relation between $\overline{D}$ and D* given by Equation (3).

The increased departure of the feldspar binary from ideality at lower temperature means that an Arrhenius plot of $\overline{D}(N_{or})$ *vs.* $1/T$ will not be a straight line, especially at lower temperatures. This is shown on Figure 4 where calculated $\overline{D}$ *vs.* $1/T$ curves are shown for three compositions (0.2, 0.4, and 0.7 N_{Or}) for both 1 and 15 kbar. (The confining pressure affects the thermodynamic term, not the value of the self-diffusion coefficients.) The curvature of these lines is pronounced at low temperature. The experimental values for $\overline{D}$ for these same three compositions are shown on Figure 4 for 1000°C and 900°C. The 900°C experimental data are about as far below the

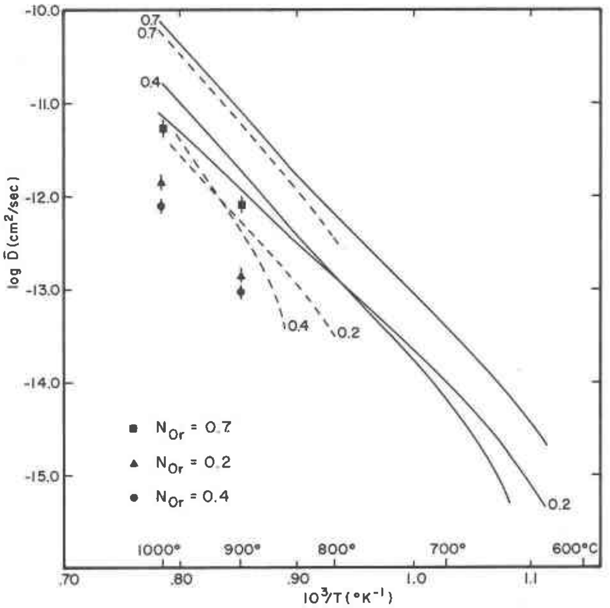

Fig. 4. Arrhenius plot of experimental and calculated $\overline{D}$'s for 0.7, 0.4, and 0.2 mole fraction Or. The solid lines represent the calculated values obtained using the thermodynamic mixing data for 1 kbar, and the dashed lines are for mixing at 15 kbar. The symbols are the experimentally determined values for these compositions at 1000°C and 900°C.

calculated values for this temperature as are the 1000°C data. Within this narrow temperature interval, the activation energy for inter-diffusion is approximately the same for the experimental and calculated results. The "average" $\overline{D}$ values determined at 600° and 650°C from the homogenization of cryptoperthite lamellae (Brady and Yund, 1983) also agree reasonably well with the lowest calculated $\overline{D}$ at these temperatures. Thus, based on Equation (3), there appears to be a fairly good agreement between the inter-diffusion and self-diffusion data over the range from 1000°C to 600°C.

Conclusions

The results of this and previous studies permit us to make the following observations concerning alkali diffusion in feldspar. (1) There is no significant dependence of the diffusion rate on either the confining pressure (to 15 kbar) or the water content of the feldspar. (2) Alkali diffusion in feldspar is anisotropic, with diffusion normal to (001) about 10 times faster than that normal to (010) and with that parallel to [100] and [011] being intermediate. (3) There is good agreement of the inter-diffusion coefficients determined experimentally from diffusion couples with the values calculated from the self-diffusion coefficients determined from bulk isotopic exchange. This provides the first, although incomplete, verification of the relation between $\overline{D}$ and D* discussed by Manning (1968) and Brady (1975). (4) The extrapolation of $\overline{D}$ values to lower temperature must take into account the non-linearity of the Arrhenius plot which is a consequence of the increase in non-ideality of the feldspar binary.

Acknowledgments

We wish to thank John Brady and Andreas Kronenberg for helpful suggestions and discussions during the course of this work, and Duncan Mardon for doing one of the electron microprobe traverses. This research was supported by NSF EAR70-26370 (Earth Science Section).

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Manuscript received, October 25, 1982;
accepted for publication, April 25, 1983.