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Alkali Feldspar Unit-Cell Parameters at Liquid Nitrogen Temperatures: Low Temperature Limits of the Displacive Transformation

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Abstract

Unit-cell parameters of analbite, low albite, high sanidine, maximum microcline, and adularia have been determined at room and liquid nitrogen temperatures. In all feldspars b and β are unchanged in this temperature interval, while a undergoes significant contraction. Interaxial angles show little change in low albite, maximum microcline, and high sanidine, though analbite displays a significant increase in α and γ (and thus an increase in obliquity) with decreasing temperature. An increase in γ of adularia from 90.00° to 90.07° may indicate a low-temperature displacive transformation from a monoclinic to a triclinic phase.

Introduction

The distribution of aluminum and silicon among the tetrahedrally coordinated equipoints in the alkali feldspar structure and the size of the alkali cations that occupy the interstices of the Al-Si-O framework help determine the symmetry of the feldspar structure (see, *e.g.*, Smith, 1970). The topologic symmetry of the alkali feldspar structure is $C2/m$. However, distortions of the tetrahedral framework and/or differences in the distribution of aluminum and silicon amongst the tetrahedral sites may reduce the symmetry to triclinic, as is the case for microcline and analbite.

High-temperature X-ray crystallographic studies of alkali feldspars show that continuous changes occur as a function of increasing temperature. The high-temperature X-ray powder diffraction experiments of Grundy and Brown (1969) and Stewart and von Limbach (1967) determined the magnitude of these modifications in low albite to analbite. High temperature single crystal data on microcline to 1200°C have been presented by Grundy and Brown (1967). Continuous modifications of the feldspar structure as a function of temperature may lead to changes in symmetry. Upon heating, analbite approaches monoclinic symmetry continuously, and at 980°C a displacive transformation takes place from $C\bar{1}$ to $C2/m$ (Grundy and Brown, 1967). Other similar displacive transformations have been found in feldspars that have an Al-Si distribution consistent with monoclinic symmetry (Kroll and Bambauer, 1971; Grove and Ito, 1973; Nager, Bambauer, and Hoffmann, 1970).

The purpose of this low temperature study was to determine the magnitudes of continuous changes in cell dimensions of alkali feldspars having different structural states. Two monoclinic alkali feldspars were observed to see if a displacive transformation could be induced at low temperatures. Three triclinic alkali feldspars were studied at low temperature to determine the lower attainable limits of the continuous changes in lattice geometry.

Experimental

Specimens of five alkali feldspars—analbite, low albite, high sanidine, maximum microcline, and adularia—were generously provided by Dr. Guy Hovis (Hovis, 1974; Thompson, Waldbaum, and Hovis, 1974). From each of these samples an untwinned, equi-dimensional single crystal approximately 200 μm in diameter was selected. Optical examination showed all five crystals to be clear, colorless, and unaltered, while X-ray photographs revealed each feldspar to have sharp and unsplit diffraction maxima. A list of specimens' localities, compositions, and structural states is given in Table 1.

Each feldspar crystal was mounted and centered on our four-circle, computer-controlled Picker diffractometer. The orientations of twelve strong reflections with 2θ greater than 35° ($MoK\alpha$ radiation), representing all octants of reciprocal space, were measured at both positive and negative 2θ. Room temperature (23°C) unit-cell parameters were refined from this orientation data using the Picker least-squares orientation program (Busing and Levy, 1967). In all cases these cell parameters agreed within experimental error with values determined from powder diffraction data (Hovis, 1974; Thompson *et al.,* 1974; Orville, 1967; Hovis, unpublished data).

(Liquid nitrogen temperature (77K) was achieved and maintained using a Cryo-Tip refrigerator system manufactured by Air Products and Chemicals, Inc. The crystal is mounted on a copper pin and is surrounded by a thin-walled beryllium vacuum shroud. The copper pin maintains contact with a small chamber of liquid nitrogen, which refrigerates the pin and crystal by conduction. The crystal temperature was monitored continuously using a copper-constantan thermocouple positioned beneath the specimen in the refrigeration pin, and was found to vary no more than 2°C from the minimum of −198°C. The feldspar crystals were first allowed to equilibrate for two hours at liquid nitrogen temperature. Experimental procedures for determining unit-cell parameters at low temperature were identical to those used at room temperature. After twelve diffraction maxima were measured, the initial two reflections were remeasured to confirm that the feldspar crystals had not altered in orientation. In all cases the initial and final orientations agreed within 0.02°.

Results

Unit-cell parameters and their estimated standard deviations for five alkali feldspars at 23°C and −198°C are presented in Table 1. In all cases *b* and β remain unchanged as temperature is lowered. The *a* dimension is significantly shortened at liquid nitrogen temperature in all the feldspars studied. An increase in obliquity is observed in analbite by a positive change in both α and γ. A similar change in the γ of adularia is observed and indicates a departure from monoclinic symmetry at low temperatures. Microcline, sanidine, and low albite show little change in interaxial angles during the 221°C temperature change, indicating that the room temperature structures probably represent frameworks with configurations near the lower limit of continuous change. Of the five feldspars studied, the interaxial angles of analbite show the greatest changes.

Conclusions

Our results are consistent with previous high temperature studies of alkali feldspars. With decreasing temperatures the triclinic minerals increase in obliquity, and cell parameters decrease. The rate of

TABLE 1. Specimen Sources, Compositions, and Unit-Cell Parameters

Sample (Reference)	$\frac{n_{or}}{n_{or}+n_{ab}}$	T (°C)	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)
Adularia St. Gotthard, Switz. (Hovis, 1973; UNT-7007)	0.882	23	8.551(3)	12.971(4)	7.210(3)	90.00(3)	116.02(3)	90.00(2)
		-198	8.528(3)	12.965(4)	7.210(3)	90.00(3)	116.05(3)	90.07(2)
Maximum Microcline Hugo pegmatite, S.D. (Orville, 1967)	0.881	23	8.578(3)	12.952(4)	7.228(2)	90.64(2)	115.94(1)	87.75(2)
		-198	8.545(4)	12.947(6)	7.219(4)	90.60(4)	115.92(2)	87.67(3)
High Sanidine Laacher See, Germany Heated 500 hours @ 1052°C, 1 atm. (Hovis, 1973; UNT-7002)	0.847	23	8.545(2)	13.040(3)	7.173(2)	90.00(2)	116.00(1)	90.00(2)
		-198	8.523(3)	13.041(4)	7.178(4)	90.00(2)	116.00(2)	90.00(2)
Low Albite Amelia Courthouse, Va. (Thompson et. al., 1973)	0.016	23	8.132(2)	12.768(4)	7.160(2)	94.21(2)	116.58(2)	87.68(2)
		-198	8.114(4)	12.771(7)	7.161(4)	94.21(4)	116.61(3)	87.67(3)
Analbite Heated Amelia low albite 522 hours @ 1060°C, 1 atm. (Thompson et. al., 1973)	0.016	23	8.169(4)	12.881(4)	7.112(3)	93.37(3)	116.49(3)	90.30(4)
		-198	8.160(4)	12.876(12)	7.105(4)	93.62(3)	116.50(3)	90.38(3)

* Parenthesized figures represent the estimated standard deviation (esd) in terms of least units cited for the value to the immediate left, thus 8.551(3) indicates an esd of 0.003.

contraction of cell edges and change of interaxial angles decreases at lower temperatures. Both the low temperature equilibrium forms of potassium and sodium feldspar undergo little change in interaxial angles over the temperature region from 23°C to −198°C, suggesting that the continuous changes which take place at elevated temperatures have nearly reached their lower limits at room temperature. In contrast, the high temperature polymorph of albite has not reached the lower bounds of its continuous change at room temperature, but increases significantly in obliquity at liquid nitrogen temperature. The experimentally determined change in γ found for the adularia is small but greater than one standard deviation and may indicate a low temperature displacive transformation from monoclinic to triclinic symmetry.

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References

Busing, W. R., and H. A. Levy (1967) Angle calculations for 3- and 4-circle X-ray and neutron diffractometers. *Acta Crystallogr.* **22,** 457–464.

Grove, T. L., and J. Ito (1973) High temperature displacive transformations in synthetic feldspars. *Trans. Am. Geophys. Union,* **54,** 499.

Grundy, H. D., and W. L. Brown (1967) Preliminary single crystal study of the lattice angles of triclinic feldspars at temperatures up to 1200 degrees. *Schweiz. Mineral. Petrogr. Mitt.* **47,** 21–30.

———, and ——— (1969) A high temperature X-ray study of the equilibrium forms of albite. *Mineral. Mag.* **37,** 156–172.

Hovis, G. (1974) A solution calorimetric and X-ray investigation of Al-Si distribution in monoclinic potassium feldspars. In, *The Feldspars. Proc. NATO Adv. Study Inst.,* University of Manchester Press, England, pp. 114–144.

Kroll, H., and H. U. Bambauer (1971) The displacive transformation of (K,Na,Ca)-feldspars. *Neues Jahrb. Mineral. Mitt.* **1971,** 413–416.

Nager, H. E., H. U. Bambauer, and W. Hoffmann (1970) Polymorphie in der Mischreihe (Ca,Sr) $Al_2Si_2O_8$. *Naturwissenshaften,* **57,** 86–87.

Orville, P. M. (1963) Alkali ion exchange between vapor and feldspar phases. *Am. J. Sci.* **261,** 201–237.

——— (1967) Unit-cell parameters of the microcline-low albite and the sanidine-high albite solid solution series. *Am. Mineral.* **52,** 55–86.

Smith, J. V. (1970) Physical properties of order-disorder structures with special reference to feldspar minerals. *Lithos,* **3,** 145–160.

Stewart, D. B., and D. von Limbach (1967) Thermal expansion of low and high albite. *Am. Mineral.* **52,** 389–413.

Thompson, J. B., Jr., D. R. Waldbaum, and G. L. Hovis (1974) Thermodynamic properties related to ordering in end-member alkali feldspars. In, *The Feldspars. Proc. NATO Adv. Study Inst.,* University of Manchester Press, England, 218–248.

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