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# Preliminary Single-Crystal Study of the Lattice Angles of Triclinic Feldspars at Temperatures up to 1200°C

By H. D. Grundy and W. L. Brown (Manchester)\*)

With 5 figures in the text and 2 tables

Abstract. The reciprocal lattice angles of six plagioclases and a maximum microcline were measured at a series of temperatures up to 1200°C on a Buerger precession camera. The crystals were mounted by sintering on a thermocouple without cement and heated in a gas flame.

The angles of the plagioclases change smoothly with temperature,  $\alpha^*$  changing most and  $\beta^*$  least; the angles of microcline do not change with temperature. Plots of the change of  $\alpha^*$  against  $\gamma^*$  for a sequence of temperatures are straight lines; the effect of heat on these angles for low and high-albite is similar to the effect of the substitution of potassium on the angles measured at room temperature.

The feldspars have been extensively studied at room temperature, but until recently little was known about their properties at elevated temperatures. The change in the lattice angles of anorthite with temperature is known from early optical work (RINNE, 1914; SCHNAASE, 1936 — see also Figs. 4 and 5) and expansion measurements have been made on various feldspars using dilatometric methods (Kôzu and UEDA, 1933). The first systematic X-ray study at elevated temperatures was made by MACKENZIE (1952) on albite-rich feldspars — he measured the angular separation of the lines 111 and 111 on diffraction charts as a function of temperature. Since then lattice angles for albite and anorthite and complete parameters for a basic labradorite at elevated temperatures have been published (BROWN, 1962; GUBSER et al., 1963; STEWART et al., 1966), and lattice parameters for two plagioclases have been discussed

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(STEWART and VON LIMBACH, 1964). BLOSS (1964) determined the change of extinction angle with temperature in anorthite.

During this preliminary study, single crystals of plagioclase feldspars selected from material of known composition and a microcline were examined on a Buerger precession camera. Each crystal was mounted on a Pt/Pt-13% Rh thermocouple by sintering at temperatures below



Fig. 1. Plot of  $\alpha^*$  against temperature for all plagioclases except anorthite:  $\circ$  albite, F9; + oligoclase, F44;  $\triangle$  and esine, E24;  $\Diamond$  labradorite, F70;  $\times$  bytownite, F71. The symbol  $\bullet$  is for material similar to F70 from same locality calculated from data given by STEWART et al. (1966). Anorthite has not been plotted because of overlap, see Fig. 4.

their melting points (usually 1100°C) for 10—30 mins. in an R. F. heater. This method of mounting avoided the use of cement which might have produced compositional changes in the crystals. The crystals were heated in a coalgas/nitrogen/air or coalgas/oxygen flame (for temperatures in the ranges 100—700°C and 700—1500°C respectively) similar to that described by GUBSER et al. (1963). With the exception of F70 two crystals of each feldspar were mounted, with which a-, b- and c-axis precession photographs were taken ( $\mu = 15^{\circ}$ , Mo K $\alpha$ ). Room-temperature photographs were taken before and after mounting by sintering and also after completion of the high-temperature photographs at high tempera-



Fig. 2. Plot of  $\beta^*$  against temperature for all plagioclases: symbols as in Fig. 1, and, in addition,  $\Box$  anorthite, F 72.

ture were generally taken using high-speed Polaroid film to reduce exposure time to 5—10 mins. and hence the total period of heating. The reciprocal lattice angles  $\alpha^*$ ,  $\beta^*$ ,  $\gamma^*$  were measured directly from the precession photographs and are accurate to within  $\pm 5'$ , except for microcline where the accuracy is  $\pm 10'$  due to lineage. The temperatures are accurate to  $\pm 30^{\circ}$ C in the range 200—400°C and  $\pm 10^{\circ}$ C in the range  $400-1200^{\circ}$ C.

The materials studied are shown in Table 1 and their lattice angles at different temperatures given in Table 2 and depicted in Figs. 1—5. As can be seen from Figs. 1—4, the variation of the reciprocal lattice angles with temperature is smooth within experimental error in all cases. STEWART et al. (1966) have suggested that there are discontinuities in the curve of molecular volume against temperature for a calcic labradorite

from Lake County, Oregon, but no such discontinuities could be detected in the plots of  $\alpha^*$  and  $\gamma^*$  against temperature for material from the same locality (F 70). The values of these angles calculated from the data of STEWART et al. are also plotted in Figs. 1, 3 and 5, from which the great



Fig. 3. Plot of  $\gamma^*$  against temperature for all plagioclases, symbols as in Figs. 1 and 2.

similarity of results is apparent. The exposure times were not long enough to determine whether the b-reflections were present or absent.

For all plagioclases the angle  $\alpha^*$  changes most with temperature,  $\gamma^*$  slightly less and  $\beta^*$  very much less (Figs. 1—4). The effect of temperature on all three angles is greatest for low-albite and decreases progressively with increasing anorthite content. F 71 is exceptional in that the changes in these angles with temperature (especially  $\alpha^*$ ) are greater than for plagioclases of similar composition — the b-reflections were present and sharp at room temperature but were so much weaker at higher temperatures that it was not possible to tell whether they were absent or not. The lattice angles of microcline do not change with temperature within experimental error (Table 2).



Fig. 4. Plot of  $\alpha^*$  against temperature for anorthite F72 and data from literature.  $\Box$  F72.

Fig. 4 is a plot of  $\alpha^*$  against temperature for F 72 and for published data on anorthite by RINNE (1914), SCHNAASE (1936) and GUBSER et al. (1963). The values for  $\alpha^*$  at elevated temperature are consistently greater for F 72 than those given by GUBSER et al. for ostensibly the same material though they agree with those of RINNE; this may be due to slightly different composition or structural state or both. The c-reflections of unheated material from F 72 are strong and only slightly diffuse on photographs taken at room temperature but are diffuse at room temperature after mounting by heating to 1350—1400°C for 30 mins. They become diffuse on raising the temperature and are absent on photographs taken at 400°C or above (compare BROWN et al., 1963). The noteworthy lack of change in  $\alpha^*$  between  $-170^\circ$ C and about 300°C (RINNE, 1914 and Fig. 4) may be connected with the disappearance of the c-reflections. The b-reflections remain sharp and strong on all photographs at temperatures up to  $1220^{\circ}$ C, so that anorthite becomes and remains bodycentred up to that temperature. No sign of flattening of the curve of  $\alpha^*$  against temperature above 900°C was found by us or by GUBSER et al. — the flattening found by SCHNAASE between 900—1000°C (Fig. 4) may be due to different material or inaccurate temperature determination.

Fig. 5 is a plot of  $\alpha^*$  against  $\gamma^*$  for temperatures at 200 degree intervals read from the smooth curve in Figs. 1—4. The variation of  $\alpha^*$  against  $\gamma^*$  with temperature is linear or nearly so for all plagioclases;



Fig. 5. Plot of  $\alpha^*$  against  $\gamma^*$  at 200 degree intervals: from below 26, 200, 400, 600, 800, 1000°C (and 1200°C in case of anorthite). Same symbols as Figs. 1 and 2. Data for labradorite from STEWART et al. (1966) as Figs. 1 and 3.  $\checkmark$  albite S 502 from BROWN (1962) at 25 and 1000°C.  $\bullet$  anorthite from Miyake Jima, Japan, at 25 and 1000°C (approximate composition An<sub>90</sub>).  $\square$  average value for microcline, 400. The values at the corners of the parallelogram ABDE from MacKENZIE and SMITH (1962). Ab<sub>62</sub>Or<sub>38</sub> from GOLDSMITH and LAVES (1961). Dotted curve ... data from SCHNAASE (1936).

microcline shows no changes in these angles with temperature. The slopes of the lines for the plagioclases are roughly parallel to the line for the anorthoclases (high-albite—sanidine join) at room temperature (MACKENZIE and SMITH 1955; BROWN 1960). The slopes decrease slightly with increasing anorthite contents. The line for low-albite is of such slope that a continuation would lead to microcline (cf. BROWN, 1962, p. 362). The effect of increasing temperature on  $\alpha^*$  and  $\gamma^*$  of low-albite is similar to the effect on these angles at room temperature of substitution of potassium in low-albite (the metastable low-albite—microcline series, GOLDSMITH and LAVES, 1961), a temperature rise of 1000°C producing about the same change as the substitution of about 30% Or (Fig. 5). The effect on  $\alpha^*$  and  $\gamma^*$  of high-albite on increasing the temperature is also similar to that of substituting about 30—40% Or (MACKENZIE 1952, LAVES 1952).

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<b>_</b>	av.	10	1

No.	Feldspar	Compos An	ition ir Ab	ı wt. % Or	Rock type	Locality
F 9	Albite <sup>1</sup> )	0.15	99.26	0.59	Alpine vein	Schyn-Schlucht, Grisons, Switzerland
F 44	Oligoclase <sup>2</sup> )	28.2	69.3	2.5	Pegmatite	Bakersville, Mitchell Co., N.C.
E 24	Andesine <sup>3</sup> )	49.0	47.2	3.8	Anorthosite	Essex County, N. Y.
F 70	Labradorite <sup>2</sup> ), <sup>4</sup> )	67.5	<b>31.6</b>	0.9	Lava	Lake View, Oregon
F 71	Bytownite <sup>2</sup> )	75.7	23.7	0.6	Anorthosite	Crystal Bay, Minnesota
F 72	Anorthite <sup>2</sup> ), <sup>5</sup> )	95.4	3.1	1.5	Cavity in Lava	Vesuvius, Italy
400	Microcline <sup>6</sup> )	$\mathbf{not}$	analys	sed	Pegmatite	Anjanabiana

<sup>1</sup>) Analysis by WEIBEL (1958), see also BROWN (1960).

<sup>2</sup>) Analysis by E. TAYLOR and H. D. GRUNDY.

<sup>3</sup>) Sample from EMMONS et al. (1953), new no. 9.

<sup>4</sup>) Similar material to that used by STEWART et al. (1966).

<sup>3</sup>) S 140, same sample as used by BROWN et al. (1963) and by GUBSER et al. (1963).

<sup>6</sup>) Sample not analysed because of presence of exsolved albite. Albite-free specimens were chosen from thin sections for single-crystal work.

#### Table 2

F 9, Albite from Schyn-Schlucht, Grisons, Switzerland. Ano.15

т°С	α*	Τ°C	β*	т°С	· γ*
$25^{1}$	86° 20'	25	$63^\circ$ $34'$	25	90° 16'
$25^{2}$	86 17	25	63 33	<b>25</b>	$90 \ 16$
$25^{3}$	86 16	25	63 32	<b>25</b>	90 16
210	86 22	250	63 39	200	90 21
400	86 32	450	63  43	380	90 28
585	86 48	610	63 - 46	600	90  43
700	86 59	—			
800	87 18	800	63 47	810	90 54
880	87 - 25		—		
1000	87 39	1010	64 04	1000	91 09

т°С	α	*	Τ°C	β	*	т°С	γ*	
26 <sup>1</sup>	86°	11′	26	$63^{\circ}$	36'	26	88°	45'
26 <sup>2</sup>	86	10	26	63	34	26	88	46
$26^{3}$	86	11	26	63	<b>35</b>	26	88	<b>45</b>
210	86	19	200	63	32			-0
<b>415</b>	86	36	390	63	39	400	88	<b>59</b>
600	86	38	580	63	39	610	89	05
795	87	00	800	63	<b>45</b>	810	89	<b>21</b>
990	87	22	1000	63	57	1010	89	31

# F 44, Oligoclase from Bakersville, N. C. An<sub>28</sub>

E 24, Andesine from Essex Co., N. Y. An<sub>49</sub>

Τ°C	α*		т°С	β	*	Τ°C	γ	*
26 <sup>1</sup>	86°	02'	26	$63^{\circ}$	46'	26	88°	27'
$26^{2}$	86	10	26	63	<b>45</b>	26	88	<b>23</b>
26 <sup>3</sup>	86	<b>05</b>	26	63	44	26	88	27
180	86	11	190	63	48	210	88	<b>28</b>
400	86	20	390	63	47	390	88	34
600	86	25	590	63	50	600	88	41
810	86	49	830	63	<b>56</b>	810	88	55
1030	87	02	1010	63	55	1020	89	06

F 70, Labradorite from Lake View, Oregon. An $_{68}$ 

T°C α*		•	т°С	β*		Т°С	$\gamma^*$	
26 <sup>1</sup>	85°	59'	26	63°	50'	26	87°	<b>49'</b>
26 <sup>2</sup>	86	00	26	63	51	26	87	45
$26^{3}$	86	00	26	63	51	26	87	<b>45</b>
180	86	01	190	63	55	200	87	49
390	86	08	400	64	00	410	87	<b>53</b>
600	86	18	580	<b>64</b>	00	610	88	03
820	86	30	810	<b>64</b>	04	800	88	13
1000	86	<b>45</b>	1000	64	03	1000	88	<b>26</b>

F 71, Bytownite from Crystal Bay, Minnesota. An<sub>76</sub>

Τ°C	α	ĸ	Τ°C	β*	T°C	Y	•
261	$85^{\circ}$	51'	not me	asured	26	87°	37'
26 <sup>2</sup>	85	51	,	,	<b>26</b>	87	40
26 <sup>3</sup>	85	50	,	,	26	87	37
200	85	57	, ,	,	200	87	43
400	86	05	,		400	87	49
500	86	06	,	,	-	_	-
600	86	18	,	,	600	88	04
700	86	25	,	,			2
800	86	33	,		800	88	12
1000	86	44	,	,	1000	88	32

т°С	α*	Τ°C	, β*	т°С	γ*
261	85° 55'	26	64° 00'	26	87° 09'
26 <sup>2</sup>	85 54	26	64 03	26	87 07
$26^{3}$	85 55	26	64 03	26	87 07
180	85 57	200	64  05	220	87 16
460	86 07	405	64 06	430	87 22
500	86 08				
540	86 10	<u></u>		<u></u>	
600	86 10	570	64 07	630	87 32
660	86 17				
710	86 18				
820	86 21	795	64 09	830	87 39
960	86 28			960	87 48
1000	86 26		_		—
1010	86 27	1010	64 10		
1080	86 32			1060	87 46
1220	86 37	1170	64 14	1210	87 59

# F 72, Anorthite from Vesuvius, Italy. An\_{95}

#### 400, Microcline from Anjanabiana

Τ°C	α*	Т°С	β*	Τ°C	$\gamma^*$
251	<b>9</b> 0° <b>19'</b>	25	$64^\circ$ $05'$	25	$92^\circ$ $17'$
$25^2$	90 19	25	64 03	<b>25</b>	92 12
$25^{3}$	90 19	<b>25</b>	64 08	<b>25</b>	92 07
180	90 16		_	2 <del></del>	
400	90 21			400	92 06
600	90 21	570	63 58	580	92 04
800	90 17	800	64 04	800	92 06
1020	90 16	1000	64 07	1010	<b>92</b> `00

1. Unheated specimen

2. Mounted specimen

3. Specimen on completion of high-temperature photography

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