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High-temperature homogenization behaviour of some sodic microperthites

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SUMMARY. While undergoing 'dry' homogenization at 980 °C the $\bar{2}01$ X-ray reflections of sodic low-albite–microcline microperthites show that phase compositions recapitulate (but in a reverse time-sense) the unmixing history of ordered feldspars about a solvus with a critical composition near 26 mole per cent $KAlSi_3O_8$.

HOMOGENIZATION in air ('dry' as opposed to hydrothermal) is a useful technique for obtaining the bulk composition of unmixed alkali feldspars, even though it is subject to systematic errors of disputed origin (Parsons, 1968; Jones *et al.*, 1969). The present paper describes an effect noted previously (Parsons, 1968; Czamanske, 1965) concerning intensity changes in the $\bar{2}01$ reflections with duration of heating, which appear to be explicable in the light of more recent work by Bachinski and Müller (1971).

Starting materials and techniques. The natural microperthite alkali feldspars used come from the Loch Ailsh syenite pluton in the Assynt district of the North-west Highlands of Scotland, described by Parsons (1965). Compositions obtained by XRF and flame photometry are given in Table I. Complete analyses of specimens 65 and 170 are given by Parsons and Smith (1974). All the feldspars are turbid and somewhat sericitized; they were separated magnetically and using heavy liquids. Localities for these samples together with the nomenclature of the intrusive units and their field relationships are given in Parsons (1965).

The X-ray diffraction patterns shown in the figures were made using a Phillips diffractometer with smear mounts. The composition-sensitive $\bar{2}01$ reflections are shown. No 2θ measurements are involved (note the diffuseness shown in fig. 1).

Because structural state may influence the homogenization *behaviour* of these perthites the degree of order of the K-phases as estimated on the criteria of Parsons and Boyd (1971) and of Wright (1968) is given in Table I. Specimens 170 and 70 were used to make the patterns figured as types 2c and 3b respectively in Parsons and Boyd (1971, fig. 1). The technique used for the Wright method is stated by Parsons (1973). The Na-phases are all close to low-albite; $\bar{2}01$ spacings of both phases approximate to those of the pure end-members.

These microperthites have a largely lamellar structure (see plates in Parsons, 1965) and vary slightly in coarseness of exsolution, the more sodic example, 170, exhibiting a finer intergrowth (mean lamellar thickness *c.* 10 μm) than the other specimens (25 μm). Specimen 70 has a 'bleb'-type texture (Parsons, 1965, plate 1B). Material used for the heating experiments recorded in fig. 1 was crushed initially in a ball-mill and individual fragments ranged downwards from 150 μm in largest dimensions. Other experiments were carried out on material crushed more finely in an agate mortar (< 50 μm). A second type of starting material (fig. 2) was a series of mechanical mixtures of weighed amounts of a natural maximum-microcline (Galicia, Spain, bulk composition $\mathrm{Ab_{0\cdot4}\,An_{0\cdot2}\,Or_{99\cdot4}}$) and a low-albite (Rischuna, Switzerland, $\mathrm{Ab_{98\cdot4}\,An_{0\cdot8}\,Or_{0\cdot8}}$), both analysed samples kindly supplied by L. R. Van der Plas. These mixtures were ground together in an agate mortar to a maximum grain size of 50 μm.

TABLE I. *Bulk composition and structural state data for sodic microperthites from the Loch Ailsh syenite complex*

Specimen No:	170	70	26	67d	65
Intrusive phase (Parsons, 1965):					
	S1	*S2*	*S2*	*S3*	*S3m*
Mole % Ab	71·7	69·2	71·0	68·5	67·9
Or	23·1	26·3	26·0	29·6	30·4
An	5·2	4·4	3·0	1·9	1·7
Ordering parameters of K-phase:					
131–1$\bar{3}$1 reflection type (Parsons and Boyd, 1971):					
	2c	*3b*	*4*	*4*	*4*
2θ Cu-$K\alpha_1$ (Wright, 1968):					
060	41·782	41·792	41·810	41·836	41·817
$\bar{2}$04	50·595	50·599	50·554	50·540	50·547
$\bar{2}$01	21·005	21·005	21·005	20·975	20·985

Heating was carried out in tightly crimped but unwelded gold capsules in a con tainer that held the capsules clustered around the junction of a chromel-alumel thermocouple, which was lowered rapidly into a preheated vertical tube furnace. This arrangement brought the samples to 950 °C in < 2 min and to the experimental temperature (980 °C) in about 4 min. Temperatures were recorded on a Honeywell recording potentiometer. Quenching, into water, was rapid.

Observations. Fig. 1 shows the appearance of the $\bar{2}$01 X-ray reflections of this suite of perthites after ball-mill crushing and heating at 980 °C for the stated times. Whereas the appearance of the $\bar{2}$01 reflections of the unheated crystals are roughly the same in all cases (the intensity of the reflections from each phase is, of course, roughly proportional to the amount of each phase, provided each phase is compositionally homogeneous) there is a variation in behaviour with time, which is related to the bulk composition. In the most sodic example (170) the Na-phase reflection remains the most intense during the homogenization process. The more potassic examples show a reversal in relative intensity of the reflections during homogenization, the initially weaker K-phase reflections becoming stronger with time until the Na-rich phase is represented only by a step on the high-2θ side of a diffuse $\bar{2}01$ reflection, and, finally by a reflection with its maximum skewed towards K-feldspar relative to the peak position on completion of homogenization. The intermediate composition, specimen 70, shows intermediate 'symmetrical' behaviour. This behaviour change seems to be related to bulk composition and although specimen 26, which has almost the same bulk composition as specimen 70, behaves like the more potassic examples, it appears that the change occurs near to 26 mole % $KAlSi_3O_8$. The difference between specimens 70 and 26 might be due to bad analysis, the higher anorthite content of 70, or K-phase structural state, since 70 contains some monoclinic material (Table I).

In an attempt to locate the composition at which the behaviour change occurred more precisely and without these imponderables a series of weighed mixtures of an analysed maximum-microcline and a low albite was made (both < 1 % An). These mixtures behaved differently to the microperthites (fig. 2). In all compositions the $\bar{2}01$ reflection for low-albite remained essentially at constant 2θ while gradually decreasing in intensity, while a second strong sharp reflection moved towards the final 2θ position for the homogenized material. For short experiments (30 min) there did appear to be a change in behaviour within the range 29 to

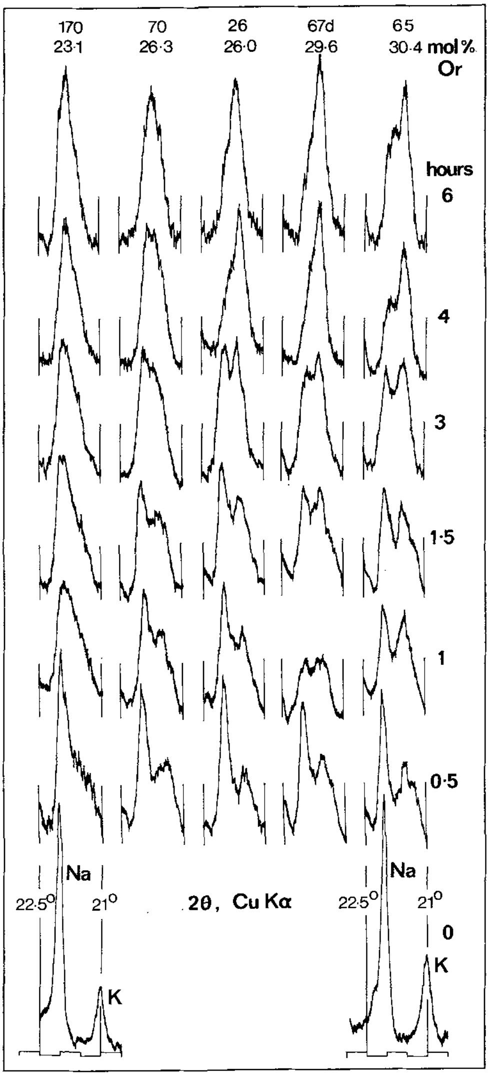

FIG. 1. X-ray diffractometer traces of $\bar{2}01$ reflections of perthitic alkali feldspars (Specimen Nos. on top line) of various bulk compositions (indicated in mole % $KAlSi_3O_8$) after various heating times at 980 °C. Two unheated examples are shown, with the Na- and K-rich phases labelled. Note how the $\bar{2}01$ reflection of the K-phase becomes dominant after four hours heating in the potassic examples, while the Na-rich reflection remains dominant in the sodic examples. This behaviour change takes place near 26 mole % $KAlSi_3O_8$.

19 mole % $KAlSi_3O_8$, but reflections with a sense of skew like that of specimen 170 never *persisted* even for compositions with < 20 mole % $KAlSi_3O_8$.

The mixed materials were crushed to < 50 μm, finer than the material used to obtain fig. 1 (< 150 μm). The rate of the homogenization process was greatly increased relative to the perthitic material, even though the lamellar width in the latter (< 25 μm) was less than the grain size. Fine crushing of the microperthites similarly increased the homogenization rate, although material crushed to < 50 μm still showed the asymmetric effects of fig. 1, a skewed single reflection being obtained after only 30 min. Ultra-fine material obtained by allowing a suspension to evaporate (< 10 μm) homogenized very rapidly and gave a single symmetrical $\bar{2}01$ reflection after 30 min at 980 °C.

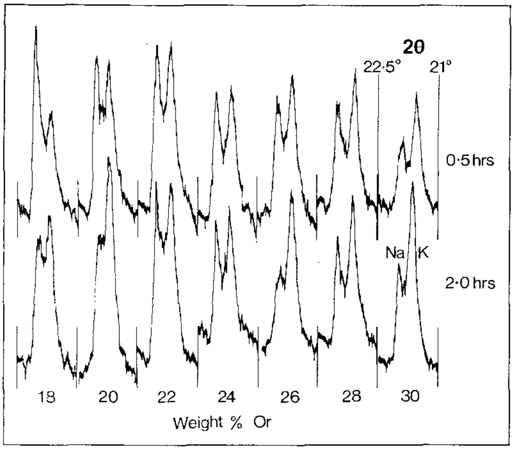

FIG. 2. Diffractometer traces of $\bar{2}01$ reflections from mixtures of weighed quantities of low albite and a maximum microcline after stated heating periods at 980 °C. Note that although after 30 min. the traces suggest a behaviour reversal like that in fig. 1, after 2 hours the effect has disappeared and all the patterns indicate a K-rich phase with a subordinate reflection for residual pure low albite. The patterns for unheated mixtures are like the lower examples of fig. 1.

Discussion. It is clear from the experiments using mixtures and those involving progressive reduction in particle size of perthitic crystals that the effect depicted in fig. 1 is seen only in material of a grain-size such that appreciable numbers of naturally formed phase-boundaries persist. In lamellar perthitic crystals these are likely to be coherent boundaries (i.e. with a continuous Al–Si framework) at least in part, as discussed by Brown and Willaime (1974). Particle size during homogenization greatly affects homogenization rate; persistence of coherent boundaries may affect the *direction* from which the final $\bar{2}01$ peak position of the homogenized material is approached (contrast the behaviour shown in figs. 1 and 2).

The change in relative reflection intensities shown by the more potassic examples in fig. 1 was described by Czamanske (1965) for feldspars in the bulk composition range 37 to 45 wt % Or, and the difference in behaviour between sodic and potassic examples in the present study recorded by the writer (1968). I then offered the explanation that the difference in behaviour was due to structural state (and therefore symmetry) differences in the K-phases; recent work suggests a better explanation.

The perthitic feldspars used all possess a highly ordered Al–Si distribution, in both Na and K-phases. Solvi for highly ordered feldspars have been obtained by Bachinski and Müller (1971) by two techniques, which yield critical compositions of 27 and 24 mole % $KAlSi_3O_8$. The behaviour change between sodic and potassic sides of the present fig. 1 takes place near to 26 mole % $KAlSi_3O_8$. In addition the relative intensities of the reflections for K- and Na-rich phases after, for example, 3 to 4 hours at 980 °C (fig. 1) are the relative intensities that would be obtained for feldspar pairs at equilibrium in the immediately subcritical region, for bulk compositions distributed on either side of the critical composition of an asymmetric solvus (like those of Bachinski and Müller, 1971) with a critical composition near 26 mole % $KAlSi_3O_8$. (Parsons and Smith, 1974, have obtained solvus curves for specimens 170 and 65 of the present study; these are essentially the same as Müller's curve.) The layout of fig. 1, with longest heating times at the top giving a sensibly homogeneous single phase, approximates to a representation of a series of feldspar pairs in equilibrium at successively lower temperatures with lowest temperature at the bottom (the diffraction patterns of the present examples are a little more diffuse than feldspar pairs produced by direct synthesis). The perthitic feldspars homogenize as though recapitulating (in a reversed time-sense) the unmixing history of ordered feldspars about a solvus that conforms with those shown by Bachinski and Müller (1971). It should be noted that this is *not* the solvus about which the feldspars actually unmixed; they will have been less well ordered at the inception of unmixing, which will have commenced at a considerably lower temperature than the critical temperature of the microcline–low-albite solvus. However, the ordered framework persists in a metastable state during these homogenization experiments, and very probably for much longer periods. Although $\bar{2}01$ reflections for these feldspars homogenized at 1000 °C for 1485 hours are sharp, reflections more sensitive to order (060, $\bar{2}04$; Wright, 1968) are diffuse and suggest the persistence of ordered structure even after long heating periods, as does the persistence of a miscibility gap to *c.* 900 °C in Bachinski and Müller's (1971) experiments, and Parsons and Smith's (1974) experiments on specimens 65 and 170.

Conclusions. The changes in intensity of X-ray reflections described in this paper can be explained if homogenization is essentially a stepwise reversal of unmixing. This behaviour is seen only if naturally formed (partly coherent?) phase boundaries persist in the crushed starting material. Material made up of separate grains of albite and K-feldspar does not show this effect. Hence the direction from which the final homogeneous composition is approached depends on particle size in relation to the coarseness of perthitic intergrowths in the starting materials. Rate is greatly increased for fine particle sizes, and alkali interchange must be more rapid from grain to grain (i.e. through the vapour in the capsule) than across interfaces between phases in perthitic intergrowth. The critical composition of the metastable low-albite–maximum-microcline solvus is located by the present interpretation at 26 mole % $KAlSi_3O_8$.

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Note added in proof: There is a typographical error in the value of Mole % Or given in the full analysis of specimen 170 by Parsons and Smith (1974). The correct value is given in the present paper. As noted by Parsons and Smith the An values (calculated from available Ca) are probably too high; if available Al is used a maximum An content of $< 2\%$ is obtained.