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## SYNTHESIS OF BIOTITE AND ITS HYDROTHERMAL ALTERATION

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**Abstract.** Biotite was synthesized from gel of a chemical composition of this mineral at  $T = 500^{\circ}\text{C}$  and  $p_{\text{H}_2\text{O}} = 1$  kbar, in an environment of varying composition and pH. Then the alteration of synthetic and natural biotites in the presence of pure water vapour and with an addition of  $\text{AlCl}_3$  or  $\text{KCl} + \text{AlF}_3$  was investigated under the same  $T-p_{\text{H}_2\text{O}}$  conditions. The products of alteration of biotite are (Mg, Al) – serpentine, andalusite, chlorite and muscovite. Their presence and the sequence of formation depend on the experimental conditions.

### INTRODUCTION

Biotites, one of the commonest minerals in the earth's crust, have been the subject of intensive research in recent years. The fundamental work dealing with the synthesis of trioctahedral micas was written by Smith and Yoder (1956), who paid particular attention to the polymorphic varieties of these minerals, proposing a convenient nomenclature to distinguish the individual modifications  $1M$ ,  $3T$ ,  $2M_1$ . Wones and Eugster (1965) studied the conditions of synthesis of trioctahedral micas of variable chemical composition and found that the partial pressure of oxygen affects the occupancy of the octahedral sites by  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ , Mg, Al and other elements no less than other thermodynamic factors. Worth noting are also the papers concerned with phase equilibria between biotites and other layer silicates. For example, Jamanata and Bodine (1971) showed experimentally that there is an equilibrium between phlogopite, chlorites and (Mg, Al) – serpentines of variable composition. They also found that the transformation of chlorite into biotite depends largely on the concentration ratio of potassium and magnesium in the gaseous phase. It was also established experimentally (Kotelnikova *et al.*, 1977) that at the increased  $p_{\text{H}_2\text{O}} - T$  parameters, chlorite transforms readily into biotite when the brucite sheet in the structure of chlorite is artificially disturbed by acid

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treatment because in this way potassium passes easily from the gaseous phase into the interlayer spaces.

Under natural conditions, the structure of biotite can be transformed both by degradation and aggradation (Lucas, 1962; Kossovskaya, 1966). On one hand, the processes of degradation of the structure of biotite take place in the weathering crusts and are attended by the transformation into montmorillonite or kaolinite (Stoch, Sikora, 1976; Koldaev *et al.* 1974), or into muscovite + chlorite + fibrolite, at acid leaching in the metamorphic zones of sedimentary rocks (Korikovskij, 1967; Lygina, Kotov, 1973). On the other hand, extensive transformation of chlorite into biotite occurs in the greenschist facies during the processes of regional metamorphism (Tuner, 1948, and others). All the papers dealing with this problem analyse, as a rule, the thermodynamic aspects of phase equilibria of the initial and new-forming minerals whereas very little is as yet known about the structural relations between biotite and the new-forming minerals. Therefore, the present authors carried out experimental studies of the synthesis of biotite and its alteration in a hydrothermal environment of varying chemical composition.

## EXPERIMENTAL AND RESULTS

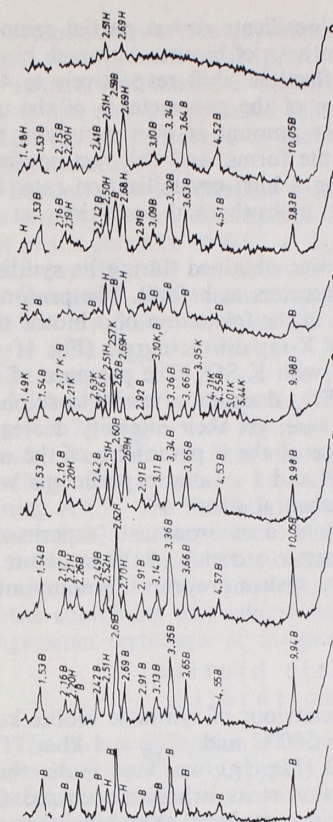
The first experiments showed that, because of its coarseness, natural biotite did not readily undergo hydrothermal alteration. Therefore, biotite was synthesized from gel in order to obtain a homogeneous, fine-dispersive material. The gel was prepared by Tikhomirova and Volkova (1974) and subjected to chemical analysis at the Institute of Experimental Mineralogy of the Academy of Sciences of the USSR. Its chemical composition was: 35.47%  $\text{SiO}_2$ , 2.15%  $\text{TiO}_2$ , 19.42%  $\text{Al}_2\text{O}_3$ , 21.08%  $\text{Fe}_2\text{O}_3$ , 10.77%  $\text{MgO}$ , 0.17%  $\text{MnO}$ , 0.24%  $\text{CaO}$ , 7.68%  $\text{K}_2\text{O}$ , 0.53%  $\text{Na}_2\text{O}$ , 2.26%  $\text{H}_2\text{O}^+$  (analysed by Tikhomirova and Volkova). This material was in the form of rusty-brown homogeneous powder which displayed only faint  $\alpha\text{-Fe}_2\text{O}_3$  lines ( $d_{104} = 2.69 \text{ \AA}$  and  $d_{110} = 2.51 \text{ \AA}$ ; Fig. 1a) in its X-ray powder pattern. Biotite which was expected to form from this gel would have a chemical composition similar to biotites of the P group acc. to Marakushev and Tatarin (1965) or to biotites occurring in metamorphic schists and gneisses of the amphibolite facies, containing garnets (Perchuk, 1973).

In some experiments also natural biotite ( $< 2 \mu\text{m}$  fraction), derived from Scandinavian pegmatites, was used. It was the polymorphic modification 1M of the following chemical composition: 35.57%  $\text{SiO}_2$ , 3.00%  $\text{TiO}_2$ , 17.86%  $\text{Al}_2\text{O}_3$ , 21.21%  $\text{Fe}_2\text{O}_3$ , 8.30%  $\text{MgO}$ , 1.04%  $\text{CaO}$ , 10.00%  $\text{K}_2\text{O}$ , 0.14%  $\text{Na}_2\text{O}$ , 3.58%  $\text{H}_2\text{O}^+$ .

Biotite or gel of a composition of biotite was placed in platinum capsules in a reactor designed by Kotov and Kopeikin (1972) and kept under the constant conditions of  $T = 500^\circ$  and  $p_{\text{H}_2\text{O}} = 1 \text{ kbar}$  in the presence of pure water, HCl ( $\text{pH} = 1$ ), as well as 0.5 N aqueous  $\text{K}_2\text{CO}_3$ , KCl and  $\text{K}_2\text{SO}_4$  solutions. After the run was finished, the reactor was cooled (about 5 min.), the pressure decreased, and the resulting material was subjected to X-ray analysis (DRON-1,  $\text{CuK}_\alpha$ , 35 kV, 18 mA, Ni filter, slits: 0.5, 1.0, 0.25, time constant 2 sec., goniometer angular speed  $2^\circ/\text{min.}$  with the sample rotating in its plane at 60 r.p.m.).

For some experiments two platinum capsules were put simultaneously in the reactor: one with biotite obtained by synthesis with  $\text{K}_2\text{SO}_4$  or KCl, the other with natural biotite. These materials were kept at a pressure of water vapour with an addition of KCl,  $\text{AlCl}_3$  or  $\text{AlF}_3$  under the same  $T - p_{\text{H}_2\text{O}}$  conditions as before.

\*  $1 \text{ \AA} = 0.1 \text{ nm}$  (SI).





—579). The appearance of this framework aluminosilicate causes partial removal of Al, Si and K from the gel, thus affecting the synthesis of biotite. Although biotite yields its characteristic lines, the 020 and 060 reflections shift respectively to 4.56 and 1.540 Å, testifying to a substantial expansion of the parameter *b* of the unit cell of biotite. This is presumably due to a larger amount of iron occupying the octahedral sites. Simultaneously, however, hematite forms, and this fact confirms the previous statement that this mineral, showing a high crystallization rate, has a tendency to metastable formation also under hydrothermal conditions at increased alkalinity.

The highest degree of crystallinity of biotite was obtained during its synthesis with additions of KCl and K<sub>2</sub>SO<sub>4</sub> (*T* – *p*<sub>H<sub>2</sub>O</sub> parameters as before). The prolongation of the reaction time from 1 to 3 days results in the formation of a biotite that yields narrower and more symmetrical lines in the X-ray diffractogram (Fig. 1f–i). This is particularly pronounced for the reactions with K<sub>2</sub>SO<sub>4</sub>. The presence of the sulphate ion, formed during the hydrolysis of K<sub>2</sub>SO<sub>4</sub>, does not give rise to sulphate minerals. The hematite lines also appear in this case, yet their intensity decreases as the experiment is prolonged. A certain increase of the *b* parameter of the unit cell of biotite during 3-day experiments with KCl and 1–3-day experiments with K<sub>2</sub>SO<sub>4</sub> suggests that more iron occupies the octahedral sites.

In conclusion, it can be stated that a weakly acid environment (experiments with an addition of KCl and K<sub>2</sub>SO<sub>4</sub>) promotes better ordering of the structure of biotite while the presence of excess potassium in the system prevents this component from passing into the water vapour phase.

#### Alteration of synthetic biotite

The first stage involved investigation of the behaviour of synthetic biotite kept in an atmosphere of pure water vapour at *T* = 500°C and *p*<sub>H<sub>2</sub>O</sub> = 1 kbar. The biotite, synthesized earlier in the presence of KCl (Fig. 1g), was kept under these conditions for 3 days, but no perceptible degradation of its structure occurred. On the contrary, the intensity of its diffraction lines increased and they became more symmetrical, which suggests that the material acquired a higher degree of crystalline perfection. At the same time, the biotite preserves the modification 1M. This fact indicates primarily that potassium present in the interlayer spaces of biotite is relatively strongly bound up with its structure and does not pass to the gaseous (water vapour) phase. The intensity of hematite lines (2.71 and 2.52 Å) decreases slightly, which presumably testifies to its partial dissolution. All the above considerations are also true of biotite synthesized in the presence of K<sub>2</sub>SO<sub>4</sub>.

Hydrothermal treatment of biotite (obtained by synthesis with K<sub>2</sub>SO<sub>4</sub> – Fig. 1i) in a medium containing AlCl<sub>3</sub> brings about significant alteration of this mineral already after 1 day. The diffraction pattern shows only its most intensive lines: 9.96, 4.51, 3.34, 2.62, 2.42 Å. Instead, there appear reflections due to two new-forming phases. One is characterized by the basal reflections *d*<sub>001</sub> = 7.08 and *d*<sub>002</sub> = 3.52 Å, as well as by general reflections: *d*<sub>200</sub> = 2.57, *d*<sub>202</sub> = 2.37, *d*<sub>040</sub> = 1.998, *d*<sub>060</sub> = 1.537, *d*<sub>020</sub> = 4.51 Å, which permit assigning it to (Mg, Al) – serpentines, presumably of the structural type B (acc. to Zvyagin *et al.*, 1966). The other phase is characterized by the reflections 5.54, 3.33, 2.78 and 2.23 Å and can be described as andalusite (ASTM, card no. 16–602). Alongside of these reflections, faint lines 14.2, 4.98 and 4.44 Å have been noted in the X-ray diffraction pattern, which testifies to the presence of other new-formed minerals with a layer structure. More intensive lines of these minerals were recorded when investigating the products of alteration of natural biotite (see below).

Prolongation of the experiment to 4 days under the same conditions leads to better development of (Mg, Al) – serpentine and andalusite. At the same time, the structure of biotite is destroyed almost completely. Moreover, the faint lines due to the above-mentioned mineral admixtures with a layer structure disappear and so do the reflections of hematite which was present in a small amount in the initial material.

Potassium was added to the system in the presence of AlF<sub>3</sub> to find whether this would prevent the rapid breakdown of the structure of biotite. It appeared that in the presence of KCl + AlF<sub>3</sub> biotite is preserved both after 3-days (Fig. 2d) and 7-day (Fig. 2e) treatment. Yet, the presence of fluorine in the system leads to the formation of its separate phases. The most pronounced in X-ray diffraction patterns is a phase which on the basis of the characteristic reflections (*d*<sub>111</sub> = 3.69, *d*<sub>120</sub> = 3.20, *d*<sub>022</sub> = 3.04, *d*<sub>112</sub> = 2.94, *d*<sub>130</sub> = 2.49, *d*<sub>103</sub> = 2.39, *d*<sub>023</sub> = 2.36, *d*<sub>123</sub> = 2.11, *d*<sub>220</sub> = 2.06 Å) can be identified as topaz (ASTM, card no. 12–765). The other phase, on the basis of reflections: *d*<sub>110</sub> = 3.26, *d*<sub>101</sub> = 2.59, *d*<sub>111</sub> = 2.23, *d*<sub>210</sub> = 1.71, *d*<sub>220</sub> = 1.64 Å, can be defined as magnesium fluoride, probably with an admixture of iron. Prolongation of the treatment to 7 days leads to the synthesis of (Mg, Al) – serpentine (reflections 7.01, 3.50, 2.37 Å) as in the medium containing AlCl<sub>3</sub>. The formation of the two-layer structure of (Mg, Al) – serpentine as a result of transformation of the three-layer structure of biotite is a rule, irrespective of the form in which aluminium has been introduced into the system (aluminium chloride or fluoride). Besides, in the case of AlF<sub>3</sub> addition, fluorine reacts with biotite combining with silicon and aluminium (synthesis of topaz), as well as with magnesium (synthesis of magnesium fluoride).

#### Alteration of natural biotite

The X-ray diffraction pattern of the natural biotite used for investigation is shown in Figure 3a. As appears from Figure 3b, this biotite does not undergo any appreciable alteration in an environment of pure water vapour (cf. similar behaviour of synthetic biotite under the same experimental conditions). When AlCl<sub>3</sub> is introduced into the system, biotite essentially transforms after 1-day treatment into three minerals: (Mg, Al) – serpentine, andalusite and muscovite. Judging by the preserved intensive reflections of biotite, however, its structure is not completely destroyed, which seems to be due primarily to the large size of its flakes. In contrast to the alteration of synthetic biotite, a small amount of chlorite also forms from natural biotite.

Prolongation of the reaction to 4 days results in the better development of chlorite, which fact is reflected in a higher intensity of its diffraction lines 14 and 4.70 Å (Fig. 3d). The amount of new-forming muscovite virtually does not change whereas the content of biotite decreases.

Comparing the experiments with synthetic and natural biotites, it will be noticed that the alteration product of the latter contains a large amount of andalusite already after one day (cf. the intensity of reflections 5.54, 5.58 and 2.78 Å in Figs. 2b and 3c). Prolongation of the reaction time fails to increase its content in relation to the other new-forming minerals (Fig. 3d). At the same time, the content of (Mg, Al) – serpentine in the alteration product tends to increase at the expense of the destruction of biotite.

The results of experiments in a medium containing KCl + AlF<sub>3</sub> (Fig. 3e, f) are similar for synthetic and natural biotites (cf. Fig. 2d, e). In both cases, the principal phases formed in the prolonged experiment (7 days) are (Mg, Al) – serpentine, topaz, magnesium fluoride ± muscovite, andalusite (Figs. 2e and 3f).



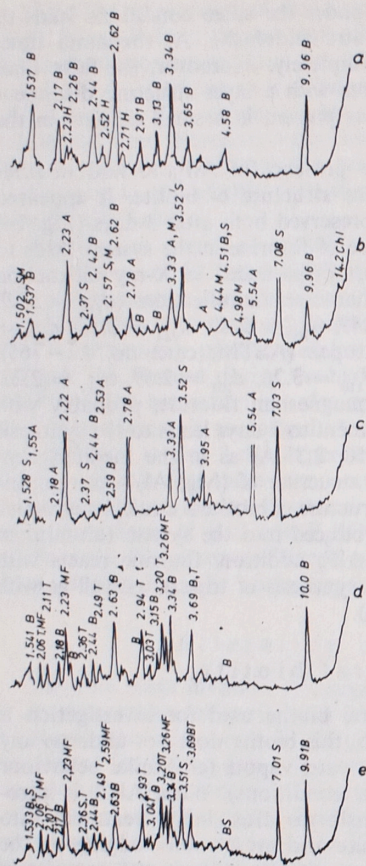


Fig. 2. X-ray diffractograms of the products of synthetic biotite alteration in medium of water vapour

a - pure water vapour, after 3 days, b - with  $\text{AlCl}_3$ , after 1 day, c - with  $\text{AlCl}_3$ , after 4 days, d - with  $\text{AlF}_3 + \text{KCl}$ , after 3 days, e - with  $\text{AlF}_3 + \text{KCl}$ , after 7 days. B - biotite, S - (Mg, Al)-serpentine, A - andalusite, T - topaz, MF -  $\text{MgF}_2$  (other symbols as on the Fig. 1)

## DISCUSSION

The above studies have shown that at  $p_{\text{H}_2\text{O}} = 1$  kbar and  $T = 500^\circ\text{C}$ , the synthesis of biotite from gel of a stoichiometric composition of this mineral proceeds more readily in the presence of potassium in an acid medium with  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$  anions than synthesis in a pure water vapour medium. An alkaline medium (addition of  $\text{K}_2\text{CO}_3$ ), on the other hand, causes aluminium as an amphoteric element to become to a higher degree an analogue of silicon (Frank-Kamenetski *et al.*, 1973 d). This leads to the synthesis of a framework silicate - kalsilite, deteriorating the conditions of formation of biotite.

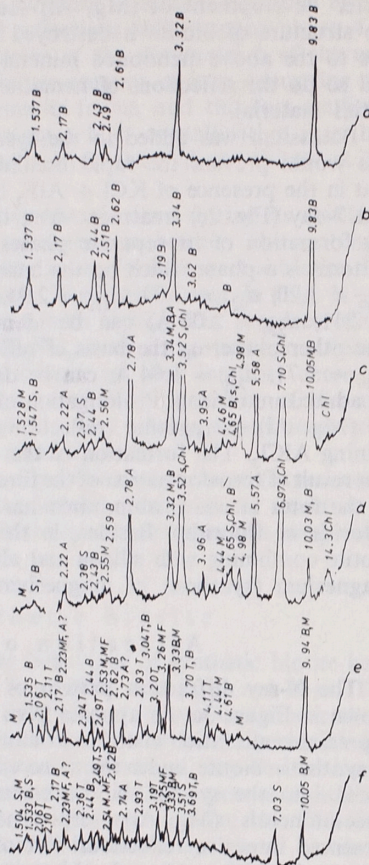


Fig. 3. X-ray diffractograms of the initial natural biotite (a) and products of its alteration in medium of water vapour (b-f)

b - pure water vapour, after 3 days, c - with  $\text{AlCl}_3$ , after 1 day, d - with  $\text{AlCl}_3$ , after 4 days, e - with  $\text{AlF}_3 + \text{KCl}$ , after 3 days, f - with  $\text{AlF}_3 + \text{KCl}$ , after 7 days. M - muscovite, Chl - chlorite (other symbols as on the Fig. 1 and 2)

In the presence of aluminium chlorides and fluorides, under hydrothermal conditions ( $p_{\text{H}_2\text{O}} = 1$  kbar and  $T = 500^\circ\text{C}$ ), both synthetic and natural biotites appear to be unstable. This is due not only to an increased aluminium content but also to the fact that at higher  $p_{\text{H}_2\text{O}} - T$  parameters  $\text{AlCl}_3$  and  $\text{AlF}_3$  hydrolyze with the formation of an acid environment containing HCl and HF. Such an environment favours the removal of potassium from the interlayer spaces of biotite and, if the reaction time is prolonged, also from the new-formed muscovite. In consequence, the weakening of the interlayer bonds occurs, attended by the passing of the three-layer structure of biotite into the two-layer structure of metastable (Nelson, Roy, 1958) (Mg, Al)-serpentine, and the precipitation of andalusite in the form of a separate phase. The presence of some excess potassium in the system, when KCl and  $\text{AlF}_3$  are added, slows down the degradation of the structure of biotite, preventing potassium from passing from the structure of biotite into the gaseous phase.

Slight differences in the composition of the initial materials affect the nature of the new-forming minerals, with the structure of biotite being degraded in the presence of  $\text{AlCl}_3$  and  $\text{AlF}_3$ . Thus, small amounts of chlorite and muscovite form at the expense of natural biotite after 4-7 days, while during the treatment of synthetic biotite these minerals appear only as transitional, unstable phases.

To summarize, it can be stated that at elevated temperatures and pressures in an acid hydrothermal environment, magnesium and iron are fixed in the 1:1 layer structure of (Mg, Al)-serpentine, a trioctahedral analogue of kaolinite. The synthesis of (Mg, Al)-serpentine instead of purely aluminium kaolinite is due to the instability of the latter under the experimental conditions:  $T = 500^\circ\text{C}$  and  $p_{\text{H}_2\text{O}} = 1$  kbar (Frank-Kamenetski *et al.*, 1973 b).

The above results represent the processes of alteration of biotite in which magnesium and iron play an active role. Under the natural conditions, in the low-temperature zones of hydrothermal alteration of rocks and in the hypogene zone, the common product of alteration of the three-layer structure of biotite is also a two-layer structure, appearing in the form of minerals of the kaolinite group. A feature differentiating the conditions discussed is that in the natural environment magnesium and iron are removed from the system in the form of carbonates (dolomite, siderite) or oxides (hematite, magnetite, maghemite), or even as ions, and the low mobility of aluminium and silicon favours the formation of kaolinite, aluminium oxides, as well as  $\text{SiO}_2$  minerals.

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## SYNTEZA BIOTYTU I JEGO HYDROTHERMALNE PRZEOBRAŻENIA

### Streszczenie

Syntetyzowano biotyt z gelu o składzie chemicznym tego minerału, w temperaturze  $T = 500^\circ\text{C}$  i ciśnieniu  $p_{H_2O} = 1 \text{ kbar}$ , w środowisku hydrotermalnym zawierającym  $K_2CO_3$ ,  $KCl$  i  $K_2SO_4$ . Stwierdzono, że synteza zachodzi najlepiej w środowisku słabo kwaśnym, w obecności potasu w fazie gazowej (dodatek  $KCl$  lub  $K_2SO_4$ ). W środowisku alkalicznym (dodatek  $K_2CO_3$ ), oprócz biotytu, w znacznej ilości tworzy się kalsilit, co świadczy o stosunkowo łatwym wchodzeniu glinu w tetraedry, ponieważ pierwiastek ten, jako amfoter, w znacznym stopniu jest analogiem krzemu.

W obecności  $AlCl_3$  w tychże samych warunkach  $T - p_{H_2O}$ , zarówno biotyt syntetyczny jak i naturalny są nietrwałe i przechodzą w andaluzyt,  $(Mg, Al) -$

serpentyń i muskowit. Muskowit łatwiej tworzy się z biotytu naturalnego. Obecność w układzie fluoru (dodatek  $AlF_3$ ) prowadzi do powstania, oprócz  $(Mg, Al) -$ serpentyń i muskowitu, samodzielnych faz zawierających fluor: topazu i  $MgF_2$ . Proces niszczenia struktury biotytu zostaje zwolniony przez dodanie do układu potasu (w formie  $KCl$ ). Obecność potasu w fazie gazowej zabezpiecza przed usuwaniem tego pierwiastka z przestrzeni międzypakietowych biotytu.

Przeprowadzone badania modelują te procesy hydrotermalnych przeobrażeń biotytu, kiedy magnez i żelazo biorą aktywny udział w przeobrażeniach fazowych, co prowadzi do syntezy dwuwarstwowej struktury  $(Mg, Al) -$ serpentyń, fazy metastabilnej, która z upływem czasu przechodzi w chloryt.

### OBJAŚNIENIA FIGUR

Fig. 1. Dyfraktogramy rentgenowskie gelu wyjściowego (a) i produktów syntezy, w środowisku pary wodnej (b—f)

b — czystej pary wodnej, przez okres 1 d, c — czystej pary wodnej, przez okres 4 d, d — z dodatkiem  $HCl$  przez okres 1 d, e — z dodatkiem  $HCl$  przez okres 4 d, f — z dodatkiem  $KCl$  przez okres 1 d, g — z dodatkiem  $KCl$  przez okres 3 d, h — z dodatkiem  $K_2SO_4$  przez okres 1 d, i — z dodatkiem  $K_2SO_4$  przez okres 3 d. B — biotyt, H — hematyt, K — kalsilit.

Fig. 2. Dyfraktogramy rentgenowskie produktów przeobrażenia syntetycznego biotytu, w środowisku pary wodnej

a — czystej pary wodnej, po okresie 3 d, b — z dodatkiem  $AlCl_3$  po okresie 1 d, c — z dodatkiem  $AlCl_3$  po okresie 4 d, d — z dodatkiem  $AlF_3 + KCl$  po okresie 3 d, e — z dodatkiem  $AlF_3 + KCl$  po okresie 7 d, S —  $(Mg, Al) -$ serpentyń, A — andaluzyt, T — topaz, MF —  $MgF_2$  (inne oznaczenia jak na fig. 1).

Fig. 3. Dyfraktogramy rentgenowskie biotytu naturalnego (a) i produktów jego przeobrażenia, w środowisku pary wodnej (b—f)

b — czystej pary wodnej, po okresie 3 d, c — z dodatkiem  $AlCl_3$  po okresie 1 d, d — z dodatkiem  $AlCl_3$  po okresie 4 d, e — z dodatkiem  $AlF_3 + KCl$  po okresie 3 d, f — z dodatkiem  $AlF_3 + KCl$  po okresie 7 d. M — muskowit, Chl — chloryt (inne oznaczenia jak na fig. 1 i 2).

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## СИНТЕЗ БИОТИТА И ЕГО ГИДРОТЕРМАЛЬНЫЕ ПРЕОБРАЗОВАНИЯ

### Резюме

Биотит синтезирован по гелю состава этого минерала в условиях  $p_{H_2O} = 1 \text{ kbar}$ ,  $T = 500^\circ\text{C}$  в  $HCl$ ,  $K_2CO_3$ ,  $KCl$ ,  $K_2SO_4$  — содержащих гидротермальных средах. Установлено, что наилучший синтез биотита достигается в слабо кислой среде при наличии в паровой фазе  $K^+$  (добавки  $KCl$  или  $K_2SO_4$ ). В щелочной среде (добавка  $K_2CO_3$ ), кроме биотита, по гелю в большом количестве синтезируется калсilit, что свидетельствует о преимущественном вхождении  $Al$  в тетраэдры, поскольку этот элемент, как амфотер, в большей степени становится аналогом кремния. В присутствии добавки  $AlCl_3$  при прочих одинаковых условиях, как синтетический, так и природный биотиты не устойчивы и замещаются андалузитом,  $7 \text{ Å} (Mg, Al) -$ серпентином и мусковитом. Muskowit лучше формируется по природному биотиту. Наличие фтора в системе  $(AlF_3)$  приводит к формированию по природному и синтетическому биотитам самостоятельных фторсодержащих фаз, таких как топаз



и  $\text{MgF}_2$  ( $\pm(\text{Mg}, \text{Al})$ )—серпентин, мусковит). Процесс разрушения биотита в этом случае замедляется при добавке калия в систему (в форме  $\text{KCl}$ ). Наличие в этом случае калия в паровой фазе предохраняет от вытеснения этого элемента из межпакетных позиций структуры биотита. Опыты моделируют тот процесс гидротермальных изменений биотита, когда  $\text{Mg}$  и  $\text{Fe}$  принимают активное участие в фазовых преобразованиях, что ведет к синтезу 1:1 структуры  $7 \text{ \AA}(\text{Mg}, \text{Al})$ —серпентина — метастабильной фазы, который с течением времени переходит в  $14 \text{ \AA}$ —хлорит.

## ОБЪЯСНЕНИЯ К ФИГУРАМ

Фиг. 1. Дифрактограммы исходного геля и продуктов синтеза:

*a* — исходный гель; *b* — биотит, синтезированный в чистой водной среде, 1 сут., *c* — то же, 4 сут., *d* — то же, с добавкой  $\text{HCl}$ , 1 сут., *e* — то же, с  $\text{K}_2\text{CO}_3$ , 1 сут., *f* — то же, с  $\text{KCl}$ , 1 сут., *g* — то же, 3 сут., *h* — то же, с  $\text{K}_2\text{SO}_4$ , 1 сут., *i* — то же, 3 сут. *H* — гематит, *B* — биотит, *K* — кальсилит

Фиг. 2. Дифрактограммы, иллюстрирующие поведение синтетического биотита в различных гидротермальных средах

*a* — биотит, обработанный чистой  $\text{H}_2\text{O}$  — паровой фазой, 3 сут., *b* — тот же, с  $\text{AlCl}_3$ , 1 сут., *c* — то же, 4 сут., *d* — то же, с  $\text{KCl} + \text{AlF}_3$ , 3 сут., *e* — то же, 7 сут. *S* —  $(\text{Mg}, \text{Al})$ —серпентин, *A* — андалузит, *T* — топаз, *MF* — фторид магния (другие обозначения см. на фиг. 1)

Фиг. 3. Дифрактограммы, иллюстрирующие поведение природного биотита в различных гидротермальных средах

*a* — исходный биотит; *b* — тот же, обработанный чистой  $\text{H}_2\text{O}$  — паровой фазой, 3 сут.; *c* — тот же, с  $\text{AlCl}_3$ , 1 сут.; *d* — то же, 4 сут.; *e* — тот же, с  $\text{AlF}_3 + \text{KCl}$ , 3 сут.; *f* — то же, 7 сут. *M* — мусковит, *Chl* — хлорит (другие обозначения см. на фиг. 1 и 2)