PHYSICAL METHODS OF IDENTIFICATION OF THE FELDSPARS FROM GRANITIC PEGMATITES★

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Received September 26, 2006

The mineral samples analysed in this paper are K, Na, Ca aluminosilicates known as feldspars and belong to the granitic pegmatites developed in the Conțu-Negovanu area, in the Southern Carpathians (Romania). Some of the samples were subjected to X-ray powder diffraction analysis and the obtained data enabled the identification of the precise feldspar terms, as well as some of their structural characteristics. The determined refractive indices and the respective birefringence data, interferometrically determined for yellow Na radiation were plotted on a determinative chart, confirming the previous determined chemical composition of the feldspar terms. The IR spectra also display characteristic features of the determined feldspar terms.

Key words: pegmatites, feldspars, X-ray powder diffraction, main refractive indices, IR spectra.

I. INTRODUCTION

Pegmatites are wholly crystalline igneous or metamorphic rocks, usually very coarse grained and with major constituents which include minerals of extreme textural variations, especially in grain size. Giant crystals, with dimensions measured in meters and weighting several tons may occur in pegmatites, but the average grain size is centimetric. Pegmatites are known to range from acid to basic types (*i.e.* silica-rich to silica-poor), the granitic type (acid) being the most abundant in the world, including Romania. The pegmatite bulk composition is represented by the following major constituents: quartz, feldspars, micas (mainly muscovite and biotite), garnet, tourmaline, apatite; often less-abundant but very diverse and interesting minerals occur too, being referred to as accessory

★ Paper presented at the National Conference on Applied Physics, June 9–10, 2006, Galați, Romania

minerals. Therefore, the granitic pegmatites are an important source of minerals with classical use, such as: optical quartz and fluorite, ceramic and dental feldspars, refractory spodumene, sheet micas, gemstones (beryl, chrysoberyl, tourmaline, zircon, topaz, garnet *etc.*), but also, a source of a broad spectrum of hi-tech metals with general long-range technological potential: Li, Rb, Cs, Be, Ga, Sc, Y, REE, Sn, Nb, Ta, U, Th, Hf *etc.*

2. SAMPLES, ANALYTICAL AND THEORETICAL METHODS

Thirteen feldspar samples (F5, F6, F16, F17A, F25, F32A, F51, F52, F53, F54, F71, F72 and F89), collected from the Conţu-Negovanu pegmatites (Southern Carpathians), were investigated in order to obtain information on their identity, structural and optical characteristics. These feldspars developed in different mineral assemblages, displaying various physical and chemical properties. Generally, feldspars may be classified chemically as members of the ternary system $NaAlSi_3O_8$ (albite, Ab) – $KAlSi_3O_8$ (K-feldspar, *e.g.* sanidine, orthose, microcline, Or) – $CaAl_2Si_2O_8$ (anorthite, An). Compositions between $NaAlSi_3O_8$ and $KAlSi_3O_8$ are referred to as alkali feldspars and those between $NaAlSi_3O_8$ and $CaAl_2Si_2O_8$ as plagioclase feldspars. A purely chemical definition of plagioclases can be given in terms of Ab-An "molecular" percentages, but specific names are also used to denote the six compositional ranges (%) showing the anorthite percentage contents into which the series has been divided. These terms are: albite (0–10% An), oligoclase (10–30% An), andesine (30–50% An), labradorite (50–70% An), bytownite (70–90% An) and anorthite (90–100% An).

The previous study of the feldspar physical properties (colour, lustre, cleavage *etc.*), the optical features observed on slides in polarized light (mainly twinning), along with the mineral assemblage characteristics, enabled us to perform a preliminary identification. Thus, some of the investigated samples may be referred to as alkali feldspars: K-feldspars (microcline) and Na-feldspars (albite). Other samples, essentially Na-rich, but containing also rather important amounts of Ca, are assigned to the plagioclase isomorphic series, being represented mainly by albite and oligoclase terms, which usually occur in this type of rocks.

In order to perform the above mentioned investigations, six feldspar samples were carefully selected under binocular magnifying lenses and then turned into fine grained powders (F32A, F51, F52, F53, F54 and F89). Some of these samples and the other ones altogether were prepared in slides for optical studies at the polarizing microscope. The analyzed specimens were cut parallel to the optical axis, so that the flat plate can be investigated perpendicularly to one of the main planes of the crystal.

Wide Angle X-Ray Diffraction (WAXD) analysis was performed on a TUR M-62 diffractometer, using the Ni-filtered Cu-Kα radiation (λ = 0.1518 nm). The working conditions were 36 kV and 20 mA, the goniometer speed of 0.5°/min. All the difractograms were investigated in the range of 4–60°, 2θ degrees, at room temperature. The diffraction data were processed using a computer program for lattice parameter refinement (Holland & Redfern, 1997).

The main refractive indices of the studied feldspars were determined using a Rayleigh interferometer, etallonated with monochromatic radiation provided by a Na lamp (λ = 589,3 nm). A polarizer with its transmission direction parallel to one of the main axes of the analyzed crystalline layer has been introduced in the measure beam of the interferometer. An identical polarizer and a glass plate with the same thickness as the crystalline layer have been introduced in the comparison beam of the interferometer.

The specimen thickness was microscopically estimated by the distance on which the microscope tube must be translated in order to obtain good images for the both faces of the crystalline plate.

The displacement of the mobile zero-fringe obtained in white light relative to zero fringe of a fix fringe-system allows the evaluation of the supplementary introduced pathway by the anisotrope layer versus the glass plate. The relative displacement of fringes is expressed by the order of fringe *k*, from the fix fringe-system that superposes with the zero fringe of the mobile system:

$$\left(n - n_g\right)L = k\lambda \tag{1}$$

In (1) n is one of the main refractive indices of the anisotrope plate in which light propagates along a main direction; n_g represents the refractive index of the compensatory glass plate; k is the interference order that measures the displacement of the fringes, and λ is the light wavelength. The refractive index was estimated using the formula:

$$n_i = n_g + \frac{k\lambda}{L}; \qquad i = a, b, c \tag{2}$$

The polarized light measurements were made using the method described in [8–10]. In order to identify the main directions in the anisotrope crystal, two identical crossed polarizers were used.

One of the main refractive indices has been measured by counting the displacement of the fringes relatively to the central fringe of the fixed fringe system. The second main refractive index has been estimated after a rotation of the polarizer from the measured beam with 90 degree around the light propagation direction. Then the orientation of the anisotrope layer has been modified in such a way that light propagates along the other main direction. A common value is obtained of the two measurements.

IR spectra were obtained using a SPECORD M80 spectrometer, in KBr pills, in the spectral range 200–4000 cm^{-1}.

3. RESULTS AND DISCUSSION

X-ray powder diffraction patterns are first of all employed for the fully identification of the feldspar type. The peaks 131, $1\,\bar{3}\,1$, 060 and $\bar{2}\,04$ are of particular interest, being also used to establish some ordering aspects and structural state of the feldspars. Comparing the *d*-space data of the diffraction-peak positions and the obtained *hkl* reflections with the respective IMA files data, low albite was identified in the sample F53, oligoclase in the samples F32A and F54 and intermediate microcline in the samples F51, F52 and F89. Their unit-cell parameters *a*, *b*, *c* and α, β, γ, along with the cell volume *V* are listed in Table 1.

Although most of the diffraction peaks in the samples F51, F52 and F89 match those of the intermediate microcline, two $\bar{2}\,01$ peaks appear instead of only one, showing that these samples are in fact mixed species of two phases of sufficiently different compositions (Deer *et al.,* 1992), one of them being that of the intermediate microcline. Since the second $\bar{2}\,01$ peak is characteristic to the albite, it seems that the K-feldspar phase (microcline) and to a lesser extent the Na-feldspar phase (albite) coexist to a certain degree in each of the three samples.

In polarized light, these samples display substitution textures, involving a fine-scale intergrowth of the sodium-rich and potassium-rich phases, characteristic for the perthite. Therefore, the samples F51, F52 and F89, assigned to an intermediate temperature range of structures, may be safely referred to as intermediate microcline-perthite, term which designates both the structural and compositional state of this type of K-feldspar.

The intermediate microcline (IM), which describes the best the structural state of these feldspars, is a triclinic K-feldspar structural form, showing less ordering than the fully ordered low-temperature structural form called low or

Table 1

The unit-cell parameters of the investigated feldspar samples

sample	***a* [Å]**	***b* [Å]**	***c* [Å]**	**α [°]**	**β [°]**	**γ [°]**	**V [Å³]**
F-53	8.12	12.56	7.21	92.70	116.77	89.24	655.61
F-32A	8.00	12.27	7.09	84.89	116.37	89.20	662.50
F-54	8.16	12.87	7.10	94.31	116.60	89.31	–
F-51	8.54	12.93	7.23	–	115.45	–	–
F-52	8.65	12.66	7.44	95.66	–	–	–
F-89	8.66	12.59	7.44	–	–	–	–

maximum microcline (LM). Generally, the structure of microcline is typical of a framework silicate in which tetrahedra of $(Si,Al)O_4$ are linked to one another by sharing oxygens in all directions. Thus, in the microcline structure there are four distinct tetrahedral sites labeled $T_1(0)$, $T_1(m)$, $T_2(0)$ and $T_2(m)$. The microcline displays a large range of tetrahedral Al-Si order-disorder patterns, indicating locally variable conditions of cooling rate, pressure, deformation, chemical composition, presence and composition of fluids *etc.* In low microcline, the Al, Si atoms are fully ordered, Al being restricted entirely to the $T_1(0)$ site. The intermediate microcline may present varying degrees of ordering, showing the different degrees of Al occupancies in the tetrahedral sites; usually, the symbol for the Al occupancy corresponds to the symbol for the tetrahedral sites: $t_1(0)$, $t_1(m)$, $t_2(0)$ and $t_2(m)$. The degree of departure from the primordial monoclinic symmetry of the microcline species, which became triclinic on cooling, is called *obliquity* or *triclinicity* and a measure of this is obtained from the diffraction data, by the equation:

$$\Delta = 12.5\ (d_{131} - d_{1-31}) \tag{3}$$

whereas the Al amount in the $T_1(0)$ site may be calculated with the relation:

$$t_1(0) = 2\theta_{Cu(-204)} - 2\theta_{Cu(060)} \tag{4}$$

For the investigated K-feldspar samples, the obliquity (Δ) and the Al amount in the $T_1(0)$ site, calculated by means of the equations (3) and (4) are given in Table 2. These data show low values, confirming the intermediate crystallization temperature of this type of feldspars.

Table 2

The $d_{(131)}$ and $d_{(1-31)}$ space data, obliquity (Δ), $2\theta_{(-204)}$ and $2\theta_{(060)}$ data and the $t_1 0$ Al amount in the $T_1(0)$ site in the investigated K-feldspars

sample	$d_{(131)}$	$d_{(1-31)}$	$\Delta = 12{,}5(d_{(131)} - d_{(1-31)})$	$2\theta_{(-204)}$	$2\theta_{(060)}$	$t_1 0 = (2\theta_{(-204)} - 2\theta_{(060)})$
F-51	3.02	2.98	0.5	50.5	41.9	8.6
F-52	3.03	2.99	0.5	50.3	41.5	8.8
F-89	3.04	2.99	0.62	50.6	41.4	9.0

As for the albite, two structural modifications can occur: a low and a high form, both triclinic, the former being appropriate to formation at low temperature and the latter, at high temperature and very rapid cooling. In high albite the Si, Al distribution is highly disordered, whilst in low albite there is complete ordering of Al into the $T_1(0)$ site. In the investigated samples, the diffraction data match those of the low albite, identified only in sample F53. For the low albite, the Al, Si distribution is evaluated with a determinative diagram (Kroll & Ribbe,

1980) built up for the plagioclase feldspars, in which the (131) line separation is used plotted against the anorthite content (An %). According to this method, using the X-ray powder patterns for Cuk_{α}, the relation $\Delta 131 \equiv 2\theta_{(131)} - 2\theta_{(1-31)}$ can be calculated and the $t_1(0)$–$t_1(m)$ amount of Al can be estimated directly from the diagram.

In the last two samples (F32A and F54) the oligoclase was identified, as a term of the *albite-anorthite* plagioclase isomorphic series. Unlike the alkali feldspars, where on cooling the unmixing of Na and K and the ordering of Al and Si may develop independently of each other, in plagioclases the *M* and *T* cations do not migrate independently of each other and there must be a linkage between Na-Si and Ca-Al for charge balance to be preserved. Since Al and Si readily diffuse within the tetrahedral framework, fine scale microstructures develop and consequently, the lattice parameters represent averages over domains within the bulk crystal that may have distinctly different structures and chemical compositions. Therefore, it is not possible to determine the true Al, Si distribution in plagioclases, and only average ones are estimated (Kroll, 1983). A measure of the relative degree of the average Al, Si order in plagioclases may be obtained using the (131) line separation plotted against the anorthite content An (%), as presented above for the albite. In this diagram, the oligoclase samples are situated in the proximity of the 0.7 line of the $t_1(0) - t_1(m)$ amount of Al, showing them to be low plagioclases.

Some of these samples were investigated in polarized light and the direct study of the specific twinning patterns – which is the easiest way of feldspar identification – revealed the presence of the polysynthetic twins, which fully identifies the plagioclase feldspars. Being closely related to the chemical composition, the main refractive indices were calculated, in order to separate the albite and the oligoclase terms within the plagioclase isomorphic series (Table 3). Plotted on a determinative chart (Deer *et al*., 1992) (Fig. 1), the measurements of the main refractive indices showed that the investigated samples fall within the albite range (F6, F16, F17A, F71 and F72) and the oligoclase range (F5 and F25).

Table 3 contains also the birefringence data for the pairs of main axes, which match the already published values [4]. From Table 3 it results that $n_a \neq n_b$, so the analyzed feldspars are biax anisotropic crystals and the refractive indices are in the following correlation:

$$n_a < n_b < n_c \tag{5}$$

Relation (5) suggests that optical axes are situated in aoc main plane.

Some feldspar IR spectra contain characteristic vibration bands, as follows:

F51 (Kfeldspar): 3450 (m); 1640; 1625 (w); 1142(i); 1050 (i); 1015 (i); 775 (m); 725 (m); 650 (m); 580 (i) 540 (m); 460 (s); 425 (i); 375 (m); 325 (m).

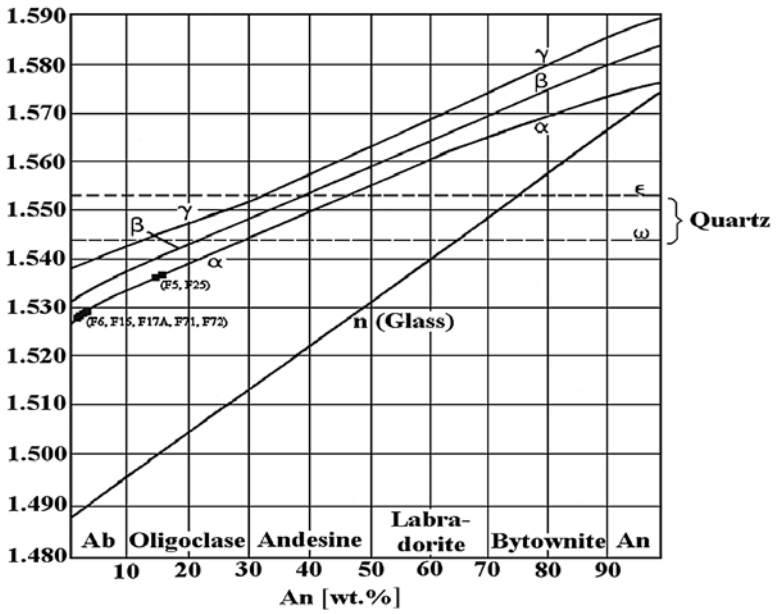

Fig. 1 – Plagioclase feldspar determinative chart (Deer *et al.*, 1996).

Table 3

The main refractive indices and the corresponding birefringence of some feldspar samples

Sample	n_a	n_b	n_c	Δn_{ab}	Δn_{bc}	Δn_{ac}
F5	1.5360	1.5422	1.5482	0.0062	0.0060	0.0122
F6	1.5275	1.5315	1.5375	0.0040	0.0060	0.0100
F16	1.5275	1.5315	1.5376	0.0040	0.0060	0.0100
F17A	1.5278	1.5318	1.5378	0.0040	0.0060	0.0100
F25	1.5362	1.5423	1.5480	0.0061	0.0057	0.0118
F71	1.5279	1.5319	1.5379	0.0040	0.0060	0.0100
F72	1.5280	1.5318	1.5379	0.0038	0.0061	0.0099

F 54 (oligoclase): 3450 (w); 1135 (i) 1162(i); 1100 (i); 1025 (i); 1008 (i); 775 (m); 762 (m); 700 (m); 640 (m); 575 (m); 535 (m); 470 (m); 425 (m); 400 (m); 375 (m).

F 53 (albite): 3450 (w); 1165 (i); 1100 (i); 1040 (i); 1025 (i); 990 (i); 785 (m); 760 (m); 748 (m); 725 (m); 650 (m); 590 (i); 465 (i); 440 (i); 400 (i); 375 (i); 335 (m).

The wave-numbers are expressed in cm^{-1} and in parentheses are indicated the intense (i); moderate intense (m), weak (w) and shoulder (s) infrared bands.

4. CONCLUSIONS

In the analysed samples, the specific feldspar terms were fully identified using the different methods of investigation.

a) The X-ray powder diffraction data enabled the identification of intermediate microcline as a K-feldspar term and also of the plagioclase terms oligoclase and albite.

b) The study of the refractive indices assigned some of the investigated samples to the albite and oligoclase range. The main refractive indices and the birefringence values of the investigated feldspar samples (interferometrically determined for yellow radiation of Na) showed that these are biax anisotropic crystals and their optical axes are situated in the aoc main plane.

c) The IR spectral bands revealed characteristic features of the feldspar terms oligoclase, albite and K-feldspar (microcline).

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