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Structural Investigation of K-Feldspar KAlSi₃O₈ Crystals by XRD and Raman Spectroscopy:

An Application to Petrological Study of Luc Yen Pegmatites, Yen Bai Province, Vietnam

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K-feldspars in pegmatites from Luc Yen gem mining area, Yen Bai province, Vietnam were studied by X-ray fluorescence, X-ray powder diffraction and the Raman spectroscopy. Chemical analysis determined the K-feldspars in the form of $(K_{0.8909}Na_{0.0388}Ca_{0.002}Pb_{0.0042}Cs_{0.0024}Rb_{0.0338})(Al_{0.9975}Fe_{0.0053}Ti_{0.0004})Si_{2.988}O_8$. Both X-ray powder diffraction and Raman spectroscopy indicated Luc Yen K-feldspars as orthoclase phase. Together with the values of Al content of the T1 tetrahedral sites in orthoclase, it is understood that Luc Yen pegmatites are of young ages (Cenozoic) and shallow intrusive types.

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1. Introduction

Luc Yen is a well-known gem mining area for ruby, sapphire, spinel, tourmaline, and green K-feldspar, located in Yen Bai province in the north of Vietnam, 270 km northwest from the capital city Hanoi. Green K-feldspars can be found together with tourmalines in pegmatite bodies, and they have been of scientific interests as many of them possess distinct transparency which led to the recognition as one of famous gems from the area [1]. From petrological point of view, feldspar is an important aluminium tectosilicate mineral in pegmatite as whose chemical composition and structural state can bring information on geochemical signatures, petrogenetic relationships with evolved pegmatite origin and processes of formation. The structural state refers to the Al and Si distribution in tetrahedral site of the framework structure which depends on the temperature of crystallization. Above 450 °C, K-feldspar crystallizes in monoclinic system with the disordered Al–Si distribution in tetrahedral site forming the orthoclase member. At lower temperature, K-feldspar crystal shows an ordered Al-Si distribution of the microcline polymorph with triclinic symmetry [2]. Determination of the certain phase of feldspar can help petrologist understand mineralization condition and petrological processes in pegmatite.

2. Materials and methods

Totally, eight K-feldspar crystals from Luc Yen were collected for this study. All samples possess the apple

green colour and good transparency. All samples vary from 1 cm to 2 cm in size, with one exception is a crystal sized up to 6 cm (Fig. 1). Chemical analyses were obtained by X-ray fluorescence (XRF) for 4 samples. Major elements were determined on fused glass disks. These were prepared by mixing 0.4 g of "dry basis" sample powder with 5.2 g of LiBO₄ flux mixture. The mixture was poured into a crucible (95% Pt-5% Au), and heated to 1000 °C in a furnace with space for four crucibles for the simultaneous preparation of glass disks. Trace elements were analyzed in powder pellets prepared by mixing thoroughly 6 g of sample powder with wax-C (Hoechst) added as a binding agent. Major and trace elements were analyzed in a sequential spectrometer BRUKER SRS3000, with an end window Rh target. Structural state of feldspar was studied by means of the Raman spectroscopy and X-ray powder diffraction



Fig. 1. K-feldspar crystal from Luc Yen pegmatites.

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(XRD). The Raman spectra were collected for all eight samples with a Jobin Yvon LabRam HR 800 spectrometer coupled with a Si-based CCD (charge-couple device) detector; samples were excited by a 514 nm green Ar^+ ion laser. XRD measurements were done for four samples on Seifert XRD 3000 TT powder diffractometer with Cu K_{α} radiation. The data were then refined with the Rietveld method using FPS software.

3. Results and discussion

The average composition of the samples obtained from XRF data can be expressed into the K-feldspar formula as following:

 $(K_{0.8909}Na_{0.0388}Ca_{0.002}Pb_{0.0042}Cs_{0.0024}Rb_{0.0338})$

$$\times (Al_{0.9975}Fe_{0.0053}Ti_{0.0004})Si_{2.988}O_8.$$

The concentration of sodium in the feldspar samples is very low and calcium content is negligible in all cases. Remarkable among minor and trace elements are Pb, Cs, Rb, Fe, and Ti.

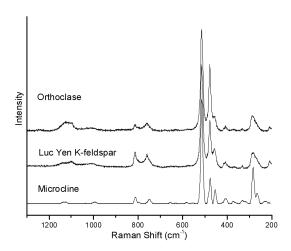


Fig. 2. Representative Raman spectra of Luc Yen K-feldspar indicates that its structure belongs to orthoclase category.

The Raman spectra obtained from Luc Yen K-feldspars and RRUFF database are shown in Fig. 2. The literature to date on the Raman spectra of K-feldspars shows that the two polymorphs of K-feldspar, i.e. microcline and orthoclase can be distinguished by the characteristic triplet in the range 450–515 cm⁻¹ and doublet in the range 250–290 cm⁻¹ [3].

The two ranges are both distributed to Si-O/Al-O stretching mode. In orthoclase, Si and Al both occupy the two crystallographically distinct tetrahedral sites, T1 and T2, whilst in microcline Al is found only on T1 sites. The disordering arrangement of Al in orthoclase associated with the present of the mirror plane in monoclinic symmetry results in the spreading of bond lengths and leads to the spread of frequency and broaden bands as well as less-resolved triplet and doublet in orthoclase Raman spectrum [4]. The Raman spectrum of Luc Yen

<i>a</i> [Å]	b [Å]		,	$V [\mathring{\rm A}^3]$	
	12.9745(5)				
	12.9730(2)				
8.5795(4)	12.9810(3)	7.1976(2)	116.112(2)	719.78	0.78
8.5585(3)	12.9785(3)	7.1950(2)	116.099(2)	717.71	0.79

K-feldspar indicates that the samples fall into the orthoclase category.

Experimentally determined XRD powder diffraction patterns also identified Luc Yen K-feldspar samples as individual phase of orthoclase. The refined cell parameters of 4 orthoclase samples are shown in Table I. According to a study of Kroll and Ribbe [5], the relations among Al content (t_1) of the T1 tetrahedral sites (i.e., t_1 = the number of Al atoms occupying T1 tetrahedral sites) and lattice parameters can be expressed by the following equation:

$$\sum t_1 = 2t_1 = \frac{b - 24.8095 + 74.9054c*}{-3.3261 + 19.5012c*}.$$

The Al content of the T1 tetrahedral sites vary from 0.78 to 0.82 (please see Table I, $\sum t_1 = 0.78-0.82$) which corresponds to orthoclase hosted in pegmatites of young ages (usually Cenozoic) and normally shallow-level intrusive [6]. This is consistent with our absolute K/Ar dating data in Luc Yen K-feldspar which show the absolute age of 30.58 Ma.

4. Conclusion

Structural investigation of K-feldspar from Luc Yen pegmatites by XRD and Raman spectroscopy indicate that K-feldspar from the area is of individual orthoclase phase. The Al content of the T1 tetrahedral sites calculated from lattice parameters leads to an understanding that the orthoclase samples were hosted in pegmatites of young ages (Cenozoic) and shallow intrusive types. This is confirmed by the K/Ar absolute age determinations.

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