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Fluid inclusion evidence of second immiscibility within magmatic fluids (79AD eruption of Mt. Vesuvius)

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ABSTRACT. — Foid-bearing syenite cognate xenoliths represent fragments of the upper peripheral parts of the K-phonolitic portions of the 79 AD magma chamber, wrenched during the explosive eruption. Abundant multiphase fluid inclusions are hosted within K-feldspar and coexist with rare silicate melt inclusions. This gives evidence to the exsolution of a magmatic volatile-rich phase from the peripheral parts of the silicate magma chamber. The characterization of daughter mineral assemblage of these fluid inclusions, by SEM-EDS and Raman spectroscopy, indicates two ubiquitous main components: Na-K chlorides (halite + sylvite) and Na-Ca carbonates (calcite ± nahcolite). Microthermometric experiments indicate nearly magmatic trapping temperatures (760°C to 830°C) of the homogeneous chloride-carbonate liquid. Cooling of such liquid produces two immiscible melt phases (chlorides and carbonates) at 455-435°C. This suggests that a hypersaline-carbonate fluid, exsolved from the silicate magma, can further experience another unmixing event that would occur in essentially «post-magmatic» environment.

KEY WORDS: *Vesuvius, foid-bearing syenites, fluid inclusions, immiscibility, exsolution.*

RIASSUNTO. — Tra gli xenoliti presenti nei depositi dell'eruzione esplosiva del 79 d.C. sono presenti frammenti di sieniti a feldspatoidi che sono state interpretate come derivanti dall'erosione delle porzioni periferiche delle parti alte K-fonolitiche della camera magmatica. I K-feldspati di queste rocce ospitano numerose inclusioni fluide multifase, che testimoniano l'essoluzione di una fase fluida acquosa di origine magmatica dalle porzioni periferiche della camera magmatica. La caratterizzazione delle associazioni di minerali figli contenuti in queste inclusioni, svolta utilizzando microscopia elettronica a scansione (SEM-EDS) e spettroscopia Raman, ha rilevato due componenti principali sempre presenti in queste inclusioni: una associazione dominata dai cloruri (halite + silvite) ed una associazione dominata dai carbonati (calcite ± nahcolite). Gli esperimenti microtermometrici hanno evidenziato temperature di intrappolamento prossime a quelle magmatiche (760°C to 830°C) e composizione omogenea del fluido intrappolato. Durante il raffreddamento, è stato osservato che una goccia di fuso si separa dal liquido omogeneo e e si smescola, tra 455°C e 435°C, ripartendosi in due fusi (cloruri e carbonati?). Il comportamento di queste inclusioni suggerisce che un fluido ipersalino, essolto dal magma, può subire un'ulteriore fase di smescolamento che avverrebbe in condizioni essenzialmente «post magmatiche».

PAROLE CHIAVE: *Vesuvio, sieniti a feldspatoidi, inclusioni fluide, immiscibilità, essoluzione.*

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INTRODUCTION

During cooling and crystallization of a silicate magma, one or several events of the exsolution of globules of a separate new immiscible fluid phase are likely to occur. Processes of magmatic immiscibility (melt-fluid, fluid-fluid, fluid-gas) are clearly recorded by fluid inclusion studies (Roedder, 1992). Evidence for direct evolution of a hypersaline fluid phase from a silicate melt is shown by the heterogeneous trapping of melt and fluid with variable proportions (Roedder, 1992; Frezzotti, 1992, 2001). Obviously, immiscible phase separation during cooling and crystallization of the magma is an inherently fugitive phenomenon and fluid inclusions may provide the only remaining evidence of this process. Fluid inclusions can both prove the existence of immiscible phase separation and constrain the compositional signature of the process (Roedder, 1984; Lowenstern, 1994; Kamenetsky *et al.*, 2003). The evolution of magmatic fluids from Vesuvius magma chambers is recently reported by Fulignati *et al.* (2001) and Gilg *et al.* (2001).

The Vesuvius magmatic system is characterized by the presence of shallow reservoirs hosted within the carbonate Mesozoic basement (dolostone and limestone) (Barberi and Leoni, 1980). The Vesuvius magma chamber before the 79AD eruption consisted of two compositionally distinct portions with a volatile-rich (H_2O, Cl, F) K-phonolitic magma capping phonotephritic magma (Cioni *et al.*, 1995). The outer margin of the 79 AD magma chamber, consisting of crystallized magmas (clinopyroxenites to foid-bearing K-syenites), continuously grades into carbonate country rocks through a skarn shell and thermometamorphic marble (Fulignati *et al.*, 2004). Foid-bearing syenite cognate xenoliths, representing the upper peripheral parts of the 79AD magma chamber, often host hypersaline fluid inclusions testifying the exsolution of a non-silicate, volatile-rich phase from the phonolitic magma at crystallizing front of the 79AD Vesuvius magmatic reservoir (Fulignati *et al.*, 2004). In the case of our study we have rare opportunity to describe a further stage of immiscibility of the exsolved magmatic volatile-rich phase «caught in the act».

ANALYTICAL TECHNIQUES

Microthermometric experiments on fluid inclusions were carried out using a Linkam TS 1500 heating stage (Dipartimento di Scienze della Terra, Pisa). Heating rates range from 5 to 30°C/min. Different cooling rates (50°C/min, 20°C/min, 10°C/min, 3°C/min) were used to record fluid immiscibility and crystallization of daughter minerals within inclusions. The accuracy of the measurement was around ±10°C controlled by the melting point of $K_2Cr_2O_7$, silver and gold.

Major and trace elements of whole-rock samples were analysed by XRF (Philips PW 1480 spectrometer) at Dipartimento di Scienze della Terra, University of Pisa.

SEM-EDS analyses were performed using a Philips XL30 apparatus equipped with EDAX DX4 (Dipartimento di Scienze della Terra, Pisa), at 20kV accelerating energy and about 0.1 nA beam current. The inclusions were opened by fracturing the host crystals in air. The samples were carbon coated and analyzed immediately after, with the aim to prevent as much as possible the lost of the daughter minerals, following the procedure reported by Metzeger *et al.* (1977). The spectra collected on daughter minerals in opened fluid inclusion often show peaks originated by excitation of the host sanidine or other daughter minerals in the same inclusion. In order to prevent interferences and to identify the peaks characteristic of a single mineral phase, several analyses were made on each solid phase in different positions.

Raman analyses were performed with a confocal Labram multichannel spectrometer of the Jobin-Yvon instruments (Dipartimento di Scienze della Terra, Siena). The excitation line at 514.5 nm was produced by an Ar^+ laser. The

Raman intensity was collected with a Peltier-cooled CCD detector. The beam was focused to a spot size of about 1-2 μm using an Olympus 100X lens. The scattered light was analyzed using a Notch holographic filter with a spectral resolution of 1.5 cm^{-1} at a grating of 1800 grooves mm.

Results

The studied foid-bearing syenites have a bulk rock K-phonolitic compostion (Table 1) and consist of a network of euhedral crystals of K-feldspar (Or_{87-85} Ab_{12-14}); other constituents are leucite, nepheline, K-pargasitic amphibole, garnet ($Andr_{72-68}$ $Gross_{23-27}$) and biotite (Mg# 55-46) (Table 2).

TABLE 1

Representative analyses of major and trace elements of foid-bearing syenites

	SAN-5	SAN-7
SiO_2 (wt.%)	57.48	60.96
TiO_2	0.29	0.17
Al_2O_3	18.76	19.75
Fe_2O_{3tot}	2.31	1.66
MnO	0.09	0.05
MgO	0.16	0.28
CaO	3.17	2.94
Na_2O	3.99	3.39
K_2O	12.45	9.47
P_2O_5	0.03	0.02
L.O.I. (925°C)	0.76	1.31
Nb (ppm)	63	26
Zr	504	162
Y	16	7
Sr	399	501
Rb	417	334
Ce	124	53
Ba	158	339
La	85	33
Ni	6	3
Cr	11	2
V	48	26
Co	2	2
Cu	8	12
Zn	47	20

L.O.I.= loss on ignition

Abundant multiphase fluid inclusions are hosted within K-feldspar crystals. Based on petrographic observations, the shape and orientation of inclusions is consistent with entrapment during crystal growth, and thus the studied inclusions are identified as primary, following criteria provided by Roedder (1984) and Goldstein (2003). They consist of a deformed vapor bubble, small amount of interstitial aqueous liquid, clear daughter minerals of cubic shape (halite and sylvite), other rounded and tabular daughter minerals (sometimes birefringent), and also opaque crystal(s) (Fig. 1a, b). Rare inclusions contain significant component of clear silicate glass (Fig. 1c). In such inclusions one or several fluid-bearing globule(s) are also present, and the glass/globule ratio is highly variable.

Microthermometric experiments

Heating/cooling experiments were carried out only on the multiphase fluid inclusions. During heating, solid phases begin to dissolve at about 100°C and cubic daughter minerals melt completely between 515°C and 570°C (Fig. 2) with a modal value at about 540°C (Fig. 3). The low temperatures of melting argue for a non-silicate composition of daughter minerals (chlorides?). Small tabular daughter minerals (carbonates?) still remain until about 700°C. The narrow range in temperature of phase transformations suggests a fairly constant bulk composition of studied inclusions and their daughter phases. If the trapped fluid is approximated by a simple NaCl aqueous solution the corresponding salinities would be in the range 62-66 wt.% $NaCl_{equiv}$ (Sterner *et al.*, 1988). However, because the inclusions contain complex daughter mineral assemblage the above estimate should have large errors. The remaining vapor bubble homogenizes at about 810°C (Fig. 2, 3). During subsequent

TABLE 2

Representative analyses of sanidine, biotite, amphibole and garnets of foid-bearing syenites

	sanidine	sanidine	sanidine	sanidine	sanidine	sanidine	sanidine	sanidine	sanidine	sanidine
SiO_2	64.98	64.28	64.75	64.45	64.50	64.51	64.52	64.86	64.60	64.64
Al_2O_3	18.81	18.92	18.75	18.80	18.95	18.77	19.00	18.64	19.18	19.11
FeO_{tot}	bdl	0.06	0.10	0.16	bdl	0.13	0.06	0.04	bdl	0.09
CaO	bdl	0.06	bdl	0.04	bdl	0.08	0.12	bdl	0.06	0.20
Na_2O	1.53	1.68	1.57	1.61	1.65	1.53	1.63	1.48	1.39	1.55
K_2O	14.68	15.00	14.83	14.94	14.90	14.98	14.68	14.98	14.77	14.41
tot	100	100	100	100	100	100	100	100	100	100
An	0.0	0.3	0.0	0.2	0.0	0.4	0.6	0.0	0.3	1.0
Ab	13.7	14.5	13.9	14.0	14.4	13.4	14.4	13.1	12.5	13.9
Or	86.3	85.2	86.1	85.8	85.6	86.2	85.1	86.9	87.2	85.1

	biotite	biotite	biotite	biotite	biotite	amphibole	amphibole	amphibole	amphibole	amphibole
SiO_2	37.92	38.10	38.14	38.10	37.41	37.04	36.96	37.00	36.99	37.45
TiO_2	2.83	2.76	2.27	2.16	2.64	1.75	1.54	2.00	1.57	1.37
Al_2O_3	15.14	14.65	14.39	14.33	14.30	14.35	13.77	13.82	14.03	13.85
FeO_{tot}	19.05	21.53	22.13	22.97	23.80	22.31	23.03	22.60	22.03	23.08
MnO	1.02	1.03	1.10	1.06	0.85	1.33	1.27	1.48	1.13	1.27
MgO	13.12	11.59	11.13	10.94	10.39	6.32	6.30	6.26	5.96	5.92
CaO	bdl	0.22	0.08	bdl	bdl	12.05	12.09	11.94	12.29	12.04
Na_2O	0.47	0.17	0.55	0.30	0.21	1.71	1.70	1.87	1.70	1.66
K_2O	10.45	9.96	10.22	10.14	10.39	3.14	3.33	3.03	3.50	3.36
tot	100	100	100	100	100	100	100	100	100	100
Mg#	55	49	47	46	44	34	33	33	32	31

	garnet	garnet	garnet	garnet	garnet	garnet	garnet	garnet	garnet	garnet
SiO_2	34.85	34.99	34.87	34.41	34.97	35.63	35.24	34.83	34.01	34.03
TiO_2	2.47	2.16	2.51	2.88	2.29	0.74	0.83	2.51	3.18	3.19
Al_2O_3	7.26	6.50	6.88	6.61	5.61	4.94	4.53	5.15	5.36	5.15
$Fe_2O_{3\,tot}$	19.88	20.71	19.99	21.33	21.89	23.70	25.04	22.29	22.70	22.81
MnO	1.99	2.41	1.80	1.92	1.82	1.21	1.99	1.21	1.85	1.69
MgO	0.11	0.17	bdl	0.08	0.30	bdl	bdl	0.25	0.29	0.28
CaO	33.45	33.06	33.94	32.77	33.13	33.79	32.36	33.75	32.61	32.84
tot	100	100	100	100	100	100	100	100	100	100
Andr.%	68	69	70	72	74	76	77	77	78	79
Gross.%	27	25	26	23	21	21	18	19	17	16
Spess.%	4	5	4	4	4	3	5	3	4	4
Pyrope%	0	1	0	0	1	0	0	1	1	1

bdl= below detection limit

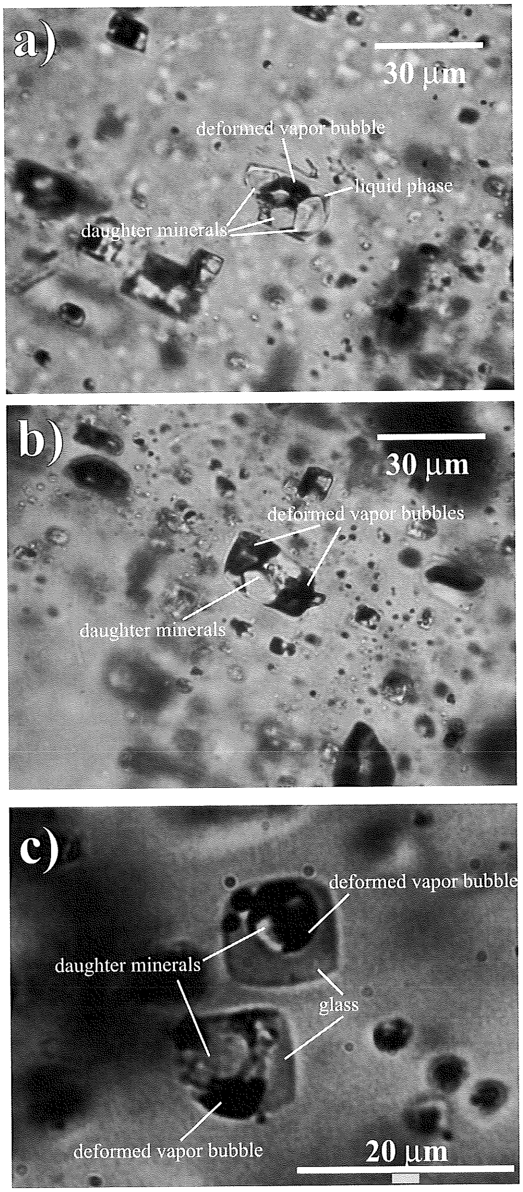

Fig. 1 – Photomicrographs of: a and b) multiphase fluid inclusions in K-feldspar crystals of foid-bearing syenites; c) multiphase silicate melt inclusions in K-feldspar crystals of foid-bearing syenites. Plane polarized light.

cooling, vapor bubble nucleates and progressively increases in size (Fig. 4). At about 500°C a bleb of melt is visible. In the interval 455-435°C this phase undergoes further unmixing, followed by formation of at least two liquids (Fig. 4). Globules of one liquid float freely in the matrix of another liquid, change their shape and size, coalesce and split apart continuously down to 100-150°C (Fig. 4). The movements of globules slow down with decreasing temperature until final solidification at 40-50°C. This behavior of the inclusions was observed in all experiments (~30) and the results can be reproduced for a single inclusion many times. The change in the cooling rate does not affect the behavior of fluid inclusions and the temperatures of unmixing.

Daughter mineral identification

Daughter minerals in the multiphase fluid inclusions are suggestive of the main compounds that constitute the trapped fluid. The SEM-EDS study on opened fluid inclusions (Fig. 5) reveals the presence of a complex daughter mineral assemblage in which the most abundant phases are halite and sylvite; other phases are calcite, fluorite, sulfates (anhydrite and glaserite) and sulfides (pyrite), and a very small tungsten-bearing phase (oxide?) is also identified (Fig. 5, Table 3) (see also Fulignati *et al.*, 1998). Daughter minerals are furthermore characterized by a laser Raman spectroscopy study. This confirms the ubiquitous occurrence of calcite in all inclusions investigated, whereas sulfates (thenardite, glaserite), nahcolite and an unidentified fluoride are also found (Table 3). During Raman spectroscopic measurements we noted spontaneous dissolution of small daughter crystals and movement of the bubble. We argue that this behavior is due to a sudden increase of temperature caused by the interaction of the laser beam with these crystals (Fe-chlorides? M.L. Frezzotti, pers. comm.). Raman spectroscopy also reveals the presence of CO_2 in the vapor bubble.

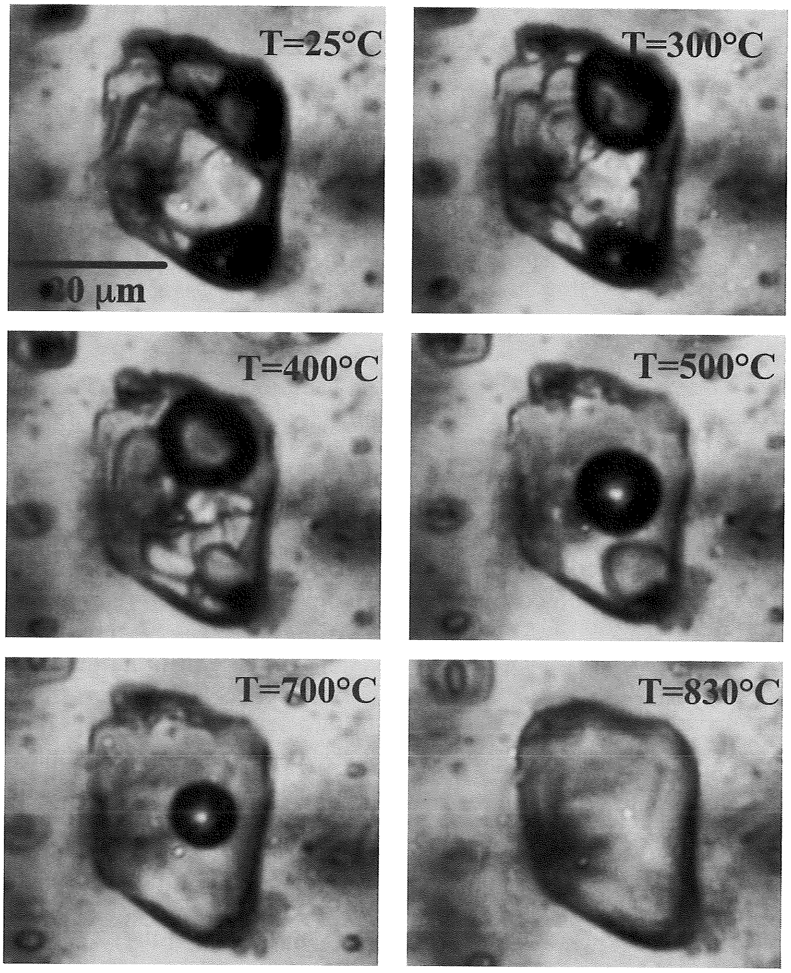

Fig. 2 – Pictures showing the behavior of multiphase fluid inclusions during heating experiments. Note at 25°C the strongly deformed vapor bubble and the abundance of daughter minerals. The liquid phase is not observable. At 300°C and 400°C the vapor bubble is rounded. Note the smoothed edges of most of the daughter minerals (chlorides), indicative of the progressive dissolution of the salts. At 500°C, only one daughter mineral is visible (halite). At 700°C all solid phases are disappeared and the bubble size is significantly reduced. At temperature higher than about 810°C the inclusion reaches the total homogenization in liquid phase.

DISCUSSION AND CONCLUSION

Foid bearing syenites of the 79 AD Vesuvius eruption are considered to be formed in the upper parts of the magma chamber (Fulignati *et al.*, 2004). The foamy appearance of these rocks can be interpreted to result from crystallization of K-feldspar crystals in the presence of exsolved fluids accumulating under the roof of the magma chamber (Lowenstern and Sinclair, 1996). Clear evidence for the processes of volatile-phase exsolution is represented by the coexisting multiphase melt (silicate glass and volatile-rich globules) and

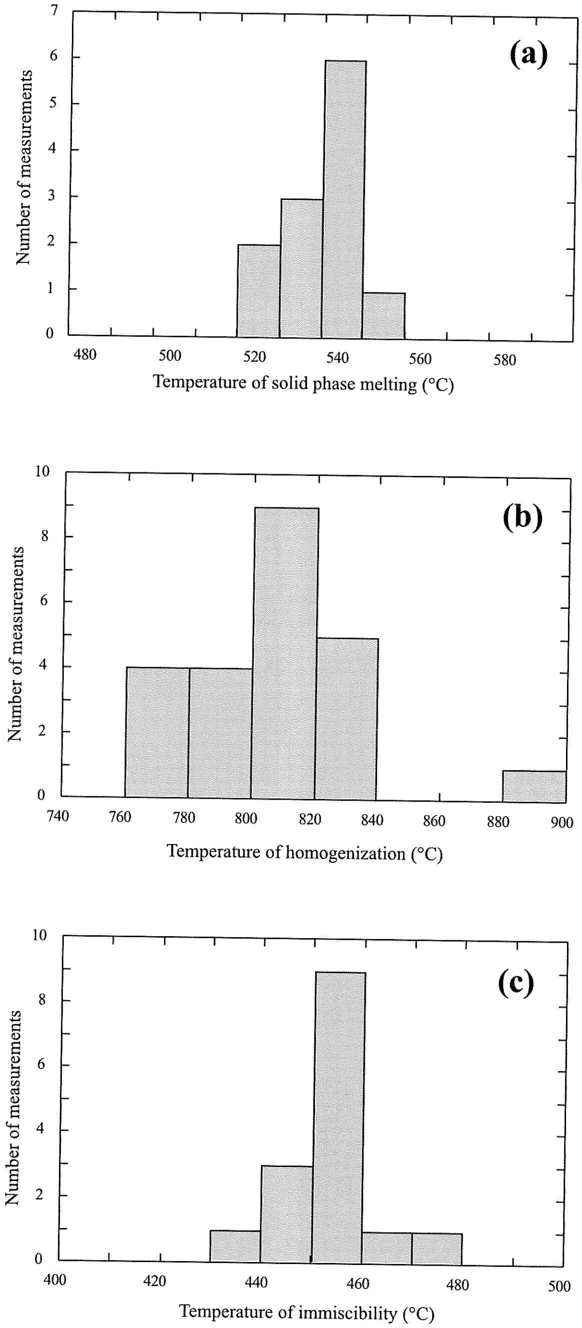

Fig. 3 – Histograms of: (a) temperature of final melt of daughter minerals; (b) temperature of homogenization of vapor bubble; (c) temperature of unmixing of two melts in the inclusions during cooling after the total homogenization.

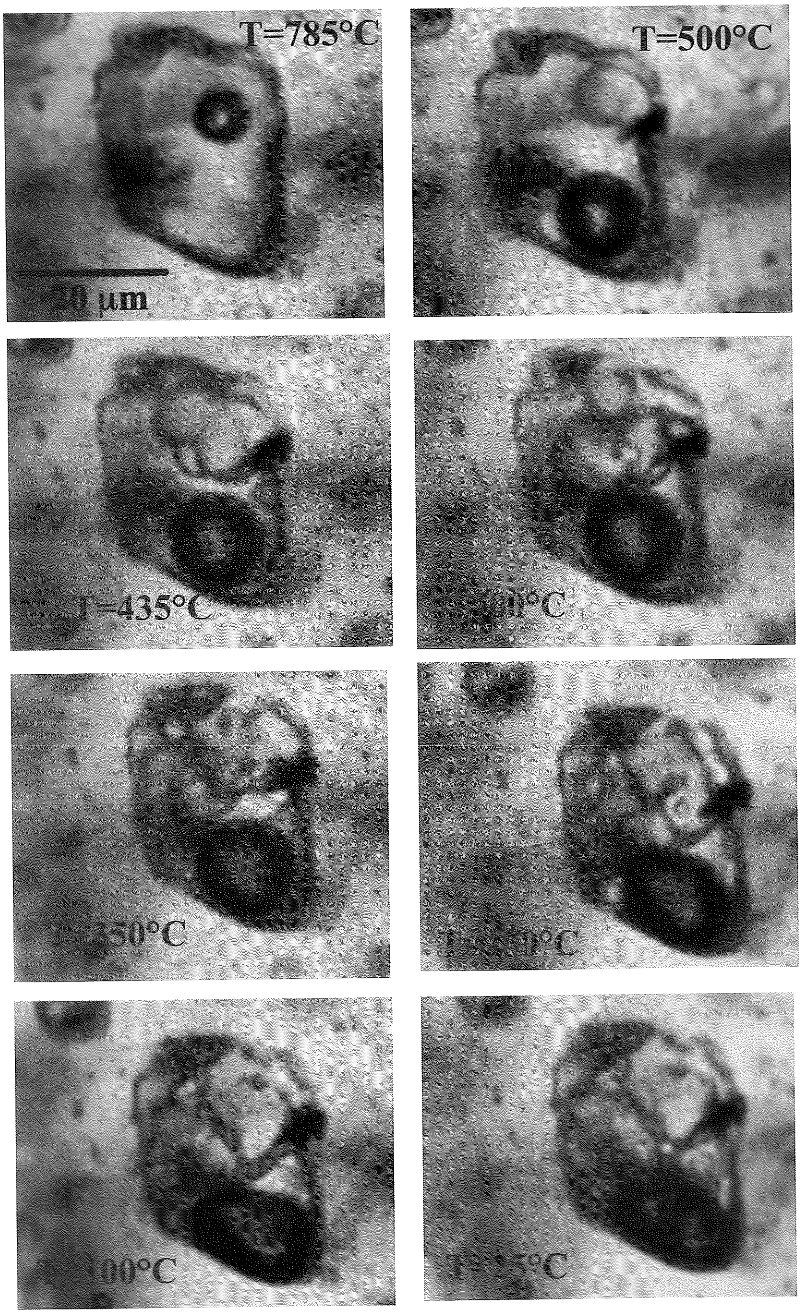

Fig. 4 – Pictures showing the behavior of multiphase fluid inclusions during cooling after the total homogenization. At 785°C the vapor bubble is nucleated. At about 500°C a bleb of melt appears. The globules visible at 435°C and 400°C represent melts unmixed from the bleb upon further cooling (probably chloride-bearing melt and carbonate-bearing melt).At lower temperatures the melts begin to crystallize until final solidification.

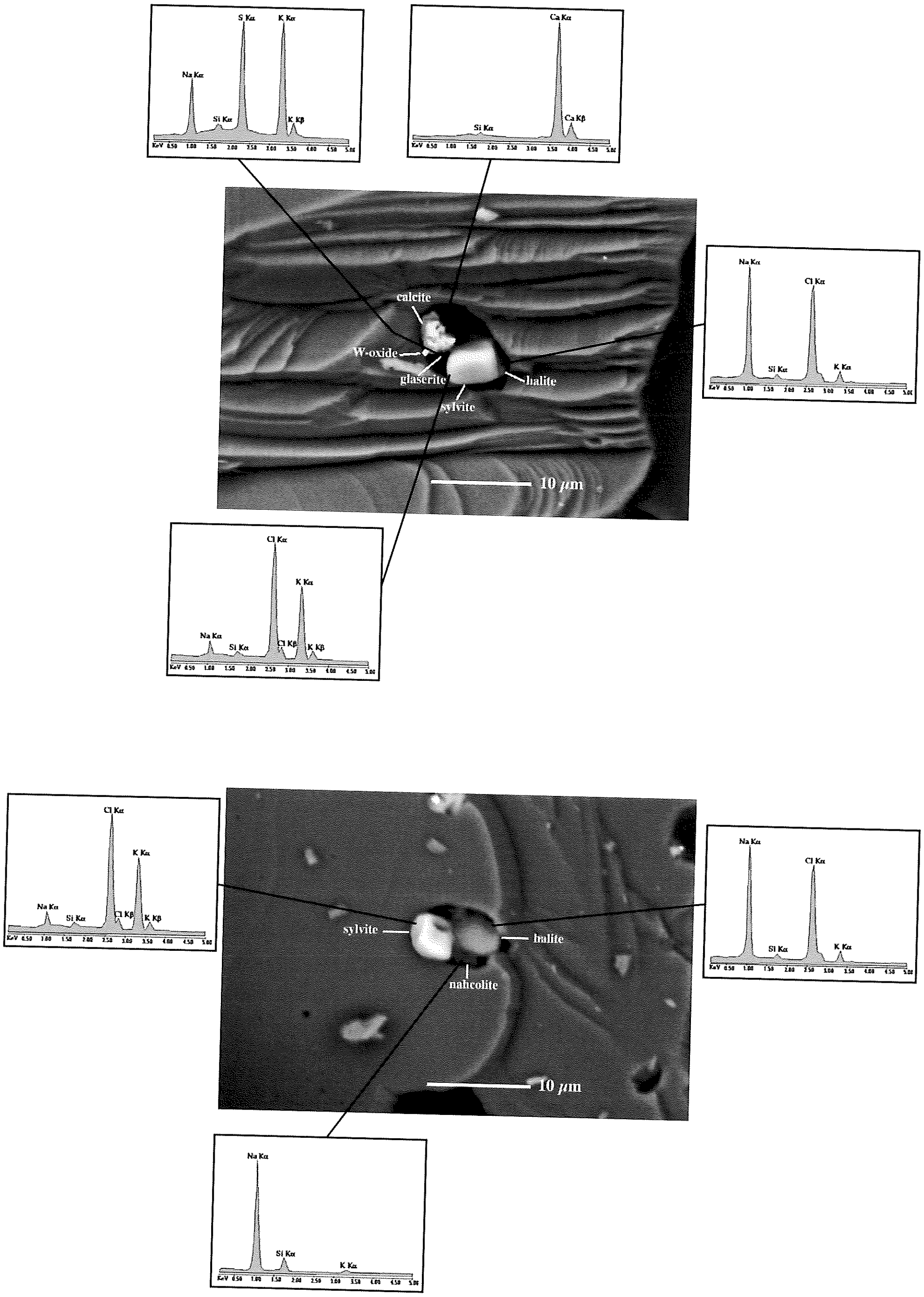

Fig. 5 – Secondary electron microscope images of opened multiphase fluid inclusions and EDS spectra of some daughter minerals.

TABLE 3

Daughter minerals identified by Laser Raman spectroscopy and SEM-EDS analyses

Daughter minerals			
Laser Raman spectroscopy		SEM-EDS	
calcite	[$CaCO_3$]	*halite*	[NaCl]
nahcolite	[$NaHCO_3$]	*sylvite*	[KCl]
thenardite	[Na_2SO_4]	*calcite*	[$CaCO_3$]
glaserite	[$(K, Na)_2SO_4$]	*nahcolite*	[$NaHCO_3$]
unknown fluoride	[?F]	*glaserite*	[$(K, Na)_2SO_4$]
		anhydrite	[$CaSO_4$]
		fluorite	[CaF_2]
		pyrite	[FeS_2]
		tungsten oxide	[W?]

fluid inclusions, analogously to what has been reported by Fulignati *et al.* (2001) in foid-bearing syenites of the 472 AD Vesuvius eruption. In particular, the strong variability of the glass/globule(s) ratio, found in the multiphase melt inclusions, is interpreted as a strong evidence for that silicate melt and essentially non-silicate fluids coexisted and were trapped heterogeneously (Roedder, 1992; Frezzotti, 1992, 2001; Lowenstern, 1995; Kamenetsky *et al.*, 2003). Similar melt inclusions were also described by Clocchiatti and Nativel (1984) in syenites of Cilaos. The exsolved fluids are represented in our samples by abundant multiphase fluid inclusions. The investigation of these inclusions can thus provide insights into the composition of magmatic fluids present during crystal growth.

The fact that all inclusions show similar phase transformations at a given temperature during heating experiments suggests that they have fairly homogeneous composition of the fluid trapped. Magmatic origin of this fluid is supported by high homogenization (trapping) temperatures of about 810°C that are very close to magmatic crystallization temperatures of 850-900° C estimated for the 79 AD phonolitic magma (Cioni *et al.*, 1995). During cooling a bleb of melt unmixes into two conjugate melts. It is worth noting that the observed unmixing occur under experimental conditions at 1 atm and extrapolation to natural systems should be taken with caution. Nevertheless, we may extrapolate this behavior to natural systems in which a hydrosaline-carbonate fluid, originally homogeneous at magmatic temperatures, may undergo unmixing in subsolidus environment. This is suggested by repeated experiments at different cooling rates showing that unmixing occurs inevitably at certain temperature, i.e. with no relation to the cooling rate. On the other hand, recent studies of melt inclusions have broadened the understanding of melt-fluid evolution in pegmatite systems, showing that the continuous miscibility between water and complex silicate melt is possible at magmatic temperature, and a separation into two coexisting melts can be induced by simple cooling (Breiter *et al.*, 1997; Thomas *et al.*, 2000; Sirbescu and Nabelek, 2003).

The characterization of daughter mineral assemblage of the studied multiphase fluid inclusions in foid-bearing syenites indicates two ubiquitous main components: a chloride-bearing association (halite + sylvite) and a carbonate-bearing association (calcite ± nahcolite). This allows interpreting unmixed phases as globules of the Na-K chloride melt set in the matrix of Na-Ca carbonate melt. The occurrence of immiscibility between chloride and carbonate components in magmatic environments is also reported by Keller and Krafft (1990), Mitchell (1997), Fulignati *et al.* (2001) and Kamenetsky *et al.* (2002).

Processes of exsolution of an aqueous fluid phase during late magmatic differentiation play important role in the generation of many magmatic-hydrothermal ore deposits (Hedenquist and Lowenstern, 1994). The results of this work suggest that the exsolved hypersaline-carbonate fluid, immiscible with the silicate magma, could further experience another unmixing event that would occur in essentially «post-magmatic» environment (after the crystallization of the foid-bearing syenite). We anticipate that immiscibility between low viscosity, highly fugitive non-silicate melts may significantly influence partitioning of metals (already scavenged from silicate magmas) in the essentially hydrothermal environment and affect significantly the style and composition of ore mineralization.

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