

**EXPERIMENTAL INVESTIGATIONS ON THE PYROMETAMORPHIC FORMATION
OF PHOSPHOROUS-BEARING OLIVINES
IN PARTIALLY MOLTEN METAPELITIC GNEISSES**

by

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Abstract

In this study we perform experimental investigations on the formation of phosphoran olivine in partially molten metapelitic gneisses and compare the results to olivines recovered from a presumably La-Tène (450-15 B.C.) age sacrificial place near Ötz, Northern Tyrol, Austria, where immolation of ritual offerings took place. The investigated metapelitic gneiss samples, used in these experiments, have the protolith assemblage biotite + plagioclase + quartz. Experimental investigations have been performed at 1 bar and temperatures from 900-1300°C in graphite crucibles using rock cubes of these metapelitic gneisses from the firing site as starting materials with bones added as a source of phosphorus. Olivine forms by incongruent melting of biotite at $T > 1000^\circ\text{C}$ through the reaction $\text{biotite} + \text{quartz} \Leftrightarrow \text{olivine} + \text{Ti-magnetite} + \text{K-rich melt}$. In experiments where bone material was sandwiched between two rock cubes, the assemblage olivine + whitlockite + Ti-bearing magnetite + plagioclase + glass formed at the interface with the bone layer. The systematics of Mg, Fe, Si, and P variations in newly formed olivine and comparison with literature data indicate that phosphorus is incorporated into olivine via the coupled substitution $2\text{P} + (\square)\text{M}_{1,2} \Leftrightarrow 2\text{Si} + (\text{Mg, Fe})\text{M}_{1,2}$. The experimental results suggest that temperatures in excess of 1000°C and strongly reducing conditions are necessary for the formation of phosphoran olivine in burning sites. This is consistent with conditions of formation obtained from the rare natural occurrences of phosphoran olivine.

Introduction

Olivine with significant P_2O_5 contents (>2 wt. %) has been described from very few terrestrial and extraterrestrial occurrences such as a syenitic breccia pipe from Pine Canyon, Utah (AGRELL et al., 1998), native iron-bearing rocks from West Greenland (GOODRICH, 1984) and pallasite meteorites (BUSECK & CLARK, 1984).

Olivine and especially fayalite is commonly found in slags and may occasionally show minor P₂O₅ contents of <1 wt% (MÜLLER et al., 1988; HEIMANN et al., 1988). TROPPEL et al. (2004) describe unusually phosphorus-rich olivines, containing up to 8.8 wt.% P₂O₅ from partially molten metapelitic gneisses recovered from a presumably La-Tène (450-15 B.C.) age sacrificial place near Ötz, Northern Tyrol, Austria, where immolation of ritual offerings took place. Slags associated with prehistoric burning sites have been well documented from Tyrol since the early 20th century from two localities namely the Goldbichl near Igls and from Ötz in the Ötztal Valley (HEIBEL, 1938). Prior to their recognition as sacrificial sites, HEIBEL (1938) interpreted the burning sites to be the result of prehistoric forest or bogfires. In contrast to Tyrol, prehistoric sacrificial burning sites are more common in Bavaria where they have been documented since the late 19th century (WEISS, 1997). One of their characteristics is the abundant presence of bone fragments indicating the ritual immolation of a variety of animal species such as cow, sheep, goat and pig (WEISS, 1997).

The biotite-plagioclase gneiss underlying the burning site near Ötz is the dominant rock-type of the polymetamorphic Austroalpine Ötztal Complex (ÖC). The mineral assemblage of the metapelites investigated in this study formed during the dominant Variscan metamorphic event with peak metamorphic PT-conditions of 550-600°C and 5-7 kbar (HOINKES et al. 1997; TROPPEL & RECHEIS, 2003). TROPPEL et al. (2004) showed that during partial melting, narrow melt layers form within the gneisses which are interpreted as former layers of biotite where partial melting was initiated. Concomitant with the melt layers, foamy patches of dark glassy material formed on the surface of the rocks. In the contact area between glassy material and residual rock the assemblages olivine + plagioclase + whitlockite and/or clinopyroxene + plagioclase + melt form depending on the local bulk composition. In the residual rock the assemblage olivine + Ti-bearing magnetite + clinopyroxene + plagioclase is present.

Textures indicate that olivine und Ti-bearing magnetite form within the former biotite layers according to the reaction:

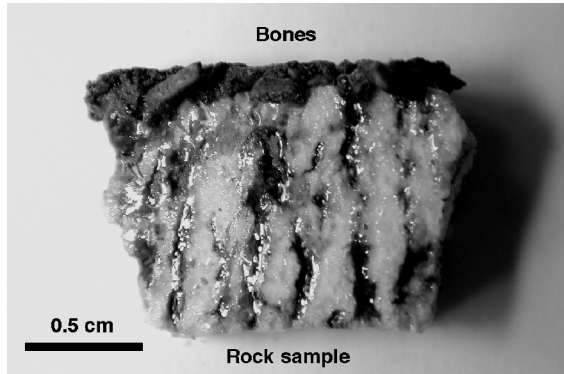


The breakdown of natural biotite at high temperatures and very low pressures has been reported so far from partially fused metapelites and granites (MAURY & BIZOUARD, 1974; LE MAITRE, 1974; GRAPES, 1986; BREARLEY, 1987a,b). Due to the likeliness of disequilibrium biotite breakdown prevailing in a short-duration firing process at very low pressures (GÜTTLER et al., 1989; TIKHOMIROVA et al., 1989; VEDDER & WILKINS, 1969; NAKAHIRA, 1965) and the fact that biotite-involving equilibria are strongly depend upon $f\text{O}_2$, it is very difficult if not impossible to deduce temperatures and oxygen fugacity during the firing process.

Very few experimental investigations simulating pyrometamorphic processes have been available and are mainly concerned with the formation and development of disequilibrium textures due to the breakdown of hydrous phases (e.g. CULTRONE et al., 2001; BREARLEY & RUBIE, 1990). In this study, we perform firing experiments at 1 bar to investigate the formation of phosphorus olivines and compare the results with the observations of TROPPEL et al. (2004) from the sacrificial burning site near Ötz in Tyrol.

Experimental methods

To place constraints on the temperature of formation of phosphorus olivines, melting experiments at 1 bar were conducted in a box furnace. To be as close as possible to the observations, we designed simple experiments, where fO_2 was only approximated to the CCO buffer but not fixed. For this study we used samples of unmelted biotite-plagioclase-gneisses from the burning site near Ötz which were cut into cubes of approximately 1 cm edge length. Most experiments were performed in graphite crucibles with the rock cube placed on top of a layer of crushed chicken bones (Fig. 1). These rock-bone aggregates were then subjected to temperatures between



900°C and 1300°C with run durations from 90 to 480 minutes (cf. Table 1). Most experiments were quenched by quickly removing the crucible from the furnace.

Fig. 1

Photograph of an experimental run product from a 1000°C experiment. The dark layer on top of the rock cube consists of bone material. The reflections on the glassy surface of the rock cube indicate that melting took place already.

Run #	T(°C)	Time (min)	Crucible	Bones	Relict primary assemblage	Secondary assemblage
AR-1	1000	285	C	no	-	-
AR-2	1000	340	Pt	no	-	-
AR-5	900	300	C	yes	Bt + Pl + Kfs + Qtz	melt + Pl
AR-6	1000	315	C	yes	Qtz + Pl	melt + Ol + Mgt + Whit
AR-9	1100	180	C	yes	Qtz + Pl	melt + Ol + Mgt + Whit + Pl
AR-7	1200	120	C	yes	Qtz + Pl	melt + Mgt + Whit
AR-8	1300	90	C	yes	Qtz	melt
AR-10	1300	210	C	no	-	-
AR-11	1300	180	C	yes	-	-
AR-12	1100	480	C	yes	Qtz	melt + Ol + Mgt + Whit + Pl
AR-13	1200	420	C	yes	Qtz	melt + Ol + Mgt + Whit + Pl

Mineral abbreviations: Bt: biotite; Pl: plagioclase, Kfs: K-feldspar; Qtz: quartz; Ol: olivine; Mgt: magnetite, Whit: whitlockite. Pt: Pt-crucible; C: graphite crucible.

Table 1

Run conditions of the experiments

Although olivine occasionally formed, quenching and the presence of bone material on only one side of the rock cube did not lead to sufficient mineral reactions at the interface between the rock and the bone layer and thus experiments were conducted where bone material was sandwiched between rock cubes.

Therefore, in order to allow a more intimate contact between bone and rock and thus to enable a stronger reaction, bone material was sandwiched between two rock slabs in the two experiments AR-12 and AR-13. Instead of quenching, these experiments were cooled slowly from 1100°C and 1200°C down to 500°C and 700°C with cooling rates of 60°C/hour and 120°C/hour to allow slow crystallization from the melt. After the experiment the cubes were embedded in epoxy resin and polished for electron microprobe and scanning electron microscope analysis. To investigate the role of crucible material during firing, two experiments at 1000°C were performed without the addition of bones: one in a graphite crucible and the other in a Pt crucible. Secondary electron (SE) images of the rock cubes from the experiment in the Pt-crucible at 1000°C showed almost no melting textures on the surface (Fig. 2A) and therefore the experiments in the Pt crucible were not pursued any further. In contrast, in the experiments with the graphite crucible, visible melting took place at the surface of the rock cube (Fig. 2B), indicating that more reducing fO_2 conditions facilitate a higher degree of partial melting. In addition, the presence of bone material to the rock cubes lead to complete melting of the rock cubes (AR-10, AR-11) at temperatures of 1300°C as shown in Figure 3.

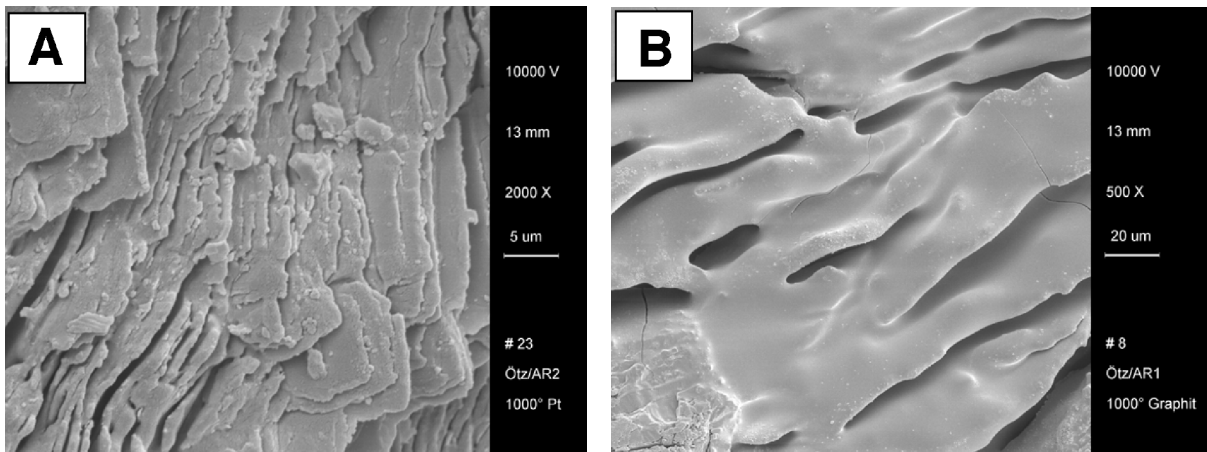
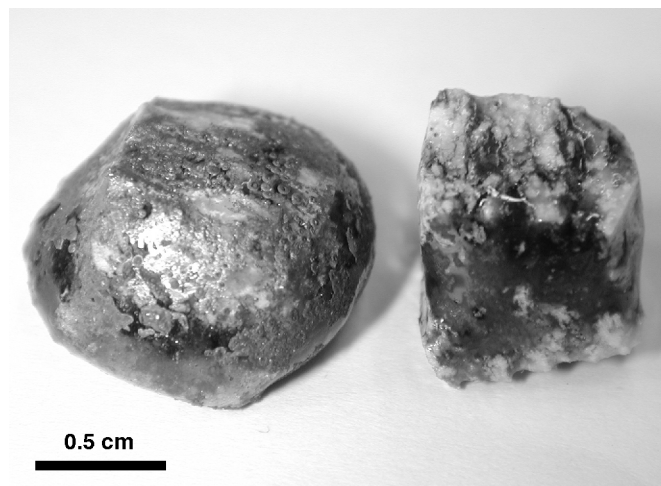


Fig. 2

Secondary electron (SE) image of the surface of rock cubes from experiments at 1000°C. (A) Experiment in a Pt crucible (AR-1). The sharp edges of the biotites do not indicate a significant degree of melting. (B) Experiment in a graphite crucible (AR-2). The rounded edges and open spaces indicate a considerable degree of melting.

Fig. 3

Comparison between two experiments at 1300°C from an experiment in a graphite crucible with bone material added (AR-11, left) and without bone material (AR-10, right). The addition of bone material to the experiments leads to a strong increase in melting during the experiments.



Experimental results

Textural relations and petrography

Petrographic investigations show that in the experiment at 900°C (AR-5) biotite is still stable and almost no textural changes take place. Granitic melting occurs in plagioclase - K-feldspar - quartz domains and adjacent to biotite-rich domains. At 1000°C (AR-6), no biotite is present anymore and instead, the assemblage olivine + Ti-bearing magnetite + melt forms. Textures significantly change due to the formation of large amounts of melt whose modal amount increases considerably towards the contact between gneiss and bone layer. Plagioclase and quartz are still present. At 1100°C (AR-9), the amount of melt further increases, leaving almost no primary plagioclase behind. Upon quenching, plagioclase needles crystallize from the melt. Above 1200°C (AR-7, AR-8), the rock is almost completely molten with only quartz still present. The slowly cooled experiments in which bone material was sandwiched between two rock slabs (AR-12, AR-13) show very similar textures and mineral assemblages as observed from the sacrificial burning site as shown in Figures 4A,B. In the former bone layer, whitlockite and melt are present (Fig. 4A) whereas along the interface between bone layer and rock cube, olivine + Ti-magnetite + plagioclase + melt have formed (Fig. 4B).

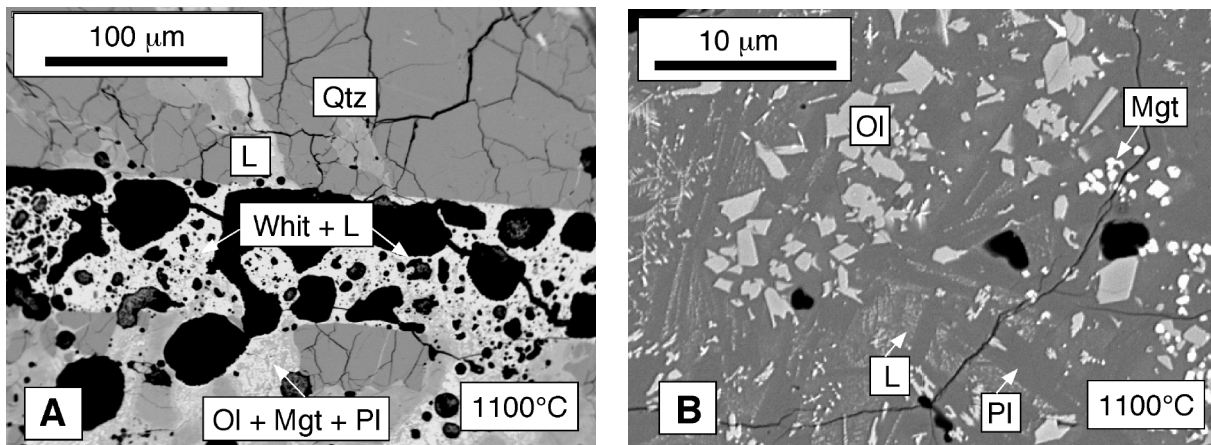


Fig. 4

Backscatter electron (BSE) images of a slowly cooled experiment in a graphite crucible at 1100°C (AR-12).

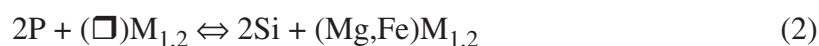
(A) The layering of the former bone layer is still visible and contains the assemblage whitlockite (Whit)+ melt (L). Small injections of melt veins into the adjacent rock cubes are also visible. Within the melt pockets the assemblage olivine (Ol) + magnetite (Mgt) + plagioclase (Pl) + melt (L) occurs.

(B) The former biotite sites now contain the assemblage olivine (Ol) + Ti-bearing magnetite (Mgt) + melt (L).

Mineral chemistry

Electron microprobe analyses of minerals were performed on an ARL - SEMQ microprobe at the Institute of Mineralogy and Petrography at the University of Innsbruck. Analytical conditions were 15 kV and a sample current of 20 nA on brass. The analyses were obtained with a NORAN-Voyager EDS system, which was calibrated with synthetic elemental standards. The counting times ranged from 40 to 200 seconds, depending on the volatility of the material analysed.

The chemical data of the phases from the slowly cooled experiments at 1100°C and 1200°C are shown in Tables 2 - 4. Olivines from the experiment at 1100°C (Table 2) show a wide range in P₂O₅-concentrations from 0.18 to 1.19 wt.% along with significant variations in their Fe/Mg-ratios (Fo₃₀Fa₇₀ to Fo₅₀Fa₅₀). Compared to olivines from the burning site, the experimentally produced olivines extend to more Fe-rich compositions but do not contain as much P₂O₅ (Table 2). As is the case for olivines from the burning site, analyses of experimentally grown olivines show a negative correlation between P and Si apfu and also between P and total cation sums. Both correlations, however, are not as pronounced as those observed for olivines from the burning site probably due to the lower phosphorus contents (cf. TROPPER et al. 2004). The element variations in olivine indicate the following substitution mechanism



similar to that encountered in olivines from the burning site at Ötz (Fig. 5A). The analyses listed in Table 2 also show in some cases increasing Ca contents with increasing P which cannot be related to contamination by whitlockite, because no whitlockite has been found adjacent to olivine in the experiments (Fig. 4A). Figure 5B shows the Si-P co-variation which also indicates that P-substitution in the olivines from all natural studies considered here (GOODRICH, 1984; BUSECK & CLARK, 1984; AGRELL et al. 1998; BRUNET, 1995) is due to the same coupled substitution (2).

SiO ₂	34.32	33.00	34.52	34.27	33.73	35.88	33.73
Al ₂ O ₃	0.35	0.35	0.91	0.61	0.53	n.d.	0.53
TiO ₂	0.35	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FeO	38.75	47.41	42.94	44.73	40.41	22.74	40.41
MgO	23.27	15.52	20.73	17.92	20.51	35.88	20.51
MnO	1.63	1.69	1.39	1.39	1.39	0.86	1.39
CaO	n.d.	0.72	0.54	0.75	1.16	0.22	1.16
P ₂ O ₃	0.18	0.47	0.61	0.63	1.19	1.21	1.19
Total	98.50	99.16	101.64	100.30	98.92	97.53	98.92
Si	0.99	1.00	0.98	1.00	0.98	0.98	0.98
Al	0.01	0.01	0.03	0.02	0.02	n.d.	0.02
Ti	0.01	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Fe ²⁺	0.94	1.20	1.02	1.09	0.98	0.51	0.98
Mg	1.01	0.70	0.88	0.78	0.89	1.43	0.89
Mn	0.04	0.04	0.03	0.03	0.03	0.02	0.03
Ca	n.d.	0.02	0.02	0.02	0.04	0.01	0.04
P	<0.01	0.01	0.01	0.02	0.03	0.03	0.03
Cations	2.99	2.98	2.98	2.97	2.97	2.98	2.97

Basis of formula calculation: 4 oxygens; n.d. not detected

Table 2

Representative microprobe analyses of P-bearing olivine

The chemical analyses of the interstitial glass from the slowly cooled experiments at 1100°C and 1200°C are shown in Table 3. Si contents range from 54.4 to 58.6 wt.% SiO₂. In contrast, Al shows the strongest variation from 8.9 to 18.5 wt.% Al₂O₃, depending on the local bulk composition from which the melt formed.

Phosphorus contents are substantial and may reach 1.3 wt.% P₂O₅. Feldspar compositions of newly-grown feldspars from the slowly cooled experiment at 1100°C range from An₄₄ to An₅₇ (Table 4). Table 5 shows chemical analyses of whitlockites from the slowly cooled experiments at 1100°C and 1200°C which is very similar to whitlockites reported by TROPPER et al. (2004).

Table 3
Chemical composition of the interstitial glass

SiO ₂	53.46	53.23	55.48
TiO ₂	n.d.	n.d.	n.d.
Al ₂ O ₃	27.68	28.56	25.94
Fe ₂ O ₃	0.72	0.36	1.10
FeO	n.d.	0.36	n.d.
MgO	n.d.	n.d.	n.d.
CaO	11.96	11.53	9.23
Na ₂ O	4.71	4.55	6.00
K ₂ O	0.41	0.43	0.93
Total	99.25	99.02	98.68
Si	2.44	2.44	2.53
Ti	n.d.	n.d.	n.d.
Al	1.49	1.54	1.40
Fe ³⁺	0.02	0.01	0.04
Fe ²⁺	n.d.	0.01	n.d.
Mg	n.d.	n.d.	n.d.
Ca	0.59	0.57	0.45
Na	0.42	0.40	0.53
K	0.02	0.03	0.05
End-members			
Anorthite	0.57	0.57	0.44
Albite	0.41	0.41	0.51
K-feldspar	0.02	0.02	0.05

Basis of formula calculation: 5 cations and 16 charges; Fe²⁺/Fe³⁺ calculated based on charge balance considerations; n.d. not detected.

Discussion

These experimental investigations have shown that the interaction of bone material and metapelitic gneisses during partial melting leads to the breakdown of biotite and the formation of P-rich olivines + whitlockite + plagioclase + K-rich glass. The unusually high degree of partial melting observed in biotite-gneisses found at the sacrificial burning site may have two reasons:

	1100°C	1100°C	1100°C	1200°C	1200°C
SiO ₂	54.41	57.43	55.61	59.02	58.62
TiO ₂	2.58	2.10	3.25	0.74	0.56
Al ₂ O ₃	9.46	10.49	8.91	18.15	18.51
FeO	16.51	11.78	12.78	7.67	8.06
MnO	0.65	0.41	0.58	n.d.	n.d.
MgO	1.42	2.06	1.41	1.65	1.76
CaO	5.55	8.67	8.97	1.24	2.25
Na ₂ O	1.40	1.41	1.20	3.96	4.75
K ₂ O	3.96	3.46	2.91	4.91	3.87
P ₂ O ₅	1.01	1.00	1.28	0.23	n.d.
Total	96.96	98.97	97.20	97.57	98.48

	1100°C	1100°C	1200°C	1200°C
CaO	46.20	45.58	48.59	48.99
P ₂ O ₅	44.80	43.98	45.24	44.91
SiO ₂	1.42	1.07	1.15	0.61
FeO	n.d.	0.86	0.75	0.71
MgO	3.35	3.66	3.21	3.20
Na ₂ O	1.77	2.38	0.65	0.68
K ₂ O	0.48	0.52	n.d.	0.13
H ₂ O ^a	0.84	0.83	0.85	0.84
Total	98.86	98.88	100.66	100.07
Ca	8.83	8.80	9.16	9.32
P	6.77	6.71	6.74	6.75
Si	0.51	0.39	0.41	0.22
Fe	n.d.	0.13	0.11	0.11
Mg	0.89	0.98	0.84	0.85
Na	0.61	0.83	0.22	0.23
K	0.11	0.12	n.d.	0.03
OH	1.00	1.00	1.00	1.00
cations	17.62	17.83	17.51	17.48

Basis of formula calculation: 28 O+OH+F+Cl; n.d. not detected; ^acalculated

Table 5
Representative microprobe analyses of whitlockite

Table 4
Representative microprobe analyses of feldspar

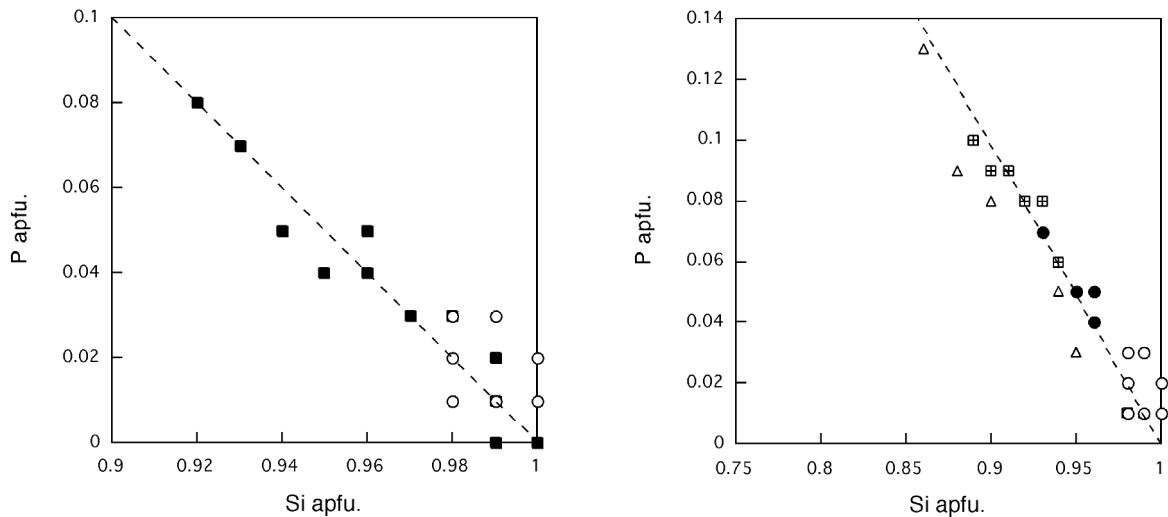


Fig. 5

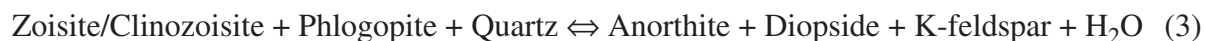
(A) Correlation between Si a.p.f.u. and P a.p.f.u. of the phosphorous olivines from the slowly cooled experiment at 1100°C (open circles). For comparison, the data from the low phosphorus portion of the olivines from the burning sit near Ötz are shown with black squares. (B) Correlation between Si a.p.f.u. and P a.p.f.u. of the phosphorous olivines from the slowly cooled experiment at 1100°C (open circles). For comparison, the data from the literature (GOODRICH, 1984: open square with grid; BUSECK & CLARK, 1984: open diamond; AGRELL et al. 1998: open square) are also shown. The dashed line indicates the $2P + (\square)M_{1,2} \Leftrightarrow 2Si + (Mg, Fe)M_{1,2}$ substitution.

- (1) an increase in the firing temperature exceeding that of biotite stability at 1 bar according to reaction (1) possibly boosted by the addition of fat from the animals carcasses. Temperatures of wood fires are usually in the range 650-900°C and rarely exceed 1000°C (RYE, 1981). It is only by blowing air into the fire or by adding organic substances, that temperatures can be raised significantly beyond 1000°C. The presence of organic material (fat, oil) from the animals and the wood is also likely to provide a reducing environment;
- (2) a decrease in the meltig temperature of the biotite-gneisses as a result of the addition of phosphorus derived from the apatite of the bones.

Our experimental investigations indicate that the temperature of olivine formation due to reaction (1) must have exceeded 1000°C at fO_2 conditions near the CCO buffer and are in agreement with temperature estimates from pallasite meteorites (1143-1359°C). The occurrence of phosphoran olivine and whitlockite in meteorites with compositions similar to those encountered in the experiments and the rocks at the firing site (GOODRICH, 1984; BUSECK & CLARK, 1984; AGRELL, et al. 1998) further indicates a similarity with the experimental conditions.

In summary, the chemical and experimental data presented above strongly indicate olivine growth under disequilibrium conditions. Although phosphoran olivine did form in the experiments the extent of P-incorporation into olivine is much smaller compared to the olivines from the burning site at Ötz. Olivines with P_2O_5 contents similar to those found in the experiments do occur in Ötz but are restricted to microdomains more distant to the rock/bone interface. Clearly, local variations in firing temperature, oxygen fugacity, bulk phosphorus and the geometry of the bone-rock aggregates must have controlled the P-incorporation in olivine.

In terms of the mineralogy, the most notable difference between the experimentally grown assemblages and those from the rocks at the burning site is the absence of clinopyroxene in the experiments. Since many rock samples show strong alteration of plagioclase to albite and zoisite/clinozoisite, the presence of clinopyroxene in the partially molten rocks could be ascribed to a reaction involving zoisite/clinozoisite:



The absence of clinopyroxenes in the experiments could thus be due to the lack of plagioclase alteration in the samples used in this investigation

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