

Experimental Data on the Thermal Reduction of Phosphorus and Iron and Their Significance for the Interpretation of the Impact Reworking of Lunar Materials

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Abstract—Experiments simulating impact melting and vaporization were performed to study variations in the valence state of P on the Moon. It was shown that P changes its valence and is partially transformed from oxidized (phosphate) to reduced (P⁰) species at high temperatures typical of the impact process. The reduction of P occurs concurrently with Fe reduction from silicates. The metallization of Fe and P is due to the effect of thermal reduction only. This supports the hypothesis of R. Hunter and L. Taylor that P⁰ produced from lunar phosphates (apatite and whitlockite) interacted with reduced Fe to form typical P-bearing minerals of lunar highland rocks, schreibersite and Fe–P alloys.

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INTRODUCTION

Experimental studies of impact melting and vaporization of rocks, meteorites, minerals, and their mixtures demonstrated that siderophile elements (Fe, Ni, Pt) can be reduced from oxidized to zero-valent (metallic) species, perhaps, owing to simple thermal reduction at temperatures of >2000°C [1–3]. We attempted to test this hypothesis for other heterovalent elements, which show lithophile or siderophile properties depending on the environment and conditions. Among such elements is phosphorous, which forms compounds of various valence states on the Moon.

Similar to K, REE, Zr, Hf, U, Th, and other elements, P behaves as a typical incompatible element during the magmatic fractionation of lunar basalts. The main P-bearing minerals of various lunar basalts are apatite Ca₅[PO₄]₃(Cl,F) and whitlockite Ca₃(PO₄)₂. By contrast, the main P-bearing mineral of lunar anorthosites, in particular, Apollo 16 samples, is schreibersite, (Fe,Ni)₃P, whereas apatite and whitlockite are scarce. Taylor and Hunter [4] noted that schreibersite usually forms intergrowths with an Fe–Ni metallic phase, which also strongly accumulates P (from 0.02 to 1.5 wt %). Phosphide grains often occur in a P-rich Fe–Ni phase. It should be noted that all these

P-bearing minerals are typically localized in anorthosite breccias, i.e., in shock-metamorphosed rocks. Taylor and Hunter found a correlation between P and Cl contents in the anorthosite breccias. Under terrestrial conditions, Cl mainly occurs in akaganeite, FeO(OH,Cl), which is formed by interaction of lawrencite FeCl₂ (primary Cl-bearing mineral under lunar conditions) with atmospheric water. Similar to schreibersite, lawrencite (akaganeite) is closely associated with the Fe–Ni phase of anorthositic rocks. The impact-melted breccia 66095 (known as Rusty Rock) from Apollo 16 material is characterized by the highest contents of P and Cl among lunar samples [5, 6]. Similar to other samples of anorthosite breccias, P and Cl are incorporated in minerals spatially associated with the Fe–Ni phase. It is noteworthy that neither apatite nor whitlockite was found in sample 66095 [6]. In addition to the Fe–Ni phase, lawrencite (akaganeite), and schreibersite, the sample contains troilite, sphalerite, and other Zn-, Pb-, and Cl-bearing phases. The rock is enriched in Cl, F, Br, S, volatile metals (Zn, Cd, Pb, Bi), and other elements. It was emphasized that lawrencite (akaganeite) and schreibersite were mainly found in the breccia matrix, where they are often related to impact-melted (glassy) rock fragments.

The formation of the Rusty Rock and the metal–schreibersite–troilite–lawrencite (MSTL) assemblage is an intriguing and controversial problem in lunar mineralogy and petrology. There are three hypotheses to explain the origin of the MSTL assemblage: (1) Fumarole hypothesis, which suggests that P, S, C, F, volatile metals Zn, Pb, Cd, and other elements, which were released by fumarole–volcanic activity, penetrated brecciated (therefore, permeable) highland rocks and reacted with metal to form the observed mineral assemblages in the matrix of gabbro–anorthosite impactites. (2) Meteorite hypothesis, which connects the presence of the Fe–Ni phase, troilite, schreibersite, and volatiles in the lunar rocks to the supply of meteoritic (or cometary) impactor material. The highland breccias are, correspondingly, variably contaminated by meteoritic material. (3) Impact–metamorphic hypothesis, according to which numerous meteorite (and comet) impacts on the lunar highlands gave rise to the thermal mobilization (liberation) of volatiles from the target rocks. These volatiles migrated subsequently into the highly permeable sequences of the impact rocks. Post-impact thermal metamorphism was accompanied by chemical reactions and condensation of Fe, P, S, and Cl compounds in the breccia matrix to form the MSTL assemblage.

The impact–metamorphic hypothesis was proposed and justified by Hunter and Taylor [5, 6]. This hypothesis seems to be the most plausible, because it provided an explanation to numerous observations within a common concept. Hunter and Taylor believed that thermal metamorphism is the main postimpact process of volatile mobilization on the Moon. The cornerstone of this hypothesis is the postulate of an “external” meteorite–cometary source of heating of the lunar rocks and a local (lunar) source of volatiles with no meteoritic contamination. This suggestion is justified by the occurrence of the major portion of observed lawrencite (akaganeite), schreibersite, and associated Fe–Ni phase in the breccia matrix. The authors seem to direct the reader to the suggestion that the impact process provoked the conversion of P and Fe into the reduced form. They did not exclude a contribution from meteorite material, although they argued that it is difficult to determine in particular cases whether metallic Fe, troilite, and schreibersite were of a local (lunar) or meteoritic origin. Hunter and Taylor suggested that this question can be solved on the basis of textural and morphological characteristics of the phases. In particular, lunar schreibersite forms irregular segregations around large metallic aggregates or occurs as dispersed particles in the breccia matrix. Meteoritic schreibersite occurs mainly as inclusions in a metal.

Nevertheless, Hunter and Taylor believed that the main source of reduced P is the lunar material represented by volatile-rich KREEP basalts, which are ubiquitous in highland breccias. In the impact processes, the phosphates of KREEP basalts were decomposed and the released volatile P and Cl interacted with metallic

Fe forming schreibersite and lawrencite (akaganeite). This concept was supported by the study of Delano and Ringwood [7], who showed that the abundances of siderophile elements (including P) in lunar highlands are similar to those in low-Ti mare basalts, ruling out the meteorite source of P. The opponents of Hunter and Taylor pointed out that there is no evidence for the impact–metamorphic mobilization of P in Apollo 14 samples with abundant KREEP material [8]. However, Hunter and Taylor argued that the absence of schreibersite and lawrencite (akaganeite) in the Apollo 14 rocks could be related to weak impact reworking.

The main problem in the impact–metamorphic formation of the MSTL assemblage is the mechanism of volatile release and P reduction. Only one of several existing suggestions was experimentally tested. The possibility of volatile mobilization during thermal metamorphism was demonstrated in the experiments of Cirlin and Housley [9], who heated a basalt–apatite mixture up to 880°C in vacuum. During this experiment, Cl was released and precipitated as lawrencite crystals (FeCl₂) on an iron foil. Crystals of troilite and pure iron were also found in the condensate. The experiments showed that Cl and S can be extracted from the basalt–apatite mixture at subsolidus temperatures and transported in a vapor phase; their reaction with Fe produced the aforementioned minerals. Taylor et al. [10] conducted annealing experiments directly with Apollo 16 breccias at temperatures of 875°C for 6–20 days. During the long-term annealing, the content of metallic Fe in the breccia increased at the expense of FeO from the silicate matrix. Simultaneously, phosphorous escaped from the metal to form whitlockite. These authors believe that the whitlockite was produced by the reaction of oxygen released during Fe reduction with P from the metal and schreibersite. It is important that P was not released and transported in the experiments of Cirlin and Housley, whereas P was oxidized in the experiments of Taylor et al. However, Hunter and Taylor [5, 6] argued that the schreibersite–lawrencite assemblage was presumably formed during the impact-related decomposition of phosphates (apatite and whitlockite), which were initially present in lunar highland rocks.

The problem of the mechanism of phosphate reduction was comprehensively considered in the scope of the impact–metamorphic hypothesis by Goldstein et al. [11–13]. It was shown that P can be reduced during interaction between whitlockite and orthopyroxene at 700–1150°C and an oxygen fugacity of 10⁻²⁶–10⁻¹³ atm (below the QFM buffer). This reaction produces Ca clinopyroxene and an Fe–P alloy. The P content in the metal can be as high as 2 wt %. Based on a comparison of the obtained data with the composition of P-bearing Fe in lunar breccias, these authors concluded that the formation of metallic Fe in the Moon occurred under more reducing conditions than those of the experiment.

Therefore, it was supposed that such conditions could arise locally owing to the uneven distribution of reducing agents, such as implanted carbon and hydrogen of solar wind or carbon and sulfur impurity from a meteorite source. Finally, the authors arrived at the conclusion that the presence of a dispersed reducing agent in lunar regolith is required to reduce P from whitlockite (or apatite). Another problem with this reaction is related to its occurrence in a solid state. Since the reaction proceeds under subsolidus conditions, its kinetic parameters are limited by the diffusion rates of elements and its efficiency depends on the presence of interfaces. The experiments also demonstrated that rapid quenching is necessary for the preservation of reduced P, because it can again be transformed from the metallic to phosphate form during slow cooling. This indicates that the formation of the lunar breccias with reduced P must include short-term cooling.

As can be seen from the literature review, all the existing concepts on the release and transportation of volatiles, as well as P reduction in lunar materials, have not yet been reliably supported by experiments. It should be noted that the available experimental data provide no evidence for the direct relation of P reduction with the impact process. It was supposed that P was reduced in the cooling impactites during the postimpact stage. However, the experimental data of Goldstein and others showed that the only experimentally supported reaction of phosphorous reduction during the postimpact stage could proceed efficiently only in a particular silicate environment fertilized with a certain amount of reducing agents. Thus, the problem of P reduction in the lunar highland rocks and related formation of the MSTL assemblage remains open.

PROBLEM FORMULATION AND HIGH-TEMPERATURE EXPERIMENT

The solution of the problem of P reduction must presumably be sought not in the stage of subsolidus thermal metamorphism but during a higher temperature stage directly related to the impact melting and vaporization of lunar rocks. The intimate association of schreibersite with metallic iron can serve as evidence for this suggestion, because the formation of metals in lunar rocks usually requires higher temperatures than the melting temperature of basalts [1]. Our study was based on the general assumption on the temperature dependence of the valence state of an element. This dependence can be illustrated by the equilibrium curves of the oxidation–reduction of Fe and P (Fig. 1). The $\log(P_{O_2})$ – T diagram shows the field of conditions characteristic of the postimpact thermal metamorphism of highland rocks [13]. It can be seen that a temperature increase within a wide range of redox conditions is sufficient to move from this field into the stability field of zero-valent Fe and P.

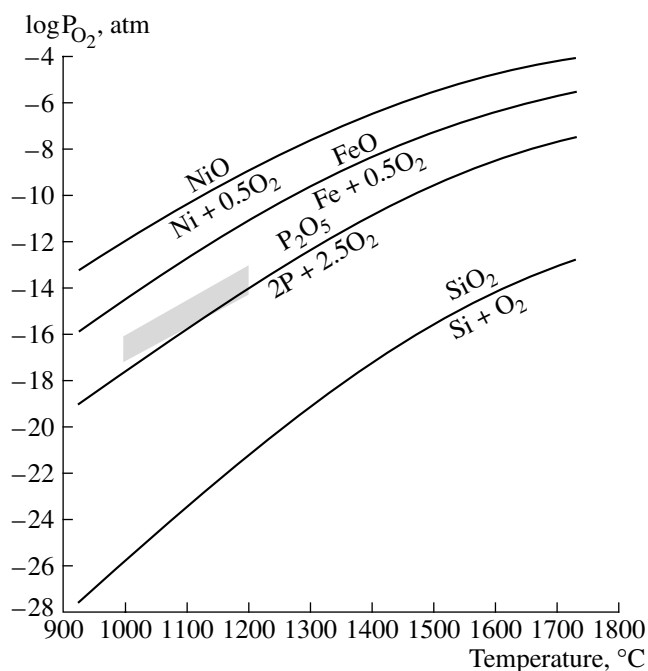


Fig. 1. The dependence of Fe and P redox reactions on oxygen pressure and temperature. The shaded field indicates the supposed conditions of the formation of lunar highland breccias according to [13].

In order to study the temperature influence on P reduction, we performed experiments simulating the impact melting and vaporization of model P-bearing mixtures and apatite. It was assumed that materials similar to the Rusty Rocks could be formed by a cometary impact. Therefore, the starting mixture used in our experiments roughly corresponded to the composition of the cometary impactor and the basaltic target [14]. The mixture consisted of basalt, chondrite (Murchison), and “cometary volatiles” in equal proportions. The latter component was prepared from S, $C_{10}H_8$, $Na_2SO_4 \cdot 10H_2O$, NH_4NO_3 , $NH_4H_2PO_4$, $Co(NO_3)_2 \cdot 6H_2O$, and KBF_4 , and its H : C : N : S proportions approximately corresponded to those in the dust grains of comet Halley [15]. Minor amounts of chlorides of volatile metals (Pb, Zn, Cd) and Pt were added to the “cometary” material. The bulk composition of the starting mixture was the following (wt %): 2.80 H, 4.33 S, 11.37 C, 1.48 Cl, 4.45 N, 0.30 P, 0.04 B, 0.28 F, 41.28 O, 8.83 Si, 1.32 Ti, 2.10 Al, 9.28 Fe, 0.09 Mn, 3.43 Mg, 1.65 Ca, 3.15 Na, 0.32 K, 0.08 Cr, 0.52 Zn, 0.53 Cd, 0.53 Pb, 0.54 Pt, 0.34 Ga, 0.09 Co, and 0.88 of other elements.

In addition to the model P-bearing sample, samples of natural apatite and a peridotite–staffelite mixture (64 wt % peridotite and 36 wt % staffelite) were used in the experiments. The natural staffelite (carbonate-fluorapatite) was taken from the Kovdor deposit; it differed from typical staffelite in high $FePO_4$ content. The apatite and peridotite–staffelite mixture were used to check

the occurrence of thermal reduction in samples which, unlike the cometary mixture, contained no internal reducing agent. The runs with these samples, which differed from the cometary mixture in that they contained no reducing agents such as $C_{10}H_8$ and S^0 , were aimed at distinguishing the pure temperature effect. The experiment with apatite had to verify or refute the hypothesis of Hunter and Taylor on the formation of reduced P species from apatite. As was mentioned above, these authors provided compelling evidence that the reduced phosphorous of lunar rocks could be produced by the impact process from the phosphates of lunar highland materials rather than from a meteorite impactor. The experiment with the peridotite–staffelite mixture was conducted to examine the possibility of the concurrent thermal reduction of P and Fe.

The experiments were performed on a laser apparatus using the technique described in [16]. The Nd-glass laser had the following parameters: wavelength $\lambda = 1.06 \mu\text{m}$, a pulse energy of 600 J, a power density of $\sim 10^6\text{--}10^7 \text{ W/cm}^2$, and a pulse duration of $\sim 10^{-3} \text{ s}$. The characteristic evaporation temperature is 4000–5000 K under such conditions. Experimental samples were mounted in a sealed cell with an internal volume of $\sim 500 \text{ cm}^3$. The experiments were performed in He at 1 atm. The experimental conditions ruled out any redox influence of the environment on the melting and vaporization of samples. It should be kept in mind that laser experiments do not exactly reproduce the impact process; in particular, there is no shock compression. However, laser experiments are appropriate to simulate

high-temperature reduction and evaporation, because these processes occur during the release of shock loading.

A laser beam, $\sim 3 \text{ mm}$ across, melted and evaporated a few tens of milligrams of the sample. A flat screen was installed on the scattering path of vapor and melt droplets at a distance of 7–8 cm from the sample. Quench condensation and formation of a condensate film took place on the surface of the screen. The condensate consisted of agglomerated nanometer-sized particles forming a loose film, a few microns thick [16]. The thickness of the condensate layer was determined from the time of its Ar ion milling and compared with the equivalent thickness of silver milling. The equivalent thickness of the film was $10^3\text{--}10^4 \text{ \AA}$. In addition, melt droplets were formed by the dispersion of liquid ejected by the vapor jet from the crater cavity and quenched on the screen surface. Morphologically similar droplets were described in [1].

According to calculations, vapor from the evaporation spot reaches the screen within $<10^{-4} \text{ s}$, which is at least an order of magnitude less than the duration of the laser pulse. This difference provides a sequential formation of the condensate film simultaneously with the evaporation. It is therefore necessary to take into account the time series of radiation energy, because it controls the temperature and chemical composition of vapor sequentially arriving from the evaporation zone. It is known that the time series of laser pulse energy is sharply asymmetrical [17]. The highest pulse intensity is observed from the initial moment to $\sim 20\%$ of the total duration. Then, the radiation intensity decreases gradually for $\sim 80\%$ of the total pulse duration. Correspondingly, the maximum sample temperature is reached at the initial moment of the laser pulse, and the first vapor portions are presumably formed at the highest temperature.

The chemical composition of the condensate was measured by X-ray photoelectron spectroscopy. The analysis was performed layer by layer, with a step of Ar ion milling of the condensate film equivalent to the milling depth of silver of 50–300 \AA . The accuracy of the measurement of spectral line position was $\pm 0.1 \text{ eV}$, which allowed us to reliably identify states corresponding to different types of element bonding. In addition, the method yielded the concentrations of elements in the condensate and their valence state, which presumably reflects the valence state of elements in the residual melt. Since the condensate was analyzed layer by layer, its chemical composition was presented by the diagrams of element concentrations as functions of depth in the condensate layer. The total thickness of the condensate layer was taken as 100%. Such diagrams display the dynamics of changes in the chemical composition of vapor, which was supplied from the zone of sample melting and evaporation: the composition of the condensate surface (origin in the diagram) corresponds to the final portions of vapor, and the deep zones corre-

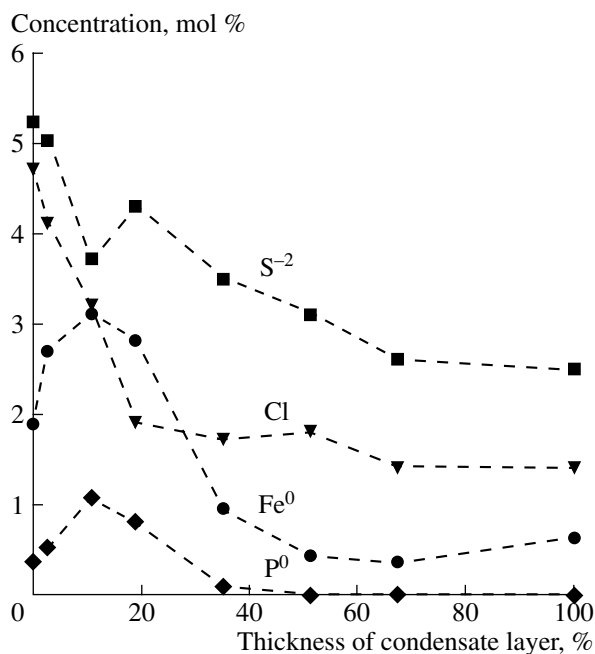


Fig. 2. Concentrations of reduced Fe, P, and S species and Cl in the layers of condensate obtained by the vaporization of the “cometary” mixture.

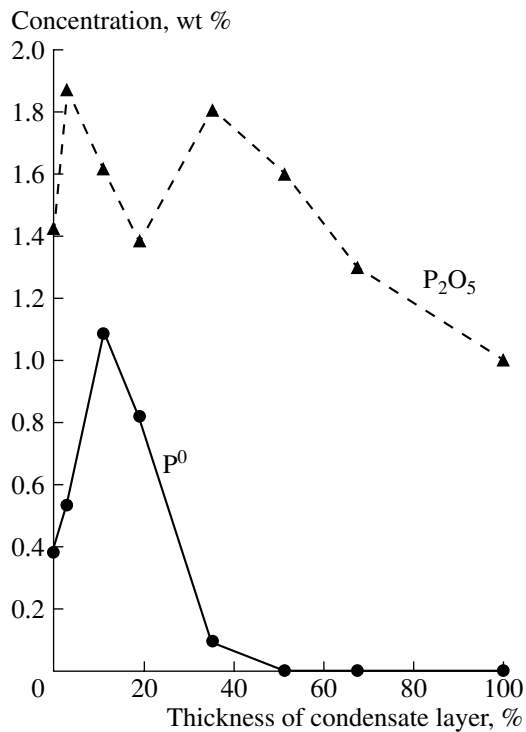


Fig. 3. Concentrations of reduced and oxidized P species in the layers of condensate obtained by the vaporization of the "cometary" mixture.

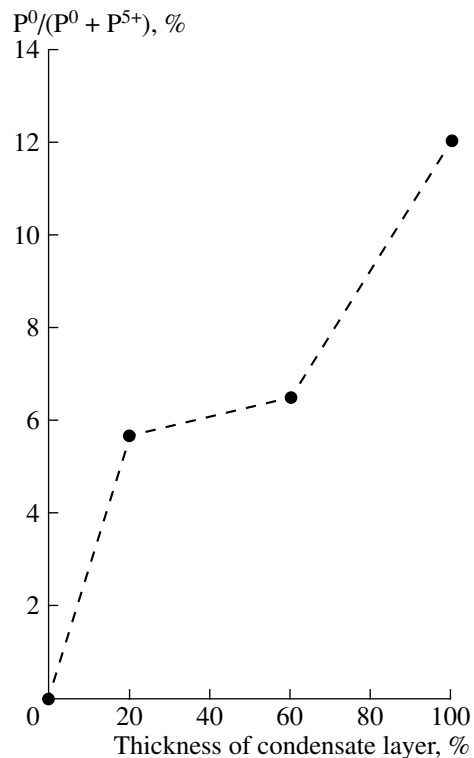


Fig. 4. Relative amount of reduced P (degree of reduction) in the layers of condensate obtained by apatite vaporization.

spond to the first vapor portions characterized by the highest temperatures.

EXPERIMENTAL RESULTS

Before describing the results of condensate analysis, it should be noted that all P in the starting mixture was in an oxidized state (phosphate). The same is true for Fe, which occurred in an oxidized form only in the starting peridotite–staffelite mixture and was mostly oxidized in the model cometary mixture, because the Murchison chondrite contains no more than ~1 wt % Fe⁰.

The layer-by-layer analysis of condensate from the experiment with the cometary–basaltic mixture clearly indicated the presence of reduced (zero-valent) P, with the highest content of P⁰ in the upper condensate zones (Fig. 2). The fraction of P⁰ in the total P content was as high as ~45% in some layers (Fig. 3). Zones with the highest contents of zero-valent P were also enriched in Fe⁰, S⁻², and Cl (Fig. 2). Iron in these layers was reduced by up to 30%. These zones also showed elevated contents of volatile metals [14]. The reason why the reduced forms of P, Fe, S, and volatile elements are accumulated in the upper low-temperature layers is unclear, but their spatial association is probably not accidental and strongly resembles the association of elements and their forms observed in the Rusty Rock sample.

The experiments with apatite confirmed that zero-valent P can be directly produced during the high-temperature melting and vaporization of this mineral. Figure 4 shows the relative contents of P⁰ in the condensate from apatite. It can be seen that the highest contents of reduced P (up to ~12% of total P) occur in the high-temperature lower zone of the condensate. It should be noted that the major portion of P remained in an oxidized state and was present in the condensate as Ca₂P₂O₇, which testifies to the high stability of this Ca phosphate at high vaporization temperature.

In the experiments with peridotite–staffelite mixture, reduced P and Fe species were observed in the condensate. Figures 5 and 6 compare the X-ray electron spectra of Fe 2p_{3/2} electrons for the starting mixture and for one condensate layer. Similar data are shown for P in Figs. 7 and 8: X-ray electron spectra of P 2s-electrons for the starting mixture and for a condensate layer (Fig. 8). It can be seen that the starting mixture contains oxidized forms of Fe, Fe²⁺ and Fe³⁺, whereas the spectrum of the condensate indicates the presence of zero-valent Fe. Phosphorous occurs in an oxidized state in the starting mixture, and part of the phosphorus is reduced to the zero-valent form in the condensate. The distribution of observed Fe and P forms is nearly uniform throughout the condensate layers (Figs. 9, 10). On average, zero-valent Fe accounts for 33% of all Fe forms in the condensate, and the fraction of zero-valent P in the total P content is 26%. The presence of the

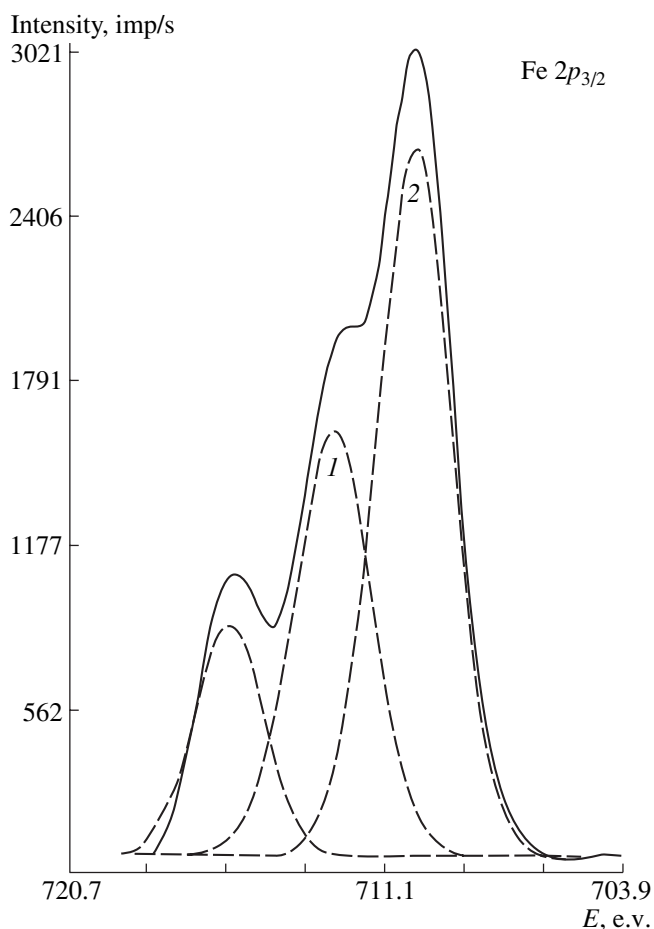


Fig. 5. X-ray electron spectrum of Fe $2p_{3/2}$ electrons in the starting peridotite-staffelite mixture (solid line). The dashed lines show the deconvolution of Fe states in the mixture: (1) Fe³⁺ as FePO₄ and (2) Fe²⁺ from olivine and pyroxene in Fe–O–Si bonds.

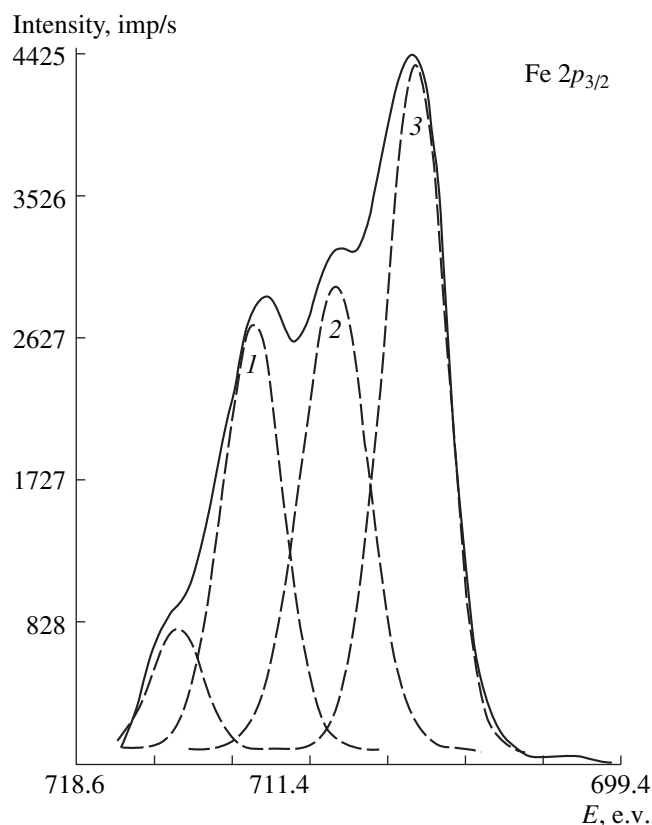


Fig. 6. X-ray electron spectrum of Fe $2p_{3/2}$ electrons in a layer of condensate obtained by the vaporization of the starting peridotite-staffelite mixture. (1) Fe³⁺ as FePO₄, (2) Fe²⁺ as FeO, and (3) reduced Fe as Fe⁰.

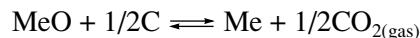
phosphate form of ferric Fe indicates its stability at high temperature. The metallic iron in this experiment was presumably formed by reduction from silicates. Similar to the experiment with apatite, the condensate contains Ca₂P₂O₇. The relatively high content of this calcium phosphate in the experiments with the peridotite-staffelite mixture is probably not accidental and reflects an important feature of the high-temperature vaporization of apatite-group minerals.

DISCUSSION

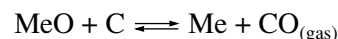
Summarizing the obtained experimental results, we arrive at the conclusion that, at the high temperature of the impact processes, P changes its valence and is partly converted into a reduced state. The reduction of P occurs concurrently with Fe reduction. The simultaneous reduction of Fe and P could be caused by the thermal reduction effect only.

It is highly probable that the presence of potential or internal reducing agents (for example, H₂ and C of solar

wind) in lunar regolith provides an additional contribution to P and Fe reduction, which increases with increasing temperature. Proceeding from general thermodynamic considerations, the exchange reactions



and



will be shifted to the right-hand side with increasing temperature, because the entropic effects of the reactions are high and positive and, hence, $\Delta G(\text{reaction})$ will decrease according to the equation $d\Delta G(\text{reaction})/dT = -\Delta S(\text{reaction})$. It is therefore reasonable to expect that such redox reactions will be efficient in the high-temperature impact process. However, the experiments with apatite and the peridotite-staffelite mixture showed that the presence of internal reducing agents is not necessary, and P and Fe can be reduced by the simple mechanism of thermoreduction decomposition with formation of Fe⁰, P⁰, and free oxygen. The latter easily

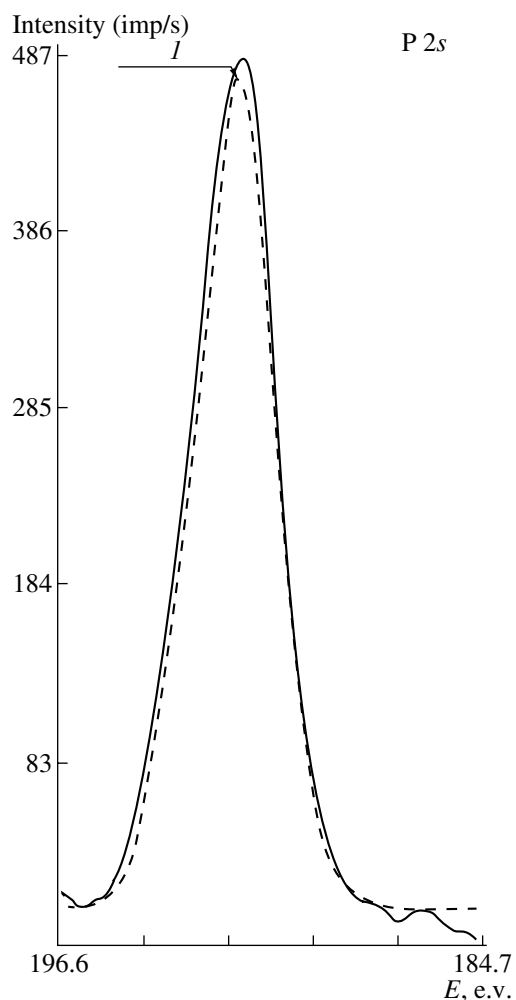


Fig. 7. X-ray electron spectrum of P 2s electrons in the starting peridotite-staffelite mixture. (1) P^{5+} in the form of Ca and Fe phosphates.

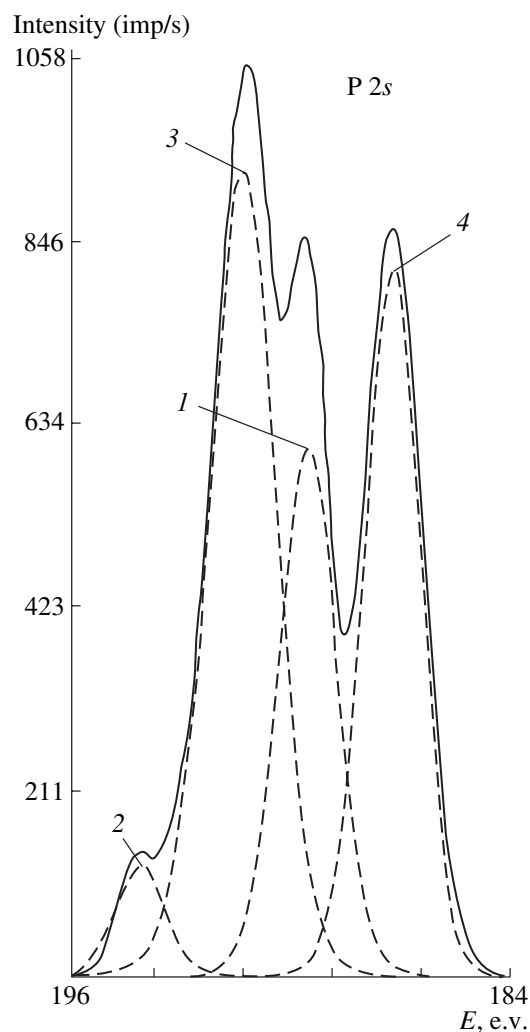


Fig. 8. X-ray electron spectrum of P 2s electrons in a layer of condensate obtained by the vaporization of the peridotite-staffelite mixture. (1) P^{5+} as $Ca_3(PO_4)_2$, (2) P^{5+} as P_2O_5 , (3) P^{5+} as $Ca_2P_2O_7$, and (4) reduced P as P^0 .

escapes into space under lunar conditions. Thus, the temperature factor can provide the reduction of P and Fe both by direct thermoreduction and in reactions involving internal reducing agents. It should be noted that the amount of Fe^0 and P^0 produced in an exchange redox reaction can be limited by the amount of reducers in the system. Therefore, we believe that reducing agents, such as C, H_2 , and others, play a minor role in the formation of elementary Fe and P on the Moon.

The obtained results shed light on possible changes in the valence state of P and Fe in lunar impact processes. They provide the first experimental evidence for P reduction at high temperatures corresponding to impact-evaporation phenomena. In addition, these experiments support the hypothesis of Hunter and Taylor that lunar phosphates (apatite and whitlockite) could be sources of P^0 , which probably reacted with reduced Fe to form schreibersite and Fe-P alloys. The experimental data also demonstrate that the presence of

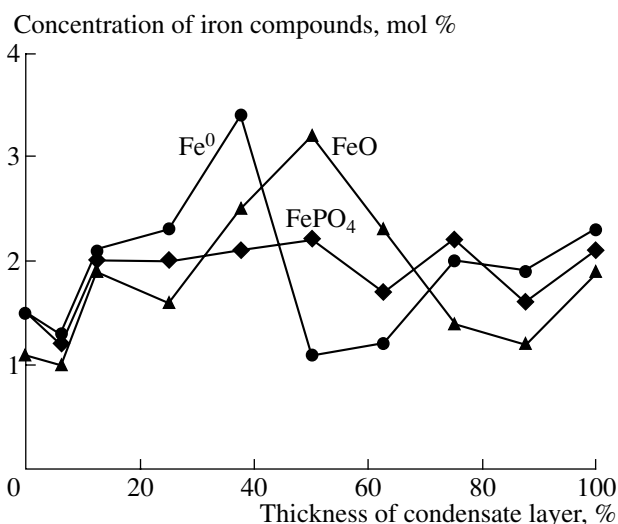


Fig. 9. Concentrations of the reduced and oxidized forms of Fe in the layers of condensate obtained by the vaporization of the peridotite-staffelite mixture.

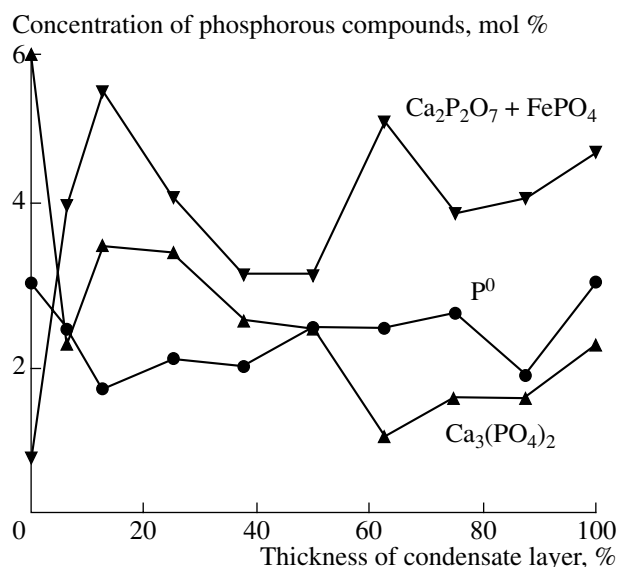


Fig. 10. Concentrations of the reduced and oxidized forms of P in the layers of condensate obtained by the vaporization of the peridotite–staffelite mixture.

reducing agents, such as implanted carbon and hydrogen or impurities of carbon and sulfur from a meteoritic source, is not necessary to form metallic iron or Fe–P alloys, as was supposed by Goldstein et al. [11–13].

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