TEMPERATURE-TIME RELATIONSHIPS FROM LUNAR TWO PHASE METALLIC PARTICLES (14310, 14163, 14003)

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Two-phase α-kamacite + γ-taenite and α-kamacite + schreibersite particles, separated magnetically from soil samples 14163 and 14003 and two-phase α-kamacite + schreibersite particles, removed mechanically from lunar rock 14310, have been examined metallographically and with the electron microprobe. These particles have been thermally metamorphosed during lunar residence. The temperatures at which these two-phase structures finally came into interface equilibrium have been deduced by comparing their measured interface compositions with the experimentally determined Fe-Ni or Fe-Ni-P equilibrium diagrams. For several of the metal-schreibersite particles an estimate has been made of the time required to form the measured concentration-distance relationship around particles of a known size using a simple diffusion-growth theory. The two-phase metal particles that have been reheated in lunar fragmental rocks show final equilibration temperatures in the range 500—600°C. Several phosphide-metal particles appear to require maximum growth times of 1 month to 30 yr in this temperature range.

The particles extracted from lunar rock 14310 have nickel and cobalt contents similar to iron meteorites or chondrites. A final equilibration temperature of 700°C is proposed for the metal/phosphide particles after cooling from the solidus during a period of about one month.

The Ni and Co contents of many metallic particles in 14310 are consistent with a meteoritic origin. It is probable that the silicate portion of 14310 had its origin, at least in part, as pre-Imbrium soil and breccia in which metal populations of both meteoritic and non-meteoritic Ni:Co content are always present.

1. Introduction

The metallographic structures, silicate associations and bulk compositions of a large number of metal particles from the Apollo 14 soil were studied by Goldstein et al. [1]. The majority of the metal fragments have Ni and Co contents corresponding to the range appropriate to meteoritic metal. This metal appears to have been liberated to the soil after undergoing thermal metamorphism within the rocks that now constitute the Fra Mauro formation. As a consequence of this thermal metamorphism it has lost its original meteoritic microstructure and undergone limited redistribution of trace elements. This metal had its meteoritic origin in the predominantly chondritic projectiles that bombarded and accumulated in the pre-Imbrium lunar crust.

In addition, among the metallic samples there were a small number of complex two-phase particles containing kamacite (α Fe) + taenite (γ Fe) and also some phosphide-metal structures. Most of these two-phase particles also appear to have been reheated within the lunar rocks or soil. The distribution of nickel and phosphorus between the two phases of these metal particles can provide a measure of the last temperature of heat treatment or equilibration for the metal and consequently for the silicate material in which it was incorporated.

In this paper we report analyses of the individual phases of a number of two-phase metallic α + γ and phosphide-metal particles as well as estimates of the effective temperatures and, in some cases, times of equilibration.
2. Procedure

A number of two phase metallic particles were separated magnetically from the $>125\mu\text{m}, <1\text{ mm}$ size fraction of lunar soils 14003,18 and 14163,165. In addition, through the courtesy of Dr. L.S. Walter, we were able to examine four of the larger fragments of metal ($>0.2\text{ mm}$ across the smallest dimension) that were mechanically removed from lunar rock 14310. All the particles were mounted in epoxy, polished and chemically analyzed with the electron microprobe. Special attention was given to the compositions of the two phases at their contact interfaces since these values may be taken to represent the frozen-in local equilibrium compositions corresponding to the most recent heat treatment of the metal. Point analyses were made at carefully selected locations within the phases and usually a series of analyses were made along a line crossing the interface. Estimates of the effective temperatures of final equilibration are made by comparing the compositions of the two coexisting phases at their contact interfaces with measured or interpolated tie-line relationships in the appropriate Fe-Ni [2] or Fe-Ni-P [3] equilibrium diagrams.

It is not possible to give a detailed account of the complex cycle of thermal events that preceded the final equilibration of the particles. However a general indication of the highest temperature to which the metal particles have been heated may sometimes be obtained by considering the extent to which the meteoritic structure, overall morphology, and phosphorus content appear to have been altered by thermal processes.

3. Results and discussion

The structural details, interface compositions and bulk analyses are collected in table 1. The specimen numbers, descriptions and bulk compositions for the lunar soil samples are those of Goldstein et al. [1].

3. 1. $\alpha - \gamma$ structures

Specimens #28.9 and #26.27 (table 1) contain significant quantities of phosphorus ($>0.15\text{ wt\%}$) and particles of taenite, called isothermal taenite [4] are observed in the microstructure. These particles were exsolved from the kamacite upon reheating and contain P and Ni contents given by the Fe-Ni-P ternary phase diagram at the temperature of reheating. The interface compositions of #28.9 and the taenite composition of #26.27 are plotted as triangles on the Fe-Ni-P phase diagram, fig. 1. An equilibration temperature of $\sim650^\circ\text{C}$ is indicated for both particles from the ternary equilibrium.

The composition of the $\alpha$ and $\gamma$ phases in specimens #27.12, #28.1 and #M-9, which contain insignificant amounts of P, are plotted as rectangles on the Fe-Ni binary diagram (fig. 1). In specimens #27.12 and #28.1 the metal again appears to have had a period of residence at high temperatures $\sim550-650^\circ\text{C}$. It should be noted that the $\alpha$ phase shows a final equilibration temperature lower than that of the $\gamma$ phase. This is a similar effect to that observed at $\alpha/\gamma$ interfaces in iron meteorites where, because of higher diffusion rates in the $\alpha$ phase, the Ni content of the $\alpha$ phase changes to reflect the decreased temperature. However the Ni content of the $\gamma$ phase changes only in the immediate vicinity of the $\alpha/\gamma$ interface and this change cannot be measured with the electron probe. It is not possible from the tie-line values to be certain of the maximum reheating temperatures for these specimens. We are inclined to place more reliance on the lower limits of estimated equilibration temperature since the conditions of reheating did not cause a significant increase of their phosphorus contents.

![Fig. 1. Temperature composition diagram for the binary Fe-Ni diagram and for the $\alpha/\alpha + \gamma$ and $\gamma/\alpha + \gamma$ boundaries at the three-phase corners of the $\alpha + \gamma + \text{Ph}$ field in the ternary Fe-Ni-P diagram. The symbols $\Delta$ and $\Box$ represent the $\alpha/\gamma$ interface compositions of several $\alpha + \gamma$ two-phase particles in the ternary and binary systems respectively. The size of the symbol represents the error in the composition determination.](image-url)
### Table 1
Description of two phase metallic particles (14310, 14163, 14003).

<table>
<thead>
<tr>
<th>Specimen no.</th>
<th>Bulk composition (wt%)</th>
<th>Description of specimen</th>
<th>Interface compositions for 2-phase structures (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ni Co P</td>
<td></td>
<td>α phase</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Ni Co P S Type Ni Co P S</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Final equilibration temp. (°C)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>α - γ structures</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>28.9</td>
<td>6.5 0.45 0.2</td>
<td>isothermal taenite particles ~ 3 μm:</td>
<td>5.4 0.45 0.23 &lt;0.02 γ 10.7 n.a. 0.06 n.a.</td>
</tr>
<tr>
<td>26.27</td>
<td>7.7 0.4 0.16</td>
<td>one particle of clear taenite</td>
<td>n.a. 0.4 0.16 &lt;0.02 γ 12.5 n.a. 0.1 n.a. γ ~650</td>
</tr>
<tr>
<td>27.12</td>
<td>6.1 0.45 0.05</td>
<td>isothermal taenite particles ~ 5 μm:</td>
<td>5.8 0.45 0.05 &lt;0.02 γ 14.7 n.a. &lt;0.02 n.a.</td>
</tr>
<tr>
<td>28.1</td>
<td>6.5 0.6 &lt;0.02</td>
<td>one particle of clear taenite</td>
<td>6.5 0.6 &lt;0.02 &lt;0.02 γ 20 n.a. &lt;0.02 n.a.</td>
</tr>
<tr>
<td>M9</td>
<td>8.5 0.75 &lt;0.02</td>
<td>blocky martensite tempered to α + γ, fig. 6 (Goldstein et al., 1972)</td>
<td>7.2 0.75 &lt;0.02 &lt;0.02 γ 36 n.a. &lt;0.02 n.a.</td>
</tr>
<tr>
<td>26.12</td>
<td>6.9 0.75 &lt;0.02</td>
<td>chondritic α-γ-plessite (16% Ni): not reheated.</td>
<td>5.8 0.75 &lt;0.02 &lt;0.02 γ 50 n.a. &lt;0.02 n.a.</td>
</tr>
</tbody>
</table>

Phosphide-metal particles

| 28.4         | 5.4 0.35 2.9           | laths of phosphide, ~3.5 μm wide, in a globule. | 3.6 0.35 0.4 <0.02 Ph 10.0 n.a. 15.5 n.a. | ~600 | Fe-Ni-P |
| 23.20        | 5.7 0.3 4.0            | laths and rim of phosphide, in a globule | 4.3 0.4 0.3 <0.02 Ph 11.2 0.05 16.7 0.1 | ~550 | Fe-Ni-P |
| M7.1         | 7.8 0.65 1.7           | phosphide precipitates ~ 10 μm in poly-crystalline α. Small amount of FeS | 5.9 0.7 0.1 <0.02 Ph 20 0.02 16.6 0.05 | ~500 | Fe-Ni-P |
| 22.5         | 13.9 0.8 0.3           | γ globule rimmed with phosphide, swathing α at phosphide metal interface | 5.9 n.a. 0.12 n.a. Ph 18.2 n.a. | 16.1 n.a. | ~500 | Fe-Ni-P |

Meteoritic Widmanstatten structure

| M3.1         | 6.6 0.45 0.1           | swathing α=6.4 Ni: Widmanstatten band α=7.4 Ni, γ=30.4 Ni (max). | 6.4 0.45 0.17 <0.02 α-γ 7.4 n.a. 0.11 n.a. | 550 | Fe-Ni-P |
It may be noted, by reference to table 1, that the \( \alpha \) and \( \gamma \) interface compositions of the chondritic \( \alpha \cdot \gamma \cdot \) plessite specimen #26.12 lie in the temperature range of the binary Fe-Ni system that has not been investigated experimentally, < 500°C. The \( \alpha/\gamma \) equilibration temperatures for the specimen, \(< 350°C\), are taken from thermodynamically calculated [2] extrapolations of the high temperature phase boundaries. The chondritic particle #26.12 does not show any metallographic or compositional evidence of reheating and this is in agreement with the identification of #26.12 as a particle from one of the meteoritic projectiles that produced local cratering in the vicinity of the Apollo 14 landing site. Since such particles have never been incorporated into the lunar crust, they have escaped thermal metamorphism and so retain their meteoritic structure as well as their meteoritic ratio of Ni and Co. Thus, we have found that most of the two-phase \( \alpha + \gamma \) meteoritic metal at the Apollo 14 landing site has been thermally processed to a greater or lesser extent in the rocks of the lunar crust. However, it does not appear necessary for such metal to have been actually melted during residence in the lunar crust.

3. 2. Phosphide-metal particles

Specimens #28.4, 23.20, M7.1 and 22.5 (table 1) constitute the group of samples that have been described [1] as phosphide-metal particles. The phosphides are distributed as bars or irregularly shaped precipitates within the \( \alpha \) or \( \beta \) phase of the metallic matrix but are often present in a rim around the periphery of the particle.

In addition there is the occasional presence of minor sulfides within the phosphide precipitates.

The Ni, P and Co compositions at the \( \alpha/\text{Ph} \) interfaces were determined from microprobe point analyses conducted across the two phases. Decreasing Ni concentrations were observed in the \( \alpha \) phase as the \( \alpha/\text{Ph} \) interface was approached, indicating that the phosphides grew from the solid state on cooling. The \( \alpha \) and phosphide phase compositions of the 4 particles are superimposed on the 550°C Fe-Ni-P isotherm, fig. 2, from which it may be seen that all of the data is consistent with the tie-lines in the \( \alpha + \text{Ph} \) phase field at a temperature in the region of 550°C. The measured P contents of the \( \alpha \) phase, as reported in table 1, indicate that particle #28.4 actually has an equilibration...
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Fig. 2. Fe-Ni-P isotherm at 550°C. The Ph/α interface compositions of the 4 phosphide-metal two-phase particles are plotted across the 2-phase α + Ph field on the diagram.

temperature closer to 600°C while particles # M7.1 and #22.5 have an equilibration temperature closer to 500°C.

One can obtain an indication of the time necessary for phosphide growth if one assumes that all the growth occurs isothermally, for example at the equilibration temperature. The calculation scheme for phosphide growth according to Goldstein and Ogilvie [5], was adopted for our analyses of particles # 28.4 and #M7.1. The assumed equilibration temperatures were 600 and 500°C respectively and the measured Ni contents for α, phosphide and the bulk as given in table 1 were used. The ternary Fe-Ni-P diffusion coefficient in α, $D_{Ni}^α$, as given by Heyward and Goldstein [6], where:

$$D_{Ni}^α (0.5 \text{ wt}\% \text{Ni}, 1.2 \text{ wt}\% \text{P}) = 0.62 \exp (-51500/RT)$$

was used for the calculation. For particle # 28.4 we calculated that it would take approximately 1 month at 600°C to grow the measured phosphide particle size of 3.5 μm. For particle # M7.1 we found that it would take approximately 50 yr at 500°C to grow the measured phosphide particle size of 10 μm. Presumably the depth of burial and the temperature of the surrounding silicate determine the equilibration times. If the major amount of particle growth occurred at higher temperatures, then the growth time would be correspondingly smaller. Therefore the isothermal calculations represent the maximum time of growth.

The bulk analyses of the phosphide-metal particles as well as the α - γ particles show meteoritic proportions of Ni and Co (> 5 wt% Ni, < 1.0 wt% Co). However there is a significantly increased amount of phosphorus in many of these particles with respect to that of meteoritic metal (> 0.05 wt%). This increased P content comes about by thermal metamorphism of the meteoritic metal during residence of the particles in the lunar fragmental rock and soil.

The increased content of phosphorus may arise by one of two processes. On the one hand phosphorus may be reduced from lunar phosphate and enter the metal. This process has been discussed by Taylor and Heyman [7] for chondritic metal and by Goldstein et al. [1] for lunar metal. On the other hand phosphorus from previously existing meteoritic schreibersite (phosphide) may redissolve in the metal and thereby increase its content of phosphorus in metallic solid solution.

The schreibersite may have been originally in the form of phosphide precipitates within or at the outer edge of the fragments of meteoritic metal. This process can occur either in the solid state at high temperatures or by melting. The absence of dendritic and eutectic structures suggests that if melting did occur the metal was cooled slowly from the liquid. The well developed phosphide precipitates likewise indicate slow cooling in the solid state.

3.3. Meteoritic Widmanstatten structure # M3.1

Sample # M3.1, which contains ~6.6 wt% Ni, 0.45 wt% Co and 0.1 wt% P, and is illustrated in fig. 11b of Goldstein et al. [1], is probably a particle with an incomplete Widmanstatten structure from a stony meteorite. Fig. 3 gives a sketch of the particle structure. The kamacite bands of the Widmanstatten structure

Fig. 3. Sketch of the structure of sample #M3.1. The Ni (6.3–15 wt%) and P (0.03–0.17 wt%) contents of selected α, α + γ and γ areas in the structure are noted.
are separate grains of α delineated by grain boundaries. Regions of swathing kamacite surround the incomplete Widmanstatten structure and a thin band of taenite-γ lies along one edge of the particle and is shaded dark in fig. 3.

Superimposed on fig. 3 are the average Ni and P contents at a selection of α and α + γ areas in the structure. These compositions were determined from a series of microprobe point analyses conducted at 2 μm intervals across the specimen. All of the phosphorus is present in solid solution in the kamacite and there are no detectable phosphides. Regions of low (6.3%–6.4%) nickel, such as the small metal nodule and the swathing kamacite around the left side of the incomplete Widmanstatten pattern are free of precipitates. By contrast the areas of higher nickel content (6.7–7.6 wt%), such as the kamacite bands of the incomplete Widmanstatten structure, show micron-sized particles of a nickel-rich, phosphorus-free isothermal taenite. Many of the particles of isothermal taenite are too small to give a reliable probe analysis, but a carefully conducted point analysis on the largest particle of isothermal taenite gave a nickel content of 30.4 wt% with 0.02 wt% P.

It has not been possible to obtain unambiguous tie-line relationships on specimen #M3.1. However we observe that isothermal taenite has formed within high Ni (6.7–7.6 wt%) kamacite bands but has not formed in the low Ni (6.3–6.4 wt%) swathing kamacite. These two composition ranges must lie respectively in the α + γ and α phase fields of the Fe-Ni-P equilibrium diagram at the effective reheating temperature. Fig. 4 shows an enlarged portion of the 550°C isothermal section for Fe-Ni-P with the measured Ni and P values of the kamacite in #M3.1 superimposed. It may be seen that at this temperature the composition of the swathing kamacite lies in the α phase field whereas the composition of the kamacite bands lie in the α + γ field. An equilibration temperature of 550°C is therefore indicated for this particle. The composition of the only measured isothermal taenite particle (30.4 wt% Ni-0.02 wt% P) is also consistent with a tie line in the α + γ field at 550°C.

3. 4. Metal extracted from lunar rock 14310

Lunar rock 14310 has received a great deal of attention because it is one of only two Apollo 14 rocks weighing over 50 g which have an igneous texture. Several investigators have suggested that it has differentiated [8, 9] from a more primary magma. On the other hand attention has been drawn to the chemical similarity between 14310 and Fra Mauro breccias [10], and soils [11, 12] and to the possibility that rock 14310 was produced by the remelting of this type of pre-existing material [12, 13].

Metal and troilite are found in association with other opaque phases between the silicates and in the mesostasis. El Goresy et al. [14a] have noted that in rock 14310 metallic iron-nickel particles may be observed with a range of nickel contents (1.5–37 wt%). The Ni content of the metal phase is correlated with the mineral assemblage with a preference of Ni for the metal phase associated with troilite [14b]. Of the 22 metal grains analyzed by El Goresy, about half have meteoritic Ni-Co contents. However, data are not available for the size of those metal particles.

Our own investigations were conducted on four particles of metal >0.2 mm in thickness that were extracted mechanically from rock 14310. The metal was therefore completely divorced from the minerals with which it was associated in the rock, and all specimens showed greater or lesser degrees of mechanical cracking, probably resulting from the extraction process. Bulk analyses were obtained for all four specimens by averaging a number of microprobe point analyses and these bulk compositions are recorded in table 1.

In each case the nickel and cobalt values when compared to the Ni-Co criterion of Goldstein and Yakowitz [15], would permit a meteoritic origin for the metal. This result is consistent with the concept of...
of 14310 as a remelted soil or fragmental rock. However the P contents of the metal are higher than those normally found in meteoritic metal (>0.05 wt%) and vary from 0.2 to 0.3 wt%. The metal in samples 1 and 2 is homogeneous and free from phosphide precipitates. One of the two phosphide-containing specimens (4) is illustrated in fig. 5. A microprobe trace was made across the largest, 5 μm wide, lathlike precipitate in specimen 3 (table 1) and the uncracked lathlike precipitate (#1) near the center of the particle 4 (fig.5). Point analyses in the other uncracked phosphide laths of particle 4 (fig.5) gave confirmatory values but analyses in the large, rounded, and rather badly cracked phosphide (#2) near the edge of the specimen, gave variable nickel results in the range 14.3–16.2 wt%.

Fig. 6 shows that the coexisting phase compositions for the uncracked, well crystallized, phosphide laths in both specimens 3 and 4 may be superimposed satisfactorily on the 700°C isothermal of the ternary Fe-Ni-P system. Experimentally Doan and Goldstein [3] have produced equilibrated phosphide crystals of 5 μm in size by step cooling from 900°C over a time period of one month. Therefore we would argue that basalt 14310 cooled from the solidus region to temperatures below 700°C in time periods of about a month.

However, attention has already been drawn to the presence in specimen 4 of what appears to be two generations of phosphide. The larger, rounded, inclusion appears to have a higher nickel content than the smaller, lathlike precipitates. Our analyses of the large rounded phosphide (#2) show variations of nickel content but, unfortunately, the phosphide is cracked and the analyses may not be entirely reliable. However, if the higher nickel content of the large rounded phosphide can be accepted as genuine it would seem to indicate that the material has undergone a more complex cycle of events than a simple uninterrupted cooling from the molten silicate condition.

El Goresy et al. [14a] drew special attention to a metallic inclusion of about 10 μm diameter which is surrounded in part by sulfide and contains small rounded grains ~ 2 μm in diameter of schreibersite in the Fe-Ni metal, one of which contains 28 wt% Ni. The matrix Fe-Ni metal is γ containing about 15.5 wt% Ni, 1.1 wt% Co and 0.09 wt% P. The Ni content is reduced to about 5–7 wt% (α phase) at the metal phosphide interface. The Ni depletion and Ni interface values argue for an equilibration temperature during cooling of about 450°C according to a reasonable extrapolation of the Fe-Ni-P diagram. The small size of the phosphide and the low P content of the particle also argue for lower effective equilibration tempera-
tures than we measured in our 14310 particles, #3 and #4. El Goresy et al. [14a] also report a very small submicron Ni-poor phosphide coexisting with the Ni-rich schreibersite. Quantitative microprobe analysis was not possible because of the small size of the phosphide. We have not found any phosphides of this type in our own investigations which have been conducted on large, specially extracted, particles of metal.

The presence of schreibersite in lunar rocks has been proposed as a criterion for meteorite contamination. More recently El Goresy et al. [14a] have re-examined this criterion and conclude that it is invalidated by their discovery of metal phosphide associations in lunar rock 14310 as outlined above. However the overall Co content of the metal-phosphide particle discussed by El Goresy [14a] is also less than 1 wt%, if the Co poor sulfide and phosphide phases are considered. Therefore this phosphide metal particle as well as the phosphide metal particles we have examined in lunar rock 14310 satisfy the cobalt criterion of meteoritic origin [14].

The high P content of the schreibersite-containing metal particles can come about by thermal metamorphism of the meteoritic metal during residence of these particles in the material which formed the basaltic rock. As discussed previously, phosphorus can enter the metal phase by reduction of lunar phosphate or solution from previously existing meteoritic schreibersite. Ridley et al. [16] argue that the presence of schreibersite in rock 14310 is probably due to the high overall phosphorus content of this rock. However because of the presence of a major amount of meteoritic metal we suggest that a meteoritic origin is more probable.

In addition, our studies of the texture of the 14310 metal phosphide particles indicates a meteoritic origin for schreibersite. The complex structure of particle 4 for example, shows two generations of phosphide. This type of structure argues most strongly for the high temperature dissolution of P from previously formed meteoritic phosphides, followed by a slow cooling sequence in which phosphide exsolution occurs as laths.

The presence of meteoritic metal as a major fraction of the metal particles in 14310 indicates that a source of such metal must have been available before melting. We have found from our studies of the metal particles in the Apollo 14 soils [1] that most of the metal was meteoritic, originating in the projectiles that bombarded the pre-Imbrium lunar crust and were accumulated and incorporated in the fragmental rocks and soil. Some of this material is included in basalt rock 14310. Thus our results are consistent with the concept of 14310 as a remelted soil or fragmental rock.

4. Conclusions

(1) Most of the two phase metal particles from soils 14003 and 14163 have been thermally processed within the fragmental rocks and soil. The α - γ particles had equilibration temperatures between 500 and 650°C while the phosphide-metal particles had equilibration temperatures between 500 and 600°C.

(2) The phosphide-metal particles were either reheated at high temperatures or remelted. Phosphide growth calculations indicate effective periods of residence within the lunar crust of from 1 month to 50 years at temperature of 500 to 600°C.

(3) Phosphide-metal equilibration in rock 14310 occurred at about 700°C. The cooling period of about 1 month from the melting point to 700°C was obtained.

(4) A major fraction of the metal particles examined in rock 14310 has a meteoritic origin and the most likely source of the metal is from the fragmental rocks and soil. Our results are consistent with the concept that rock 14310 was produced, at least in part, by the remelting of pre-Imbrium lunar material.

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References