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X-RAY STUDY OF PCP FROM THE MURCHISON CM CARBONACEOUS CHONDRITE

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Abstract: We have performed X-ray diffraction measurements and electron-microprobe analyses of PCP particles in the Murchison CM carbonaceous chondrite and determined the relative abundances of minerals in PCP. Type-I PCP, which is rich in Fe and S and occurs as rounded, massive forms, shows high abundance of tochilinite and variable amounts of cronstedtite and magnetite. Tochilinite shows very strong 002 reflection at 5.4 Å and very weak 001 reflection at 10.8 Å, indicating a higher Fe content in octahedral sites. Comparison of the observed X-ray diffraction pattern of type-I PCP with the calculated X-ray patterns of tochilinite in various compositions shows that Fe occupies more than 90% of the octahedral sites in tochilinite in PCP, which is quite different from terrestrial tochilinite. Type-II PCP, which occurs mainly in the matrix as fibrous needle clusters, is composed mainly of cronstedtite with a disordered stacking sequence along the c-axis. The other major component of type-II PCP differs between non-clastic and clastic portions. In the former, it is tochilinite, whereas in the latter, it is a mixed-layer mineral made of cronstedtite and tochilinite layers. Relative abundances of minerals in type-II PCP indicate that the proportion of the mixed-layer mineral increases with a decrease of tochilinite; thus the mixed-layer mineral is presumed to be a secondary alteration product of tochilinite. Therefore, the enrichment of the mixed-layer mineral in type-II PCP in clastic portion suggests that aqueous alteration in the clastic portion has advanced relative to that in the non-clastic portion and that dynamic processes such as mixing and fragmentation of the rocks might have assisted to activate the aqueous alteration reactions. Our experiments reveal that Murchison is a mixture of multiple lithic phases that have experienced different degrees of aqueous alteration and the alteration might have been enhanced by cataclastic effects probably induced by impacts on the meteorite parent body.

Introduction

Poorly-characterized phase (PCP; defined by Fuchs et al., 1973) is a major constituent of CM carbonaceous chondrites. It comprises up to 30 vol% of the matrix (Bunch and Chang, 1980) and typically ranges in size from 20 to 200 µm. It occurs mainly in the matrix and, in lesser amounts, in chondrules. It can be divided into two types: rounded to subrounded, massive inclusions (type I) and fibrous needle clusters (type II) (Bunch and Chang, 1980; Barber, 1985). It is rich in Fe and O and contains variable amounts of Al, Ni, Si, Mg, Cr, and P (Tomeoka et al., 1989). Despite the original definition, some characteristics of PCP are revealed by transmission electron microscope (TEM) studies (e.g., Barber 1981; Tomeoka and Buseck, 1983, 1985; Barber et al., 1983; Mackinnon and ZOLENSKY, 1984). Barber (1981) was the first to report that amongst the PCP there was an unknown layer phase with a layer spacing of approximately 10.8 and 5.4 Å, rich in Fe, Ni, S and thought to contain oxygen. Mackinnon and Zolensky (1984) designated the unknown layer phase to be tochilinite based on X-ray diffraction data. Tomeoka and Buseck (1985) studied PCP by TEM and found that it is a complex intergrowth of 1) an Fe-Ni-S-O layered mineral (tochilinite), 2) cronstedtite, 3) minor amounts of submicron grains of magnetite, chromite, and a mineral containing Fe, Ni, Cr, and P. They redefined the acronym PCP as "Partly Characterized Phases". We use this definition for the rest of the paper.

In contrast with mineralogical and structural identification, the origin and alteration process of PCP still remain unknown. In this study, we performed X-ray experiments on various forms and compositions of PCP in the Murchison CM chondrite by using a Gandolfi X-ray camera which enables us to obtain bulk X-ray diffraction data from a single PCP particle. Our goal is to estimate relative abundances of phases comprising PCP and to see how the abundances change in the course of aqueous alteration reactions.

2. Material and Experimental Methods

Polished thin slices with thickness of ~100 μ m were prepared from two meteorite chips of Murchison. PCP particles in these polished slices have been studied using a reflected-light optical microscope and an electron-microprobe analyzer (JEOL 733 Superprobe) equipped with wave-length-dispersive X-ray spectrometers (WDS). WDS analyses were obtained at 15 kV accelerating voltage and 10 nA beam current with a focused beam ~3 μ m in diameter. A defocused beam 50 μ m in diameter was also used to determine bulk composition of individual PCP particles. The correction for quantitative chemical analysis was made by the ZAF method.

After petrographic characterization, portions of PCP approximately $50\times50\times50~\mu m$ in size were extracted from the thin slices under a binoscope using an edged tool. The extracted PCP were mounted on a thin glass fiber with $10~\mu m$ in diameter and exposed to Cr K α X-ray in a Gandolfi camera. The X-ray generator was operated at 35 kV and 20 mA. It usually took 36–72 hours to obtain a X-ray powder diffraction photograph of PCP. The X-ray photograph was scanned by a micro-densitometer and transfered to a computer in order to read precise peak positions and to calculate integrated intensities.

The Gandolfi method (Gandolfi, 1967) appears to be the most appropriate method for characterization of phases and determination of the relative abundances of these phases in PCP. This method can obtain a precise X-ray powder photograph from a small particle of $50-100~\mu m$ in diameter. Most of PCP particles range in diameter $50-100~\mu m$, thus bulk mineralogical information of individual PCP particles is available by this method. Similar information about PCP can also be estimated from the bulk chemical composition of PCP by separating the bulk composition into several mineral compositions (e.g., McSween, 1987). This method is useful to estimate the approximate mineral abundances in PCP, but is not valid if minerals in the PCP have heterogeneous compositions or if a mixed-layer mineral is present. The X-ray experiments of this study reveal that a mixed-layer mineral is one of the major components of PCP.

3. Results

3.1. Lithology of Murchison

Murchison is a regolith breccia, as demonstrated by the fact that it shows brecciated texture and contains solar-implanted noble gases (Black, 1972). It consists of mainly two lithologies: non-clastic and clastic portions. In the non-clastic portion (Fig. 1a), many chondrules show circular shape and have fine-grained rim material. PCP particles are the dominant component in the matrix. They occur as a well-recognized spheroidal shape in back-scattered electron images, indicative of large compositional difference mainly in Fe, between PCP (Fe rich) and the matrix (Fe poor). The non-clastic portion does not seem to be affected by post-accretionary cataclastic effects and corresponds to "primary accretionary rock" in the CM chondrites, as described by Metzler et al. (1992).

In the clastic portion (Fig. 1b), chondrules are principally absent and mineral fragments, mainly forsterite, are dispersed. The clastic portion appears to have experienced

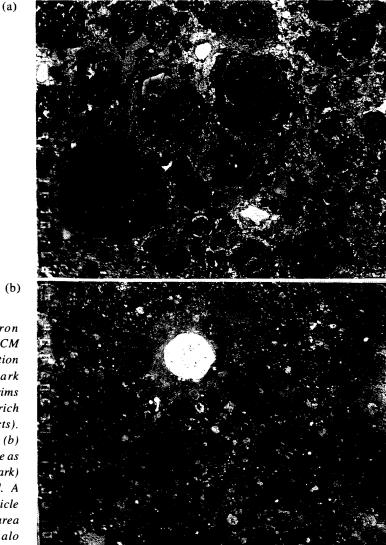


Fig. 1. Back-scattered-electron (BSE) images of the Murchison CM chondrite. (a) Non-clastic portion consisting of chondrules (dark rounded objects), fine-grained rims around chondrules, and matrix rich in PCP particles (bright objects). Width of the image is 3 mm. (b) Clastic portion at the same scale as (a). Many mineral fragments (dark) and PCP (bright) are dispersed. A rounded large type-I PCP particle is present in the upper central area of the image and a bright halo spreads from the PCP.

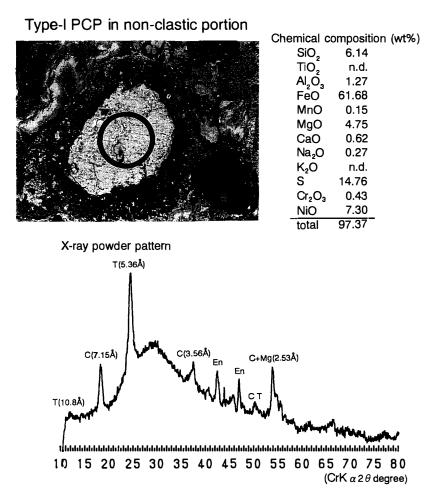


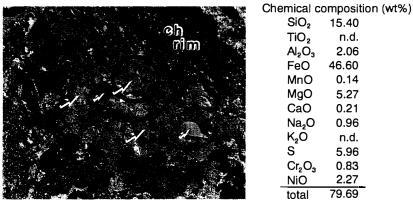
Fig. 2a. A BSE image of type-I PCP and its chemical composition and X-ray powder pattern. Width of the image is 300 µm. The circle in the BSE image is an approximate portion where X-ray diffraction is measured. The chemical composition is obtained by defocused beam analysis. The BSE image shows typical texture of type-I PCP particle: it is massive, subrounded and has a fine-grained rim. The X-ray pattern shows that tochilinite (T) is dominant mineral in type-I PCP. The other abundant phases are cronstedtite (C) and magnetite (Mg). Enstatite (En) is also contained in type-I PCP, but it is rarely observed.

mixing and fragmentation induced by dynamic processes such as impacts. The abundance of anhydrous minerals in this portion is apparently lower than that in the non-clastic portion. PCP particles occur in the matrix, but are less abundant than in the non-clastic portion. The compositional difference between the PCP and material around the PCP is not distinct, unlike the PCP in the non-clastic portion. In the three thin slices used in this study, two are dominated by the clastic lithology and one by the non-clastic lithology.

3.2. Type-I PCP

Type-I PCP particles (Fig. 2a) occur both in non-clastic and clastic portions and contain large amounts of Fe (~70 wt% in FeO) and S (~15 wt% in S), and small but





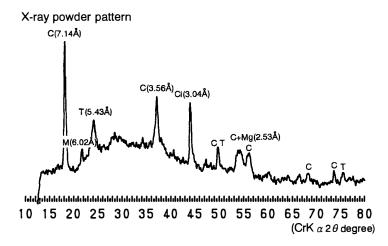


Fig. 2b. A BSE image of type-II PCP particles (indicated by large arrows) in the non-clastic portion. Also shown are chemical composition and X-ray powder pattern of the PCP indicated by a circle. Width of the image is 300 µm. The BSE image shows that type-II PCP particles occur outside the chondrule rim (Ch rim) and coexist with mineral fragments of mainly olivine (indicated by small arrows). Chemical composition shows that, compared with type-I PCP, Si and Mg are more abundant and Fe, S, and Ni are less abundant. The X-ray pattern shows that the most dominant mineral is cronstedtite (C) and next are tochilinite (T) and calcite (Cl). Magnetite (Mg) and a mixed-layer mineral are also identified.

variable amounts of Ni, Si, Mg, Al, and Ca (Fig. 2a). Type-I PCP in the clastic portion (Fig. 1b) sometimes shows a bright halo around the PCP in back-scattered electron images. This halo spreads over the clastic matrix material and is richer in Fe and S relative to the portions outside the halo. This appears to suggest that the halo has been generated by leaching of Fe and S from the PCP due to aqueous alteration after the clastic matrix material was produced.

Relatively large particles of type-I PCP (>70 μ m in diameter) were taken from both non-clastic and clastic portions and their X-ray diffraction patterns were recorded. A representative X-ray pattern of type-I PCP is shown in Fig. 2a. This shows a very

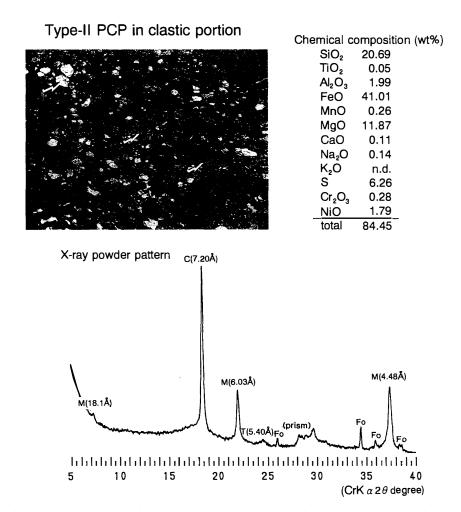


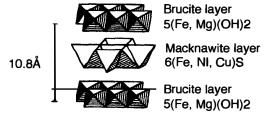
Fig. 2c. A BSE image of clastic portion where type-II PCP are dispersed. Width of the image is 2 mm. The BSE image shows that type-II PCP particles in the clastic portion have a variable appearance; some are well recognized particles (indicated by a large arrow), whereas most are formless and grade into the matrix (indicated by small arrows). Chemical composition and X-ray powder pattern are obtained from the circled portion of PCP. The composition is basically similar to that of type-II PCP in the non-clastic portion. In contrast, the X-ray pattern is quite different. The mixed-layer mineral (M) is much more abundant in type-II PCP in the clastic portion. Basal reflections of mixed-layer mineral appear at 18.1 Å, 6.03 Å, 4.48 Å in this X-ray pattern. Prism reflection of cronstedtite is clearly observed at around 28–30 degrees. Minor amounts of forsterite (Fo) is also observed and it may be fine-grained mineral fragments which are dispersed throughout the clastic portion.

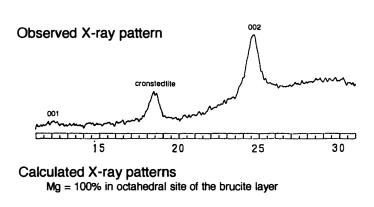
strong 5.4 Å, strong 7.2 Å, 3.6 Å and 2.5 Å, and many smaller reflections, indicating that the most abundant mineral is tochilinite and the other major minerals are cronstedtite and magnetite. The 5.4 Å reflection is assigned to the 002 reflection of tochilinite, since tochilinite has 10.8 Å basal spacing (Organova et al., 1973). But, in the X-ray pattern, the 001 reflection from the 10.8 Å basal spacing is very weak (Fig. 2a). This might be explained by the fact that the mackinawite and brucite (or amakinite) layers of tochilinite (Fig. 3) are predominantly occupied by Fe in their cation sites, weakening the 001 re-

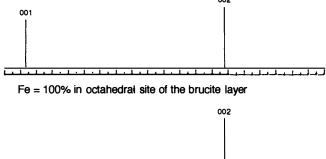
flection due to 5-Å periodicity of Fe-rich layers in the structure.

X-ray intensity calculations using a structure model of tochilinite (Organova et al., 1973) were conducted to estimate Fe^{2+} occupancy in the octahedral sites of the tochilinite in PCP. Figure 4 shows that the calculated intensity of the 001 reflection of tochilinite decreases with an increase of Fe^{2+} in the octahedral sites. The intensity of the 001 reflection in the observed X-ray pattern (Fig. 4) is calculated to be ~1% relative to that of the 002 reflection, suggesting that Fe^{2+} occupies more than 90% of octahedral sites in PCP tochilinite. Such high Fe^{2+} occupancy has never been reported from terrestrial tochilinite

Fig. 3. Structure of tochilinite from Mackinnon and Zolensky (1984). Tochilinite consists of a stack of alternating brucite and mackinawite layers with 5.4×2 Å interlayer spacings along c-axis.







001 15 20 Crκα 30

Fig. 4. Observed and calculated X-ray patterns of tochilinite in the range of 11–31 degrees (CrKα). The observed X-ray pattern is taken from type-I PCP and shows very weak intensity of 001 reflection relative to 002 reflection. The calculated X-ray patterns are from two end-members of tochilinite; Mg=100% and Fe=100% in the octahedral sites. The X-ray pattern of Fe = 100% is almost identical to that of type-I PCP.

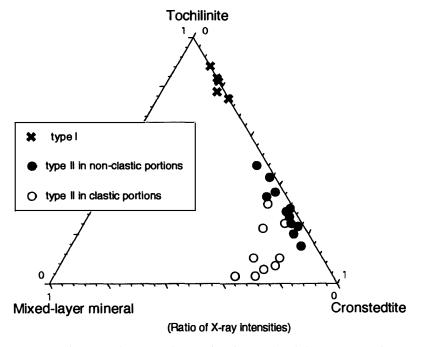


Fig. 5. A ternary diagram showing relative abundance of tochilinite, cronstedtite, and the mixed-layer mineral in PCP in Murchison. The relative abundance is expressed as ratios of X-ray intensities of the three minerals and the following reflections are used for the calculation of intensity ratios: 002 reflection of tochilinite, 001 of cronstedtite, and 003 of mixed-layer meneral. The diagram indicates that type-I PCP are rich in tochilinite and type-II PCP are rich in cronstedtite. Among type-II PCP, the mixed-layer mineral is significantly more abundant in PCP in the clastic portion.

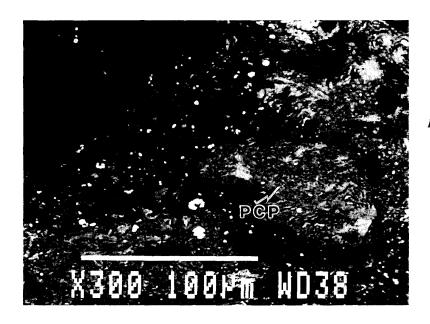


Fig. 6. A BSE image showing fine texture of type-II PCP particles that are composed of intimate intergrowth of fibrous minerals. Bright fibers are probably rich in tochilinite and dark fibers are cronstedtite. This fibrous PCP is commonly observed in non-clastic portions, but scarcely observed in clastic portions.

(cf., Organova et al., 1973; Zolensky and Mackinnon, 1986) and indicates that the physico-chemical conditions of formation of PCP tochilinite may be different from those

of terrestrial tochilinite (Zolensky et al., 1989). Relative abundances of minerals in type-I PCP estimated from X-ray intensities are summarized in Fig. 5.

3.3. Type-II PCP in non-clastic portion

Type-II PCP particles constitute most of the matrix in the non-clastic portion of the Murchison meteorite (Figs. 1a, 2b and 6). They have high Fe (\sim 55 wt% in FeO), medium Si (\sim 15 wt% in SiO₂) and S (\sim 10 wt% in S), and low Mg, Ca, Al, and Ni contents (Fig. 2b). The compositions are somewhat variable among type-II PCP and basically show a negative correlation of Si and Mg against Fe, S, and Ni contents.

X-ray powder patterns of type-II PCP in the non-clastic portion commonly show a very strong 7.1–7.2 Å, strong 3.6 Å, variable 6.0 Å, 5.4 Å, and 3.0 Å, and many other small refractions (Fig. 2b). In contrast to type-I PCP, the major mineral is cronstedtite and next is tochilinite. Magnetite and calcite are commonly detected. The 02/prism reflection appears at around 4.5 Å, suggesting that cronstedtite may have a disordered stacking sequence or cylindrical morphology (Devouard and Baronnet, 1995). The mean basal spacing, d (001), of cronstedtite in type-II PCP is 7.14 ± 0.03 Å. It is known that the basal spacing decreases from 7.3 Å for pure serpentine, in which all tetrahedral sites are occupied by Si⁴⁺, due to an increase of Fe³⁺ in tetrahedral sites. The 7.14 ± 0.03 Å basal spacing approximately corresponds to Si⁴⁺: Fe³⁺ =3:1 in the tetrahedral sites, analogous to Al³⁺ substitution in tetrahedral sites (Shirozu, 1958).

The 5.4 Å reflection of tochilinite shows peak broadening which could be due to the low crystallinity of tochilinite in type-II PCP (Fig. 2b). The 6.0 Å reflection may be 003 basal reflection of a tochilinite-cronstedtite mixed-layer mineral, and its intensity is consistently low in type-II PCP in the non-clastic portion. The identification of a mixed-layer mineral in the X-ray powder pattern is explained in detail in the following section. Relative abundances of the minerals in type-II PCP in non-clastic portion are summarized in Fig. 5.

3.4. Type-II PCP in clastic portion

Type-II PCP particles in the clastic portion (Fig. 2c) have high Fe (~50 wt% in FeO) and Si (~20 wt% in SiO₂), medium S (~10 wt% in S) and Mg (~12wt% in MgO) contents, and variable but small amounts of Ni, Al,and Ca. The difference in composition between type-II PCP in the clastic portion and that in the non-clastic portion is not distinct, but the former tends to have higher Si and Mg contents.

X-ray diffraction patterns of type-II PCP in the clastic portion are quite different from those in the non-clastic portion (Fig. 2c); the 7.1–7.2 Å peak is very strong, the 6.0 Å peak is strong, and the 5.4 Å peak is very weak. The most major mineral is disordered cronstedtite, similar to the type-II PCP in the non-clastic portion. However, the next major mineral is a mixed-layer mineral of cronstedtite and tochilinite whose main reflection is observed at 6.0 Å (Fig. 2c). Presence of magnetite and calcite is often recognized. Relative abundances of these minerals are shown in Fig. 5.

A mixed-layer mineral has been found in Murchison as an unknown layered mineral having serpentine-type (S) and brucite-type (B) structual repeats, "SBB-type", by TEM studies (Mackinnon and Buseck, 1979; Akai, 1980; Mackinnon, 1982) and previ-

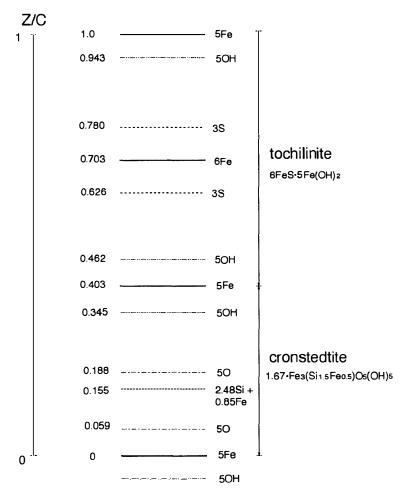


Fig. 7. One dimensional structure model of the mixed-layer mineral of tochilinite and cronstedtite. In this model, all cation sites of tochilinite are assumed to be occupied by Fe²⁺ and 25% of tetrahedral sites in cronstedtite are occupied by Fe³⁺.

Table 1. Integrated intensities of basal reflections from the mixed-layer mineral.

	001	002	003	004	005	006	007	008
Observed*	7	n.o.	100	19	6	n.o.	n.o.	n.o.
Calculated*	7	3	100	31	8	3	3	0

^{*} Type-II PCP in the clastic portion.

ous studies reveal that it has ~18 Å interlayer spacing. Mackinnon and Zolensky (1984) proposed that it may be a regularly interstratified mineral of cronstedtite and tochilinite. However, since then, no work has been made to confirm the identification of this mixed-layer mineral. This is partly because of the difficulty in obtaining precise diffraction data by TEM-SAED method (e.g., Mackinnon, 1982). In this study using the Gandolfi

^{* 1:1} regular interstratification of tochilinite and cronstedtite (structure model in Fig. 7).

method, the basal reflections of the mixed-layer mineral can be observed at 18.0 Å, 6.0 Å, 4.5 Å (Fig. 2c) and 3.6 Å and integrated intensities of each reflection are determined (Table 1) by means of profile-fitting technique using a spilit-type pseudo-Voigt function (Young and Wiles, 1982). We also calculated the intensities of the basal reflections, I_{00i} , using a one-dimensional structural model of the mixed-layer mineral (Fig. 7). The following equations are used for calculation:

$$I_{00l} = Lp |F_{00l}|^2,$$

 $F_{00l} = \sum_{n=1}^{N} f_n \exp(2\pi i \cdot l z_n),$

where Lp is the Lorlentz polarization factor, F_{00l} is a structural factor, N is the number of atoms in a unit cell, f_n is an atomic scattering factor, and z_n is an atomic parameter. Table 1 shows the results of the calculations. It shows a good agreement between the observed intensity distribution and the calculated one, demonstrating that the mixed-layer mineral in PCP is a 1:1 regular interstratification of cronstedtite and tochilinite.

4. Discussion

Our X-ray experiments reveal that PCP in the Murchison CM chondrite are mainly composed of five minerals; cronstedtite, tochilinite, the mixed-layer mineral, magnetite, and calcite. This identification generally confirms the TEM results of previous works (BARBER 1981; TOMEOKA and BUSECK, 1983, 1985; BARBER et al., 1983; MACKINNON and ZOLENSKY, 1984; ZOLENSKY et al., 1993). Our experiments, however, show that the relative abundances of minerals are considerably different among the PCP. Figure 5 shows the relative abundances of cronstedtite, tochilinite, and the mixed-layer mineral in all PCP analyzed in this study. A clearer distinction is observed in the abundance of cronstedtite between type-I and -II PCP, being much more abundant in type-II PCP. If type-I and -II PCP particles have a common precursor and type-II PCP particles are an alteration product of type-I PCP, PCP particles that have an intermediate abundance of cronstedtite between type-I and -II PCP are expected to occur, but such a PCP particle has not been found among the PCP studied so far (Fig. 5). This implies a difference in origin between type-I and -II PCP. In this regard, it has been reported that different precursor minerals have altered to different types of PCP, kamacite to type-I PCP (Fuchs et al., 1973; TOMEOKA and BUSECK, 1985) and melilite and troilite to type-II PCP (LEE and Greenwood, 1994; Degawa et al., 1995). These precursor materials may remain unaltered and coexist with PCP in some occasions, although our X-ray results show that most of the PCP particles are dominated by hydrous minerals and do not contain the precursor minerals suggested by the above authors, indicating that the precursors of PCP must have been completely replaced in Murchison, which is known to be one of the moderately altered CM chondrites (e.g., McSween, 1987).

Our study reveals that the mixed-layer mineral, which is a regular intergrowth of tochilinite and cronstedtite, constitutes a variable fraction of type-II PCP. It is clearly observed that the amount of the mixed-layer mineral in type-II PCP is closely related to the lithology where the PCP particles are located (Fig. 5); type-II PCP in the clastic

portion are usually enriched in the mixed-layered mineral, while type-II PCP in the nonclastic portion constantly show a low abundance of the mixed-layer mineral. Thus, it can be deduced that conditions of aqueous alteration might have been different between the clastic and non-clastic portions. Under the former condition, the mixed-layer mineral seems to be stable, whereas under the latter condition, separate phases of tochilinite and cronstedtite seem to be stable. In Murchison, clastic and non-clastic portions are intimately mixed even in a thin slice of approximately 1 cm². We can not see a good reason why the alteration condition differs within such a small area. Thus, it must be concluded that Murchison might not have suffered from aqueous alteration in the present structural form, and would rather be a mixing product of multiple lithic phases with different alteration histories. This conclusion is consistent with the model proposed by METZLER et al. (1992) that CM chondrites are mainly composed of two different lithologies, primary accretionary rocks and clastic matrix; these two lithologies probably correspond to the non-clastic and clastic portions described in this study.

TEM observations by Tomeoka and Buseck (1985) and Zolensky et al. (1993) showed that minerals in type-II PCP were altered and replaced by newly formed minerals in the course of aqueous alteration; the mixed-layer mineral appears at the expense of tochilinite in the altered type-II PCP. This reaction is also observed in our X-ray experiments. The abundance of the mixed-layer mineral increases with a decrease of tochilinite, while cronstedtite shows a relatively constant abundance (Fig. 5). This X-ray result indicates that the mixed-layer mineral is mainly produced by replacing tochilinite, assuming that the wide range of variations in mineral abundances in type-II PCP are all ascribed to the degree of alteration. All the previous observations and our results suggest that advanced aqueous alteration of type-II PCP produces the mixed-layer mineral at the expense of tochilinite, and the abundance of the mixed-layer mineral could be a indicator of the degree of aqueous alteration. Therefore, enrichment of the mixed-layered mineral in the PCP in the clastic portion indicates that aqueous alteration might have progressed further in the clastic portion.

Electron-microscope observations show that the clastic portion appears to have suffered from mechanical reworking, *i.e.*, mixing and fragmentation probably due to impacts on the meteorite parent body. The mixed-layer mineral can not be produced only by these mechanical effects. The activity of an aqueous solution which contains various molecules and ions is necessary to promote the reaction from the mono-layer tochilinite to the mixed-layer mineral. Post-shock residual heating might have enhanced the activity of an aqueous solution. Thus, high abundance of the mixed-layer mineral in the clastic portion would be evidence demonstrating that impact-induced cataclastic effects have assisted to advance aqueous alteration in the parent body. The recent study of Brearley (1995) also shows that the fragmentation and mixing of anhydrous minerals and subsequent aqueous alteration can cause unusual mineralogy to develop, in this case the enrichment of saponite in the Bells CM chondrite.

5. Conclusion

X-ray examination of PCP particles in the Murchison CM carbonaceous chondrite using a Gandolfi camera reveals that tochilinite, cronstedtite, a mixed-layer mineral of

tochilinite and cronstedtite, magnetite, and calcite are main constituent minerals of PCP and their relative abundances vary between type-I and -II PCP and among type-II PCP. The enrichment of the mixed-layer mineral in type-II PCP in the clastic portion of the meteorite indicates that the clastic portion has experienced more advanced aqueous alteration relative to the non-clastic portion. This may be evidence that cataclastic effects, like mixing and fragmentation, have promoted aqueous alteration in the meteorite parent body.

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