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Tuite, γ-$Ca_3(PO_4)_2$: a new mineral from the Suizhou L6 chondrite

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Abstract: Tuite, γ-$Ca_3(PO_4)_2$, the high-pressure polymorph of whitlockite, was found in a shock melt vein of the Suizhou chondrite. It occurs as polycrystalline aggregates in association with coarse-grained aggregates of ringwoodite, majorite and $NaAlSi_3O_8$-hollandite. These high-pressure mineral assemblages are enclosed in the fine-grained matrix of the vein that consists of majorite-pyrope$_{ss}$ garnet, metal and troilite. This new mineral is trigonal, $R\bar{3}m$, a = 5.258 Å, c = 18.727 Å. The tuite is colourless and transparent with vitreous lustre and white streak; it is uniaxial (+) with high refractive indices (ε = 1.706, ω = 1.701) and low birefringence (0.005). Microprobe analyses of tuite yield an empirical formula $(Ca_{2.51}Mg_{0.29})_{2.80}Na_{0.28}(P_{1.01}O_4)_2$. The simplified formula, $Ca_3(PO_4)_2$, is the same as whitlockite outside the veins. Stronger reflections on synchrotron radiation X-ray powder diffraction pattern of tuite are d = 2.628 (100), 2.891 (80), 1.945 (47), 1.730 (25) and 1.567(22). The figures in brackets are relative intensities. Tuite in the shock vein of this meteorite was transformed from whitlockite *via* a shock-produced solid-state reaction, while the pressure and temperature in the shock vein attained 23 GPa and 2000°C. This new mineral and the mineral name (tuite) have been approved by the Commission on New Minerals and Mineral Names of the International Mineralogical Association.

Key-words: Suizhou meteorite, high-pressure polymorph, whitlockite, new mineral, tuite.

Introduction

The anhydrous calcium phosphate, $Ca_3(PO_4)_2$, is found in many terrestrial and extraterrestrial materials including meteorites, lunar rocks and eucrites (Griffin *et al.*, 1972; Buchwald, 1984; Delaney *et al.*, 1984; Lundberg *et al.*, 1988; Heide & Wlotzka, 1995). However, there is some controversy over the mineral names of this anhydrous phosphate, in which whitlockite or merrillite was used in different studies (Mason, 1971; Calvo & Gopal, 1975; Nickel & Nichols, 1991; Rubin, 1997). In our earlier publications, we used the name whitlockite to describe this mineral in the Suizhou meteorite (Xie *et al.*, 2001a, 2001b), but the name merrillite was used in another paper according to the advice of a referee (Xie *et al.*, 2002). Recently, the chairman of the Commission on New Minerals and Mineral Names (CNMMN) of the International Mineralogical Association (IMA), Dr. J. Grice, emphasized that the name 'merrillite' was never approved by IMA and the name whitlockite should be used (pers. comm.).

It was pointed out that the compound $Ca_3(PO_4)_2$ has four polymorphs including α'-, α-, β-, and γ-phases, in which the β-phase is stable at ambient conditions, the γ-phase at high pressure, and the α'- and α-phases at high temperatures (Sugiyama & Tokonami, 1987). Whitlockite in meteorites has a structure essentially identical to that of synthetic β-$Ca_3(PO_4)_2$ (Gopal & Calvo,1972; Prewitt & Rothbard, 1975; Dowty, 1977). The γ-$Ca_3(PO_4)_2$ was first obtained experimentally as the product of decomposition of apatite by Murayama *et al.* (1986) at about 12 GPa and up to 2300°C. The structure of synthesized γ-$Ca_3(PO_4)_2$ was subsequently determined by Sugiyama & Tokonami (1987). Recently, we reported the first natural occurrence of a high-pressure polymorph of whitlockite in the shock melt vein of the Suizhou L6 chondrite, which has the same structure as γ-$Ca_3(PO_4)_2$ (Xie *et al.*, 2001c, 2002). The mineral name (tuite) of this new phase have been approved by CNMMN of IMA. The name tuite is after Professor Guangzhi Tu, the founding director of the Institute of Geochemistry, Chinese Academy of Sciences and first President of the Chinese Society of Mineralogy, Petrology and Geochemistry, in honour of his great contribution to geochemistry research and his pioneering work in studying Chinese meteorites. The type material is deposited in the Geological Museum of the Guangzhou Institute of Geochemistry, Chinese Academy of Sciences (CAS).

This paper describes the natural occurrence, physical and optical properties, chemical composition, crystallography and possible formation mechanism of this new high-pressure phase in the Suizhou meteorite.

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Table 1. Electron microprobe analyses of tuite and whitlockite (wt %).

	Tuite				Whitlockite
	1	2	3	average	
TiO_2	0.06	0.03	0.04	0.04	0.06
FeO	0.33	0.44	0.37	0.38	0.28
MgO	3.60	3.55	3.59	3.58	3.27
CaO	45.91	46.41	46.10	46.14	46.62
NiO	0.03	0.09	0.03	0.05	0.08
Na_2O	2.78	2.82	2.81	2.80	2.57
K_2O	0.05	0.09	0.07	0.07	0.03
Cr_2O_3	0.00	0.01	0.00	0.00	0.03
P_2O_5	47.14	47.19	47.15	47.16	47.67
Total	99.90	100.63	100.16	100.22	100.61

Formula of tuite: $Ca_{17.55}(Mg_{1.89}, Fe_{0.11})_{2.00}(Na_{1.93}, K_{0.03})_{1.96}(P_{1.01}O_4)_{14}$, or $(Ca_{2.51}\ Mg_{0.29})_{2.80}Na_{0.28}(P_{1.01}O_4)_2$. Formula of whitlockite: $Ca_{17.63}(Mg_{1.72}, Fe_{0.08})_{1.80}(Na_{1.76}, K_{0.02})_{1.78}(P_{1.02}O_4)_{14}$, or $(Ca_{2.52}\ Mg_{0.26})_{2.78}Na_{0.25}(P_{1.02}O_4)_2$. The calculation bases for both formulae are: O = 56 and O = 8, respectively.

Occurrence

Polycrystalline aggregate grains of tuite were identified in a shock-melt vein of the Suizhou L6 chondrite, which fell in 1986 in Dayanpo, 12.5 km southeast of the city of Suizhou, Hubei Province, China (35°18'N, 113°35'E) (Xie *et al.*, 2001b). This chondritic meteorite consists of olivine, low Ca-pyroxene, plagioclase that has been almost totally converted to maskelynite, FeNi metal, troilite, whitlockite, chlor-apatite, chromite and ilmenite (Xie *et al.*, 2001a, 2001b). It is moderately shocked to shock stage S3-S4 (Stöffler *et al.*, 1991), and contains a few very thin shock-induced melt veins 0.02-0.09 mm in width.

The Suizhou meteorite contains about 2 % by volume of whitlockite in the chondritic portions (Xie *et al.*, 2002). It occurs as single grains of irregular shape with maximum dimension of 100 µm. The grains of whitlockite are heavily fractured, but their structure is undisturbed. The results of microprobe analyses show the empirical formula of whitlockite to be $Ca_{17.63}(Mg_{1.72},Fe_{0.08})_{1.80}(Na_{1.76},K_{0.02})_{1.78}(P_{1.02}O_4)_{14}$ or $(Ca_{2.52}Mg_{0.26})_{2.78}Na_{0.25}(P_{1.02}O_4)_2$ (Table 1). The composition of whitlockite in Suizhou is similar to that of whitlockite in many other ordinary chondrites and some achondrites (Dowty, 1977; Chen *et al.*, 1995). The features of Raman spectra of whitlockite in the Suizhou meteorite are identical to those in the Sixiangkou L6 chondrite (Chen *et al.*, 1995).

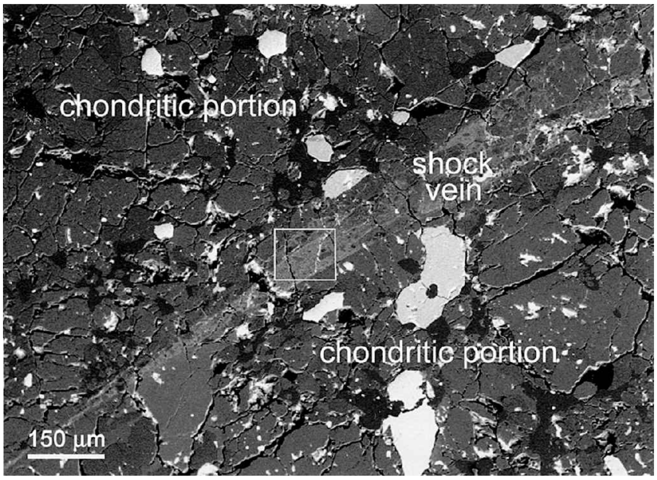

Fig. 1. Back-scattered electron (BSE) image of a shock melt vein in the Suizhou meteorite. Note a shock melt vein cutting the chondritic portion.

The shock veins in the Suizhou meteorite contain abundant high-pressure minerals (Fig. 1). Two distinct high-pressure mineral assemblages have been described (Xie *et al.*, 2001a, 2001b): (1) coarse-grained assemblage consisting of ringwoodite, low-calcium majorite, $NaAlSi_3O_8$-hollandite produced by phase transformation of olivine, low-calcium pyroxene and plagioclase respectively at high *P-T* conditions, and (2) fine-grained assemblage (the black matrix of the veins) consisting of majorite-pyrope $_{SS}$ (SS, solid solution) crystallized at high pressures and temperatures from a shock-induced silicate melt plus liquidus FeNi-FeS eutectic intergrowths. Synchrotron radiation X-ray diffraction pattern of the fine-grained matrix in a Suizhou vein shows the main diffraction lines of three constituent minerals, *e.g.* majorite-pyrope garnet (d = 2.571, 2.88, 2.451 and 1.538 Å), FeNi metal (d = 2.026, 1.170 and 1.453 Å) and troilite (d = 2.091, 2.667, 2.99, 1.934 Å). Tuite, the high-pressure polymorph of whitlockite, occurs within a shock melt vein as part of the coarse-grained mineral assemblage and is surrounded by the fine-grained vein matrix minerals (Fig. 2).

Appearance and physical properties

The two tuite grains in the Suizhou shock vein show similar tabular morphology. One is 10 x 20 µm and the other is 6 x 4 µm in size (Fig. 2). The surface of both tuite grains is very smooth and neither type shows any fractures, cleavages or particular microstructure. Tuite shows a dark grey colour on the back-scattered electron image. The lustre of tuite is vitreous and its streak is white.

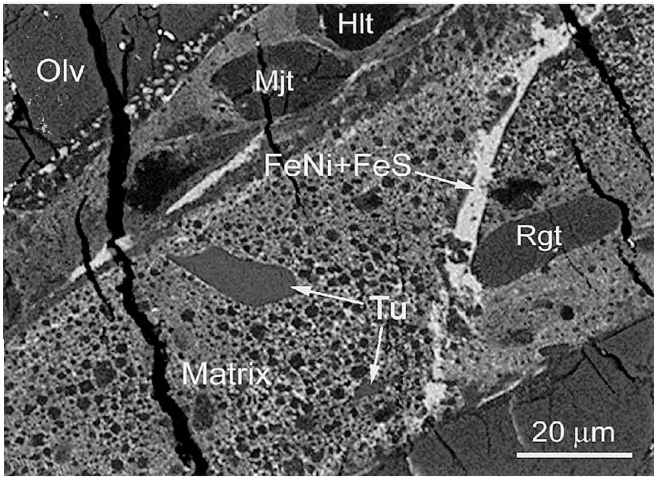

Fig. 2. Enlarged BSE image showing two tuite grains surrounded by fine-grained matrix and coexisted with other high-pressure minerals. Tu = Tuite, Rgt = Ringwoodite, Mjt = Majorite, Hlt = $NaAlSi_3O_8$-hollandite, Matrix = Fine-grained matrix consisting of majorite–pyrope garnet in solid solution plus FeNi+FeS intergrowth, FeNi+FeS = Metal+troilite veinlet.

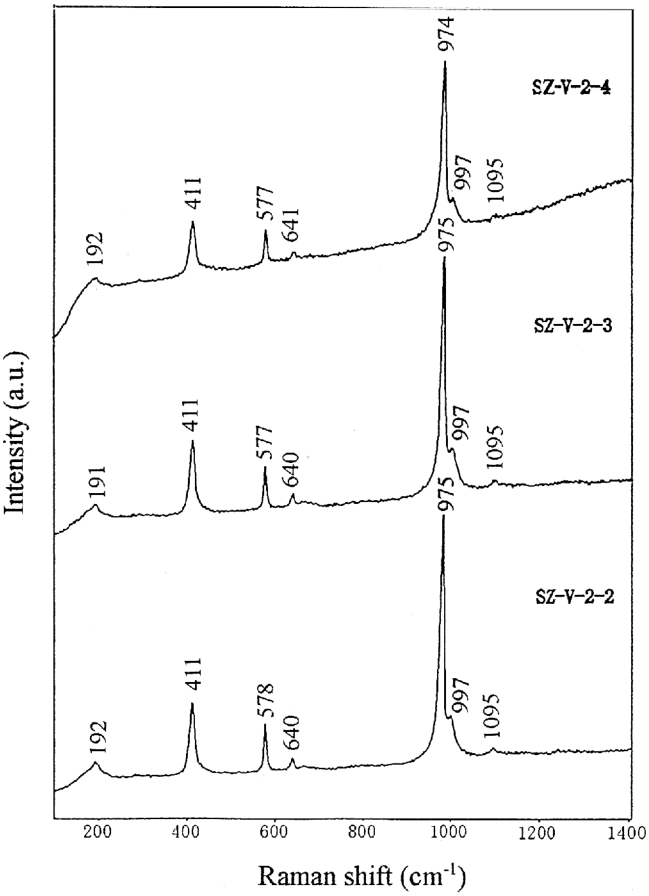

Fig. 3. Raman spectra of tuite in the Suizhou meteorite.

Optical properties

Tuite cannot be identified directly in thin sections of the Suizhou meteorite under the optical microscope in transmitted-light due to its polycrystalline nature and very small grain size. Furthermore, the presence of matrix minerals (FeNi-Metal, Fe-sulphide and garnet) underneath the tuite grains makes these tuite grains (about 5 µm in thickness) almost "opaque". Therefore, we synthesized the high-pressure γ-$Ca_3(PO_4)_2$ phase in the laboratory at 14 GPa and 1400°C using $Ca_3(PO_4)_2$ powder as starting material (the synthesis procedure was described in Xie *et al.*, 2002). The synthesized γ-$Ca_3(PO_4)_2$ crystals are colourless and transparent with grain sizes 60 to 130 µm. These crystals were used for determination of the optical properties of tuite. The refractive indices were measured using the immersion method.

The results of measurements on synthetic tuite crystals are as follows: uniaxial (+), $\varepsilon = 1.706(3)$ and $\omega = 1.701(4)$. Our results are in good agreement with the description of Murayama *et al.* (1986) for their synthetic γ-$Ca_3(PO_4)_2$ phase, which shows high refractive indices and low birefringence, and are markedly different from those of natural whitlockite from Palermo, New Hampshire, USA. This latter phase has a composition (CaO-46.90, MgO-2.53, P_2O_5-45.68 wt %) similar to that of tuite in Suizhou, but shows uniaxial (–) optical sign with refractive indices of $\omega = 1.629$ and $\varepsilon = 1.626$ (Frondel, 1941).

Chemical composition

The chemical composition of natural tuite was obtained with a Cameca SX-51 electron probe microanalyzer (EPMA) at 15 kV and 10 nA in the Institute of Geology and Geophysics, CAS. A tabular tuite grain (10 x 20 µm in size on a polished thin- section) was probed in three areas (Table 1). Results show that this mineral appears homogeneous with empirical formula $Ca_{17.55}(Mg_{1.89},Fe_{0.11})_{2.00}(Na_{1.93},K_{0.03})_{1.96}(P_{1.01}O_4)_{14}$ or $(Ca_{2.51}Mg_{0.29})_{2.80}Na_{0.28}(P_{1.01}O_4)_2$. The compositions of tuite are identical to whitlockite in the chondritic portions (Table 1), and there are no additional elements incorporated into this high-pressure phosphate phase from the surrounding matrix melt. The simplified formula for tuite may be given as $Ca_3(PO_4)_2$.

Raman and X-ray studies

The Raman spectra of tuite in a Suizhou vein are shown in Fig. 3. All three spectra are identical, showing one intense peak at 975 cm^{-1} with a shoulder at 997 cm^{-1}, two less intense peaks at 411 and 577 cm^{-1}, and three weak peaks at 192, 640 and 1095 cm^{-1}. The intense Raman peak at 975 cm^{-1} can be assigned to the $\nu1$ symmetric stretching vibration of the PO_4 group, while the peak at 1095 cm^{-1} corresponds to $\nu3$ asymmetric stretching vibration of PO_4. Peaks at 577 and 640 cm^{-1} are associated with $\nu4$ bending mode, and those at less than 410 cm^{-1} with the lattice modes. The Raman spectrum of tuite is different from that of whitlockite outside the veins in the Suizhou meteorite,

Table 2. X-ray powder diffraction data of synthetic γ-$Ca_3(PO_4)_2$ and tuite.

	Synthetic γ-$Ca_3(PO_4)_2$		Tuite	
hkl	$d_{meas.}$	I/I_0	$d_{meas.}$	I/I_0
1 0 1	4.4240	1.6	4.428	6.5
0 0 6	3.1175	4.0	3.118	5.7
1 0 5	2.8905	83	2.891	80.3
1 1 0	2.6288	100	2.628	100
1 1 3	2.4223	4.7	2.423	4.6
2 0 2	2.2120	24.3	2.214	20.3
1 0 8	2.0799	6.2	2.080	5.0
0 0 9	2.0783	10.5	2.078	12.2
2 0 4	2.0469	16.1	2.047	15.7
1 1 6	2.0097	8.2	2.009	8.0
2 0 5	1.9448	50.0	1.945	47.3
2 0 7	1.7324	3.0	1.734	4.3
0 1 10	1.7302	30.8	1.730	24.5
1 1 9	1.6303	3.1	1.627	5.9
1 2 5	1.5634	23.2	1.567	22.2
3 0 0	1.5177	18.2	1.518	18.6
2 0 10	1.4452	8.8	1.445	10.7

which has intense peaks at 956 and 972 cm^{-1}, less intense peaks at 408, 445 and 1080 cm^{-1}, and weak peaks at 178, 550, and 1026 cm^{-1} (Xie *et al.*, 2002). This suggests that a phase transformation from whitlockite to a new structure phase took place in the shock veins.

Murayama *et al.* (1986) pointed out that they examined the γ-$Ca_3(PO_4)_2$ phase by X-ray powder diffraction analysis. However, no diffraction data of this phase have been published in the literature. Therefore, we collected powder X-ray diffraction data for our synthetic tuite with a Rigaku PSPC-MDG2000 X-ray micro-diffractometer equipped with an imaging plate detector system. The recorded powder X-ray diffraction lines are listed in Table 2. The X-ray diffraction data reveal that the synthetic tuite has a trigonal unit cell with cell parameters of $a = 5.2576$ Å and $c = 18.7049$ Å, density (calc.) = 3.452 (g/cm^3) (Xie *et al.*, 2002). Our synthetic tuite phase is identical to the high-pressure γ-$Ca_3(PO_4)_2$ phase found by Maruyama *et al.* (1986) as the decomposition product of apatite at high pressures and temperatures. The structure refinements are also based on crystal structural model for the γ-$Ca_3(PO_4)_2$ phase proposed by Sugiyama & Tokonami (1987). The calculated *d*-values agree very well with the observed X-ray diffraction data (Xie *et al.*, 2002).

The powder X-ray diffraction data of natural tuite in the Suizhou meteorite were obtained by using synchrotron X-radiation with an energy-dispersive diffraction technique at the beam-line X17C of the National Synchrotron Light Source, Brookhaven National Laboratory. The operating voltage was 2.584 GeV, with a beam current of 100 mA. The X-ray beam was collimated to a size of 15 x 15 µm, and was focused onto a single tuite grain (10 x 20 µm) in a polished thin-section that was rotated systematically (ω = -30°–30°, χ = 0°–360°) to collect diffraction data. Energy dispersive X-ray diffraction data (EDXD) was acquired with an intrinsic germanium detector. The X-ray diffraction data were obtained at 2θ settings of 8° and 10°, respectively.

Although the size and thickness of the probed grain are very small and the influence of the fine-grained matrix minerals surrounding and underneath the tuite grain is very strong, we were able to obtain 17 diffraction peaks from the probed tuite grain (Table 2). The strongest X-ray diffraction lines are 2.628(100), 2.891(80), 1.945(47), 1.730(25) and 1.567(22). The figures in brackets are relative intensities. These data are in good consistence with the diffraction lines of our synthetic tuite. The trigonal unit cell parameters for tuite in the Suizhou meteorite are: $a = 5.258(1)$ Å and $c = 18.727(3)$ Å; space group $R\bar{3}m$; density (calc.) = 3.447 (g/cm^3); the figures in brackets are standard deviations in the last significant digits. The structure refinements are based on a crystal structural model for the γ-$Ca_3(PO_4)_2$ phase proposed by Sugiyama and Tokonami(1987).

Based on the Raman spectroscopic measurements and the X-ray diffraction data, we conclude that tuite has the same crystal structure as the synthesized high-pressure phase of $Ca_3(PO_4)_2$. Evidently, tuite is the first natural occurrence of a high-pressure polymorph of whitlockite.

P-T conditions

Shock-induced high pressures and temperatures led to the formation of many high-pressure minerals in the shock veins. The phase transition from plagioclase to the hollandite-structured polymorph constrains the pressure and temperature in the shock vein to 23 GPa and 2000°C (Liu, 1978; Yagi *et al.*, 1994; Gillett *et al.*, 2000; Xie *et al.*, 2001b). Such *P-T* conditions in the veins can also be obtained for the solid-state phase transformations from olivine to ringwoodite and from pyroxene to majorite (Chen *et al.*, 1996; Xie *et al.*, 2001b). Tuite in Suizhou formed through an isochemical soild-state phase transition of whitlockite, not through the decomposition of apatite as shown by the experiments of Murayama *et al.* (1986). Even though the phase diagrams in Murayama *et al.* (1986) indicate that the γ-$Ca_3(PO_4)_2$ might form at pressures as low as 12 GPa, it would still be present at 20 GPa or higher. The

P-T conditions suggested for Tuite are consistent with the P-T conditions (up to 23 GPa and 2000°C) indicated by the other high-pressure phases in Suizhou veins.

Geochemical significance

Tuite is a dense phase of whitlockite that could exist deep in the Earth. It has been demonstrated that the γ-$Ca_3(PO_4)_2$-structured phases, including tuite, have a 12-coordinated Ca(1) site and a 10-coordinated Ca(2) site with mean *M-O* distances of 2.74 Å and 2.59 Å, respectively (Sugiyama & Tokonami, 1987). These bonds are much longer than those of the 8-coordinated Ca site in garnet and diopside ($M\text{-}O \approx 2.40$ Å). Consequently, the γ-$Ca_3(PO_4)_2$-structured phases have the potential to accommodate very large lithophile elements, such as Sr and Ba, as well as rare earth elements, and persist as stable crystalline phases throughout the upper mantle (Beswick & Carmichael, 1978; Murayama *et al.*, 1986).

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