American Mineralogist, Volume 104, pages 775–778, 2019

LETTER

Discovery of asimowite, the Fe-analog of wadsleyite, in shock-melted silicate droplets of the Suizhou L6 and the Quebrada Chimborazo 001 CB3.0 chondrites

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ABSTRACT

We report the first natural occurrence and single-crystal X-ray diffraction study of the Fe-analog of wadsleyite [$a = 5.7485(4)$, $b = 11.5761(9)$, $c = 8.3630(7)$ Å, $V = 556.52(7)$ Å^3; space group *Imma*], spinelloid-structured Fe_2SiO_4, a missing phase among the predicted high-pressure polymorphs of ferroan olivine, with the composition $(Fe^{2+}_{1.10}Mg_{0.80}Cr^{3+}_{0.04}Mn^{2+}_{0.02}Ca_{0.02}Al_{0.02}Na_{0.01})_{\Sigma 2.01}(Si_{0.97}Al_{0.03})_{\Sigma 1.00}O_4$. The new mineral was approved by the International Mineralogical Association (No. 2018-102) and named asimowite in honor of Paul D. Asimow, the Eleanor and John R. McMillan Professor of Geology and Geochemistry at the California Institute of Technology. It was discovered in rare shock-melted silicate droplets embedded in Fe,Ni-metal in both the Suizhou L6 chondrite and the Quebrada Chimborazo (QC) 001 CB3.0 chondrite. Asimowite is rare, but the shock-melted silicate droplets are very frequent in both meteorites, and most of them contain Fe-rich wadsleyite (Fa_{30-45}). Although the existence of such Fe-rich wadsleyite in shock veins may be due to the kinetic reasons, new theoretical and experimental studies of the stability of $(Fe,Mg)_2SiO_4$ at high temperature (>1800 K) and pressure are clearly needed. This may also have a significant impact on the temperature and chemical estimates of the mantle's transition zone in Earth.

Keywords: Wadsleyite, iron, spinelloid, chondrite, meteorite, crystal structure, microprobe analysis, Earth's transition zone

INTRODUCTION

Most of the major rocky planet-forming materials [i.e., *M*-Si-O (*M* = Mg, Fe)] such as majorite (Tomioka et al. 2016), akimotoite-hemleyite (Tomioka and Fujino 1999; Bindi et al. 2017), wadsleyite (Price et al. 1983), ringwoodite-ahrensite (Binns et al. 1969; Ma et al. 2016; Bindi et al. 2018), and bridgmanite (Tschauner et al. 2014) have been discovered in extraterrestrial rocks that have experienced high-pressure and high-temperature collisions in outer space. Such rocks, known as shocked meteorites, represent a fascinating challenge for geoscientists because their mineralogical assemblages, and the processes they underwent, could give important indications on the mechanisms affecting planets and asteroids through the evolution of the solar system. Furthermore, the studies of shocked meteorites could give important hints for the mineralogy of Earth's deep interior, which are currently inferred from the investigation of mantle xenoliths (Collerson et al. 2000) and inclusions in diamonds (Moore and Gurney 1985; Walter et al. 2011), as well as from experimental studies of phase equilibria of silicates and oxides (e.g., Gasparik 2003; Irifune and Tsuchiya 2007).

Among the high-pressure *M*-Si-O phases, wadsleyite [the β-polymorph of $(Mg,Fe)_2SiO_4$] is considered the dominant phase in the upper portions of the transition zone (e.g., Irifune and Ringwood 1987). In addition, the transition of olivine into wadsleyite structure has been considered as the cause for the observed discontinuity in seismic wave velocities near 410 km depth (e.g., Bina and Wood 1987; Katsura and Ito 1989). Experimental works at conditions relevant for the Earth's transition zone (<1873 K) yield that the Fe content of wadsleyite seems limited to $Fa_{<35}$. Above this Fe-content, as long as no Fe^{3+} is involved, olivine directly transforms to ringwoodite. However, Finger et al. (1993) synthesized a single wadsleyite crystal with Fa_{40} at 15.2 GPa and 1973 K, which should lie within the ringwoodite stability field of the Mg_2SiO_4–Fe_2SiO_4 phase diagram (Fei and Bertka 1999). The Fe_2SiO_4 end-member requires much lower pressures to transform directly from fayalite to its high-pressure polymorph ahrensite (Ono et al. 2013). The FeO content of natural wadsleyite depends on the transformation mechanism (Sharp and de Carli 2006). Wadsleyite in ordinary chondrites, mostly coexisting with ringwoodite, shows values of Fa_{6-20} (e.g., Miyahara et al. 2008; Ono et al. 2013), whereas wadsleyite grains studied in barred olivine fragments in shock melted areas in the CB_a chondrite Gujba range

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0003-004X/19/0005–775$05.00/DOI: https://doi.org/10.2138/am-2019-6960

in composition from $Fa_{1.3}$ to $Fa_{3.7}$ (Weisberg and Kimura 2010).

Here we report the discovery of the first natural occurrence of the Fe-analog of wadsleyite, the third polymorph of Fe_2SiO_4 after fayalite and ahrensite. The new mineral was found in rare shock-melted silicate droplets embedded in Fe,Ni-metal in both the Suizhou L6 chondrite and the Quebrada Chimborazo (QC) 001 CB3.0 chondrite. It was named after Paul D. Asimow (b. 1969), the Eleanor and John R. McMillan Professor of Geology and Geochemistry at the California Institute of Technology, for his research in igneous and computational petrology and mineral physics and for exploring the behavior of materials under shock conditions. The new mineral and mineral name have been approved by the Commission on New Minerals, Nomenclature and Classification of the International Mineralogical Association (no. 2018-102). Holotype material (Suizhou) is deposited in the collections of the Museo di Storia Naturale, Università degli Studi di Firenze, via La Pira 4, I-50121, Firenze, Italy, catalog number 3238/I. Co-type material (Quebrada Chimborazo 001 CB meteorite) is preserved at the Goethe University Frankfurt, Germany.

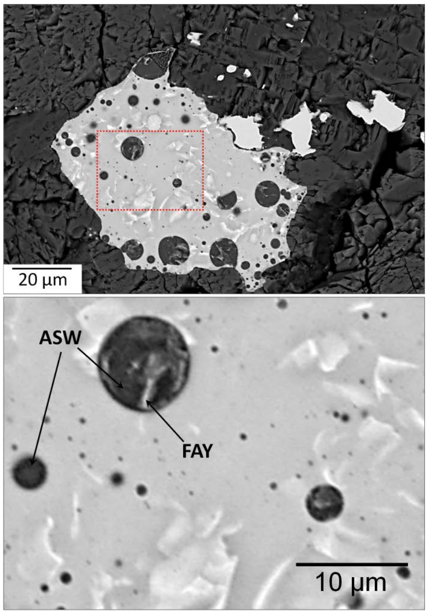


Figure 1. Top: SEM-BSE image of asimowite (in melt droplets inside the bright Fe,Ni-metal) in the Suizhou meteorite. Bottom: Enlarged region highlighted in the top image with a dashed red rectangle. ASW = asimowite, FAY = fayalite. The brightness variation in the Fe,Ni-metal are due to different Fe/Ni ratios. (Color online.)

OCCURRENCES OF ASIMOWITE IN SUIZHOU AND QC 001

Asimowite occurs in both meteorites as inclusions in very rare, shock-melted micrometer-sized droplets closely associated with olivine (Figs. 1 and 2a) and immersed in Fe,Ni-metal (likely taenite). Color, luster, streak, hardness, tenacity, cleavage, fracture, density, and optical properties could not be determined because of the small grain size. Although asimowite is certainly rare, the melt droplets of the two chondrites are very frequent, and most of them contain Fe-rich wadsleyite ($Fa_{35–45}$).

Suizhou is a shocked meteorite with occurrence of thin, less than 300 µm in thickness, shock-melt veins containing abundant high-pressure mineral polymorphs including ringwoodite, majorite, majorite-pyrope garnet, akimotoite, hemleyite, magnesiowüstite, lingunite, tuite, and xieite (Xie et al. 2011 and references therein; Chen and Xie 2015 and references therein; Bindi et al. 2017). In addition, Chen et al. (2004) reported $(Mg,Fe)SiO_3$ glass in the shock veins and suggested that it could be a vitrified perovskite.

Quebrada Chimborazo 001 represents a new member of the CB (Bencubbin-like) metal-rich carbonaceous chondrites. The CB chondrites are a rare group of meteorites having several unique characteristics: (1) high (60–80 vol%) metal abundances, (2) most chondrules have magnesium-rich compositions and non-porphyritic (cryptocrystalline or skeletal olivine) textures, (3) whole-rock chemical compositions are highly depleted in moderately volatile lithophile elements, and (4) bulk nitrogen isotopic composition is highly enriched in ^{15}N (e.g., Weisberg and Kimura 2010; Koch et al. 2016, 2017; Brenker et al. 2018). The CB chondrites contain a high abundance of metal-silicate shock melts. Silicate shock melts are enriched in FeO compared to the CB chondrules and often contain high-pressure phases (e.g., coesite, majorite, and wadsleyite).

Chemical composition

The chemical compositions of asimowite in both meteorites was first qualitatively analyzed with EDS. The analyses did not indicate the presence of elements ($Z > 9$) other than Fe, Mg, Si, and minor Cr, Na, Al, Mn, and Ca. Quantitative analyses ($n = 3$) were obtained only for asimowite from Suizhou, using the same crystal studied by X-ray single-crystal diffraction (see below). The instrument used was a JEOL-JXA 8200 microprobe (WDS mode, 15 kV, 10 nA, 1 µm beam size, counting times 20 s for peak and 10 s for background). For the WDS analyses, the $K\alpha$ lines for all the elements were used. The asimowite crystal was found chemically homogeneous within analytical uncertainties of our measurements (Table 1). The empirical formula (based on 4 oxygen atoms pfu, and assuming all Fe and Mn as divalent) is $(Fe^{2+}_{1.10}Mg_{0.80}Cr^{3+}_{0.04}Mn^{2+}_{0.02}Ca_{0.02}Al_{0.02}Na_{0.01})_{\Sigma 2.01}(Si_{0.97}Al_{0.03})_{\Sigma 1.00}O_4$. The composition of Fe-rich wadsleyite is in the range $Fa_{28–60}$ and $Fa_{30–56}$ in Suizhou and QC 001 chondrite, respectively.

EBSD (electron backscatter diffraction)

Electron backscatter diffraction techniques were applied to identify the crystal structure and study the lattice preferred orientation of the asimowite crystals within the HP/HT melt droplets of the QC 001 chondrite. The study was performed with a Zeiss Sigma VP FEG variable-pressure scanning electron microscope equipped with an HKL INCA Premium Synergy Integrated ED/EBSD system (Oxford Instruments, Oxfordshire, U.K.) and a modified Emitech K1250 cryostage at the University of Otago (New Zealand). The

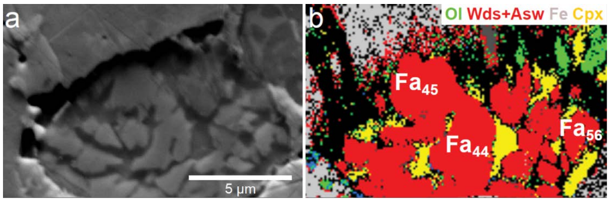


Figure 2. Electron backscatter diffraction mapping of the whole melt droplet containing asimowite from the Quebrada Chimborazo 001 CB meteorite. (**a**) Secondary electrons image; (**b**) EBSD mapping with color coding of the different phases identified via automatic indexing (Ol = olivine; Wds+Asw = wadsleyite+asimowite; Fe = Fe,Ni-metal; Cpx = clinopyroxene). The Fe_2SiO_4 component is reported on the Wds/Asw crystals as Fa contents. (Color online.)

instrument operated at 20 kV and 6 nA in a focused-beam mode with a 70° tilted stage and a variable pressure mode (25 Pa). The focused electron beam is several nanometers in diameter. The spatial resolution for diffracted backscatter electrons was ~30 nm. The EBSD system was calibrated using a single-crystal silicon standard. In Figure 2b, we report the EBSD map of a melt droplet shown in Figure 2a; color coding corresponds to different phases identified via automatic indexing.

X-ray single-crystal diffraction

A small asimowite fragment (12 × 9 × 6 μm in size) from the Suizhou meteorite was extracted from the polished section under a reflected light microscope and mounted on a 5 μm diameter carbon fiber, which was, in turn, attached to a glass rod. The fragment consists of crystalline asimowite [unit-cell values: $a = 5.7485(4)$, $b = 11.5761(9)$, $c = 8.3630(7)$ Å, $V = 556.52(7)$ Å^3, and $Z = 8$; space group *Imma*] associated to minor, fine-grained polycrystalline fayalitic olivine. Single-crystal X-ray diffraction intensity data of asimowite were collected with a Bruker D8 Venture Photon 100 CMOS equipped with graphite-monochromatized Mo$K\alpha$ radiation. The detector-to-crystal distance was 70 mm. Data were collected using ω and φ scan modes, in 0.5° slices, with an exposure time of 60 s per frame. The data were corrected for Lorentz and polarization factors and absorption using the software package APEX3 (Bruker AXS Inc. 2016). A total of 1932 unique reflections was collected. Given the similarity in unit-cell values and space groups, the structure was refined starting from the atomic coordinates reported for the *Imma* crystal structure of Fe-rich wadsleyite (Hazen et al. 2000) using the program SHELXL-97 (Sheldrick 2008). The site occupancy factor (s.o.f.) at the cation sites was allowed to vary (Fe vs. Mg for the three octahedral sites and Si vs. structural vacancy for the tetrahedral site) using scattering curves for neutral atoms taken from the *International Tables for Crystallography* (Wilson 1992). At the last stage, with anisotropic displacement parameters for all the atoms, the structure was refined to $R1 = 0.0295$ using 1932 independent reflections. As the use of neutral-atom scattering factors in refinements of Mg-Fe silicates could lead to systematic errors in refined site occupancies (Angel and Nestola 2016), a new structure refinement was carried out using ionized scattering factors. The results were nearly identical to those obtained using curves for neutral atoms, both in terms of site occupancies and bond distances. The tetrahedral site showed a mean electron number 14.0 and was thought to be fully occupied by silicon. The mean electron numbers at the three octahedral M sites were the following: 20.1 ($M1$ site), 17.5 ($M2$ site), and 21.2 ($M3$ site), corresponding to $Fe_{0.58}Mg_{0.42}$, $Mg_{0.61}Fe_{0.39}$, and $Fe_{0.66}Mg_{0.34}$, respectively. Altogether, taking into account the different multiplicity of the three octahedral sites, the refined X-ray formula can be written as $(Fe^{2+}_{1.14}Mg_{0.86})SiO_4$. Such a formula is in excellent agreement with that obtained from electron microprobe analysis.

TABLE 1. Analytical data (in wt%) for asimowite

	Mean	Range	S.D.	Probe standard
SiO_2	33.23	32.88–33.69	0.65	forsterite
Al_2O_3	1.27	1.01–1.54	0.14	albite
Cr_2O_3	1.53	0.98–1.69	0.15	chromite
FeO	44.87	43.12–46.02	0.71	fayalite
MgO	18.21	17.88–18.63	0.24	forsterite
CaO	0.56	0.29–0.80	0.08	plagioclase
MnO	0.80	0.52–1.03	0.11	bustamite
Na_2O	0.24	0.19–0.39	0.07	albite
Total	100.71	98.93–101.10		

Selected bond distances are shown in Table 2. The list of the observed and calculated structure factors and the CIF are deposited[1].

RESULTS AND DISCUSSION

Crystal-chemical remarks

The crystal structure of asimowite shown in Figure 3 is based on close-packing of cation polyhedra and features extensive edge-sharing of divalent cation octahedra. This topology provides little opportunity for bending of cation-oxygen-cation angles. The unit-cell parameters of asimowite are strongly influenced by the entry of Fe into the structure. We observed a general expansion of the unit cell from pure Mg_2SiO_4 through $(Mg_{1.50}Fe^{2+}_{0.50})SiO_4$ (Hazen et al. 2000). The assignment of Fe^{2+} substituting for Mg at the octahedral sites is required both to account for the electron density at the sites and to justify the increase of the mean bond distances relative to pure Mg_2SiO_4 and $(Mg_{1.50}Fe^{2+}_{0.50})SiO_4$ (Hazen et al. 2000).

The mean bond distances at the three octahedral sites (i.e., $M1$-O = 2.104 Å; $M2$-O = 2.103 Å; $M3$-O = 2.123 Å) are in excellent agreement with those that can be extrapolated from the data by Hazen et al. (2000) for Fe^{2+}-bearing wadsleyite. Furthermore, we confirmed the tendency for Fe to occupy $M1$ and $M3$ preferentially over $M2$ (Hazen et al. 2000; Woodland et al. 2012). These crystal-chemical considerations, together with the perfect charge balance of the formula, point to the presence of Fe (and Mn) and Cr in the divalent and trivalent states, respectively.

Although the chemical composition of asimowite is far from the ideal Fe_2SiO_4 end-member, Fe is the dominant cation at the octahedral sites of the structure. Furthermore, we always found Fe > Mg in all the microprobe analyses carried out on the crystal used for the structural investigation. Given the mean electron numbers refined at the three octahedral sites, one should write the ideal crystal-chemical formula for asimowite as $Fe_3MgSi_2O_8$ with $Z = 4$. However, we prefer to keep the simplified formula Fe_2SiO_4, to immediately indicate the solid solution with wadsleyite.

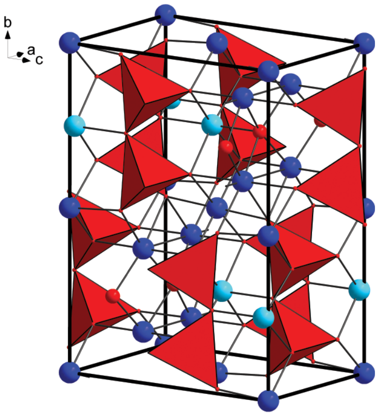
Origin

Shock conditions estimated for the Suizhou meteorite are in the P range 18–20 GPa and T range 1973–2273 K (Chen and Xie 2015). The shock conditions of CB chondrites are estimated as >19 GPa and >2273 K (Weisberg and Kimura 2010; Koch et al. 2016). Experimental data show that with increasing temperature the two-phase field of olivine and wadsleyite shifts toward more fayalitic compositions (Fei and Bertka 1999). On the basis of the coexistence of olivine and wadsleyite (Fig. 2) and according to the P-T diagram of the Allende chondrite (Agee et al. 1995), we suggest that asimowite crystallized from a Fe-enriched melt produced by a mixture of

TABLE 2. Selected bond distances (Å) for asimowite

$M1$-O4	2.0815(6)	(×4)
$M1$-O3	2.1477(9)	(×2)
mean	2.104	
$M2$-O1	2.011(2)	
$M2$-O4	2.1117(6)	(×4)
$M2$-O2	2.162(2)	
mean	2.103	
$M3$-O1	2.0690(3)	(×2)
$M3$-O3	2.1415(6)	(×2)
$M3$-O4	2.1596(6)	(×2)
mean	2.123	
Si-O3	1.6308(9)	
Si-O4	1.6447(6)	(×2)
Si-O2	1.7041(8)	
mean	1.656	

FIGURE 3. The crystal structure of asimowite. Si-tetrahedra are depicted in red. Fe-dominant $M1$ and $M3$ and Mg-dominant $M2$ cations are drawn as deep and light blue spheres, respectively. The unit cell and the orientation of the figure are outlined. (Color online.)



partial molten Fe,Ni-metal and silicate at very high temperature > 2000 K and pressure ≥15 GPa.

IMPLICATIONS

The high amount of Fe-rich ferropericlase inclusions found in diamonds of potential super-deep origin questions the bulk chemical model of the Earth (Kaminsky 2012). Although this might be due to a biased sampling of the lower mantle, we think it is worth further addressing this discrepancy. Although the discovery of asimowite in shock veins may be due to kinetic reasons as a metastable crystallization due to rapid cooling during decompression from the peak shock pressures (18–20 GPa for the Suizhou chondrite; >19 GPa for CB chondrites), new theoretical and experimental studies of the stability of $(Fe,Mg)_2SiO_4$ at high temperature (>1800 K) are clearly needed. This may also have a significant impact on the temperature and chemical estimates of the mantle's transition zone in Earth.

ACKNOWLEDGMENTS

The manuscript took advantage from the revision of Ross Angel, Chi Ma, Naotaka Tomioka, Ian Swainson, and one anonymous reviewer. The research was supported by "progetto di Ateneo 2016, University of Firenze" to L.B.

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MANUSCRIPT RECEIVED JANUARY 15, 2019
MANUSCRIPT ACCEPTED FEBRUARY 13, 2019
MANUSCRIPT HANDLED BY IAN SWAINSON

Endnote:

[1] Deposit item AM-19-56960, CIF. Deposit items are free to all readers and found on the MSA website, via the specific issue's Table of Contents (go to http://www.minsocam.org/MSA/AmMin/TOC/2019/May2019_data/May2019_data.html).