

LETTERS

Rock-forming moissanite (natural α -silicon carbide)

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ABSTRACT

We report the first occurrence of moissanite (SiC) as a rock-forming mineral (8.4 vol%) in one unique specimen of a terrestrial rock. The sample has a homogeneous, porphyritic texture, and was found as a beach pebble thought to be derived from a Tertiary volcanic province of the Aegean Sea region. The matrix is bluish-colored and consists of very fine-grained brucite, calcite, and magnesite, in which macrocrysts of quartz (25.3 vol%) and moissanite are found. Other accessory phases are phlogopite-3*T*, magnesiochromite, an Fe-rich phase, Cl-bearing brucite, Al-rich orthopyroxene, and unidentified MgFe-silicates (4 vol%). The bulk-rock composition shows a “kimberlitic” chemistry (55.8 wt% SiO₂, 28.5 wt% MgO, 1.4 wt% CaO, 18.1 wt% LOI). Colorless gemmy, and blue or black moissanite crystals are subhedral and display characteristic hexagonal symmetry (6*H* polytype). Most moissanite grains contain metallic Si and Fe-silicide (Fe₃Si₇) inclusions, and more rarely, other Fe-silicides with varying amounts of Al (≤ 24.5 wt%), Ca (≤ 8.0 wt%), Mn (≤ 6.8 wt%), Ti (≤ 16.3 wt%), and Ni (≤ 2.6 wt%). The $\delta^{13}\text{C}$ value of the moissanite is -28.1% . According to available data, the f_{O_2} stability field of SiC is five to six log units below the iron-wüstite (IW) buffer curve. Therefore, the observed Fe-bearing silicates cannot have been equilibrated with SiC under ambient pressure. Instead, our finding indicates that the rock most likely formed at the ultrahigh-pressure conditions of the upper mantle or transition zone.

INTRODUCTION

The terrestrial origin of silicon carbide (SiC = moissanite) samples has been the subject of controversial debates during the last century. Since the discovery of SiC crystals as inclusions in natural diamonds in kimberlites (Moore et al. 1986; Moore and Gurney 1989; Otter and Gurney 1989; Leung 1990) and in lamproites (Jaques et al. 1989), however, the natural occurrence of moissanite in terrestrial rocks has been widely accepted.

The first discovery of naturally formed SiC dates back to 1904, when Moissan reported its occurrence from the Canyon Diablo Fe meteorite. However, Moissan's finding was thought to be an artifact (Mason 1967) from SiC-bearing cutting tools used to prepare the meteorite samples. Since then, reports of new occurrences of natural SiC (e.g., Regis and Sand 1958; Bobrievich et al. 1957; Bauer et al. 1963; Marshintsev et al. 1967; Kaminskiy et al. 1969; Moskvitin et al. 1978; He 1984; Jaques et al. 1986; Marshintsev 1990; Filippidis 1993) have been debated vigorously and many geologists considered a natural terrestrial origin as highly improbable (e.g., Milton and Vitaliano 1984; Woermann and Rosenhauer 1985, p. 316). The

issue of a possible contamination with synthetic SiC from abrasives could have been, instead, ruled out in more recent reports of SiC found in meteorites, as measured isotopic anomalies in many trapped elements could definitely be identified as presolar in origin (e.g., Bernatowicz et al. 1987; Tang et al. 1989; Lewis et al. 1990; Daulton et al. 2002).

Typically, moissanite has been found as a constituent of kimberlitic pipes or in associated volcanic rocks. Recently, well-ordered α -SiC and β -SiC crystals were found as inclusions within diamonds (Leung 1990) and dispersed in the matrix of kimberlites from Fuxian, China (Leung et al. 1990). Leung et al. (1996) discussed the heteroepitaxial intergrowth between β -SiC and diamond along the (110) plane as a possible mechanism for the genesis of natural diamonds. Mathez et al. (1995) investigated the chemical composition of metallic Si and Fe-silicide inclusions in moissanite grains extracted from kimberlites from Yakutia, Russia. They also presented C-isotope data of moissanite grains, which were clearly distinguishable from interstellar SiC (e.g., Stone et al. 1991; Zinner et al. 1989). According to Mathez et al. (1995, p. 781–782), chemical and mineralogical data obtained from the SiC, the inclusions, and the associated minerals “...leaves little doubt that SiC occurs naturally and is present in the Earth's mantle...” and “...establish that SiC...is a widespread, albeit rare, phase in diamond-bearing rocks.”

Moissanite, besides diamond, is a potential “window” into

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the redox conditions of the Earth's mantle. The current study characterizes one unique specimen of a newly discovered rock pebble containing 8.4 vol% SiC, thought to be associated with calc-alkaline volcanic rocks. Moissanite is, to our knowledge, reported for the first time in rock-forming quantities. In contrast to previous studies, where moissanite grains were obtained from heavy mineral concentrates or as inclusions in diamonds, the present occurrence allows the study of textural relationships between SiC and the other phases using conventional polished thin sections.

SAMPLE AND METHODS

One unique specimen was found at a beach along the Turkish coast of the Mediterranean Sea, around 150 km NW from Izmir, and is most likely derived from Tertiary volcanic rocks outcropping in the area. The source outcrop, however, has not yet been located. The sample displays an unusual bluish color (Fig. 1a). It is macroscopically homogeneous and texturally isotropic. It was collected by Mr. Salvatore Musacchia and given to the first author as a "curiosity."

The sample has been analyzed by optical microscopy, powder X-ray diffraction (XRD, Philips PW 1800, $\text{CuK}\alpha$ radiation, $2\text{--}65^\circ 2\theta$), X-ray fluorescence (XRF, Philips PW 2400 spectrometer), scanning electron microscopy with energy dispersive system (FEI SEM-EDS XL30 Sirion, operating conditions 20 kV), and a Multiphase Carbon Determinator (Leco RC 412). Single-crystal data were obtained on an ENRAF NONIUS CAD4 X-ray diffractometer, using a graphite monochromator and $\text{MoK}\alpha$ X-radiation at room temperature (293 K). Electron-microprobe analyses of silicates and oxides were obtained with a wavelength-dispersive system (Cameca SX-50: operated at 15 kV accelerating potential with a 20 nA beam current, counting times were 20 s on peak and background for major elements and up to 40 s for trace elements); a synthetic SiC standard was used for moissanite, and Si and Fe metal standards were used for metallic inclusions (10 nA beam current). Physical conditions of cathodoluminescence analyses were 25 kV and 90 mA. The C-isotope ratio of the SiC was determined by flash combustion using a Carlo Erba CNS elemental analyzer coupled in continuous flow to a Micromass Optima mass spectrometer. Carbon- and O-isotope ratios of the carbonate were determined by reaction at 90°C with 100% phosphoric acid on an automated carbonate device connected to a VG-PRISM mass spectrometer. Mineral modes were determined by point counting (2400 points). Moissanite crystals were separated from the sample by etching away the matrix in a 5% HCl solution. XRD powder, XRF, SEM and Leco analyses were performed at the University of Fribourg. XRD powder, XRF, SEM and Leco analyses were performed at the University of Fribourg. XRD powder, XRF, SEM, and Leco analyses were performed at the University of Fribourg. XRD single crystal, EMPA, and CL analyses were performed at the University of Bern. Isotopic analyses were performed at the ETH-Zürich.

RESULTS

The bulk-chemical composition obtained by XRF (Table 1) shows that SiO_2 , MgO, and CaO are the only oxides present in significant quantities, all the other analyzed major and trace elements show very low concentrations. The volatiles (H_2O and CO_2) are also present in significant quantities. The principal phases are moissanite (8.4 vol%) and undeformed, xenomorphic, mm-sized and inclusion-free quartz grains (25.3 vol%), dispersed in a brucite-dominated matrix (58.2 vol%) also containing calcite, magnesite, phlogopite-3T, Cl-bearing brucite, magnesiochromite, Al-rich orthopyroxene, two unidentified MgFe-silicates, and an unidentified Fe-rich phase. Quartz and moissanite are never observed in direct contact with each other. The MgFe-silicates are reddish and yellowish in color, and have compositions intermediate between olivine and orthopyroxene. Spherical structures or "globules," ranging in diameter from a few hundred micrometers to a millimeter, are dispersed throughout the groundmass. These globules have the same mineral assemblage as the bulk but a smaller grain size,

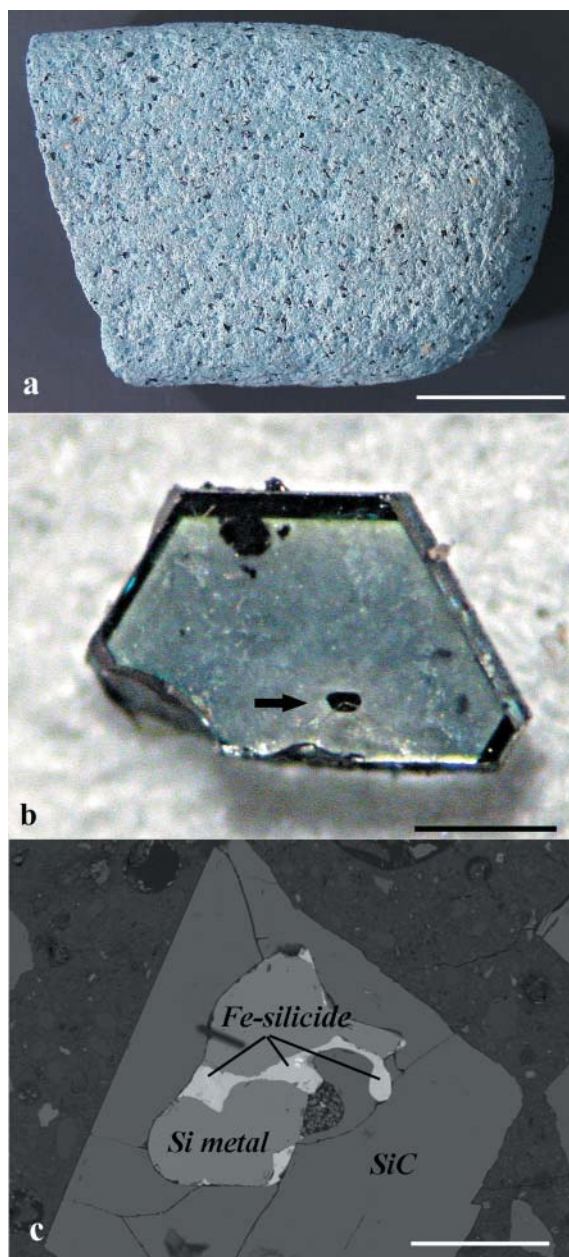


FIGURE 1. (a) Photo of the SiC-rich specimen. Black spots are moissanite crystals. Scale bar is 2 cm. (b) Gemmy, platy crystal of moissanite shown with largest (001) face. The arrow indicates a drop-shaped, black metallic inclusion. Scale bar is 300 μm . (c) Back-scattered electron image of metallic inclusion in moissanite. The gray shading in the BSE image consists mainly of pure metal Si (Table 2). The brighter areas in the middle and along the rims of the inclusion are composed of Fe-silicide. The very bright irregular white spots are likely inclusions of a third phase with higher average atomic number. Scale bar is 200 μm .

and account for 6.2 vol% of the bulk sample. Crystals of light-blue Cl-bearing brucite (1.9 vol%), a few micrometers in size, are homogeneously scattered throughout the matrix and are responsible for the bluish color of the pebble. All unidentified

phases are microcrystalline and subject of on-going studies.

Separated moissanite crystals range between 0.2 and 1.5 mm in size, are blue or black in color, and have a metallic luster. Some completely transparent gemmy crystals with brilliant subadamantine luster are present as well (Fig. 1b). In general, the crystals show well-developed crystallographic faces, but some are corroded or fractured. They have a characteristic platy or elongated, pinacoidal, hexagonal shape bounded by {100} faces (α -SiC). The unit-cell dimensions obtained with the single-crystal X-ray diffraction analysis on three grains are $a = 3.080(1)$ Å and $c = 15.12(1)$ Å (6H polytype). In a standard thin section (5×2 cm), at least 341 crystals of moissanite have been counted. Under transmitted light, moissanite ranges from colorless to light blue, or dark blue to almost black. Greenish-yellow or pinkish crystals also have been observed. Some moissanite grains are pleochroic, others are probably twinned. The optical character is always uniaxial positive. Moissanite grains are commonly associated closely with the red MgFe-silicates, which are commonly in contact with SiC crystal faces. A similar mineral paragenesis has been described by Mathez et al. (1995) for natural SiC crystals from Yakutia. The moissanite crystals are very homogeneous chemically, with none of the analyzed elements other than Si present above the detection limit (Table 2). A few moissanite grains show yellow, blue, and red cathodoluminescence. Yellow luminescence is confined to grain boundaries. Carbon-isotope data were obtained on moissanite and on carbonates. The moissanite has a $\delta^{13}\text{C}$ value of -28.1% relative to the Pee Dee belemnite standard (PDB), and the bulk carbonate yielded a $\delta^{13}\text{C}$ value of -11.9% and a $\delta^{18}\text{O}$ of -3.6% . Carbon isotope analyses have been performed also on a synthetic SiC sample and a value of -27.1% was obtained. The C-isotope signature, therefore, is not well suited to distinguish natural terrestrial SiC from synthetic SiC.

One third of the examined moissanite crystals contain metallic inclusions with rounded shapes. The average size of the inclusions is between 50 and 100 μm , with a maximum of 400 μm . Most of the inclusions occur inside the crystals. A few metallic grains have been found along moissanite crystal boundaries or dispersed in the brucitic matrix. Crystals with more than one inclusion are rare (max 10 inclusions). Pure Si is the most common inclusion. Fe-silicides are present along the Si metal-SiC boundaries (Fig. 1c), or form exsolution domains in metallic Si. Fe_3Si_7 is the most common Fe-silicide. This phase is stable at ambient pressure above 937 °C and decomposes to Si and FeSi_2 at lower temperatures (Kubaschewski 1982). Manganese and Ni substitute for Fe in various proportions in Fe_3Si_7 (Table 2). Other silicide-containing phases have variable stoichiometries and were classified as Si-Fe-Al-Ca and Si-Fe-Ti (possi-

TABLE 1. Bulk rock chemistry

	XRF	1 σ	d.l.		XRF	1 σ	d.l.		Leco
SiO ₂	55.84	0.25	0.01	Ba	b.d.	20	12	CO ₂ *	1.96
TiO ₂	0.02	0.01	0.01	Cr	31	5	5	H ₂ O*	16.19
Al ₂ O ₃	0.41	0.08	0.01	Cu	b.d.	4	4	H ₂ O ⁻	3.61
FeO	0.09	0.05	0.01	Nb	4	2	2		
MnO	0.01	0.01	0.01	Ni	35	3	3		
MgO	28.49	0.02	0.01	Pb	b.d.	4	7		
CaO	1.43	0.05	0.01	Rb	b.d.	3	3		
Na ₂ O	0.02	0.02	0.01	Sr	46	4	3		
K ₂ O	0.03	0.02	0.01	V	b.d.	6	5		
P ₂ O ₅	0.01	0.01	0.01	Y	51	3	3		
LOI	18.13			Zn	b.d.	4	3		
				Zr	13				
Total	104.48								

Note: The SiO₂ value includes metallic Si and SiC, which brings the total above 100 wt%. *At $T_{\text{max}} = 1000$ °C of the Leco instrument SiC does not break down. The listed CO₂ content is therefore considered as CO₂ from the carbonates only. Based on the modal mineralogy, the total CO₂ should be 8–9 wt% (SiC + carbonates), and approximately 1 wt% Cl should be present too. 1 σ = relative error; d.l. = detection limit; b.d. = below detection. Major and minor elements in wt%; trace elements in ppm; CO₂ and H₂O also in wt%.

bly alloys), and as $\text{Si}_2(\text{Fe},\text{Al},\text{Ca})_3$ and $\text{Si}_3(\text{Fe},\text{Al})_2$ (probably stoichiometric compounds) phases. Another metallic phase consists of Si_2Ca (tentatively stoichiometric compound) (Table 2).

ARTIFICIAL VS. NATURAL

An artificial origin of the beach pebble may be suspected considering the large quantity of SiC crystals and the presence of quartz in an ultramafic matrix. Moreover, the outcrop from which the specimen may have originated has not been located yet. There are, however, a number of indications that the material is natural. The beach where the pebble was collected is in an unpopulated region, around 40 km away from the closest village and 150 km away from the closest industrial town. A thorough investigation of the patent literature concerning SiC, as well as inquiries with different synthetic SiC producers (e.g., Timcal AG, Ticino, Switzerland) using the Acheson method (e.g., Knippenberg 1963) and their industrial customers (e.g., Smyris s.r.l. Milano, Italy), gave no indications that the pebble as a whole or the silicon carbide per se could have been a synthetic product.

In contrast, all analytical results are typical for natural moissanite.

(1) In synthetic SiC processed either with or without additives, C constitutes the major impurity phase and has a graphitic character (Backhaus-Ricoult et al. 1993, p. 2204). Other reported inclusions, all always nanometer-sized, are B_4C and B_{25}C , metallic Fe and Si, and FeSi and Ti_5Si_3 precipitates (e.g., More et al. 1986; Backhaus-Ricoult et al. 1993; Munro 1997). In the present sample, neither graphite nor any other form of C-bearing inclusions have been found.

(2) The metallic inclusions are characteristic, although not unequivocal (e.g., Lyakhovich 1980, p. 965), indicators of the natural origin of the moissanite crystals. Many silicide and alloy compositions have been observed previously in natural SiC: metallic Fe by Bauer et al. (1963); native Si and Fe by Leung (1990) and Leung et al. (1990); native Si and ferrosilicide by Marshintsev (1990); micrometer-sized metallic Si, ferrosilicide (Fe_3Si_7), and light rare earth element (LREE) rich Fe-Ti-Zr silicides (up to 16 wt% Ce and up to 4 wt% Th) by Mathez et al. (1995). Except for the LREE-rich metallic phases, all the above

TABLE 2. Electron microprobe analyses of moissanite and metallic inclusions

wt%	moissanite		1 σ	d.l.	silicon		iron silicide							
	SiC	SiC			Si	Fe ₃ Si ₇	Si-Fe-Al-Ca	Si ₂ (Fe,Al,Ca) ₃	Si ₂ (Fe,Al,Ca) ₃	Si ₃ (Fe,Al) ₂	Si-Fe-Ti	Si ₂ Ca	1 σ	d.l.
Si	69.67	70.23	0.11	0.03	100.82	52.72	37.48	32.39	33.84	47.13	41.16	56.38	0.39	0.20
Ti	b.d.	b.d.	–	0.03	b.d.	0.55	0.04	0.03	b.d.	0.15	16.27	b.d.	0.02	0.03
Cr	b.d.	b.d.	–	0.05	b.d.	0.08	b.d.	b.d.	0.06	b.d.	0.08	b.d.	0.01	0.06
Al	b.d.	b.d.	–	0.01	0.02	0.12	20.99	24.55	22.64	5.51	2.35	0.42	0.01	0.01
Fe	b.d.	b.d.	–	0.06	b.d.	37.14	31.51	33.74	34.13	45.93	37.72	0.07	0.16	0.06
Mn	b.d.	b.d.	–	0.07	b.d.	6.76	0.50	0.50	0.65	1.28	1.65	b.d.	0.02	0.06
Mg	b.d.	b.d.	–	0.03	b.d.	b.d.	0.06	0.12	b.d.	b.d.	0.01	b.d.	0.003	0.01
Ni	b.d.	b.d.	–	0.08	b.d.	2.61	2.05	1.73	1.63	0.79	1.32	b.d.	0.02	0.10
Ca	b.d.	b.d.	–	0.02	b.d.	b.d.	8.01	6.42	6.85	b.d.	0.08	40.52	0.01	0.03
Na	b.d.	b.d.	–	0.02										
K	b.d.	b.d.	–	0.02										
C	29.80*	30.04*												
Total	99.49	100.32			100.90	99.98	100.63	99.49	99.82	100.85	100.65	97.53		
Si	1.000	1.000			1.000	6.884	2.284	2.009	2.102	3.058	2.792	1.984		
Ti	0.000	0.000			0.000	0.042	0.001	0.001	0.000	0.006	0.647	0.000		
Cr	0.000	0.000			0.000	0.005	0.000	0.000	0.000	0.002	0.003	0.000		
Al	0.000	0.000			0.000	0.016	1.330	1.584	1.463	0.372	0.166	0.016		
Fe	0.000	0.000			0.000	2.439	0.966	1.052	1.066	1.498	1.287	0.001		
Mn	0.000	0.000			0.000	0.451	0.015	0.016	0.021	0.042	0.057	0.000		
Mg	0.000	0.000			0.000	0.000	0.004	0.008	0.000	0.000	0.000	0.000		
Ni	0.000	0.000			0.000	0.163	0.059	0.051	0.048	0.024	0.043	0.000		
Ca	0.000	0.000			0.000	0.000	0.342	0.279	0.298	0.000	0.004	0.999		
Na	0.000	0.000												
K	0.000	0.000												
C	1.000	1.000												
Σ cations	2.000	2.000			1.000	10.000	5.000	5.000	5.000	5.000	5.000	3.000		

Note: 1 σ = relative error; d.l. = detection limit; b.d. = below detection.

* Calculated by stoichiometry.

TABLE 3. Carbon isotopic data for SiC and associated minerals.

Mineral	$\delta^{13}\text{C}$	Description	Source
Moissanite	–28.1‰	Bluish pebble	this study
Carbonate	–11.9‰	Bluish pebble	this study
Moissanite	–24‰	Fuxian kimberlite	Leung et al. 1990
Diamond	–2.9‰ to –4.8‰	Fuxian kimberlite	Leung et al. 1990
Moissanite	–22‰ to –29‰	Yakutia kimberlite	Mathez et al. 1995
Diamond	–31‰	Yakutia kimberlite	Mathez et al. 1995
SiC	6 to 160‰	Interstellar SiC	Zinner et al. 1989
SiC	150 to 5200‰	Interstellar SiC	Stone et al. 1991
SiC	–27.1‰	Synthetic	this study

Note: Uncertainty of the measurements for this study: 0.1‰.

compounds, always at the micrometer scale, have been found in our sample (Table 2).

(3) Two unidentified MgFe-silicates, with FeO contents ranging between 3 and 8 wt%, are found throughout the matrix and in contact with SiC. According to Mathez et al. (1995), Fe-bearing silicate phases cannot be in equilibrium with SiC under conditions characteristic of the industrial process. At ambient pressure, the f_{O_2} stability field of moissanite is five to six log units below the IW buffer. Under such conditions, the Fe in Fe-bearing silicates is reduced to the metallic form and expelled from the structure. Therefore, in the system Fe-Mg-Si-O-C, the coexistence of SiC and Fe-bearing silicates is probably only possible at the ultra-high pressures conditions of the upper mantle or greater depths.

(4) The moissanite $\delta^{13}\text{C}$ value of –28.1‰ is in agreement with ranges published by Marshintsev (1990), e.g., lower than –25‰, and Mathez et al. (1995), e.g., –22 to –29‰ for Russian moissanite, as well as those of Leung et al. (1990), e.g.,

TABLE 4. EDS semiquantitative analyses

	opx 1	opx 2	mg-cr
	wt%	wt%	
SiO ₂	47.42	48.87	
Al ₂ O ₃	10.54	7.13	26.35
Cr ₂ O ₃	1.31		42.57
FeO	13.38	18.96	19.66
NiO	1.86	1.56	
MgO	25.49	23.48	11.43
Total	100.00	100.00	100.01
Si	1.717	1.805	
Al	0.450	0.310	0.951
Cr	0.038		1.031
Fe	0.405	0.586	0.504
Ni	0.054	0.046	
Mg	1.376	1.293	0.522
Σ cations	4.039	4.040	3.008

Note: opx = orthopyroxene; mg-cr = magnesiochromite

–24‰, for Chinese natural SiC. In natural diamond genesis, a strongly depleted ¹³C isotope signature has been interpreted as a primary mantle feature (e.g., Deines et al. 1993). Compared to diamonds, the values for SiC exhibit a much narrower range (Table 3). The $\delta^{13}\text{C}$ values of meteoritic interstellar SiC grains range from 150 to 5200‰, and thus are far from terrestrial values (Stone et al. 1991, see Table 3).

(5) The presence of phlogopite (e.g., Dawson and Smith 1975) and brucite (e.g., Berg 1989), the high volatile content and the presence of globules (e.g., Pell 1997), all point to a “kimberlitic environment.” The high Mg content of chromite (>11 wt% MgO, Table 4) is typical for primary mantle-derived spinel (e.g., Shulze 2001). The high Al content of the

orthopyroxene (>10 wt% Al₂O₃, Table 4) points to high equilibration temperature (probably >1200 °C) (e.g., Danckwerth and Newton 1974).

The intimate association of SiC and Fe-bearing silicates, along with the other above considerations, suggest an origin from the mantle of the present sample. The fact that such an assemblage is not reported experimentally shows the need for more detailed ultra-high pressure studies in moissanite-bearing systems.

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