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How Impossible

CRYSTALS

Came to Earth

A Short History

Before 1984, scientists thought they had a complete understanding of all the possible ways atoms and molecules can join together to make a solid. This knowledge, established nearly two centuries ago, is codified in a set of principles known as *the laws of crystallography*, which are essential for understanding and taking advantage of the physical properties of matter, whether it be making steel for a bridge, cleaving the facets of a diamond, or manipulating the electronic properties of silicon for use in integrated circuits. According to these laws, arrangements of atoms in solids are either completely random, as in the case of window glass, or crystalline, as is the case for sugar or table salt. In the case of crystalline materials, the atoms are organized in a symmetrical lattice like the square tiles in a simple bathroom tiling wherein tiles repeat in a periodic pattern (translational symmetry) that also exhibits a discrete rotational symmetry. The two cases are analogous to mosaics in which the tiles are put together either randomly or in an orderly, symmetrical tessellation. A key fact about regular tessellations, known since the ancient Egyptians, is that only certain symmetries can be obtained. The same rules apply to matter. Thus, periodic materials can only exhibit certain rotational symmetries: two-, three-, four-, and sixfold symmetry axes; five-, seven-, eight-, or higher-fold symmetry axes are strictly forbidden. Icosahedral symmetry, which includes six independent fivefold symmetry axes, is super-forbidden.

Thirty-five years ago, one of us (PJS) and Dov Levine, his student, were inspired by a curious geometric pattern invented by Sir Roger Penrose (Penrose 1974) a few years earlier. Penrose had identified a pair of tile shapes that could only fit together non-periodically, forming a self-similar pattern full of fivefold symmetric clusters of tiles (fig. 1, top). Several theorists independently speculated that there might be some analogy for solids, though in most cases it was not

made clear what this meant. The approach of Levine and Steinhardt (1984) was systematic in first identifying that the fivefold symmetry was possible and that the same quasiperiodic principle could be used to construct polyhedral units with protrusions and holes on their faces that constrain the way they join together such that the units can only fit together in a three-dimensional solid with icosahedral symmetry (fig. 1, bottom). The two theorists challenged the cen-

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Dr. Paul J. Steinhardt is the Albert Einstein Professor in Science and director of the Princeton Center for Theoretical Science at Princeton University. With Dov Levine, he introduced the concept of quasicrystals in 1984; he also developed some of the basic theory describing their physical properties and originated the search for natural quasicrystals. He is also recognized for his contributions to particle physics and cosmology.

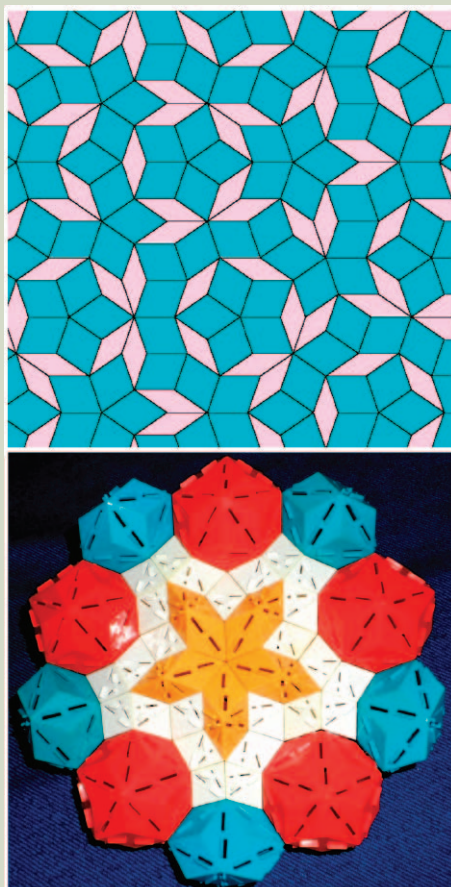


Figure 1. Top: Fragment of a two-dimensional Penrose tiling composed of two types of tiles (pale blue and pink) arranged with crystallographically forbidden fivefold symmetry. **Bottom:** Fragment of a three-dimensional icosahedral quasicrystal composed of four types of polyhedral units with holes and protrusions that constrain the way the units match face-to-face in such a way as to guarantee that all space-filling arrangements are quasicrystalline. The two-dimensional tiling at the top does not show the necessary matching rules (protrusions/dimples).

tury-old laws of crystallography and proposed a hypothetical new form of matter, which they dubbed “quasicrystal,” whose symmetries and physical properties were previously thought to be impossible. The idea took off when an Israeli materials scientist, Daniel Shechtman, and his collaborators accidentally discovered a possible example (a puzzling alloy of aluminum and manganese) in his laboratory (Shechtman et al. 1984), a discovery that ultimately earned Shechtman the Nobel Prize in Chemistry in 2011.

During the next few years, the story became complicated as several scientists, including Linus Pauling, discounted the quasicrystal model and proposed alternative explanations for Shechtman’s alloy that could explain its properties seemingly just as well. The problem boiled down to the fact that the aluminum-manganese alloy was too disordered to unambiguously distinguish among the various models. The debate was resolved in 1987 with the discovery of a different alloy with such perfect quasiperiodic structure that it could only be explained as a bona fide quasicrystal (Tsai, Inoue, and Masumoto 1987). But a new debate immediately began: Why do quasicrystals form? Are they truly stable forms of

matter like crystals? These questions motivated the search for a natural quasicrystal. Indeed, if stable quasicrystals are possible, Nature should have formed them without any human intervention, and they should have survived for eons, just like crystals.

Based on this idea, one of us (PJS) developed a systematic plan for searching for natural quasicrystals (Lu et al. 2001) using the catalogue of powder diffraction patterns collected by the International Center for Powder Diffraction Data. Their extensive database (ICDD-PDF) contains over eighty thousand patterns of synthetic and natural materials including nearly nine thousand mineral patterns. The key to the search strategy was to build a mathematical protocol for assigning quantitative figures-of-merit to the powder patterns that would separate known quasicrystals and promising quasicrystal candidates from the vast majority of powder patterns in the ICDD-PDF. Using these figures-of-merit, the search by Lu et al. (2001) ranked all the patterns in the catalogue and identified six minerals among the one hundred most promising candidates, but after a careful investigation, none of the candidates proved to be an icosahedral quasicrystal or anything remarkable. The problem, as it turned out, was that the data in the ICDD-PDF catalogue contained sufficient errors to give false positives for complex but ordinary periodic crystals.

After a year of failure, one of us (LB) had the intuition to test minerals that were not listed in the ICDD-PDF catalogue altogether but whose compositions were similar to known quasicrystals synthesized in the laboratory. This led to the discovery of a promising candidate in the mineral collection of the *Museo di Storia Naturale* (Natural History Museum) of the University of Florence that scored high on the powder diffraction test developed in Lu et al. (2001). The sample, labeled “khatyrkite” (catalogue No. 46407/G; fig. 2), was acquired by the Florence museum in 1990 from an Amsterdam

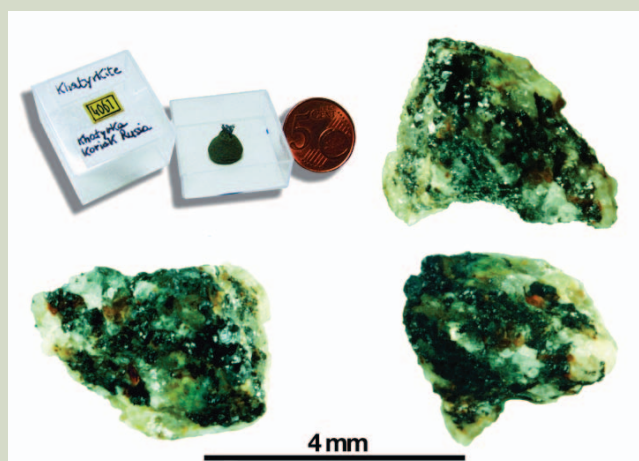


Figure 2. Different views of the original khatyrkite-bearing sample belonging to the collections of the *Museo di Storia Naturale* of the University of Florence, Italy (catalogue No. 46407/G). The lighter-colored material on the exterior contains a mixture of spinel, clinopyroxene, and olivine. The dark material consists predominantly of khatyrkite (CuAl_2) and cupalite (CuAl) but also includes granules of icosahedrite with composition $\text{Al}_{63}\text{Cu}_{24}\text{Fe}_{13}$.

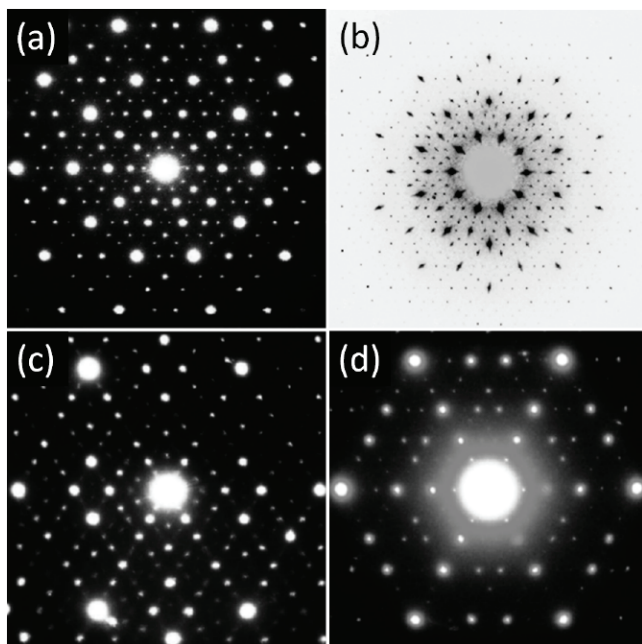


Figure 3. The unmistakable signature of an icosahedral quasicrystal consists of patterns of sharp peaks arranged in straight lines in an incommensurate lattice with fivefold (a), twofold (c), and threefold (d) symmetry. The electron diffraction patterns shown here, taken from a grain of icosahedrite, match those predicted for a face-centered icosahedral quasicrystal, as do the angles that separate the symmetry axes. In panel (b) is reported a single-crystal X-ray diffraction reconstructed precession photograph down one of the fivefold symmetry axes of the icosahedron collected on a small icosahedrite fragment dug out from a grain recovered in the 2011 Koryak expedition.

collector and catalogued as coming from the Khatyrka region of the Koryak Mountains in the Chukotka autonomous okrug (district) on the northeastern part of the Kamchatka Peninsula (Bindi et al. 2009, 2011, 2012). As first reported by Razin, Rudashevskij, and Vyalsov (1985), khatyrkite, nominally $(\text{Cu,Zn})\text{Al}_2$, is a tetragonal mineral found in association with cupalite, nominally $(\text{Cu,Zn})\text{Al}$, which is orthorhombic. In the Florence sample, khatyrkite was found to be intergrown with typical rock-forming minerals (e.g., forsterite and diopside), other metallic crystal phases (cupalite and $\beta\text{-AlCuFe}$), and a few grains of a new species, with composition $\text{Al}_{63}\text{Cu}_{24}\text{Fe}_{13}$, whose X-ray powder diffraction pattern did not match that of any known mineral. Sure enough, in a test conducted at 7 A.M. on New Year's Day 2009 in a dark Princeton laboratory, the $\text{Al}_{63}\text{Cu}_{24}\text{Fe}_{13}$ unnamed species was found to have a perfect icosahedral quasicrystalline structure (fig. 3).

The story might have ended there, if not for a puzzle. The conditions to form the mineral studied on New Year's morning were nothing like the pristine conditions used to synthesize quasicrystals in the laboratory. How had Nature done it? When one of the authors (PJS) approached the famous petrologist Lincoln S. Hollister (Princeton University) to find out how this might be possible, the response was short and to the point: *It's impossible!* The quasicrystal contained metallic aluminum, Hollister noted, which can only be separated from oxygen artificially, as in aluminum processing plants.

The sample had to be slag, a worthless manmade artifact. The apparent success had now turned to depressing failure, leading to the next chapter in the story.

For more than a year and a half, we worked to trace the origin of the Florence sample. During this period we often had the impression we were living in a fairytale; however, every bit of the story was true. As in any good fairytale, impossible things happen, and impossible barriers are overcome again and again, sometimes magically by good luck and sometimes through fanatical determination.

A letter in the museum archives explained that the box containing the sample in figure 2 had been bought in a bulk allotment from an Amsterdam collector. It seemed like a dead end, until a fortuitous meeting during a dinner in Florence eight years ago. One of us (LB) was regaling his companions with the quasicrystal story when an acquaintance at the table remembered that an older woman married to a mineral collector lived on his street in Amsterdam. When the acquaintance returned home, he asked his neighbor about the mineral dealer, and, remarkably, he was the woman's deceased husband. We hopped on a plane to Amsterdam.

The widow knew nothing of khatyrkite, but she offered to let us look through her husband's "secret diary." In it, the dealer explained that he had purchased the mineral from a man named Tim during a trip to Romania in 1987. But where had Tim obtained the mineral? We spent six weeks trying to find him and didn't get even a smidgen of a hint. Thus, we went again to meet the widow to see if she knew anything about Tim, the Romanian. She didn't. But she revealed that her husband used to keep a "secret, secret diary." That diary revealed that the dealer had actually purchased the mineral from Leonid Razin, then the director of the Institute of Platinum in St. Petersburg, Russia. It was a name we recognized.

In 1985 Razin had scientifically reported and characterized the only other genuine example of khatyrkite we knew to exist—the holotype, which was discovered near the Koryak Mountains in Far Eastern Russia and is kept in the Mining Museum in St. Petersburg. It seemed that the holotype and the Florence specimen were found together and that Razin had studied the former and sold the latter. But when one of us (PJS) tracked down Razin and called him up at his new home in Israel, Razin said he didn't remember how he had acquired the khatyrkite. Again, the trail went cold.

Out of ideas, we returned to the 1985 paper in which Razin reported the discovery of khatyrkite. The first paragraph mentioned a man named Valery Kryachko who seemed to have played a role in the discovery. Contacts told us that Kryachko was probably an untraceable rural miner who had picked up the khatyrkite while panning for minerals on behalf of the Institute of Platinum. But not long afterward, while idly perusing Russian mineralogy journals in search of more leads, we spotted Kryachko's name among the authors of a different paper from 1995. Suddenly we went from nothing to thinking maybe, maybe this is our guy. We found Kryachko in Moscow. An enthusiastic mineralogist in his sixties, he explained that Razin had indeed hired him to mine for platinum back when Kryachko was in graduate school. In



Figure 4. Members of the Koryak expedition team (from left): Bogdan Makovskii (driver), Vadim Distler (IGEM, Russia), Marina Yudovskaya (IGEM, Russia), Valery Kryachko (Voronezh, IGEM), Glenn MacPherson (Smithsonian Institution, United States), Luca Bindi (University of Florence, Italy), Victor Komelkov (driver), Olga Komelkova (cook), Alexander Kostin (BHP Billiton, United States), Christopher Andronicos (Cornell, United States), Michael Eddy (MIT, United States), Will Steinhardt (Harvard, United States). At the center is Paul Steinhardt (Princeton, United States), leader of the expedition. W. M. Steinhardt photo.

1979, he was deposited by helicopter at an obscure stream called Listvenitovyi, hundreds of miles from the nearest village, and spent several days digging through the blue-green clay. No platinum turned up in the several hundred kilograms of clay he panned, but Kryachko did find a few shiny little nuggets he couldn't identify. He delivered them to Razin and never heard about them again.

In the meantime, the early laboratory tests had destroyed most of the Florence sample except for a few micrograins. Those few micrograins, however, ultimately indicated something unexpected: the rock was most likely the remnant of an asteroid dating to the birth of the solar system! This result was obtained by an ion probe investigation of the rare oxygen isotope ($^{18}\text{O}/^{16}\text{O}$ and $^{17}\text{O}/^{16}\text{O}$) compositions, which was carried out at the California Institute of Technology (Bindi et al. 2012). The oxygen isotope measurements for the spectrum of minerals in the Florence sample were found to be spread along the carbonaceous chondrite anhydrous minerals (CCAM) line and were clearly inconsistent with the terrestrial fractionation (TF) line. Hence, the silicates and oxides in the rock sample unambiguously identify the sample as being of extraterrestrial origin with isotope values resembling the constituents of calcium aluminum-rich inclusions (CAIs) from CV3 and CO3 carbonaceous chondrites, which

are among the oldest meteorites to have formed in our solar system. The discovery was a breakthrough again: it indicated new processes important in forming the initial matter that came together to form the cores of planets. Confirming the origin of the sample and finding more samples to study suddenly took on new scientific importance.

The only way to find more samples was to return to where the original was found, one of the most remote places on the planet. Despite overwhelming barriers, on 22 July 2011, a team of ten scientists from the United States, Russia, and Italy; two drivers; and a cook/lawyer (fig. 4) gathered at the edge of the town of Anadyr, the capital of Chukotka, ready to board the odd-looking double-track vehicles that would take them across the tundra (fig. 5) and into the Koryak Mountains to the Listvenitovyi stream, 230 kilometers to the southwest. The U.S. contingent consisted of Christopher Andronicos (Purdue University), an expert on structural geology; Glenn MacPherson (Smithsonian Institution, Washington, D.C.), former chairman of the Division of Mineral Sciences at the Natural History Museum; graduate students in geoscience Will Steinhardt (Harvard University) and Michael Eddy (MIT); translator and general assistant with a background in oil exploration Alexander Kostin (BHP Billiton); and both of us. Yudovskaya, Distler, and Kryachko,



Figure 5. Top: General view of the Koryak Mountains. Bottom: Two scenes from the expedition.

the Russian contingent, contributed expertise in ore minerals and the region. Victor Komelkov and Bogdan Makovskii were the drivers and maintenance crew for the two tractor vehicles and responsible for netting fish for the team; Victor's wife, Olga Komelkova, was the cook; and Victor and Olga were accompanied by Bucks, their indomitable cat. As the team boarded the two strange vehicles with great hopes, there was no telling if they would find anything. Some on the expedition and many at home maintained doubts about whether there was any truth to the entire story of the origin of the quasicrystal-bearing rock.

The three-week adventure included broken axles, fires, bears, mosquitoes, and August snowstorms. At the Listvenitovy stream, 1.5 tons of clay and sediment were dug by hand and painstakingly panned in the same manner that gold was panned in the California gold rush. The results of this hard labor would not be known until weeks after the journey was over and the samples were studied in the laboratory.

During the next five years, ten new samples were found through a painstaking grain-by-grain search. These have firmly established that the quasicrystal and the rock containing it are definitely part of a carbonaceous chondritic

meteorite with calcium-aluminum inclusions that date back 4.5 Gya to the formation of the solar system (Lin et al. 2017 and references therein). The new meteorite find has been named Khatyrka (MacPherson et al. 2013). The name derives from the Khatyrka River, one of the main rivers draining the Koryak Mountains. That river is also the namesake of the mineral khatyrkite, which gives an added symmetry to the meteorite name. Khatyrka has been approved by the Nomenclature Committee of the Meteoritical Society, and representative specimens are on deposit at the U.S. National Museum of Natural History, Smithsonian Institution, Washington, D.C.

The Khatyrka meteoritic fragments recovered from the expedition presented a range of evidence indicating that an impact shock generated a heterogeneous distribution of pressures and temperatures in which some portions of the meteorite reached at least 5 gigapascals (GPa) and 1,200°C (Hollister et al. 2014). Among the Khatyrka fragments a second type of natural quasicrystal has been found (Bindi et al. 2015a). This quasicrystal has a composition of $Al_{71}Ni_{24}Fe_5$ and is the first known natural quasicrystal with decagonal symmetry, a periodic stacking of layers containing quasipe-

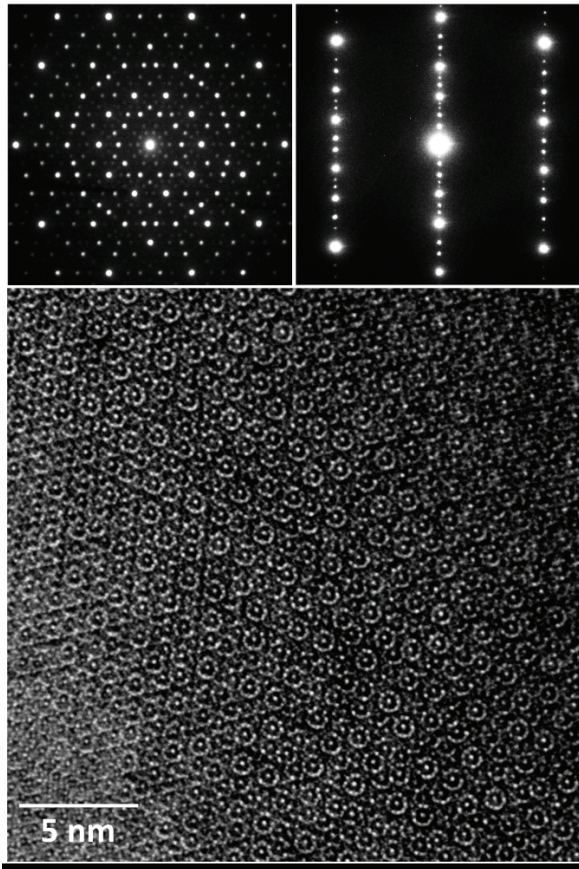


Figure 6. The top panel reports two selected area electron diffraction patterns collected down the tenfold axis (*left*) and along an axis perpendicular to the tenfold axis (*right*). The structure has layers, each of which is quasiperiodic and has tenfold symmetry; these layers are periodically arranged along the direction normal to the layers. The bottom panel is a high-resolution transmission electron microscopy (HRTEM) image showing that the real space structure of decagonite consists of a remarkable homogeneous, quasiperiodic and tenfold symmetric pattern.

riodic atomic arrangements with crystallographically forbidden tenfold symmetry (figs. 6 and 7). The mineral and its name, decagonite, have been approved by the Commission on New Minerals, Nomenclature and Classification, IMA

(Bindi et al. 2015b). As already observed for icosahedrite (Bindi et al. 2009, 2011), decagonite exhibits a high degree of structural perfection, particularly the absence of significant phason strains (fig. 7), which are a special kind of disorder particular to quasicrystals (Lubensky et al. 1986) corresponding to certain local flipping of small groups of tiles in a tessellation. Phason strains are common in synthetic quasicrystals unless they are grown under highly controlled thermodynamic conditions and with a high degree of purity. The natural quasicrystals, by contrast, are clearly formed far from equilibrium and in close contact with other phases. We think that either the mineral samples formed without phason strain in the first place, or subsequent annealing was sufficient for phason strains to relax away.

In addition to the numerous new minerals described in the Khatyrka meteorite (Bindi et al. 2014; Ma et al. 2017), particularly notable is the recent description of the possible third natural quasicrystal (Bindi et al. 2016), which coexists with icosahedrite in the same fragment (fig. 8). The new icosahedral phase has a composition $\text{Al}_{62.0(8)}\text{Cu}_{31.2(8)}\text{Fe}_{6.8(4)}$, which is outside the measured equilibrium stability field at standard pressure of the previously reported Al-Cu-Fe quasicrystal ($\text{Al}_x\text{Cu}_y\text{Fe}_z$, with x between 61 and 64 percent, y between 24 and 26 percent, and z between 12 and 13 percent). The discovery of an icosahedral Al-Cu-Fe quasicrystal with a composition far from that of any known ideal, stable quasicrystal is notable for several reasons. It is only the third example of a natural quasicrystal to be found anywhere, all from fragments of the same Khatyrka meteorite; and it is the first documented example of the coexistence of two different Al-Cu-Fe icosahedral phase compositions. Furthermore, and maybe the most stunning, it is the first example of a composition discovered in nature prior to being discovered in the laboratory.

The occurrence inside the meteorite demonstrates that quasicrystals can form naturally within a complex, inhomogeneous medium. The result not only settles the fundamental issues about quasicrystals as a form of matter that is effectively stable (e.g., over geologic time scales), but also shows that quasicrystals formed in a violent collision between asteroids at the birth of the solar system and, though once

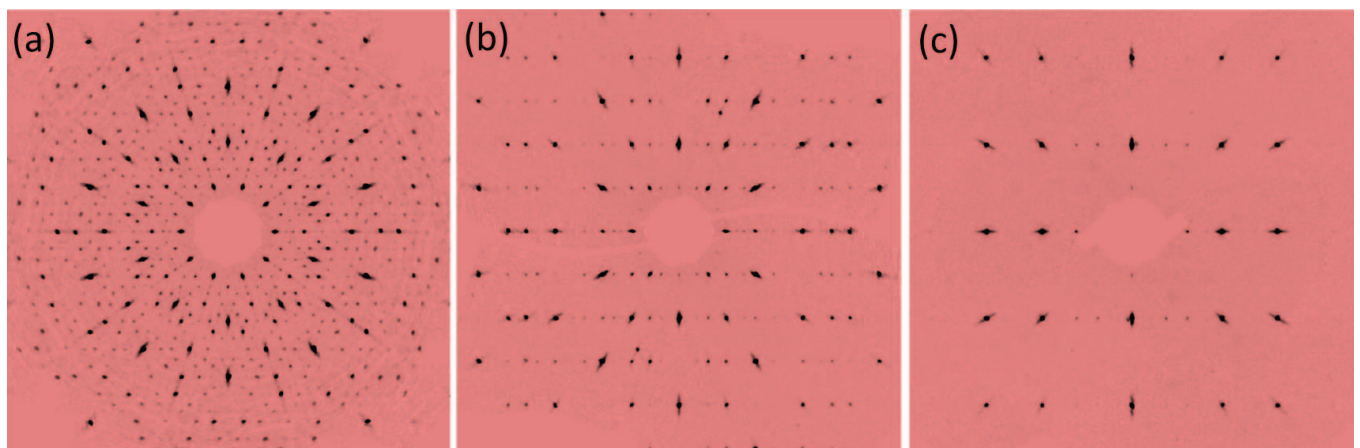


Figure 7. Reconstructed precession images along the tenfold symmetry axis (a) and perpendicular to the tenfold direction (b, c) obtained using the collected single-crystal X-ray data set (MoK α radiation) from decagonite.

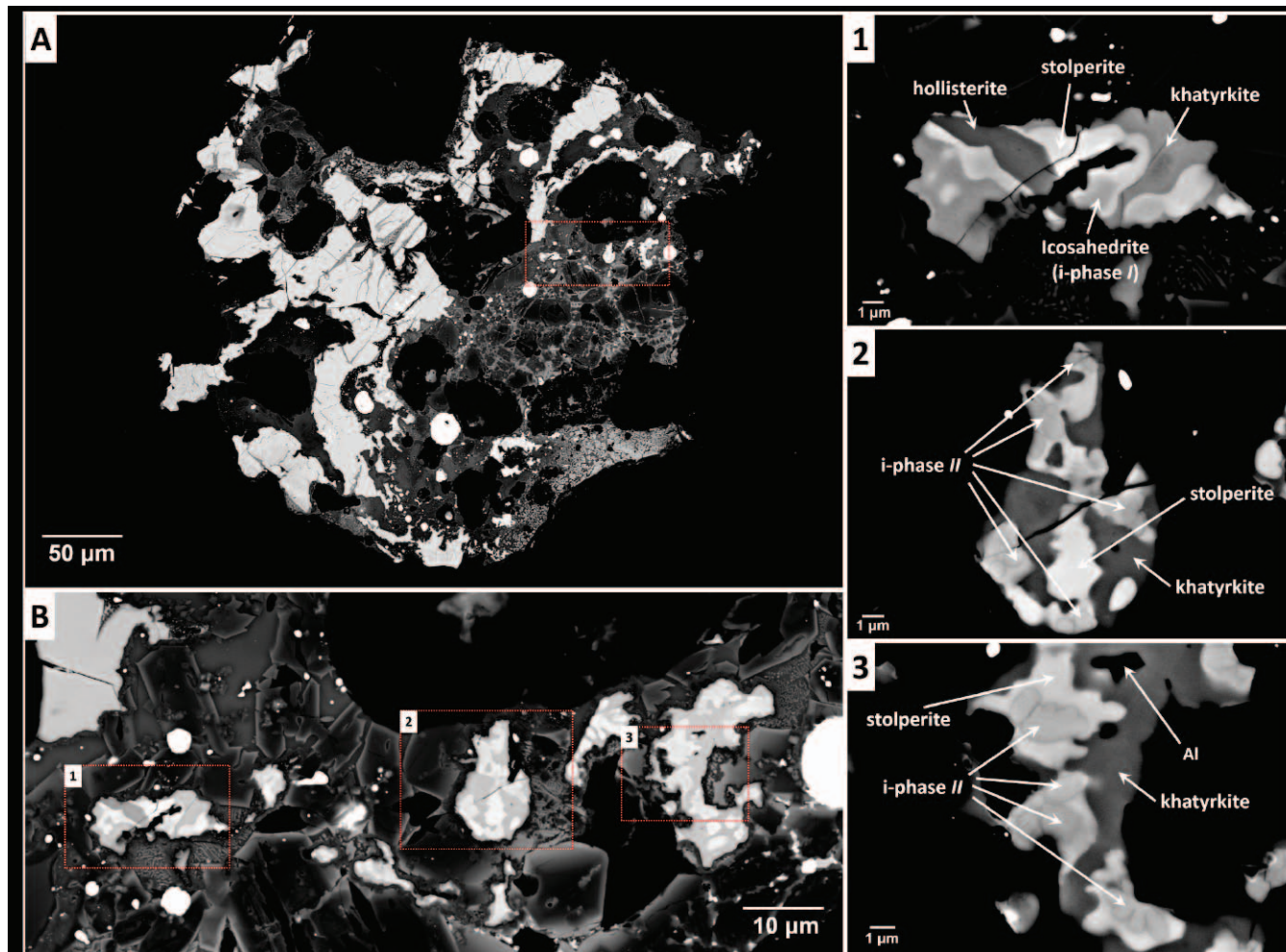


Figure 8. Backscattered electron images of one of the grains recovered in the 2011 Koryak expedition (grain No. 126A). (A) General view of the grain; red dashed box indicates the region to be enlarged in (B). (B) The area where there are the three metal assemblages containing the two different icosahedral phases; red dashed boxes (indicated as 1, 2, and 3) indicate the regions to be enlarged in panels on right. Panels 1, 2, and 3 show the different associations of minerals in the three metal assemblages. The possible third natural quasicrystal is indicated as “i-phase II.”

thought to be impossible, are probably not so rare materials in the Milky Way, and perhaps the universe.

ACKNOWLEDGMENTS

The results reported here would not be possible without the extraordinary efforts of our expeditionary team, pictured and named in figure 4, and the generosity of our anonymous donor who funded the expedition. This article reviews work spanning papers with them and with such valued collaborators as J. M. Eiler, Y. Guan, L. S. Hollister, J. Poirier, and N. Yao. Many who have given valuable advice and support include C. Ballhaus, J. Beckett, P. Bonazzi, A. E. Goresy, A. Ishiwatari, H.-C. Jeong, J. Jones, S. Menchetti, M. Morozov, G. Poirier, P. Robinson, V. Rudashevskij, P. Spry, and E. Stolper. We give special thanks to the IGEM (Moscow) for their support in organizing the expedition and collaboration. We thank John Jaszczak and John Rakovan for the invitation to write this article recounting the discovery of quasicrystals in *Nature* for *Rocks & Minerals* and for their useful reviews.

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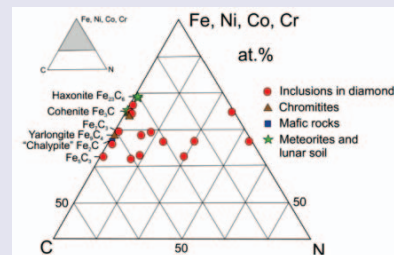
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Comparing the composition of the Earth to carbonaceous chondrites shows the nitrogen in Earth is significantly less than expected, unlike hydrogen, carbon, and most noble gases. It seems unlikely that N was lost during the formation or early history of the planet. Either N is retained in a mantle mineral during magma generation, or it is trapped in the core. So far, there has been no evidence for nitrogen reservoirs in the mantle larger than the present atmospheric N, though high concentrations of N occur in iron meteorites. In the August 2017 issue of the *American Mineralogist*, Kaminsky and Wirth report the occurrence of iron nitrides, Fe₂N and Fe₃N, and carbonitride, Fe₉(N,C)₄, as inclusions in lower-mantle diamonds from Juina area, Mato Grosso State, Brazil. They conclude that these N minerals form at the core/mantle boundary as the result of the infiltration of liquid metal, containing light elements, from the outer core into the lower mantle, and that major reservoirs for Earth's nitrogen should be expected in the core and in the lowermost mantle.



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