



Quasicrystals at high pressures and temperatures: a review

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Abstract

We summarize the results of studies on quasicrystals (QCs) at extreme conditions over the last 4 decades with particular emphasis for compositions falling in the Al-based ternary system as the closest to those of quasicrystals discovered in nature, such as icosahedrite and decagonite. We show that, in contrast with what thought in the past, both pressure and temperature act to stabilize QCs, for which a clear phase transition to either crystalline approximants or amorphous material has been limited to very few compositions only. Such stabilization is proved by the compressibility behavior of QCs that resembles that of the pure constituent metals. Additional remarks come from the experimental observation of QC formation at high pressure and temperature in both static and dynamic experiments. These results seem, in conclusion, to suggest that the occurrence of QCs in nature might be more a rule rather than an exception.

Keywords Quasicrystals · Icosahedrite · Decagonite · High pressure · Meteorites · Diamond anvil cell technique

1 Introduction

Quasicrystals (QCs), generally defined as quasiperiodic solids characterized by unconventional crystallographic rules (Levine and Steinhardt 1984), find applications in industry due to their physical properties, light absorption, reduced adhesion and friction, thermal insulation, coating on mechanical devices, etc. (Dubois 2012, 2023). The investigation of the physical and chemical properties of QCs appears also relevant in the field of planetary geology despite their rare occurrence in nature. Indeed, the Khatyrka meteorite, a CV3 carbonaceous chondrite discovered in the Koryak mountains, Eastern Russia (Bindi et al. 2012; MacPherson et al. 2013), represents, to date, the only natural sample that

has led to the discovery of icosahedral (*i*)-Al₆₃Cu₂₄Fe₁₃, then named icosahedrite, the first naturally occurring QC (Bindi et al. 2009, 2011). The discovery was followed by the finding of the second natural QC, Al₇₁Ni₂₄Fe₅, named decagonite for the internal decagonal symmetry (Bindi et al. 2015a, b), and, very recently, a third, still unnamed *i*-QC, Al₆₂Cu₃₁Fe₇ (Bindi et al. 2016). The latter QC is quite exceptional owing to its Fe-poor composition outside the stability field of QCs in the Al–Cu–Fe ternary system (Bancel 1999) and represents the first example of a composition discovered in nature prior to being discovered in the laboratory. The meteorite containing natural quasicrystals shows evidence indicating high pressures (P) and temperatures (T) (Hollister et al. 2014; Lin et al. 2017).

Besides the finding of icosahedrite as intergrowth within silicates that can be taken as proof of their chemical equilibrium, icosahedrite was reported to coexist with two high-pressure phases, i.e., stishovite and ahrensite (Bindi et al. 2012; Hollister et al. 2014). The former being stable between 8 and 60 GPa (Swamy et al. 1994; Kuwayama 2008), while the latter, representing the high-pressure (HP) polymorph of Fe₂SiO₄ (Ma et al. 2016), is stable above ~5 GPa (Yagi et al. 1987) up to ~23 GPa (Presnall 1995).

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Along with the coexistence of HP minerals, the presence of dendritic quenched textures formed by metallic liquids (Hollister et al. 2014; Lin et al. 2017) can be interpreted as evidence of the melting-driven formation of QCs that likely also led to the formation of additional Al–Cu–Fe crystalline phases such as khatyrkite, cupalite, hollisterite, kryachkoite, and stolperite (Ma et al. 2017).

To corroborate these observations, laboratory experiments were carried out. They included: (1) testing the stability of QCs at high P and T; (2) phase equilibria studies within chemical systems representative of icosahedrite, for instance, within the Al–Cu–Fe system; and (3) constraining the P–T conditions for melting of QC.

In the frame of an international conference on quasicrystals held at the Accademia dei Lincei in November 2022 (Bindi and Parisi 2023), we took the occasion to review past and recent experimental studies on the HP–T stability of synthetic QCs, with particular attention to the compositions of interest for planetary geology.

2 Al-based quasicrystals at high pressures

The general interest for QCs relies on their diverse physical properties that result in potential wide industrial applications such as hydrogen storage, hydride battery materials, and coating of soft metals (Dubois 2000, 2012, 2023). Beyond the highly debated topic whether QCs are stabilized by entropy or energy, their investigation under HP allows to monitor changes of the atomic arrangement resulting into phase transitions as well as changes in their physical properties that might be of inspiration for further industrial applications. Al-based QCs are of particular interest in this review as their composition resembles that found in nature (Bindi et al. 2009, 2011, 2015a, b, 2016). Table 1 is a summary of the QC compositions that have been investigated at high P so far, along with their elastic parameters (K_0 in GPa and K_0^I) resulting from fitting the volume data collected up to the maximum reported P with either a Murnaghan or a Birch–Murnaghan Equation of State (EoS). Table 1

Table 1 In situ HP studies on Al-bearing QC

QC	P (GPa)	Technique	Pressure medium	K_0 (GPa)	K_0^I	References
<i>i</i> -Al ₈₂ Fe ₁₈	8.3	PXRD	NaCl	–	–	Jaya et al. (1987)
<i>i</i> -Al ₇₈ Mn ₂₂	47	EDXRD	Methanol/ethanol	144 ± 13	1.9 ± 0.6	Johnson et al. (1988)
<i>i</i> -Al ₇₄ Mn ₂₆	47	EDXRD	Methanol/ethanol	148 ± 7	1.5 ± 0.3	Johnson et al. (1988)
α-AlMn (crystalline)	47	EDXRD	Methanol/ethanol	85 ± 2	2.9 ± 0.1	Johnson et al. (1988)
<i>i</i> -Al ₈₆ Mn ₁₄	28	PXRD	Methanol/ethanol	117.6 ± 16.8	6.0 ± 2.4	Sato-Sorensen and Sorensen (1989)
<i>i</i> -Al ₆₀ Li ₃₀ Cu ₁₀	20	PXRD	Methanol/ethanol/H ₂ O	70	–	Akahama et al. (1989)
<i>i</i> -Al ₆₈ Ru ₁₇ Cu ₁₅	30.8	PXRD	Methanol/ethanol/H ₂ O	150	–	Akahama et al. (1991)
<i>i</i> -Al ₆₅ Cu ₂₀ Ru ₁₅	35.8	PXRD	Si oil	128 ± 10	5.0 ± 1.0	Sadoc et al. (1998)
<i>d</i> -Al ₆₃ Cu _{17.5} Co _{17.5} Si ₂	Unk	CT	–	–	–	Kang and Dubois (1992)
<i>d</i> -Al ₆₅ Co ₁₅ Cu ₂₀	19.1	ADXRD	Methanol/ethanol	131 ± 8.0	3.8 ± 0.1	Krauss and Steurer (2004)
<i>i</i> -Al _{63.5} Cu _{24.5} Fe ₁₂	Unk	CT	–	–	–	Kang and Dubois (1992)
<i>i</i> -Al ₆₂ Cu _{25.5} Fe _{12.5}	35	EDXRD	Si oil	139 ± 6.0	2.7	Sadoc et al. (1994)
<i>i</i> -Al ₆₂ Cu _{25.5} Fe ₁₂	33.5	EDXRD	Gold	155 ± 10	2.0	Lefebvre et al. (1995)
<i>a</i> -Al _{62.8} Cu ₂₆ Fe _{11.2}	33.5	EDXRD	Gold	175 ± 16	2.0	Lefebvre et al. (1995)
<i>a</i> -Al ₆₄ Cu ₂₄ Fe ₁₂	33.5	EDXRD	Gold	175 ± 16	2.0	Lefebvre et al. (1995)
<i>i</i> -AlCuFe	50	ADXRD	Ne	113.7	4.22	Stagno et al. (2015)
<i>i</i> -AlCuFe	104	ADXRD	Ne	121 ± 3	3.87 ± 12	Stagno et al. (2021)
<i>i</i> -Al ₆₂ Cu _{25.5} V _{12.5}	20	EDXRD	Methanol/ethanol	79 ± 6	10.7 ± 1.6	Ponkratz et al. (2001)
<i>i</i> -Al ₆₂ Cu _{25.5} Cr _{12.5}	20	EDXRD	Methanol/ethanol	122 ± 2	12 ± 1	Ponkratz et al. (2001)
<i>i</i> -Al ₆₂ Cu _{25.5} Mn _{12.5}	20	EDXRD	Methanol/ethanol	116 ± 7	9.5 ± 2	Ponkratz et al. (2001)
<i>i</i> -Al _{68.7} Pd _{21.7} Mn _{9.6}	40	PXRD	Si oil	133 ± 5	5 ± 1	Amazit et al. (1997)
<i>i</i> -Al ₇₂ Pd _{18.5} Mn _{9.5}	70	ADXRD	None	100 ± 12	5.3 ± 0.9	Hasegawa et al. (1999a)
<i>i</i> -Al _{68.2} Pd _{22.8} Mn ₉	35	ADXRD	He	125 ± 1	3.9 ± 3	Decremps et al. (2006)
<i>i</i> -Al _{70.5} Pd ₂₁ Re _{8.5}	35.9	PXRD	Si oil	180 ± 10	5 ± 1	Sadoc et al. (1998)
<i>d</i> -AlNiCo	40	PXRD	–	144.3 ± 10.3	4.68 ± 0.68	Zhou et al. (1996)
<i>d</i> -Al ₇₂ Ni ₂₀ Co ₈	67	ADXRD	None	~ 120	~ 5	Hasegawa et al. (1999b)
<i>d</i> -AlNiCo	10	PXRD	–	120	5	Krauss and Steurer (2004)
<i>d</i> -AlNiCo	10	PXRD	–	121 ± 8	3.5 ± 1.4	Krauss and Steurer (2004)

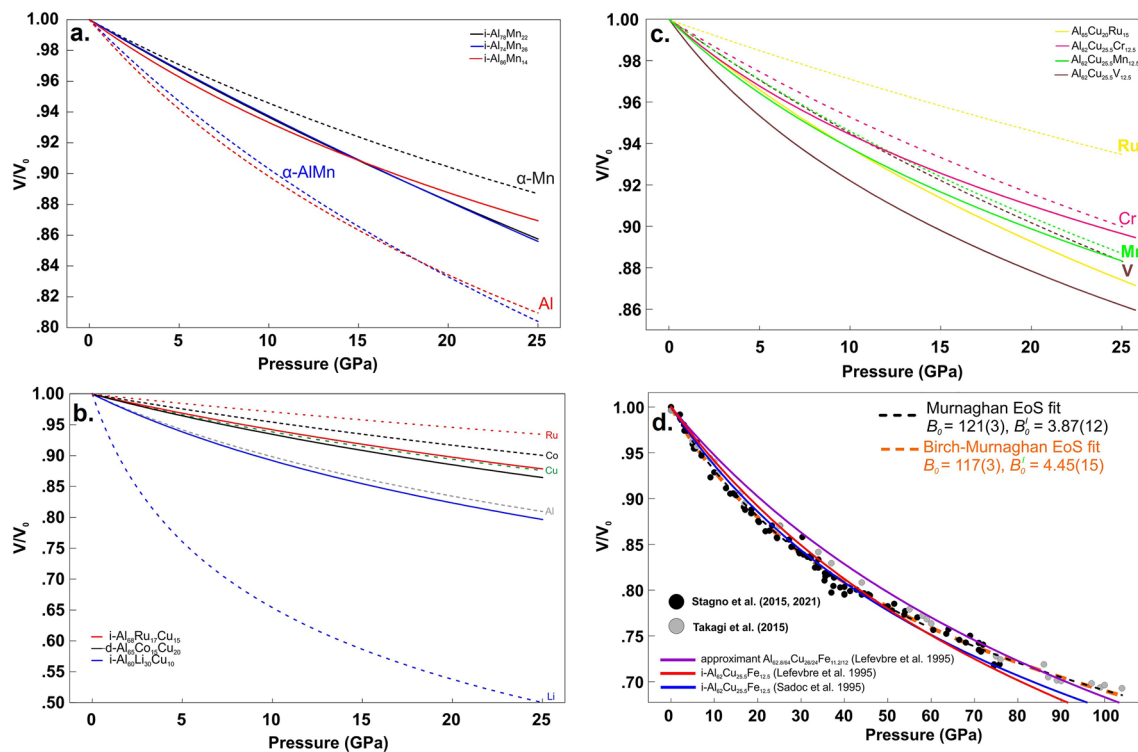


Fig. 1 Pressure–volume data for **a** QCs known within the binary Al–Mn system; **b** QCs with icosahedral and decagonal symmetry within the AlYCu system with $Y = \text{Ru, Co, and Li}$; **c** QCs within the AlCuZ with $Z = \text{Ru, Cr, Mn, and V}$; **d** QCs with icosahedral symmetry and an approximant within the system Al–Cu–Fe system. The curves

obtained from these studies are drawn within the investigated P range considering that no structural phase transformation has been shown. For each diagram, the P–V data of the pure metals are reported for comparison with references given in the text

represents, therefore, an updated version of what was given by Krauss and Steurer (2004). It reports the stoichiometry for each QC and the latest experimental data referred exclusively to binary and ternary Al-based QCs due to their similarity with both natural icosahedrite and decagonite. As it can be seen from Table 1, the bulk modulus, K_0 , varies between 79 GPa for $i\text{-Al}_{62}\text{Cu}_{25.5}\text{V}_{12.5}$ (Ponkratov et al. 2001) and 180 GPa for $i\text{-Al}_{70.5}\text{Pd}_{21}\text{Re}_{8.5}$ (Sadoc et al. 1998), the discrepancy being likely related to the different chemical composition.

A couple of years after the experimental finding of icosahedral $\text{Al}_{86}\text{Mn}_{14}$ by Shechtman et al. (1984) and the theoretical prediction by Levine and Steinhardt (1984), the first HP investigation of $i\text{-QC}$ up to 14 GPa by monitoring changes in resistivity of $i\text{-Al}_{78}\text{Mn}_{22}$ and $i\text{-Al}_{86}\text{Mn}_{14}$ was performed by Parthasarathy et al. (1986). The authors reported a QC-to-crystalline transition (plus the occurrence of pure Al) at 5 and 9.2 GPa, respectively. Later, a powder XRD study was performed on the binary $i\text{-Al}_{78}\text{Fe}_{22}$ up to 8.3 GPa by Jaya et al. (1987) using a metal anvil clamp cell and NaCl as pressure medium. They proved the stability of the $i\text{-QC}$ with an anisotropic behavior under compression without providing elastic parameters. More HP data became later available for

binary $i\text{-AlMn}$ alloys by both conventional powder XRD (Sato-Sorensen and Sorensen 1989) and synchrotron energy-dispersive XRD (Johnson et al. 1988), which allowed a much better resolution other than to shed light on the effects of composition on the bulk modulus. Figure 1a shows the volume change, V/V_0 , with the applied P (GPa) for each of the three known icosahedral QC ($i\text{-Al}_{78}\text{Mn}_{22}$, $i\text{-Al}_{74}\text{Mn}_{26}$, and $i\text{-Al}_{86}\text{Mn}_{14}$) for which a K_0 varying between ~ 118 and 148 GPa can be seen. The diagram also shows the compressibility of the two pure metals (Al EoS from Dewaele et al. 2004; Mn EoS from Fujihisa and Takemura 1995) and for the crystalline AlMn alloy (Sato-Sorensen and Sorensen 1989) for comparison. The V/V_0 of the $i\text{-QCs}$ appears related to the compressibility of the pure metals. Noteworthy, $i\text{-QCs}$ result less compressible than the crystalline $\alpha\text{-AlMn}$ (K_0 of ~ 85 GPa; Sato-Sorensen and Sorensen 1989).

Further Al-based QCs are those in the ternary Al–Y–Cu system with $Y = \text{Li, Ru, and Co}$ (Akahama et al. 1989, 1991; Krauss and Steurer 2004). Among them, $\text{Al}_{60}\text{Li}_{30}\text{Cu}_{10}$ possesses a decagonal symmetry, for which the HP behavior was investigated using diamond anvil cell (methanol + ethanol \pm water as pressure medium) both with a conventional powder diffractometer and angle-dispersive

synchrotron radiation up to about 30 GPa. Once again, the bulk moduli of the $i\text{-Al}_{60}\text{Li}_{30}\text{Cu}_{10}$, $i\text{-Al}_{68}\text{Ru}_{17}\text{Cu}_{15}$, and $d\text{-Al}_{60}\text{Li}_{30}\text{Cu}_{10}$ are observed to vary from 70 to 150 GPa and the V/V_0 is shown in Fig. 1b along with the compressibility of pure Co (Fujihisa and Takemura 1996), Cu (Dewaele et al. 2004), Ru (Anzellini et al. 2019), and Li (Hanfland et al. 1999). The dependence of the QCs compressibility from that of the pure constituents appears obvious and can be used to predict preliminarily the physical behavior of a specific QC alloy. Interestingly, Akahama et al. (1989) reported amorphization followed by crystallization in the case of $i\text{-Al}_{60}\text{Li}_{30}\text{Cu}_{10}$. This observation followed the previous report by Parthasarathy et al. (1986) of QC-to-crystal transition suggesting possible metastability of the QC out from their P–T of synthesis. However, amorphization of QCs remains limited to this Li-bearing compositions, such as $i\text{-Al}_{60}\text{Li}_{30}\text{Cu}_{10}$ and crystalline $\text{Al}_5\text{Li}_3\text{Cu}$ (Winters and Hammack 1993).

The AlCuZ ($Z = \text{Fe, Ru, V, Cr, and Mn}$; see Table 1) QCs are all characterized by icosahedral symmetry with bulk modulus comprised between 79 and 155 GPa, as determined using the DAC either with the conventional powder XRD (Sadoc et al. 1998) or in situ energy-dispersive XRD (Ponkratov et al. 2001) up to about 36 GPa, and Si oil and methanol–ethanol employed as pressure media, respectively. For comparison, the EoS of the Z elements (Anzellini et al. 2019, 2022 for Ru and Cr; Ding et al. 2007 for V; Fujihisa and Takemura 1996 for Mn) are shown in Fig. 1c along with the compressibility of $i\text{-Al}_{65}\text{Cu}_{20}\text{Ru}_{15}$, $i\text{-Al}_{62}\text{Cu}_{15.5}\text{Cr}_{12.5}$, $i\text{-Al}_{62}\text{Cu}_{15.5}\text{Mn}_{12.5}$, and $i\text{-Al}_{62}\text{Cu}_{15.5}\text{V}_{12.5}$. Once again, the compressibility of the $i\text{-QCs}$ appears mostly influenced by the elastic behavior of the pure elements. An exception is represented by $i\text{-Al}_{65}\text{Cu}_{20}\text{Ru}_{15}$ and $i\text{-Al}_{62}\text{Cu}_{15.5}\text{Cr}_{12.5}$ as consequence of the high K_0 of the latter (see Table 1). This ternary system also includes the composition of icosahedrite. Experiments at high P on Al–Cu–Fe QCs were reported by Chen et al. (1991), who proved the stability of the QC phase.

The finding of icosahedrite (Bindi et al. 2009) has been of inspiration for the experimental studies conducted on synthetic $i\text{-Al}_{63}\text{Cu}_{24}\text{Fe}_{13}$ at high pressure using in situ angle-dispersive X-ray (ADXRD) assisted by synchrotron radiation (Stagno et al. 2015). These experiments differ from what reported previously in both the higher brilliance of the synchrotron radiation, that allows a better resolution at HP within a wide d -spacing, and the choice of Ne as pressure medium rather than the less hydrostatic Si oil or methanol + ethanol \pm water (Jayaraman 1983). In addition, seven out of eight interplanar distances have been examined including the peaks (12,16) at 5.53 Å and (8,4) at 8.94 Å that are shown to be more compressible over the P range of investigation up to 76 GPa (Stagno et al. 2021) both in compression and decompression. The estimated reduction of the lattice parameter from the ambient pressure value

of 12.64 Å is by $\sim 10\%$. The compressional data on $i\text{-Al}_{63}\text{Cu}_{24}\text{Fe}_{13}$ fitted to a Birch–Murnaghan EoS do not show a significant change of the bulk modulus of 121(8) GPa (K_0' of 3.68 ± 4) at 76 GPa with respect to the 113.7 GPa (K_0' of 4.22) obtained for the same $i\text{-QC}$ compressed to 50 GPa (Stagno et al. 2015). A fit to a unique EoS results to a K_0 of 120(2) GPa and K_0' of 3.87(14). These EoS parameters are slightly smaller than 139 GPa (K_0' of 2.7) for $i\text{-Al}_{62}\text{Cu}_{25.5}\text{Fe}_{12.5}$ proposed by Sadoc et al. (1994). Figure 1d shows the variation of the volume V/V_0 as function of P for the compositions displayed in Table 1 extrapolated to the same P by assuming neither phase transitions nor decompositions for experimental data collected by Takagi et al. (2015) and refitted by Stagno et al. (2021). Such assumption is supported by the lack of evidence of either P-induced structural phase transitions or amorphization in the literature. Further, the fit of integrated data collected up to 104 GPa produces a bulk modulus of 117(3) GPa (K_0' of 4.45 ± 15), which is again consistent with the previous fit by Stagno et al. (2015). On the other hand, the extrapolated V/V_0 for the rhombohedral and pentagonal approximant phases have the same physical behavior at high P, as reflected by the same EoS parameters (Lefebvre et al. 1995).

Unfortunately, no experimental data exist to date to test the stability of decagonal $\text{Al}_{71}\text{Ni}_{24}\text{Fe}_5$ (the analogue of decagonite; Bindi et al. 2015a, b) at high P. The only available data are from Lefebvre et al. (1995) and (Hasegawa et al. 1999a, b) referred to decagonal $\text{Al}_{72}\text{Ni}_{20}\text{Co}_8$ for which a bulk modulus of 155 GPa (K_0' of 2.0) and 120 GPa (K_0' of 5) was proposed, respectively. Additional compositions are reported in Table 1 such as those in the AIPdMn and AIPdRe. AIPdMn was investigated up to 70 GPa in the absence of pressure medium (Hasegawa et al. 1999a) with a quite low bulk modulus (100 GPa and K_0' of 5.3). AIPdRe was compressed up to about 36 GPa in Si oil (Sadoc et al. 1998) for which the high bulk modulus of 180 GPa has been reported.

3 Al-based quasicrystals at high temperatures

The investigation of the behavior of QCs at high T has been classically motivated by the question whether QCs are stabilized by energy or entropy. When scaled to natural scenarios, the fact that QCs can retain their structure to high T would have dramatic implications for their possible occurrence in igneous rocks as well as in post-impact thermal cooling processes proving their stability upon slow cooling over the geological timescale. The discovery of natural QCs opened a scenario where these minerals can play a role in crystallization mechanisms driven by high P and T conditions. Synthetically produced QCs are

known to form by rapid quench from a liquid alloy, as reported by early studies by Shechtman et al. (1984) and by Ball and Lloyd (1985), who faced the issue whether the quasiperiodic structure could be intermediate between amorphous and crystalline. In fact, early HT investigations of QCs seemed to point out that these were metastable phases. At least, this was the case of *i*-Al₈₆Mn₁₄ that, once annealed at T slightly less than 400 °C for 10 min, transformed to orthorhombic Al₆Mn, the *stable* phase (as reported by Chen et al. 1985), with some relics of icosahedral QC, the *metastable* phase. An important step forward for the same QC alloy composition was represented by the observation of congruent melting induced by electron beam irradiation (Knapp and Follstaedt 1987) of both icosahedral and decagonal Al_{85.2–79.5}Mn_{14.8–20.5}, with the melting T of the latter (i.e., 965 ± 20 °C) being higher than that of the icosahedral QC (i.e., 910 ± 20 °C) but both lower than the melting T of the corresponding crystalline analogues. The same year, the solidification of an icosahedral QC with composition Al₆₅Cu₂₀Fe₁₅ was described by Tsai et al. (1987) to be exceptionally stable after annealing for 48 h at 845 °C. The year after, the structural stability of icosahedral Al₆₅Cu₂₀Ru₁₅ was reported along with its stability after annealing for 12 h at ~860 °C (Tsai et al. 1988). These observations were followed by the discovery of single decagonal QCs with compositions of Al₇₅Ni_{9.16}Fe_{9.21} and Al₇₅Ni_{9.16}Co_{9.21} (Tsai et al. 1989) for which a low thermal stability was later reported to be between 847 and 930 °C (Grushko et al. 1996). On the contrary, a similar *d*-QC with composition Al₆₅Cu₁₅Co₂₀ was shown to be stable up to 940 °C by thermal annealing but decomposed to a cubic *fcc* phase when treated with electron beam irradiation (He et al. 1991). These studies show, therefore, a common protocol consisting of quenching the QC from a not well constrained high T owing to the use of arc furnaces followed by the verification of the structural stability upon annealing as a function of T and time.

The Al–Cu–Fe system was investigated in detail at ambient P by Bancel (1999), who showed the coexistence of the icosahedral QC along with crystalline phases, such as β , λ , and ω (already described by Bradley and Goldschmidt 1939) all surrounded the icosahedral region with a narrow stability field. This field ranges Cu–Fe content from 24–12 to 26–13 atom% within ~100 °C up to ~900 °C, beyond which the alloy was proposed to melt incongruently to produce a liquid coexisting with a cubic and/or monoclinic crystalline phases and then being totally molten at ~1000 °C (Gratias et al. 1993; Bancel 1999). The relatively low melting point of the *i*-QC reflects the high Al content, the melting temperature of which is about 660 °C and ambient P. The thermal stability of the ternary Al–Cu–Fe QC allowed to study the lattice parameter a_{6D} to be investigated as a

function of T leading, therefore, to the determination of the thermal expansion coefficient of $12.3 \cdot 10^{-6} \cdot K^{-1}$ (Quivy et al. 1994).

Zhang and Lück (2003a) and Zhang et al. (2005) performed phase equilibria studies of metallurgic interest within the Al–Cu–Fe system between 560 and 900 °C at ambient P under a controlled inert atmosphere. These studies reported 27 solidification reactions from which icosahedral QC never crystallized as liquidus phase except in the case of Al₆₂Cu_{34.5}Fe_{3.5} melt composition that, after a cooling process, resulted in the formation of the icosahedral QC with composition Al_{64.2}Cu_{25.7}Fe_{10.1}.

Zhang and Lück (2003b) also described the solidification sequence for 31 starting compositions in the vicinity of icosahedrite through experiments performed at ambient pressure and temperatures above 1000 °C. Among all these compositions, Al₆₄Cu₂₃Fe₁₃, Al₆₅Cu₂₅Fe₁₀, and Al₆₇Cu_{21.5}Fe_{11.5} are those recalling the composition of icosahedrite. The solidification sequence at 1 atm for these three compositions as function of temperature (although not always clearly stated) is:

1. Liquid (Al₆₄Cu₂₃Fe₁₃) → liquid + λ -phase (Al_{74.7}Cu_{3.9}Fe_{21.4}) → liquid + λ -phase + *i*-QC-phase (Al_{63.6}Cu_{23.8}Fe_{12.6}) → λ -phase + *i*-QC-phase (Al_{63.6}Cu_{23.8}Fe_{12.6});
2. Liquid (Al₆₅Cu₂₅Fe₁₀) → liquid + λ -phase (Al_{74.7}Cu_{3.8}Fe_{21.8}) at 986 °C → liquid + λ -phase + *i*-QC-phase (Al_{65.4}Cu_{25.5}Fe_{12.1}) at 818 °C → liquid + λ -phase + *i*-QC-phase + β -phase (Al_{56.4}Cu_{40.4}Fe_{3.2}) at 671 °C → λ -phase + *i*-QC-phase + β -phase + η -phase (Al_{49.7}Cu_{49.7}Fe_{0.6}) + θ -phase (Al_{68.1}Cu_{31.7}Fe_{0.2}) at 576 °C;
3. Liquid (Al₆₇Cu_{21.5}Fe_{11.5}) → liquid + λ -phase (Al_{74.5}Cu_{3.9}Fe_{21.6}) → liquid + λ -phase + *i*-QC-phase (Al_{66.4}Cu_{21.4}Fe_{12.2}) → liquid + λ -phase + *i*-QC-phase + β -phase (Al_{56.9}Cu_{40.9}Fe_{2.2}) → λ -phase + *i*-QC-phase + β -phase + θ -phase (Al_{67.6}Cu_{32.1}Fe_{0.3}).

These phase equilibria studies and solidification paths, although delivered to the metallurgy community, have represented a useful guide to the interpretation of naturally occurring QCs as well as the run products of HP and T experiments.

It is important to remember that *i*-QCs in nature appear to retain their structure even after experiencing exposure to the highly oxidizing terrestrial atmosphere after their fall on Earth. Because of the high Al content along with other O₂-sensitive elements like Cu and Fe, the *i*-QC can be reasonably considered highly exposed to oxidation. Explorative and preliminary experiments were performed at T between 700 and 1200 °C and ambient P (Table 2) using a synthetic icosahedral quasicrystalline powder as starting material

Table 2 Experimental conditions

Run n	Date	<i>P</i> (GPa)	<i>T</i> (°C)	Time (h)	Starting material	fo ₂	Technique	Notes
GM_A1	22Dec13	0.0001	800	1	AlCuFe	Air	Gas mixing	
GM_A2	22Dec13	0.0001	900	1	AlCuFe	Air	Gas mixing	
GM_A3	23Dec13	0.0001	1000	1	AlCuFe	Air	Gas mixing	
GM_A4	23Dec13	0.0001	700	1	AlCuFe	Air	Gas mixing	
GM_A5	24Dec13	0.0001	1100	1	AlCuFe	Air	Gas mixing	
GM_A6	24Dec13	0.0001	1200	1	AlCuFe	Air	Gas mixing	
GM_A7	24Dec13	0.0001	1100	6	AlCuFe	Air	Gas mixing	

with nominal composition of Al₆₅Cu₂₃Fe₁₂ (purity ≥ 99.9%, Sigma-Aldrich) with negligible amounts of AlFe₃ resulting from the industrial synthesis preparation. The morphology of the starting material is shown in Fig. 2a along with the typical XRD pattern (Fig. 2b) and the forbidden QC rotational symmetry (Fig. 2c). Experiments were performed using a Deltech furnace and the starting powder loaded into a Pt capsule opened on the top side and placed in the hot-spot portion of the furnace. The temperature during the experiments was monitored with a Pt/Pt-10%Rh (S-type) thermocouple inserted within an alumina sleeve positioned near the top of the capsule. The samples were kept at constant target temperature for a period of 1 and 6 h. Each run was quenched by dropping the capsule into liquid nitrogen. The recovered run products (Fig. 3a–f) along with the X-ray diffraction patterns in Fig. 4 reveal a gradual exsolution of an alumina-rich layer that rims the icosahedrite particles. Powder X-ray diffraction data confirm that the structure of the starting material is preserved with the increase of temperature well above that proposed by Bancel (1999). These results shed light on the oxidation of synthetic icosahedrite consisting in the formation of refractory Al₂O₃. Further experiments consisting of heat treatment at variable oxygen fugacity would be ideal to explore the O-dependence on the formation of additional phases and the effect on the melting *T*.

4 Quasicrystals at high temperature and pressure

Despite the important role that *T* might have on structural changes of QCs and their physical properties, to date, the investigation of the stability of QCs at simultaneous HP and HT has been very limited. Both natural Al₆₃Cu₂₄Fe₁₃ icosahedrite (Bindi et al. 2009) and Al₇₁Ni₂₄Fe₅ decagonite (Bindi et al. 2015a, b) were predicted by high-temperature studies within the Al–Cu–Fe and Al–Ni–Fe system (Bancel 1999; Tsai et al. 1989; Lemmerz et al. 1994). In addition, these natural QCs are accepted to have experienced HP during shock-impact events.

A few studies describe the synthesis and stability of *i*-QC at relatively low temperature (350–840 °C) and low pressure (uniaxial compression testing; Kang and Dubois 1992) up to 1.64 GPa (Turquier et al. 2004), which cannot be directly applied to explain the occurrence of icosahedrite in the Khatyrka meteorite where typical HP-*T* minerals (i.e., stishovite and ahrensite) were found. A first indication that comes from the observation of natural QCs is their capacity to retain the icosahedral and decagonal structure at extreme conditions. Recent experimental simulations allowed to understand whether *i*-AlCuFe retains icosahedral symmetry at high *P* and *T* or, rather, it transforms either into a periodic approximant (Lefebvre et al. 1995; Quiquandon et al. 1996) or other crystalline phases (Gratias et al. 1993; Barbier et al. 1993). Subsequent experimental HP-*T* data conducted using both the multi-anvil apparatus and the diamond anvil cell either ex situ or in combination with synchrotron radiation allowed to monitor the structural evolution during compression/heating in real time. Stagno et al. (2014) reported the first experiment at hydrostatic *P* of 5 GPa and *T* up to 1500 °C using the multi-anvil apparatus combined with the in situ synchrotron radiation at Spring8 (Japan). The starting material used in this study was the synthetic quasicrystalline powder mentioned above with variable grain size ≤ 25 μm and composition of Al₆₅Cu₂₃Fe₁₂, quite similar to icosahedrite (Al₆₃Cu₂₄Fe₁₃), and both falling into the stability field of the icosahedral phase reported by Bancel (1999). Their results, supported by X-ray diffraction (in energy-dispersive mode and single-crystal XRD on the recovered QC sample), chemical and textural analyses (suggesting the role of *P* in increasing the melting *T* with no approximants formed) provided evidence of the structural stability of synthetic icosahedrite at 5 GPa up to 1400 °C. In addition, the obtained thermal expansion coefficients at 5 GPa of 4.67·10⁻⁵ A °⁻¹·K⁻¹ and 5.22·10⁻⁸ A⁻¹·K⁻¹, resulted only slightly different than those determined by Quivy et al. (1994) at ambient *P*.

A further important achievement is represented by the investigation of synthetic icosahedrite at simultaneous HP and *T* using the diamond anvil cell combined with a double-sided laser heating system assisted by synchrotron radiation

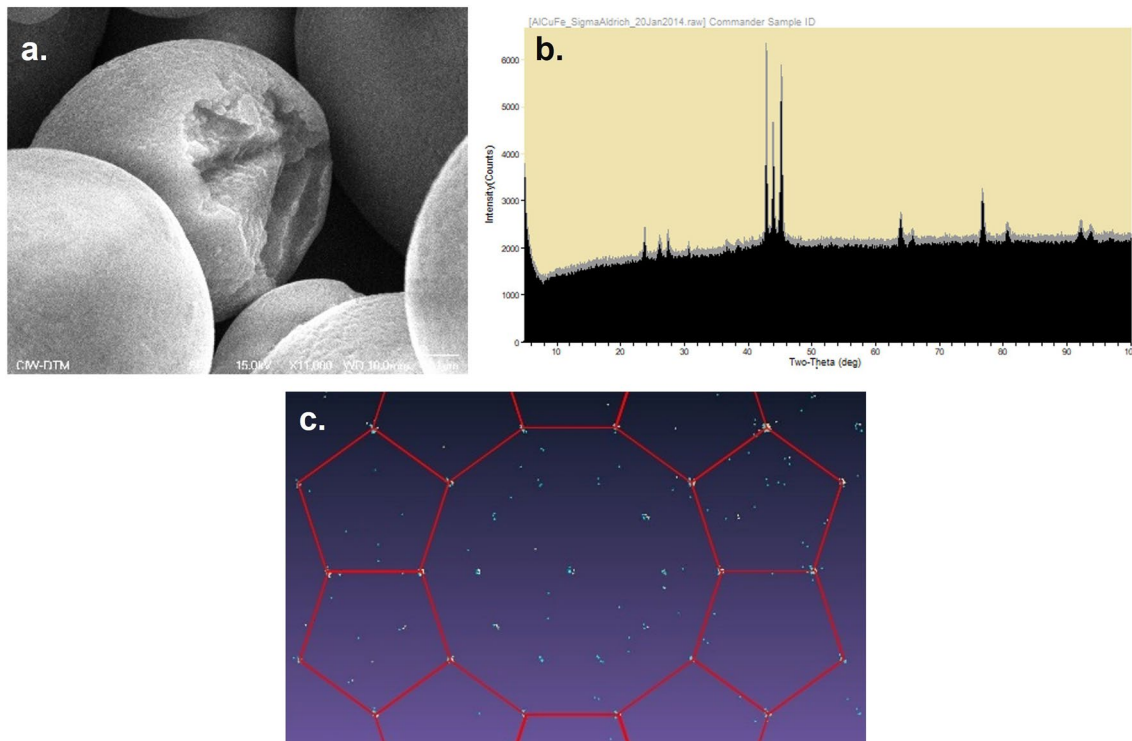


Fig. 2 Shown are **a** the morphology of the starting material by secondary electrons; **b** typical XRD pattern of synthetic $\text{Al}_{65}\text{Cu}_{23}\text{Fe}_{12}$; and **c** the forbidden QC rotational symmetry collected on the same

polycrystalline powder used as starting material for HP-T experiments by Stagno and coauthors

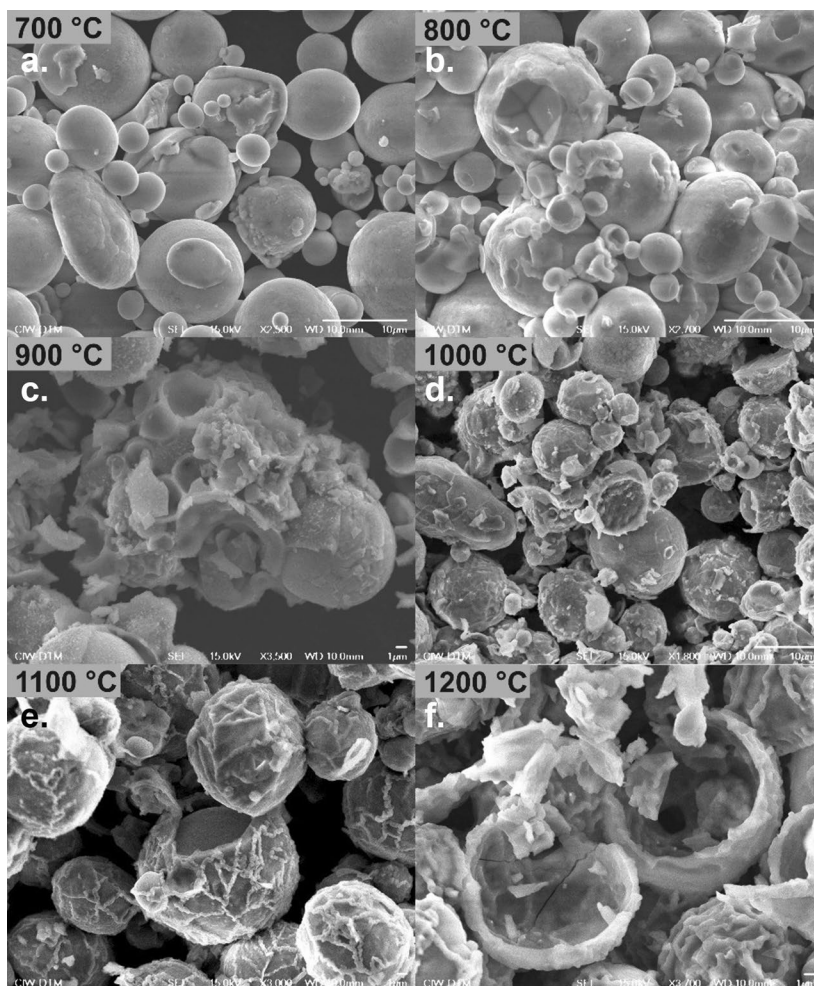
at HPCAT and GSECARS beamlines of Argonne National Laboratory (Chicago, US) described by Stagno et al. (2015). In these experiments, performed using Ne as pressure medium and angle-dispersive X-ray diffraction, the synthetic quasicrystalline powder was first compressed to ~ 42 GPa and then heated to ~ 1560 °C while collecting X-ray diffraction patterns to monitor any possible transformation or melting. The sample was then cooled down to 730 °C before being quenched. Stagno et al. (2015) reported neither amorphization nor phase transformation, rather the presence of the characteristic diffraction peaks including those at low theta-angles useful to calculate the six-dimensional lattice parameter (Bindi et al. 2011). The same result was observed after heating up to ~ 1837 °C and then quenched directly to room-temperature. The confirmation that the *i*-QC survived to such HP and T was given by the evidence of the fivefold symmetry obtained by ex situ single-crystal X-ray diffraction measurements on a micrometer-sized grain handpicked from the open cell.

A more extensive study is represented by the investigation of the phase equilibria within the Al–Cu–Fe system where synthetic $\text{Al}_{65}\text{Cu}_{23}\text{Fe}_{12}$ was used as starting material for experiments performed using the multi-anvil press within a P range of 3, 5, and 21 GPa and T from 800 °C to 1700 °C. Here, the results can be summarized with the

i-QC being stable at 3 GPa from 800 °C to 1100 °C at least in equilibrium with quenched phases that resemble those found in Khatyrka like (Fe-bearing) stolperite, cupalite, and khatyrkite \pm a melt. At 5 GPa, the stability of icosahedrite is confirmed at 1200 °C along with the same additional Al-bearing phases, although we cannot exclude that *i*-QC could be stable at 1200 °C $\leq T < 1500$ °C. At 21 GPa, the quasicrystal was recovered from runs up to 1500 °C along with the presence of both Al-bearing and liquid phases. Some important aspects are necessary to point out as the following: (1) the successful synthesis of an icosahedral QC phase at 5 GPa and 1200 °C starting from a mixture of pure Al, Cu, and Fe in relative ratios typical of icosahedrite (run PR1205r); (2) the observation of two icosahedral QCs very similar in composition (runs PR1197 and PR1205r) that matches with the occurrence of the two icosahedral QCs reported by Bindi et al. (2016) as $\text{Al}_{62.64}\text{Cu}_{24.11}\text{Fe}_{13.26}$ and $\text{Al}_{65}\text{Cu}_{20.81}\text{Fe}_{14.19}$; (3) pressure acts to stabilize the QC structure to higher T as indicated by the positive Clapeyron–Clausius slope in Fig. 6 of Stagno et al. (2017).

Another important achievement is represented by a revision of the solidification sequence proposed by Zhang and Lück (2003b). Stagno et al. (2017) proposed the following reactions sequence from 1400 to 800 °C at 3 GPa:

Fig. 3 a–f Images of the run products listed in Table 2 taken with the secondary electrons suggest the exsolution starting at 900 °C of an alumina-rich layer that rims the icosahedrite particles



Liquid ($\text{Al}_{65}\text{Cu}_{23}\text{Fe}_{12}$) [$T > 1400$ °C] \rightarrow (unconstrained) \rightarrow Liq + Fe-khatyrkite ($\text{Al}_{65.6}\text{Cu}_{19}\text{Fe}_{15.4}$) + stolperite ($\text{Al}_{48.8}\text{Cu}_{45.9}\text{Fe}_{5.36}$) + cupalite ($\text{Al}_{43.9}\text{Cu}_{54.7}\text{Fe}_{1.4}$) at 1400 °C \rightarrow Liq + Fe-khatyrkite ($\text{Al}_{66.6}\text{Cu}_{17.7}\text{Fe}_{15.7}$) + stolperite ($\text{Al}_{49.9}\text{Cu}_{44.1}\text{Fe}_{6.0}$) at 1200 °C \rightarrow Liq + Fe-khatyrkite ($\text{Al}_{66.6}\text{Cu}_{17.8}\text{Fe}_{15.6}$) + stolperite ($\text{Al}_{52.2}\text{Cu}_{40.1}\text{Fe}_{7.7}$) + icosahedrite ($\text{Al}_{60.6/62.2}\text{Cu}_{26.2/21.9}\text{Fe}_{13.2/15.9}$) at 1100 °C \rightarrow stolperite ($\text{Al}_{53.8}\text{Cu}_{40.2}\text{Fe}_{6.0}$) + icosahedrite ($\text{Al}_{66.5}\text{Cu}_{20.2}\text{Fe}_{13.3}$) at 800 °C.

The proposed reaction sequence from 1500 to 1200 °C at 5 GPa is:

Liquid ($\text{Al}_{65}\text{Cu}_{23}\text{Fe}_{12}$) [$T > 1500$ °C] \rightarrow (unconstrained) \rightarrow Liq + Fe-khatyrkite ($\text{Al}_{65.2}\text{Cu}_{19.5}\text{Fe}_{15.3}$) + stolperite ($\text{Al}_{48.2}\text{Cu}_{45.5}\text{Fe}_{6.4}$) at 1400 °C \rightarrow stolperite ($\text{Al}_{49.7}\text{Cu}_{44.2}\text{Fe}_{6.1}$) + icosahedrite ($\text{Al}_{65/62.6}\text{Cu}_{20.8/24.1}\text{Fe}_{14.2/13.3}$) [+ cupalite ($\text{Al}_{44.5}\text{Cu}_{53.9}\text{Fe}_{1.6}$) + Fe-khatyrkite ($\text{Al}_{67.2}\text{Cu}_{9.6}\text{Fe}_{23.2}$)] at 1200 °C.

Finally, the proposed reaction sequence from 1700 to 1400 °C at 21 GPa is:

Liquid ($\text{Al}_{65}\text{Cu}_{23}\text{Fe}_{12}$) at $T > 1700$ °C \rightarrow Liq ($\text{Al}_{12.2}\text{Cu}_{21.6}\text{Fe}_{66.2}$) + Al at 1700 °C \rightarrow Liq + Fe-rich cupalite ($\text{Al}_{48.8}\text{Cu}_{36.1}\text{Fe}_{15.1}$) + icosahedrite ($\text{Al}_{64.7}\text{Cu}_{20.2}\text{Fe}_{15}$) + aluminum [$\text{Al}(\text{Cu},\text{Fe})$] at 1500 °C \rightarrow stolperite ($\text{Al}_{52.8}\text{Cu}_{31.7}\text{Fe}_{15.3}$) + icosahedrite ($\text{Al}_{68.2}\text{Cu}_{22.4}\text{Fe}_{9.4}$) at 1450 °C \rightarrow icosahedrite ($\text{Al}_{65.5}\text{Cu}_{23.3}\text{Fe}_{11.2}$) at $1450 > T > 1400$ °C (7).

It must be emphasized that the experimental studies producing icosahedrite performed by multi-anvil and DAC lasted from 5 to 300 min at high P and T implying, therefore, the kinetics stability of the QC structure. This interval is well beyond that applied in experiments where the QC was either annealed or irradiated by an electron beam. Dynamic experiments certainly help to reproduce mechanism like shock impact at the origin of QC formation; however, static experiments carried out using the diamond anvil cell and multi-anvil presses are necessary to monitor structural changes under HP-T, breakdown reactions, and melting relations at HP-T. Considering the possible origin of QCs in hypervelocity impacts in outer space, both *i*-AlCuFe quasicrystal and *d*-QC were reported as run products of shock experiments during

Fig. 4 The powder X-ray diffraction patterns relative to the run products quenched from 700 to 1200 °C confirm that the crystallinity of the quasicrystalline starting material is preserved at high T under oxidized atmosphere

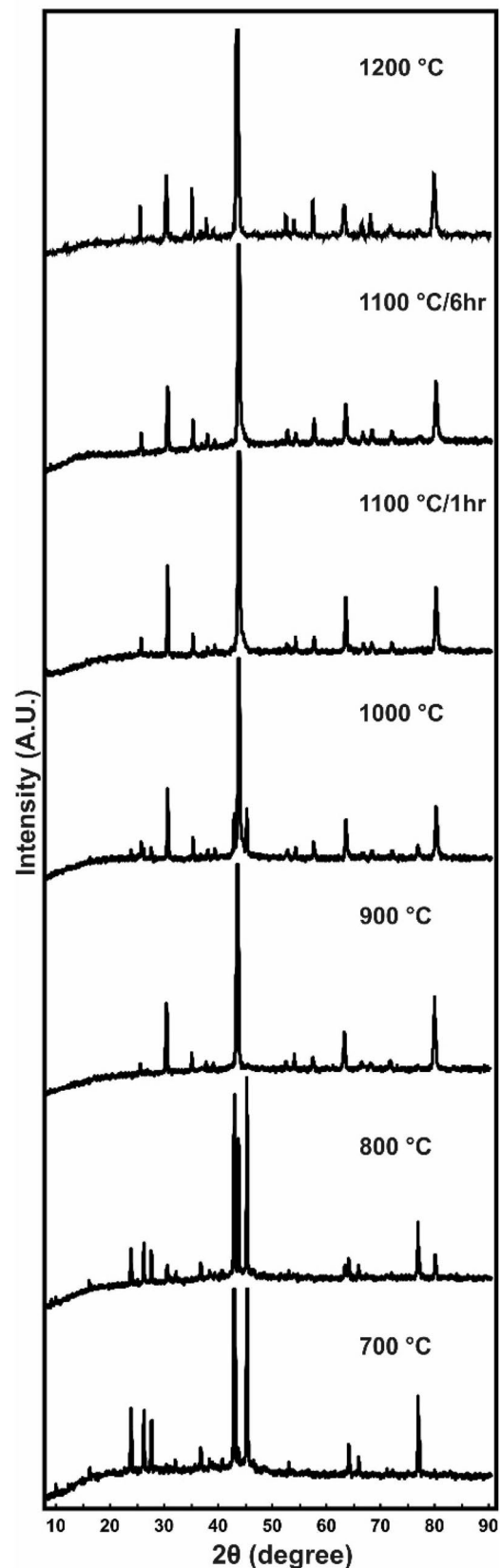
which shock pressures up to 35 GPa were generated at T of 400 °C within less than 1 μ s (Asimow et al. 2016; Oppenheim et al. 2017a, b; Hu et al. 2020). Within such a short time, both the textural contacts between the quasicrystalline phases and the Al–Cu–Fe alloys as well as their chemical compositions were reproduced as occurring in the Khatyrka meteorite. In addition, the recovered Al–Cu–Fe QCs hosted additional elements like Cr and Ni, that represents important evidence of the stability of QCs even beyond the compositional field previously proposed by Bancel (1999).

5 Further remarks

If, on one side, the last 4 decades have seen a slightly decreased interest for the study of QCs in terms of synthesis within various systems and their possible applications (Steurer 2018; Dubois 2023), the investigation of the structural stability of these materials as function of P and T remains limited to the studies reported in Table 1. From the geoscience perspective, the finding of decagonite ($\text{Al}_{71}\text{Ni}_{24}\text{Fe}_5$; Bindi et al. 2015a, b; Buganski and Bindi 2021), a second icosahedral quasicrystal ($\text{Al}_{62}\text{Cu}_{31.2}\text{Fe}_{6.8}$; Bindi et al. 2016) and proxidecagonite, $\text{Al}_{34}\text{Ni}_9\text{Fe}_2$, the first natural known periodic crystalline approximant to decagonite ($\text{Al}_{71}\text{Ni}_{24}\text{Fe}_5$; Bindi et al. 2018), all would benefit from high P studies to determine their elastic parameters as well as their behavior when compressed and heated. Similar investigations should be undertaken on those minerals reported to coexist with the natural QCs like stolperite, hollisterite, kryachkoite, cupalite, and khatyrkite. These studies would be a guide to accurately determine the P–T conditions for the stability of QCs in nature and the mechanisms for their formation. Afterall, the presence of QCs has been also proposed in more common scenarios like sedimentary rocks as consequence of the recrystallization when clay and salt react (Kharitonova et al. 2015).

Additional open questions remain on the physical and chemical properties of the liquid coexisting with the QCs or from which QCs form. Are the interatomic distances retained from solid QCs to liquids and back? Such an answer would help to understand whether QCs might have formed from a liquid alloy that migrated to the center of a planetesimal to form its core.

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Data availability Data are available

Declarations

Conflict of interest The authors declare no competing interests.

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