High-pressure neutron powder diffraction study on arsenolite deuterium inclusion compound: structure and formation kinetics

Supplementary Information

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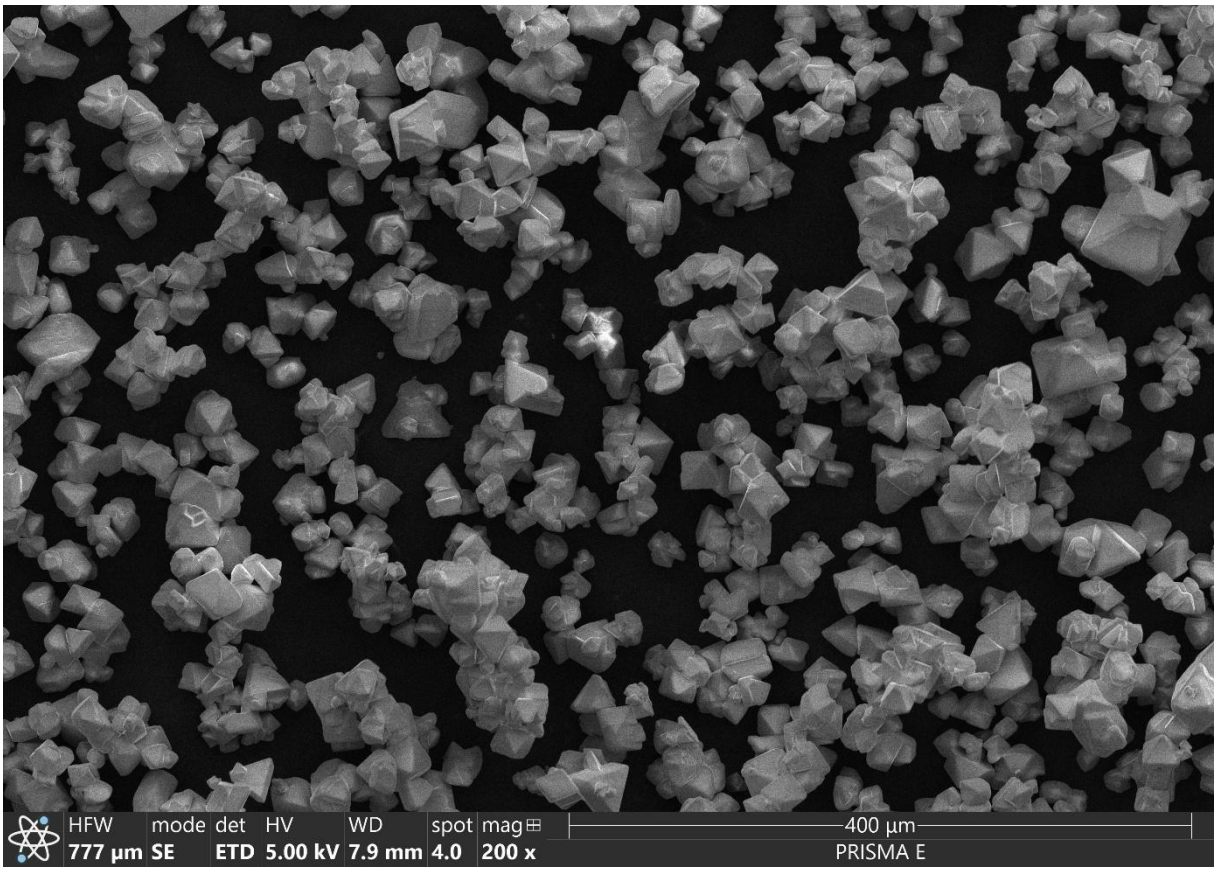

Figure S1. Scanning electron micrograph of the arsenolite powder used for the experiments presented in the article.

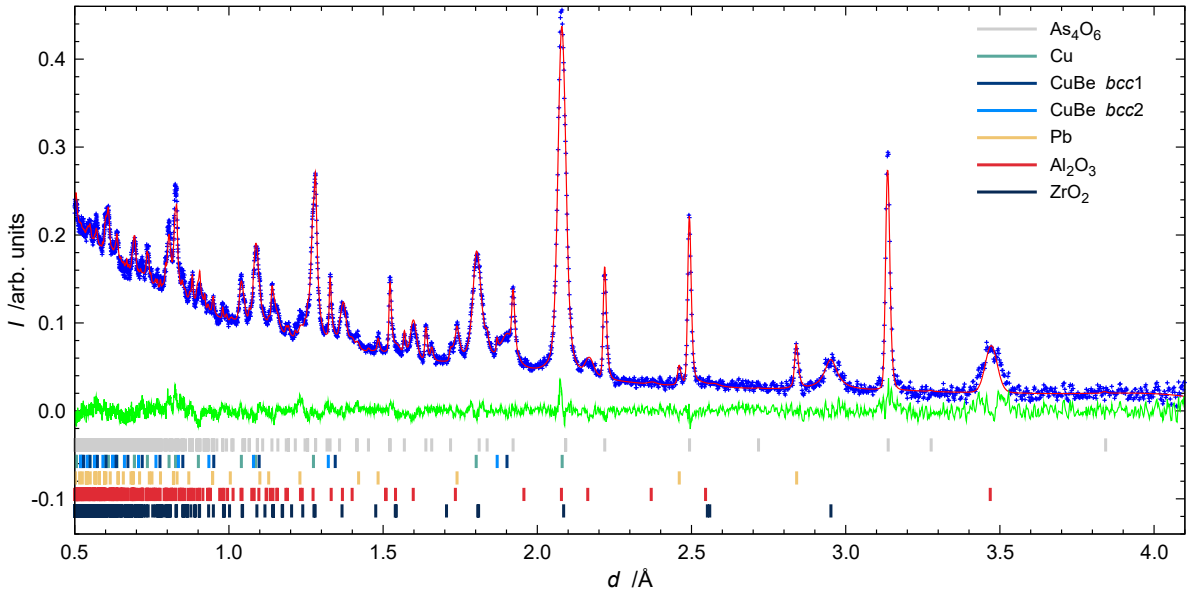

Figure S2. TOF NPD recorded at 295 K and 0.81(2) GPa. Blue crosses, red and green lines correspond to experimental points, Rietveld fit curve, and the difference between the two, respectively. Ticks at the bottom denote the position of reflections of the phases listed in the legend. R_{wp} = 4.56%; GoF = 0.90.

Table S1. Unit-cell parameters and volumes of arsenolite and its inclusion compound with hydrogen, deuterium, and helium, determined at room temperature.

p /GPa	X	$a(As_4O_6 \cdot 2X)$ /Å	$V(As_4O_6 \cdot 2X)$ /Å^3
2.07(5)	H_2	11.087[a]	1362.1[a]
1.96(2)	D_2	11.0825(6)	1361.2[b]
3.56(5)	He	10.710[b]	1228.5[c]

[a] Data from ref. 4.
[b] This study.
[c] Data from ref. 3.

Table S2. Unit-cell parameters and volumes of arsenolite and its inclusion compound with deuterium, determined by Rietveld refinement in this study. The measurements are listed in chronological order.

T /K	*p* /GPa	$a(As_4O_6)$ /Å	$V(As_4O_6)$ /Å^3	$a(As_4O_6 \cdot 2D_2)$ /Å	$V(As_4O_6 \cdot 2D_2)$ /Å^3
295(1)	0.809(18)	10.8692(3)	1284.09(10)	-	-
295(1)	1.56(2)	10.7454(13)	1240.7(4)	11.1311(9)	1379.1(3)
295(1)	1.96(2)	10.6518(4)	1208.49(16)	11.0823(6)	1361.1(2)
250(1)	1.93(5)	-	-	11.0904(12)	1364.1(4)
200(1)	1.78(3)	-	-	11.0899(11)	1363.9(4)
150(1)	1.80(4)	-	-	11.0801(11)	1360.3(4)
120(1)	1.68(2)	-	-	11.0792(7)	1359.9(3)
120(1)	0.76(2)	-	-	11.2789(8)	1434.8(3)
120(1)	0.57(5)[a]	-	-	11.2811(19)	1435.7(7)
150(1)	0.75(5)	-	-	11.2770(9)	1434.1(3)
200(1)	0.56(3)	10.9539(10)	1314.3(4)	-	-
250(1)	0.05(4)	11.0287(10)	1341.5(4)	-	-
290(1)	0.21(2)	11.0033(8)	1332.2(3)	-	-

[a] after 36 h

Table S3. Refinement indicators for different structural models of arsenolite inclusion compound with deuterium $As_4O_6 \cdot 2D_2$ at 1.96(2) GPa and 295 K. <*uvw*> denotes the crystallographic directions along which the molecules are aligned, whereas “free rot” denotes a model with freely rotating D_2 molecules

model	R_{wp}	GoF
<111>	3.36%	1.62
<110>	3.40%	1.65
<100>	3.47%	1.68
free rot	3.49%	1.69

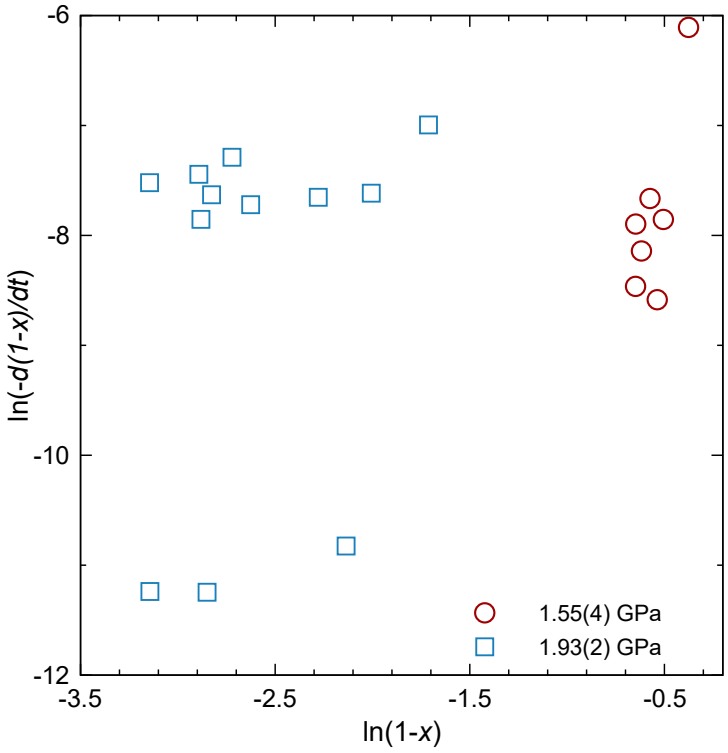

Figure S3. $\ln(-d(1-x)/dt)$ plotted as a function of $\ln(1-x)$, where x and t are conversion factor and time, respectively.

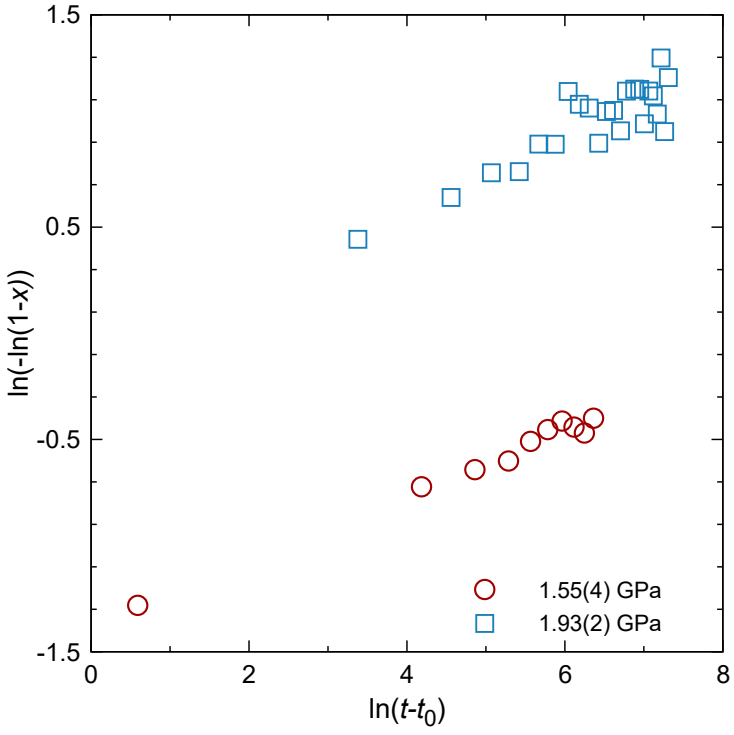

Figure S4. $\ln(-\ln(1-x))$ plotted as a function of $(t-t_0)$, where x, t and t_0 are conversion factor, time and the time between the reaction start and the beginning of diffraction measurements, respectively.