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Temperature dependent structural studies of β-pyrochlore KOs_2O_6

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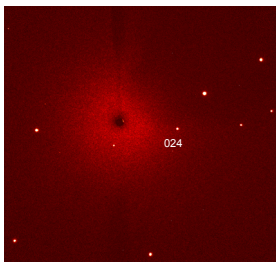
Temperature scans of selected Bragg reflections at BM01A SNBL/ESRF



Above: KOs_2O_6 at 300K; T scan; exposure time: 10 sec, Ω range: 3°; Phi range: 89° - 92°; $Phi_{024,100K}$ = 90.25°; $Phi_{024,100K}$ = 90.58°.

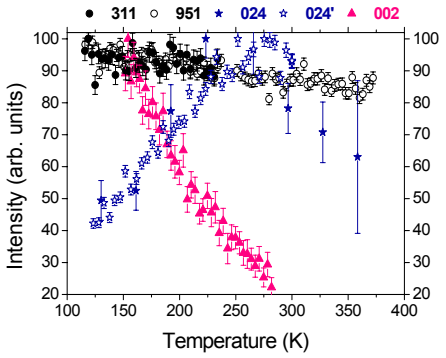
Scaling (maximum 100) and monitor correction for all scanned reflections. Phi range optimised for all scans.

→ "Normal" behavior of the "Fd$\bar{3}$m" reflections

All reflections of the 024 type (each measured in different Phi ranges) show similar intensity variation as a function of temperature

→ 024: maxima at 300 K, possible decrease of intensity towards higher temperatures, drastic decrease towards lower temperatures

→ 002: only one single temperature scan for 002 type reflections; 80 % increase of intensity from 280 K towards lower temperatures



Simulation of very small Fd$\bar{3}$m → F$\bar{4}$3m symmetry breaking

a) Starting point is a pseudo Fd$\bar{3}$m setting with F$\bar{4}$3m symmetry (simulation using PowderCell-Software):

x_{Os} = 0.875 and $U_{iso,\,Rb1} = U_{iso,\,Rb2}$

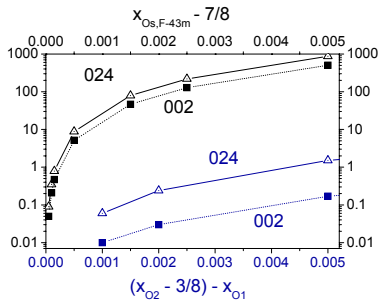
x_{O2}-3/8 = x_{O1} and $U_{iso,\,O1} = U_{iso,\,O2}$

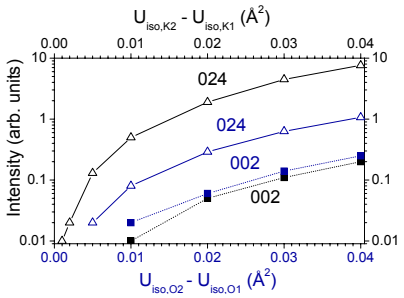
Intensity of "forbidden" 002 type and 024 type reflections is zero.

b) Breaking pseudo Fd$\bar{3}$m symmetry

(I) $x_{Os} \neq 0.875$ and (II) $U_{iso,\,Rb1} \neq U_{iso,\,Rb2}$

(III) x_{O2}-3/8 ≠ x_{O1} and (IV) $U_{iso,\,O1} \neq U_{iso,\,O2}$





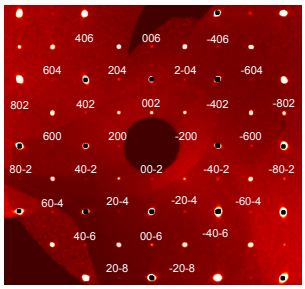
Crystal structure refinement at 100, 150 and 400 K

Superconducting (T_c= 9.6 K) β-pyrochlore KOs_2O_6 single crystals has been synthesized by encapsulation techniques [1].

Detailed single crystal X-ray diffraction studies with a home source at 300K [1] and with synchrotron radiation at the BM01A beamline at SNBL/ESRF (Grenoble), using a ONYX CCD system (λ = 0.72184 Å), at 100, 150 and 400 K [present work] show Bragg peaks (like 002 and 024) that violate Fd$\bar{3}$m symmetry.

The crystal structure of β-pyrochlore KOs_2O_6 between 100 K and 400 K is identified as **non-centrosymmetric** with space group **F$\bar{4}$3m**.

SHELXL/SADABS refinement BM01a SNBL/ESRF			
T (K)	100	150	400
a (Å)	10.0833(4)	10.0872(4)	10.1040(3)
V (Å³)	1025.2(2)	1026.4(2)	1031.5(2)
Exposure (sec)	4-8	4-8	10
Filter / Ω	no / 1°	no / 1°	robin 2 / 3°
Reflections	3928	3441	5441
Unique reflec.	437	430	436
Parameters	16	16	16
R_{int}	0.0641	0.0717	0.0680
R_{sig}	0.0298	0.0375	0.0257
R_1	0.0312	0.0345	0.0245
R_w	0.0650	0.0723	0.0543

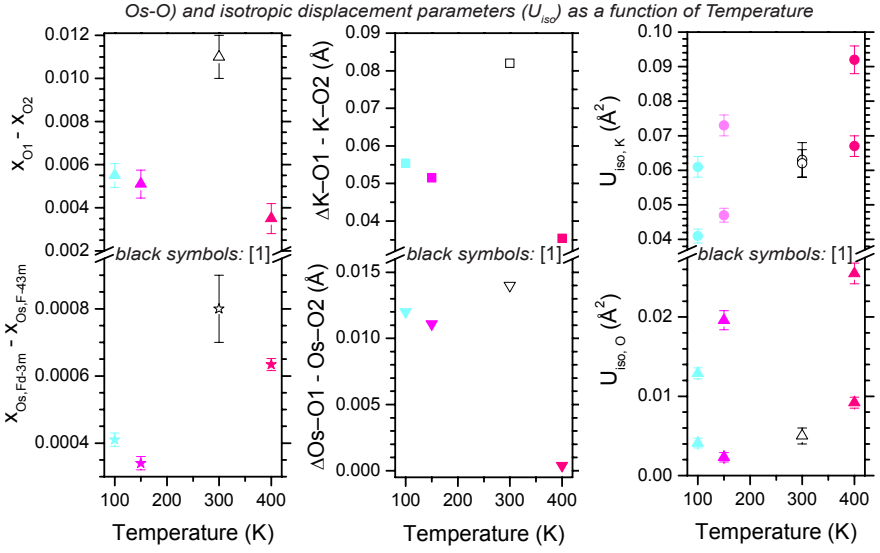


Above: KOs_2O_6 at 100K; reconstructed [h0l] layer; exposure time: 4 - 8 sec.

KOs_2O_6 F$\bar{4}$3m (No. 216)				
Atom	**Site**	**x**	**U_{iso}**	
Os	16e	0.87541(2)	0.00396(4)	100
		0.87534(2)	0.00452(5)	150
		0.87563(2)	0.00690(4)	400
K1	4c	1/4	0.041(2)	100
		1/4	0.047(2)	150
		1/4	0.067(2)	400
K2	4b	1/2	0.061(3)	100
		1/2	0.073(3)	150
		1/2	0.092(4)	400
O1	24f	0.1806(5)	0.0041(6)	100
		0.1815(4)	0.0023(6)	150
		0.1820(5)	0.0092(7)	400
O2	24g	0.5611(6)	0.0129(9)	100
		0.5616(8)	0.0196(12)	150
		0.5605(9)	0.0255(13)	400

Os: y, z = x; Rb1: y, z = 1/4; Rb2: y, z = 1/2; O1: y, z = 0; O2: y, z = 1/4, black symbols: [1]

Below: atomic coordinates (x), selected interatomic distance differences Δ (K-O and Os-O) and isotropic displacement parameters (U_{iso}) as a function of Temperature



Reference: [1] G.Schuck, S. M. Kazakov, K. Rogacki, N. D. Zhigadlo and J. Karpinski, Phys. Rev. B, 73 (2006) 144506

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