Crystal structure of terbium antimonide oxide, $\text{Tb}_9\text{Sb}_5\text{O}_5$

Jürgen Nuss and Martin Jansen*

Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, 70569 Stuttgart, Germany

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ture profile was applied: 298 → 1923 K (50 Kh⁻¹, subsequent annealing for 12 h); 1923 → 1673 K (20 Kh⁻¹, subsequent annealing for 48 h); 1673 → 298 K (20 Kh⁻¹).

Discussion

$\text{Tb}_9\text{Sb}_5\text{O}_5$ crystallize in the $\text{La}_9\text{Sb}_5\text{O}_5$ type of structure [1,2]. The atoms Tb2, Tb3 and Sb1, Sb2 are arranged in a corrugated double layer [TbSb], which can be regarded as a two-dimensional slab analogous to NaCl. Each [TbSb] fragment is sandwiched between two layers built from Tb1 and O1, O2. The NaCl-like double sheet is also a feature common to the structures of $\text{Eu}_4\text{Sb}_2\text{O}$ [3] or Sc_2Sb [4]. In the latter compound the interlayer exclusively consists of Sc atoms. In the case of $\text{Tb}_9\text{Sb}_5\text{O}_5$ this layer is composed of Tb and O atoms with the composition Tb_4O_5 , and only 4/5 of the possible metal positions are occupied by terbium. Therefore, the structure can be regarded as a defect variant of Sc_2Sb , stuffed with oxygen: $\text{Tb}_9\text{Sb}_5\text{O}_5 = \text{Sc}_{10}\text{Sb}_5\text{O}_5$. From the originally square Sc_5 pyramids, with their tips alternating up and down along the [001] direction, 4/5 transform into tetrahedra and 1/5 remain unchanged. All tetrahedral and square-pyramidal holes are filled with O atoms. The partial structure (Tb_9Sb_5) also corresponds to the inverted Hf_5Sb_9 type of structure [5]. The buckling of the NaCl-like slabs is one way to fit the dimensions of their meshes to the sandwiched sheets inbetween [6]. Here in $\text{Tb}_9\text{Sb}_5\text{O}_5$, the present of vacancies in the Tb_4O_5 sublattice, allows an additional adjustment, which is needed because of $d(\text{Tb}-\text{Sb}) = 3.143 \text{ \AA} < 2^{1/2} d(\text{Tb}-\text{O}) = 3.325 \text{ \AA}$.

Table 1. Data collection and handling.

Crystal:	dark silver block, size $0.01 \times 0.02 \times 0.02 \text{ mm}$
Wavelength:	Mo K_α radiation ($0.71073 \text{ \AA}$)
μ :	441.78 cm^{-1}
Diffractometer, scan mode:	SMART APEX II, Bruker AXS, ω
$2\theta_{\text{max}}$:	74.4°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	16398, 2211
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1615
$N(\text{param})_{\text{refined}}$:	47
Programs:	SHELXS-97 [7], SHELXL-97 [8], ATOMS [9]

Abstract

$\text{O}_5\text{Sb}_5\text{Tb}_9$, tetragonal, $P4/n$ (no. 85), $a = 9.862(1) \text{ \AA}$, $c = 8.838(2) \text{ \AA}$, $V = 859.5 \text{ \AA}^3$, $Z = 2$, $R_{\text{gt}}(F) = 0.030$, $wR_{\text{ref}}(F^2) = 0.049$, $T = 296 \text{ K}$.

Source of material

$\text{Tb}_9\text{Sb}_5\text{O}_5$ has been synthesized from terbium metal, antimony, and Sb_2O_3 . Stoichiometric amounts of the starting materials were mixed and sealed in a tantalum ampoule. The following tempera-

* Correspondence author (e-mail: M.Jansen@fkf.mpg.de)

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Tb(1)	8g	0.33120(2)	0.02250(2)	0.99800(3)	0.0049(1)	0.0073(1)	0.00766(9)	0.00007(8)	−0.00054(9)	0.00030(9)
Tb(2)	2c	¼	¼	0.33136(6)	0.0093(1)	U ₁₁	0.0057(2)	0	0	0
Tb(3)	8g	0.14585(3)	−0.04496(3)	0.65470(3)	0.0090(1)	0.0090(1)	0.0055(1)	−0.00048(9)	0.00012(9)	−0.00048(9)
Sb(1)	2c	¼	¼	0.68537(9)	0.0071(2)	U ₁₁	0.0109(3)	0	0	0
Sb(2)	8g	0.15426(4)	−0.05428(3)	0.30038(4)	0.0071(2)	0.0079(2)	0.0067(1)	−0.0003(1)	0.0004(1)	0.0001(1)
O(1)	2c	¼	¼	0.067(1)	0.012(2)	U ₁₁	0.023(5)	0	0	0
O(2)	8g	0.1284(4)	−0.0239(4)	0.9117(4)	0.009(2)	0.007(2)	0.008(2)	−0.001(1)	0.000(1)	−0.000(1)

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