

## PREPARATION OF TERBIUM MONOSULFIDE THIN CRYSTALLINE FILM

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A process is described for the growth of thin crystalline TbS films ranging in thickness from 0,3 to 1,8  $\mu\text{m}$  by flash vacuum thermal evaporation from preliminary synthesized bulk crystal of TbS. The films was grown on glass-ceramic, fused silica, sapphire and (111) single-crystal silicon. Thin films had cubic crystal structure (NaCl structure type) with lattice parameters  $a = 5,52 \text{ \AA}$ .

**Keyword:** thin film, vacuum thermal evaporation, bulk crystal, lattice parameters.

Разработана технология приготовления тонких кристаллических плёнок толщиной 0,3–1,8 мкм методом взрывного вакуумно-термического испарения предварительно синтезированного объёмного кристалла TbS. Подложками служили пластины из ситала, кварца, сапфира и монокристаллического кремния с ориентацией (111). Плёнки имели кубическую решётку (структурный тип NaCl) с параметром решётки  $a = 5,52 \text{ \AA}$ .

**Ключевые слова:** тонкая плёнка, вакуумно-термическое испарение, объёмный кристалл, параметр решётки.

Розроблено технологію готування тонких кристалічних плівок товщиною 0,3 – 1,8 мкм методом вибухового вакуумно-термічного випару попередньо синтезованого об'ємного кристала TbS. Підкладками слугували пластини із ситалу, кварцу, сапфіра й монокристалічного кремнію з орієнтацією (111). Плівки мали кубічні ґрати (структурний тип NaCl) з параметром ґрат  $a = 5,52 \text{ \AA}$ .

**Ключові слова:** тонка плівка, вакуумно-термічний випар, об'ємний кристал, параметр ґрат.

## INTRODUCTION

Sulfides of rare – earth elements have interesting thermal, magnetic, optical and other properties [1 – 11]. However not all these compound are studied well enough. Such a little studied compounds concerns monosulfide of terbium.

The purposes of the presented work was working out of technology of preparation of the crystal films of terbium monosulfide.

## EXPERIMENTAL

The crystalline TbS films ranging in thickness from 0,3 to 1,8  $\mu\text{m}$  were grown by flash vacuum thermal evaporation from preliminary synthesized bulk crystal of TbS.

The films were grown on glass-ceramic, fused silica, sapphire and (111) single-crystal silicon substrates  $15 \times 8 \times 1 \text{ mm}$  in dimentions. These substrate materials were shown to have an insignificant effect on the phase composition and crystallinity of the films. During the growth process, the vacuum in the deposition chamber was maintained at  $10^{-6} \text{ Pa}$ . The substrate temperature was varied in our experiment from 750 to 1250 K, the deposition rate – from 35 to 65  $\text{\AA}/\text{sec}$ .

The phase composition and crystallinity of the films were checked by X-ray diffraction (Ni-filtered  $\text{CuK}_{\alpha}$  radiation, continuous scan mode with a scan rate of  $4 \cdot 10^{-2} \text{ deg/S}$ ) and electron diffraction. Electron diffraction patterns were obtained in reflection at an accelerating voltage of  $(75 - 100) \cdot 10^2 \text{ V}$ . The surface of the films was examined by X-ray fluorescence analysis (Camebax-Microbeam system). Their elemental analysis was determined by electron X-ray microanalysis.

## RESULTS AND DISCUSSION

By X-ray and electron diffraction methods were investigated influence of temperature of a substrate in the range of 750 – 1250 K and also the material of a substrate on crystallinity and phase structure of prepared films.

As have shown conducted researches TbS films well grows practically on all applied substrates.

At temperatures of a substrate in the region 750 – 990 K are formed polycrystalline single-phase films, only on occasion on X-ray diffraction pattern additional weak maxima of reflexion

which are found out it is possible was to connect with presence of small quantity  $Tb_2S_3$  and  $Tb_2S_4$ . At temperatures 990 – 1140 K to presence of a texture with an axis [200] and [100] is observed. At temperature 1130 K a texture is not present and there are separate blocks of TbS monocystals with a size to  $10^{-4}$  m which limits dot reflexes are well revealed. At subsequent increase in temperature of a substrate from 1170 to 1200 K one phases of films it is broken and in the prepared films to the dress with TbS presents well revealed inclusions of  $Yb_2S_3$ .

Fig. 1 shows a typical the electronogram

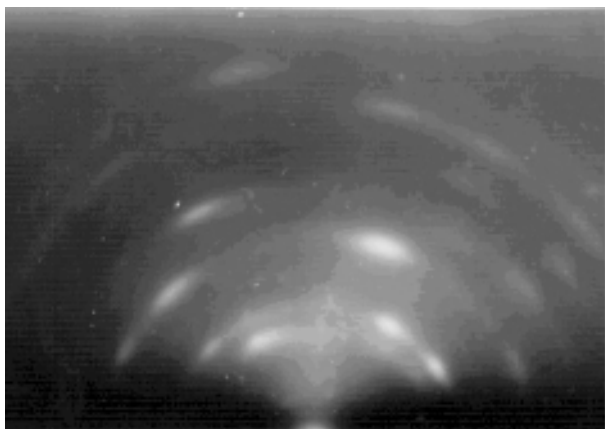


Fig.1. Electron diffraction pattern of a thin TbS film (sapphire substrate, film thickness, 1,7  $\mu$ m).

and fig. 2. X-ray diffraction pattern of an TbS film grown at a substrate temperature of 1140 K. Analysis of X-ray and electron diffraction data indicated that the film grown at this substrate temperature consisted TbS with lattice parameter  $a = 5,52 \text{ \AA}$  in good agreement with data reported for bulk samples [12].

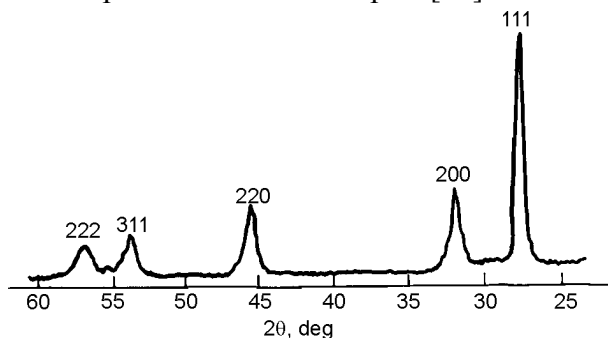


Fig. 2. X-ray diffraction pattern of a thin TbS film (glas – ceramic substrate, film thickness, 1,3  $\mu$ m).

Using the method of X-ray microanalyze it was shown that obtained films include 50.1 at. % Tb and 49.9 at.% S.

In secondary X-rays the surface of the prepared films has been removed. The atoms of

terbium and sulphur are distributed on a surface of films in regular intervals enough as it is visible from fig. 3 and fig. 4.

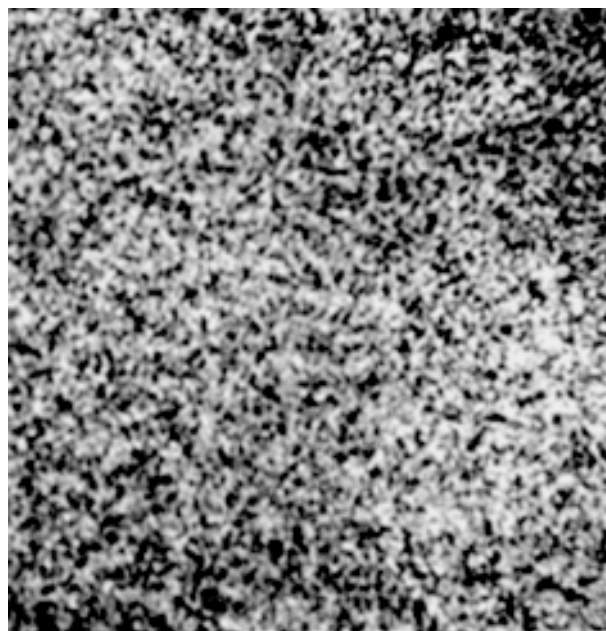


Fig. 3. The image of distribution of Tb atoms on a surface of TbS films in secondary X-rays ( $\times 400$ ).

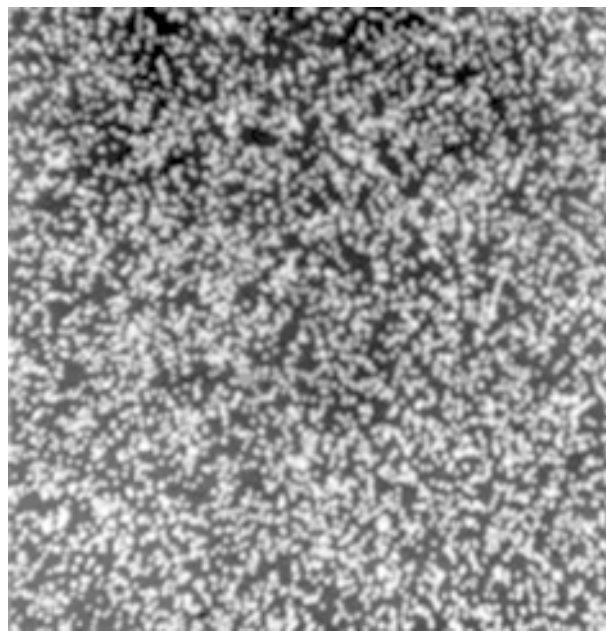


Fig. 4. The image of distribution of S atoms on a surface of TbS films in secondary X-rays ( $\times 400$ ).

At room temperature were measured specific reasistancy and thermoelectromotive force (thermo-emf) on the prepared films. In order to measure the specific resistancy by means of the compensation method, the Holl effect – using constant current and magnetic field (20000  $\text{\AA}$ ), thermo-emf – by the absolute method with correction for thermo-emf of copper. A film had specific electroresistance of an order  $0,5 \cdot 10^{-5}$  Ohm·m,

thermo-emf equaled  $\sim 4 \cdot 10^{-6}$  V/K and the Hall constant –  $\sim 2 \cdot 10^{-10}$  m<sup>3</sup>/C. This data of electro-physical measurements will well enough be coordinated with the corresponding data received on bulk crystals of TbS [12].

## CONCLUSIONS

The technology of preparation of thin crystal films of TbS is developed through flash vacuum thermal evaporation from preliminary synthesized bulk crystal of TbS. The thickness of films varied within 0,3 – 1,8 μm. The films was grown on glass-ceramic, fused silica, sapphire and (111) single-crystal silicon. Electronographical and X-ray analyze showed that thin films had cubic crystal structure of NaCl type with lattice parameters  $a = 8.91$  Å. Using the method of X-ray microanalyze it was shown that obtained films include 50.1 at.%Tb and 49,91 at.%S. At room temperature all films had specific electroresistance of an order  $0,5 \cdot 10^{-5}$  Ohm·m, thermo-emf equaled  $\sim 410^{-6}$  V/K and the Hall constant –  $\sim 10^{-10}$  m<sup>3</sup>/C.

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