Amorphous Antimony in Thin Films

Styrkas A. D.*

In Solid St. Phys RAS Chernogolovka Russia.

*Corresponding Author: Styrkas A. D. In Solid St. Phys RAS Chernogolovka Russia.

Annotation: The conditions for obtaining amorphous Sb films by electrode position from various solutions and vacuum evaporation are studied. It is shown that the explosion Sb is determined by the phase transformation into a stable phase, and not by impurities Cl.

Keywords: Sb amorphous electrolytic vacuum explosiveness mechanism.

1. PREFACE

It is considered to be the existence of Sb 4 modifications [1]: 1) crystal αSbhexagonal; 2) below – 90°C yellow, blackening in light and heating with a transition to 3) black form; and 4) "explosive" β Sb. It is believed that β Sb is a solid chlorine solution in Sb and is formed by electrolysis of chloride solutions at high current density. This statement is found in many textbooks.

2. EXPERIMENTAL PART

Polarization curves i - φ were removed galvanostatically in an inert gas atmosphere with stirring against a saturated cacalome reference electrode. Cathode – Pt polished plate area 1sm², all φ related to normal hydrogen. Analysis – amperometric titration by [2]. The cathode space was separated from the anode from anodic porous partitions. The current output was determined by weighing the cathode. Total Q passing through the cell, electricity controlled copper Q-meter with a solution of Eitel. Reflectance R of films was measured on a GOI reflectometer with Se photocell, vacuum deposition was carried out at specified distances from the crucible with Sb heated by a spiral from W to substrates of transparent polished SiO₂ for adhesion, the Color of the amorphous film treated with a weak solution of SnCl₂ and its transition to a brilliant α Sb Was noted visually. The electrical conductivity of the film during the transition from amorphous to crystalline state changed ~ 500 times. It turns out that the so-called β-form of "explosive antimony" does not contain chlorine. This is proved by chemical analysis. The existence of metastable amorphous Sb passing to α Sb under energy influences was found.

Analysis by [2] show that Sb obtained by electrode position from a solution of SbCl₃ in HCl acid in strongly acidic medium gave the absolute absence of Cl in the sediments. The explosion of the sediment is not due to the presence of Cl, but because it has an amorphous metastable structure. Its amorphous proven by X-raying. The deboeagram has a clean background with a negligible hint of Sb. Exploding gives from amorphous Sbthe αSb dust during the exothermic transition, as the explosion tetragonal, InSb obtained at low T and high P. The abundance of allotropic phases complicates the electrode position of Sbfromthe SbCl₃solutions, explosive Sbgrows at the cathode, which was explained by the presence of Cl impurities. The collected fine dust Sb also does not contain Cl. Additives to surfactant solution to increase (-) the potential of isolation such as diphenylamine, thymol, βnaphthol release potential φ from +0.2 V is shifted to -0. 85V, giving compact shiny precipitates contaminated with inclusions of organic impurities. This precipitation is also amorphous, but not explosive and do not contain Cl, and whenSb heated, blackened from charring of organic impurities captured Strip out impurities slows down the process of transition into the stable phase, and the explosion does not happen. Also in vacuum at the spraying to cold SiO₂, remote from the crucible, settled brown oily spot, but it soon as quickly as the explosion instantly turned into a brilliant crystalline film Sb too, but without the spread of dust particles. The growth of the phase provided the process with energy, the analysis gave 100% Sb, proving that it was a transition from the amorphous
phase to a stable one. With rapid condensation on cold SiO$_2$ and a small thickness of the nano-sized film, the metastable phase can exist, but passes into the crystal stable α-phase under energy influences. At a close nearby distance from the evaporator, this phenomenon can’t be observed due to the heating of the evaporator by radiation. A similar phenomenon is observed in the amorphous Sb obtained by electrolysis in the presence of surfactive substances. Shiny amorphous cathode films on Pt and Cu foil explode with sound when heated, but without sputtering due to organic impurities inhibiting the atomization. The light Sb from the sulfuric acid baths at the bend makes a sound similar to cry at the bend Sn. Covalent brittle crystallites give rise to a cry when friction. Bending of amorphous cathode sediments obtained in the presence of surfactant does not give cry. There is no sound in precipitation from sulphate solutions in which Sb$_2$(SO$_4$)$_3$ is slightly soluble. In a solution of 1.54 g/l Sb$_2$(SO$_4$)$_3$ in 10 n H$_2$SO$_4$ at a current density i below 0.5 mA/cm$^2$, crystal light Sb grows. At i=0.2 mA/cm$^2$, polishing gives a reflection coefficient R of almost 100%, as from vacuum films. With growth i R of the depositions with steeply falls. At i from 0.6; 0.7; and 1.2 mA/cm$^2$ R obtained respectively 40%; 9%; 2%, at higher i grows black loose sediment Sb with R=0 at particles crystalline and fine. The current output drops, but smoothly (from 100% at 0.2 to 12% at 10 mA/cm$^2$) At i above id not only do powders and dendrites grow, the current output drops too. Decreases the release of the Sb due to its restoration to SbH$_3$. (Fig 1) The Formation of SbH$_3$s also proven analytically.

Similarly, when large oversaturated and at high speed formation of nanoparticles of metastable phase, even non-existent in the nature of the W-phase CdTe describes the real preparation of the W phase, but not S CdTe [3], that allowed to investigate a number of optical and other interesting properties of this unknown structure. Nanoparticles of sizes less than 1 nm of phase unknown in the nature, naturally, metastable, but capable to exist. Diamond-like structure S of semiconductor CdTe has very close lattice parameters to its counterpart of semiconductor αSn, so it can infect the βSn. All connections A3B5 can have both structure S, and W, but only for CdTe structure is missing in nature! Cubic phase of CdTe can infect βSn to αSn, but tetragonal phase W CdTe can’t infects. So them easy distinguishing! Later with exposure and energy assistance, this metastable phase passes into a cubic stable S phase of CdTe and infection occurs, proving the description of the mechanism of formation of even non-existent in nature W CdTe Phase W and its the subsequent transition to a stable phase S, infecting tin. It is possible to create something that was not in nature, we must to do need conditions. So it is possible to create and metastable non-existing tetragonal In Sb by obtaining at low T and high P. Introduced under normal conditions, it explodes into the dust of the S structure. (InSb is the isoelectronic and structural analogue of the semiconductor αSn) the Transition from diamond-like covalent αSn to tetragonal metal βSn, stable phase similarly, only in the opposite structural direction, converts Sn into powder from mechanical stresses with a change in the volume of the structure by 26.6% with similar mechanical breakdowns. So is method of receiving of pure Sn powder by many times repetition of βSn→αSn and αSn→βSn, without explode.

The abundance of allotropic phases in Sb complicates electrode position. At large super saturation in solutions SbCl$_3$ grows at the cathode explosive Sb, that was explained by the presence of impurities Cl. And the collected fine dust Sb doesn’t contain Cl. Additives to the solution of surfactants to increase the -ϕ of the potential of deposition by diphenylamine, thymol, β-naphthol from +0.2 V shift to –0, 85V, giving a compact shiny precipitates contaminated with inclusions of organic impurities. Precipitation is also amorphous, but not explosive and do not contain Cl, and when heated, blackened from charring of organic impurities captured Strip out impurities slows down the process of transition into the stable phase, so the explosion does not happen.

When spraying in vacuum on cold SiO$_2$, removed from the crucible, settled brown oily spot, but it soon as quickly as the explosion instantly turned into a brilliant crystalline film Sb, but without the spread of particles. The phase growth provided the process with energy, the Analysis gave 100% Sb, proving that it is also transition from amorphous phase to stable. At rapid condensation on cold SiO$_2$ and at small thickness of the nano-sized film, the metastable phase can exist, but passes into the crystal stable α-phase under the energy effects of the phase growth. At a close distance from the evaporator, this phenomenon cannot be observed due to the heating of the evaporator by radiation. A similar phenomenon is observed in the amorphous Sb obtained by electrolysis in the presence of surfactants. Shiny amorphous cathode films on Pt and Cu foil explode with sound when heated, but without spraying due to organic impurities inhibiting the spray. The light Sb from the sulfuric acid baths at the bend makes a sound similar to the cry at the bend Sn. Covalent brittle crystallites rise rise
to a cry at the friction. Bendingo amorphous cathode sediments obtained in the presence of surfactant do not give cry. There is no sound in precipitation from sulphate solutions in which Sb₂(SO₄)₃ is slightly soluble. In a solution of 1.54 g/l sulphate Sb in 10 n acid at a current density i below 0.5 mA/sm², crystal light Sb grows. At i=0.2 mA/sm² polishing gives a reflection coefficient R of almost 100%, as from vacuum films. With growth i R with steeply fall. At i = 0.6, ; 0.7; and 1.2mA/sm² R, respectively, obtained 40%; 9%;2%, atihigher Sb grows black loose sediment with R=0 of small crystalline particles.

![Image]

**Fig 1.** Common cathodic curves (1); and partial (2): the deposition of Sb decay shows the formation of SbH₃ at φ value more than -0.15; and (3) the desorption of H₂.

The current output drops, but smoothly (from 100% at 0.2. to 12% at 10 mA/sm²) Ati above id not only do powder and dendrites grow, the current output drops. Decreases the release of the Sb due to its restoration to SbH₃. (Fig1.) the Formation of SbH₃ is proved analytically.

3. **SUMMARY**

Amorphous films were obtained by electrode position and vacuum deposition of antimony. It is proved that there is no previously considered β - phase of antimony, and the so-called explosive antimony, allegedly exploding from the content of chlorine in it is a metastable amorphous phase capable of existing in nano particle sizes and passing into a stable α-phase with energy reins and growth of particle sizes, like other metastable systems.

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