

Characteristics of Evaporated Antimony Films as a Function of the Antimony Source

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tions. When the lens was in the slightly defocused position, a relatively uniform area of vaporization, 1.93 mm in diam, was formed. However, when the lens was in the position forming the smallest focal spot size, the texture of the surface showed a considerable amount of vaporization over an area of 0.66 mm in diameter, with a deeper hole in the center of 0.2 mm in diameter. The depth of the center hole varied with the laser energy and the target material. No hole was formed in tungsten at any laser energy, while holes as deep as 0.5 mm were formed in aluminum at the higher laser energies.

We have deduced that this hole was not in existence during the laser pulse. It must have been formed as a result of a strong shock produced by the laser giant pulse, which then continued to propagate into the material after the pulse was off, and by subsequent evapor-

ation or boiling of the shock-heated material. This is substantiated by data obtained with aluminum foil, and was consistent with calculations made by Kidder.⁴ When 0.025-mm aluminum foil was used as a target material, it was found that no laser light penetrated the foil, even at energies which formed holes 0.5 mm deep in thick targets. Also, the momentum transfer data showed the aluminum foil to behave the same as thick targets. This would not have happened had the hole formed in the foil during the laser pulse and allowed some laser energy to pass through unabsorbed.

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We would like to express our appreciation for the many helpful discussions with Ray E. Kidder.

⁴ R. E. Kidder (private communication).

Characteristics of Evaporated Antimony Films as a Function of the Antimony Source*

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Thin antimony films deposited by evaporation from a PtSb source differ from films produced from elementary antimony in electrical resistance, light transmission, reaction with oxygen, and particle size. Qualitative mass spectroscopic experiments indicate that these differences may be due to antimony evaporating from PtSb predominantly as Sb₁ as distinct from the Sb₄ aggregates known to evaporate from elementary antimony.

IN some vacuum devices such as phototubes, it is necessary to evaporate a metal whose vapor pressure is so high that the device cannot be heated to the required degassing temperature. This difficulty is overcome by evaporating the metal from an intermetallic compound whose decomposition temperature is higher than the evaporation temperature of the metal to be evaporated. The vapor pressure of the other constituent(s) of the compound must, of course, be so low that only the desired metal evaporates at the decomposition temperature. This method has been used successfully in the past to evaporate tellurium from indium telluride¹ and antimony from platinum or palladium antimonide.²

During recent experiments with Sb films produced by evaporation from beads consisting, respectively, of elementary Sb and of the compound PtSb, considerable differences in the properties of the resulting deposits

were observed. In both cases, the films were formed by evaporation from a distance of about 4 cm onto a glass substrate until the light transmission of the film was reduced to 70% of the original value. On the basis of published curves³ for the relationship between light transmission (measured with a tungsten lamp) and film thickness, this light transmission corresponds to a film thickness of approximately 70 Å. The beads were heated to temperatures at which the desired light transmission was obtained within approximately 10 sec. These temperatures were about 550°C for the Sb bead and about 1000°C for the PtSb bead.

Before describing the two types of Sb films, it should be emphasized that the different properties of the films evaporated from PtSb are not caused by the presence of small amounts of Pt. This possibility was excluded by the results of spectroscopic analysis.⁴ Moreover, it was shown in control experiments that, in accordance with expectation, Pt is only evaporated if the source is heated to much higher temperatures.

The following are the most significant differences be-

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¹ A. H. Sommer, *Rev. Sci. Instr.* **32**, 356 (1961).

² *Electronics and Their Application in Industry and Research*, B. Lovell, Ed. (Pilot Press, London, 1947), p. 65.

³ G. A. Condas, *Rev. Sci. Instr.* **33**, 987 (1962).

⁴ These analyses were performed by F. R. Hughes of RCA, Lancaster, Pa.

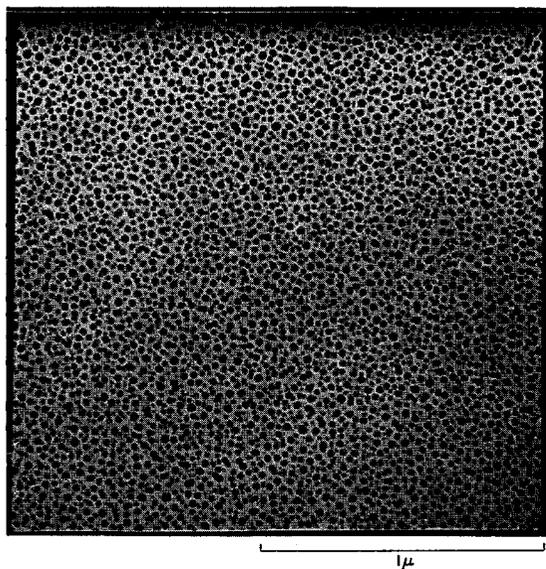


FIG. 1. Electron micrograph of Sb film deposited from Sb bead.

tween Sb films deposited from Sb beads ("film A") and from PtSb beads ("film B"):

(1) *Resistance.* When the films are deposited between two contact strips, 15 mm long and 2 mm apart, the resistance of both types is in excess of $10^8 \Omega$ at room temperature. When the films are heated to 160°C , the resistance of film B decreases to values of the order of $10^3 \Omega$, whereas the resistance of film A remains unchanged.

(2) *Light transmission.* When films having a light transmission of approximately 70% are allowed to react with an alkali metal such as cesium or potassium to

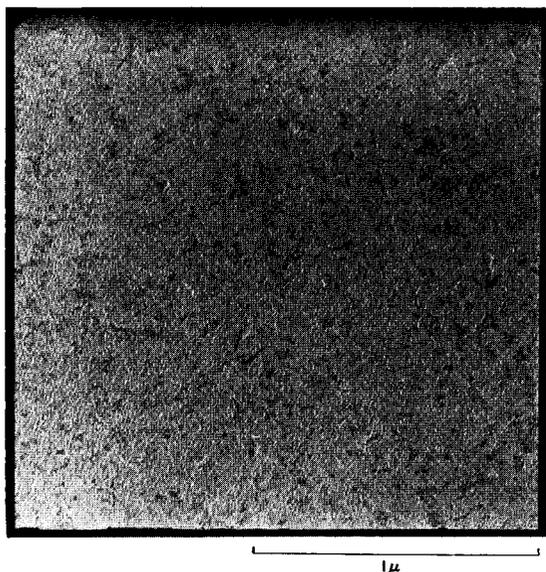


FIG. 2. Electron micrograph of Sb film deposited from PtSb bead.

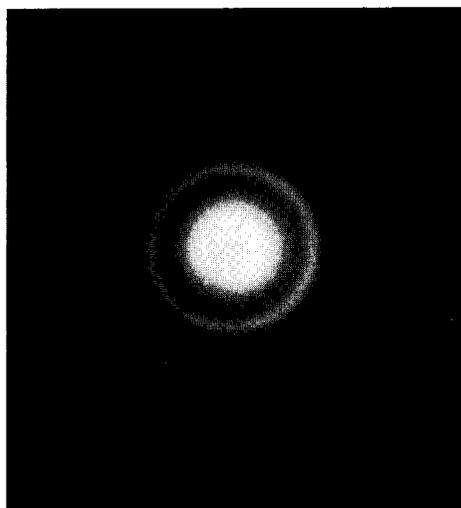


FIG. 3. Electron diffraction pattern of Sb film deposited from Sb bead.

form the photoelectric antimonide $M_3\text{Sb}$ (M being the alkali metal), the optical density of the resulting material when formed with film A is two to three times greater than when it is formed with film B. If one makes the reasonable assumption that the same $M_3\text{Sb}$ compound is formed from both substrates, one has to conclude that, for equal light transmission, film B is much thinner than film A. (The correlation between thickness and light transmission mentioned earlier³ was derived from films evaporated from elementary Sb and hence applies to films of type A.)

(3) *Effect of oxygen.* Thin films of different evaporated metals are known to vary with respect to their reactivity with oxygen. To give two examples, bismuth films are oxidized superficially by exposure to oxygen at

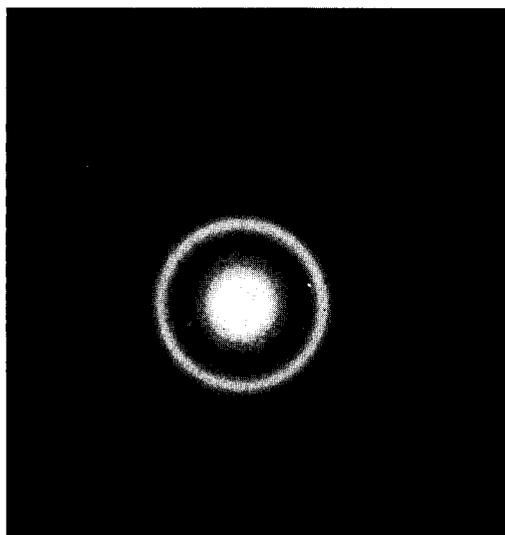


FIG. 4. Electron diffraction pattern of Sb film deposited from PtSb bead.

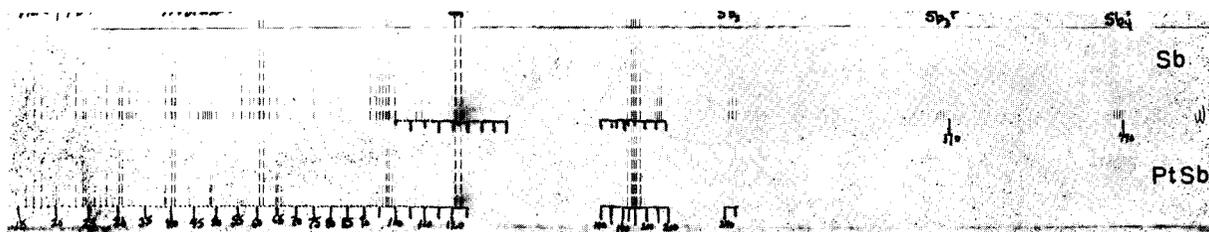


Fig. 5. Mass spectrograms of Sb ions produced by a spark between Sb (top) and between PtSb (bottom) electrodes.

room temperature and to a greater depth upon heating in oxygen; silver films, on the other hand, can be oxidized only by exposure to a glow discharge in oxygen. With antimony, it was observed that films of type A are not measurably attacked by oxygen ("measurably" being defined as accompanied by a measurable increase in light transmission) upon exposure at room temperature, at temperatures as high as 160°C, or even in a glow discharge. By contrast, it was found that Sb films of type B are readily oxidized either by heating in oxygen to 160°C or by a glow discharge. The oxidation, however, is limited to a depth of the order of 10 atoms thick.

(4) *Structure of Sb films.* In order to find an explanation for the different properties of the two film types, several samples were examined by electron microscopy and by high-energy (transmission) electron diffraction.⁵ For this purpose, the films were deposited in a conventional manner upon amorphous carbon films on copper mesh; earlier experiments had indicated that the behavior of this material as a substrate is very similar to that of glass. Thus, there is reason to believe that the results described below also apply to the films deposited on glass which were used in the experiments (1), (2), and (3).

Figures 1 and 2 show electron micrographs of samples of films A and B, respectively. It is apparent that both films consist of distinct particles, but that the particle size in film B is much smaller. The estimated diameter of type B particles is about 30 Å, whereas that of type A particles is about 350 Å. It is also apparent that the particles in film B are much more closely packed than in film A. This difference would seem to account, at least qualitatively, both for the lower resistance of type B and for its smaller thickness at a given value of light transmission. Moreover, the smaller particle size is also compatible with the readiness of B films to react with oxygen.

The electron diffraction patterns are shown in Figs. 3 and 4. They both agree with the known structure of Sb and are both diffuse, indicating that the material is amorphous. This finding is in agreement with earlier

⁵ These experiments were performed and interpreted by M. D. Coutts and C. W. Roth.

work⁶ in which it was shown that Sb films of less than 180Å thickness are amorphous. The diffraction patterns thus eliminate the possibility of interpreting the difference between A and B films by the assumption that the B film is crystalline and the A film is amorphous.

In an attempt to explain the effect of the PtSb source, the following hypothesis was put forward by Simon⁷: Although antimony is known⁸ to evaporate from elementary Sb in aggregates of the formula Sb₄, it may possibly evaporate from PtSb in the monatomic form, in a remote analogy to the formation of "nascent" hydrogen.

Because a check of this hypothesis by evaporation of Sb in the mass spectrograph was not feasible at this time, a preliminary experiment was suggested⁹ and performed¹⁰ as follows: A spark was produced in the mass spectrograph, first between two beads of Sb, then between two PtSb beads. Whatever the size of the aggregate in which the metal evaporates upon heating, the most likely ion to form in the spark is Sb₁⁺. However, it was hoped that a sufficient number of ions of larger size would form to show a difference between Sb and PtSb if, in fact, no Sb₄ is formed from PtSb.

Figure 5 shows the result of this experiment. Although the expected strong lines appear for monatomic Sb in both samples, Sb₄⁺ and Sb₃⁺ (a likely decomposition product of Sb₄) are clearly visible in the spectrogram of Sb, but completely absent in PtSb. There is some Sb₂⁺ in the PtSb pattern, but the difference in the two spectrograms certainly supports the hypothesis that antimony evaporates from PtSb in smaller (mainly monatomic) particles than from Sb. Qualitatively, this result is plausible because the Sb is bound by Pt atoms in the compound PtSb, whereas it is bound by Sb atoms in elementary antimony. If the hypothesis is correct, it may also account for the different size of the aggregates shown in Figs. 1 and 2 because the Sb₁ particles are likely to produce more nucleation centers per unit time than the Sb₄ particles.

⁶ W. Lotmar, *Helv. Phys. Acta* **18**, 369 (1945).

⁷ R. E. Simon (private communication).

⁸ R. Hultgren *et al.*, *Selected Values of Thermodynamic Properties of Metals and Alloys* (John Wiley & Sons, Inc., New York, 1963), p. 247.

⁹ By R. E. Honig.

¹⁰ By J. R. Woolston and H. H. Whitaker.