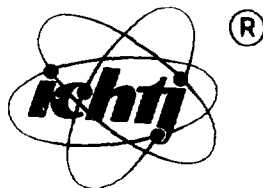


**LECH WALIŚ \* LUZJA ROWIŃSKA \* EWA PAŃCZYK**

**EVALUATION OF THE EFFICIENCY  
OF THE PROCESSES  
OF PURIFICATION OF ANTIMONY  
TO SEMICONDUCTOR GRADE PURITY**

INCT-2135/I



**INSTYTUT CHEMII I TECHNIKI JĄDROWEJ  
INSTITUTE OF NUCLEAR CHEMISTRY AND TECHNOLOGY  
ИНСТИТУТ ЯДЕРНОЙ ХИМИИ И ТЕХНИКИ**

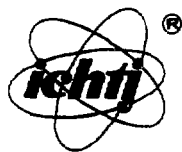
**WARSZAWA 1992**

LECH WALIŚ \* LUZJA ROWIŃSKA \* EWA PAŃCZYK

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**OCENA EFEKTYWNOŚCI PROCESÓW OCZYSZCZANIA ANTYMONU  
DO CZYSTOŚCI PÓLPRZEWODNIKOWEJ**

**ОЦЕНКА ЭФФЕКТИВНОСТИ ПРОЦЕССОВ ОЧИСТКИ СУРЬМЫ  
ДО ПОЛУПРОВОДНИКОВОЙ ЧИСТОТЫ**



**Institute of Nuclear Chemistry and Technology  
Department of Nuclear Methods of Material Engineering**

**WARSZAWA 1992**

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## ABSTRACT

A complex of techniques for purification of antimony from arsenic has been examined with the aid of radiotracer  $^{76}\text{As}$ . The investigated processes comprised vacuum distillation, zone melting and remelting of the metal under artificial slags. The purification efficiencies for the above processes were high and amounted to 94% (for 30% of the charge), 50% (for 50% of the charge) and 99.5% (for 60% of the charge), respectively. Attempts were made to determine the kinetics of the separation of arsenic from antimony by distillation.

The application of the radioactive tracer made it possible to determine rapidly the distribution of impurities after each stage of the process within a wide concentration range ( $10^{-2}$ - $10^{-7}$  g/g).

## STRESZCZENIE

Stosując znacznik promieniotwórczy  $^{76}\text{As}$  przeprowadzono badania kompleksu metod prowadzących do oczyszczania antymonu od arsenu. Badaniami objęto procesy destylacji próżniowej, topienia strefowego i przetapiania metalu pod żużłami syntetycznymi. Uzyskano wysoka efektywność oczyszczania dla wyżej wymienionych procesów, odpowiednio 94% (dla 30% wsadu), 50% (dla 50% wsadu) i 99,5% (dla 60% wsadu). Poczyniono pierwsze kroki w kierunku określenia kinetyki procesu oddestylowania arsenu z antymonu.

Zastosowanie znacznika promieniotwórczego umożliwiło szybkie określenie rozkładu zanieczyszczeń po każdej fazie procesu w szerokim zakresie stężeń ( $10^{-2}$ - $10^{-7}$  g/g).

## РЕЗЮМЕ

С применением радиоиндикатора  $^{76}\text{As}$  проведены исследования комплекса методов, в результате которых проводится очистка сурьмы от мышьяка. Исследовались процессы вакуумной перегонки, зонной плавки и переплавки

металла под слоем синтетического шлака. В этих процессах получена высокая эффективность очистки, соответственно 94% (для 30% шихты), 50% (для 50% шихты) и 99,5% (для 60% шихты). Предварительно определена также кинетика процесса дистилляционного выделения мышьяка из сурьмы.

Применение радиоиндикатора позволило на быстрое определение распределения загрязнений на каждом этапе процесса в широком диапазоне концентрации ( $10^{-2}$  и  $10^{-7}$  г/г).

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## 1. INTRODUCTION

Purification of metals to semiconductor purity grade is generally carried out by vacuum distillation and zone melting. The effectiveness of these processes was examined using the radiotracer method. Complex studies on these for the system antimony - arsenic were carried out. Antimony was chosen as the object of the studies because of its importance to semiconductor industry (manufacturing of the III-V group compounds with high carriers mobilities), and of technological difficulties in the separation of arsenic from antimony. When antimony of 99.9% purity is used as the starting material, it is difficult to obtain antimony of semiconductor purity grade by means of physical methods. Therefore preliminary purification, generally by chemical methods, is carried out. A physico-chemical method was proposed for production of antimony of 99.999% purity [1]. The method consisted in conversion of antimony oxide to chloride, distillation of the chloride from an aqueous solution, followed by recovery of antimony from chlorides by reduction with hydrogen at the temperature 1073 K. Antimony of 99.8% purity was used as the starting material in this process. In the present work, remelting under artificial slags was used as a preliminary purification technique.

## 2. EXAMINATION OF THE VACUUM DISTILLATION PROCESS

In the distillation process using a condenser of low temperature, practically all atoms which are in transient contact with the condenser do deposit on its surface. The condensation coefficients are close to unity and the composition of the condensate corresponds to that of the vapour above it. If the temperature of the condenser is sufficiently high, volatile components exhibit low condensation coefficients, which leads to further purification of the condensate [2,3]. In practice, condensers of higher temperature are more important. The increase in the condensation temperature results in a decrease of the condensation coefficients of all components, and therefore the rate of distillation is reduced. In practice, the choice of the conditions is a compromise between the efficiency of the process and the purity of the material obtained. The separation of the pair of elements Sb-As is difficult due to their thermodynamic properties.

The distillation processes were carried out using both variants of the method - with a heated and unheated condenser. The vacuum distillation was carried out in the equipment described earlier [4]. The distillation furnace was divided into six heating zones. The temperature gradients in the distillation apparatus with the heated and unheated condenser are shown in (Fig.1).

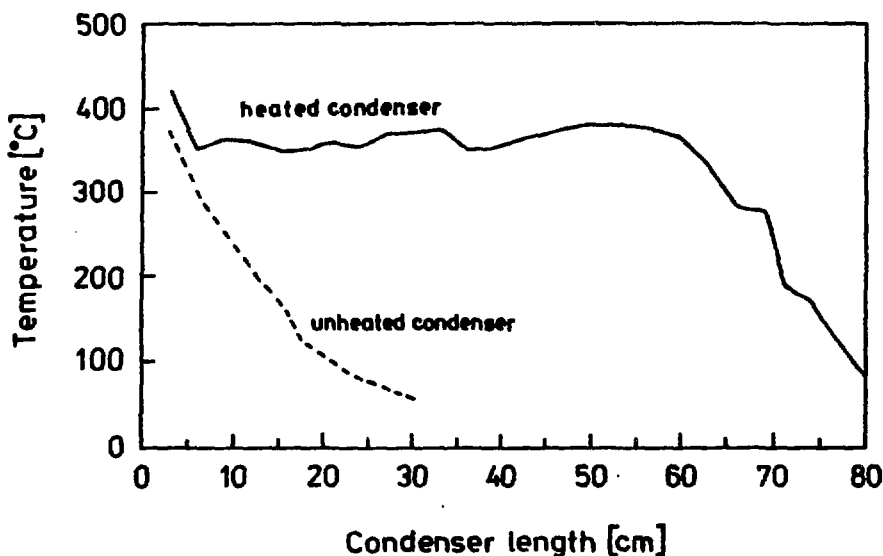


Fig.1. Temperature gradient of the condenser

The distillation process was carried out at the temperature of 933-953 K, under the pressure of  $2.5 \times 10^{-5}$  Tr. The mean distillation time was about 2 hours. The purity of the starting antimony after the addition of 0.2 g of arsenic with  $^{76}\text{As}$  tracer was 99.9 %. The radioactive tracer was introduced by repeated remelting of antimony with the arsenic placed inside the ingot. The tracer  $^{76}\text{As}$  was obtained by irradiation of 0.2 g of arsenic in the nuclear reactor EWA at Świerk with the neutron flux of  $5 \times 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$  for 15 minutes. After the distillation process the condensate was sampled and measurements of the radiation intensity were carried out. The samples were collected from a surface of  $1 \text{ cm}^2$  along the upper and the lower of the condensate, every 1 cm. Table 1 illustrates the distribution of antimony mass and the concentration of arsenic along the upper and lower part of the condenser. The distributions of the mean arsenic concentration in the



Table 1. Masses of antimony and concentrations of arsenic in antimony samples after vacuum distillation using condenser heated to 623 K

Condenser length [cm]	Sb mass [g]			As concentration in antimony [ppm]		
	top	bottom	mean	top	bottom	mean
1	1.31	2.24	1.77	3.56	8.79	6.18
2	3.56	2.26	2.91	3.41	6.83	5.12
3	1.08	1.86	1.47	4.82	8.63	6.73
4	1.79	1.77	1.78	8.43	9.89	9.16
5	1.55	1.90	1.72	5.39	9.52	7.46
6	1.38	3.17	2.27	6.24	7.89	7.07
7	1.13	2.68	1.90	6.93	10.67	8.80
8	1.93	1.97	1.95	8.26	15.8	12.0
9	1.93	2.11	2.02	10.6	18.5	14.6
10	2.43	2.24	2.33	11.5	19.4	15.5
11	2.27	2.20	2.23	13.4	12.5	12.9
12	2.08	2.05	2.06	15.2	23.6	19.4
13	1.97	2.22	2.09	15.8	24.4	20.1
14	1.74	2.59	2.17	15.0	21.1	18.1
15	1.73	1.58	1.75	15.1	25.5	20.3
16	1.34	1.17	1.28	16.4	23.6	20.0
17	1.52	1.79	1.65	15.1	24.0	19.5
18	1.80	3.30	2.55	15.1	27.8	21.4
19	1.78	3.65	2.71	22.2	27.5	24.9
20	2.54	3.48	3.01	24.5	32.4	28.5
21	3.17	2.57	2.87	26.4	40.5	33.4
22	2.41	2.11	2.25	27.8	37.7	32.8
23	1.58	2.16	1.87	34.3	47.0	40.6
24	1.60	1.80	1.70	31.7	48.5	40.1
25	1.92	1.33	1.62	31.7	57.5	44.6
26	1.70	1.31	1.50	34.8	50.3	42.6
27	1.34	1.40	1.40	42.4	54.3	48.3
28	1.31	1.02	1.16	44.0	64.5	54.2
29	1.24	0.67	0.96	50.6	73.2	61.9
30	1.14	0.95	1.04	53.5	68.4	60.9
31	1.06	0.83	0.94	61.1	81.5	71.3
32	0.99	0.65	0.82	63.0	83.7	73.3
33	0.71	0.70	0.71	74.4	110.0	92.2
34	0.59	1.45	1.02	79.7	274.1	176.9
35	0.73	1.00	0.86	94.4	396.8	245.1

heated (a) and unheated (b) condensers are shown in (Fig.2). The measurements of the radiation intensity were carried out with the aid of a NaI(Tl) detector coupled to the IN-90 gamma-ray spectrometer (Intertechnique).

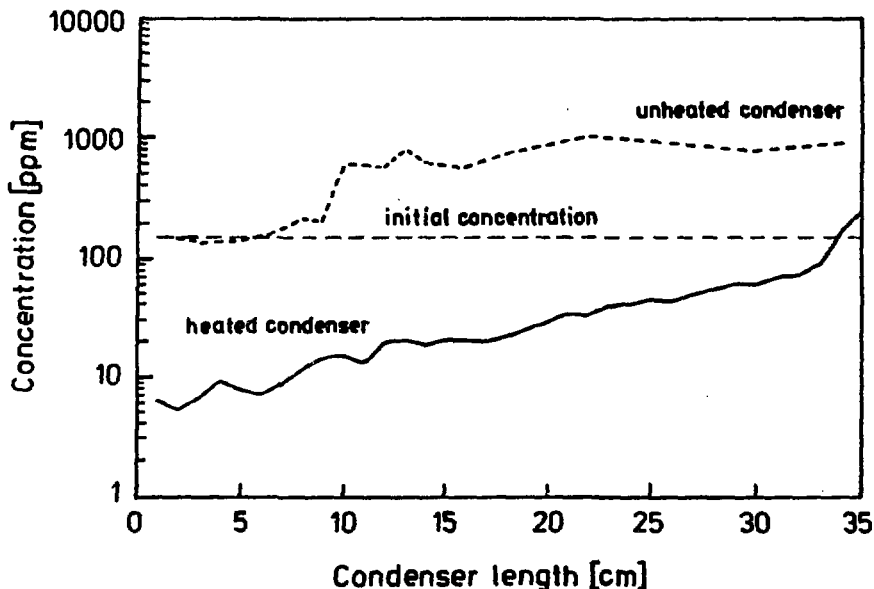


Fig.2. Distribution of As in antimony as a function of condenser length  
The content of  $^{76}\text{As}$  was determined from the integral spectrum. The counting time was 1000 s. The total mass of antimony deposited in each condenser zone (Table 2) was evaluated from the sample data (Table 1).

Table 2. Mass of antimony and mean As concentration in the condensate after vacuum distillation using heated condenser

Condenser zone [mm]	Mean As concentration in antimony [ppm]	Mass of antimony [g]
starting antimony	154	1000
I(0-100)	9	334
II(100-200)	20	341
III(200-300)	46	254

### 3. EVALUATION OF THE PURIFICATION DEGREE OF ANTIMONY FROM ARSENIC AS A FUNCTION OF THE TIME OF THE PROCESS

The distribution of impurities of many elements as a function of the condenser length is well known. This distribution has the same character, despite of different vapour pressures of the elements [5,6]. In order to evaluate the intensity of evaporation of impurities during the distillation process, the concentration of arsenic in lateral layers of the condensate was determined. The sampling point is shown in (Fig.3). A sample of the condensate labelled with  $^{76}\text{As}$  was cut out and the layers were removed (using microtome) from the sample, starting from those adjacent to the condenser wall (beginning of the distillation) deep into the sample (end of the distillation).

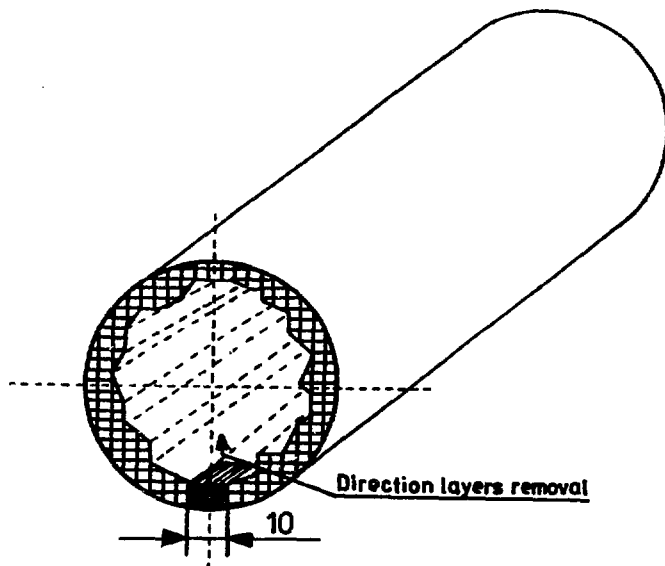


Fig.3. Position of the condensate sample examined

The thickness of the first and the final layers was 50 and 100  $\mu\text{m}$ , respectively. In total 1500  $\mu\text{m}$  of antimony layer was removed. The results are shown in (Fig.4). The sample from which the layers were removed had grown on a curved surface (the process was carried out in a graphite tube). For this reason the first layers were removed only from a part of the surface and the content of arsenic in these layers is significantly lower than in those removed from the whole surface.

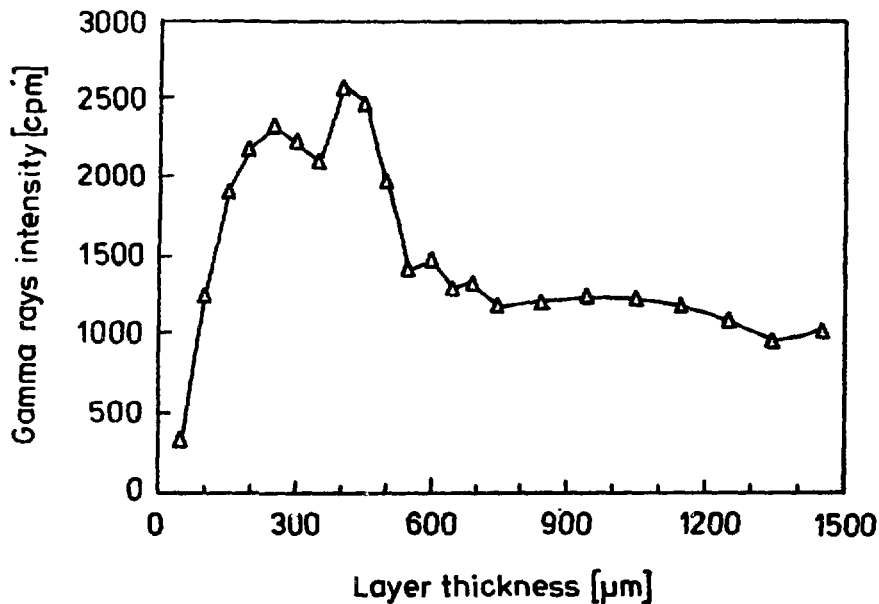


Fig.4. Distribution of arsenic in a lateral layer of antimony deposited in the condenser

4. EVALUATION OF THE KINETICS OF THE ANTIMONY DISTILLATION PROCESS

The distillation process of antimony labelled with <sup>76</sup>As was carried out under the conditions described above. The radiation intensity of <sup>76</sup>As contained in the antimony layer growing on the condenser walls, was

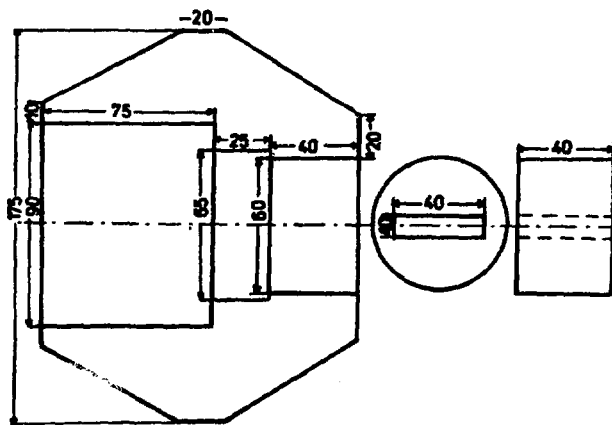


Fig.5. Lead collimator

measured during the process. The spectrometric probe consisted of a NaI(Tl) crystal of dimensions 45x50 mm (type SS-48) coupled to the Didac-800 analyzer (Intertechnique) operating in multiscaler mode. The probe was placed at the beginning of the condenser and shielded by a collimator. The shape and dimensions of the collimator are shown in (Fig.5).

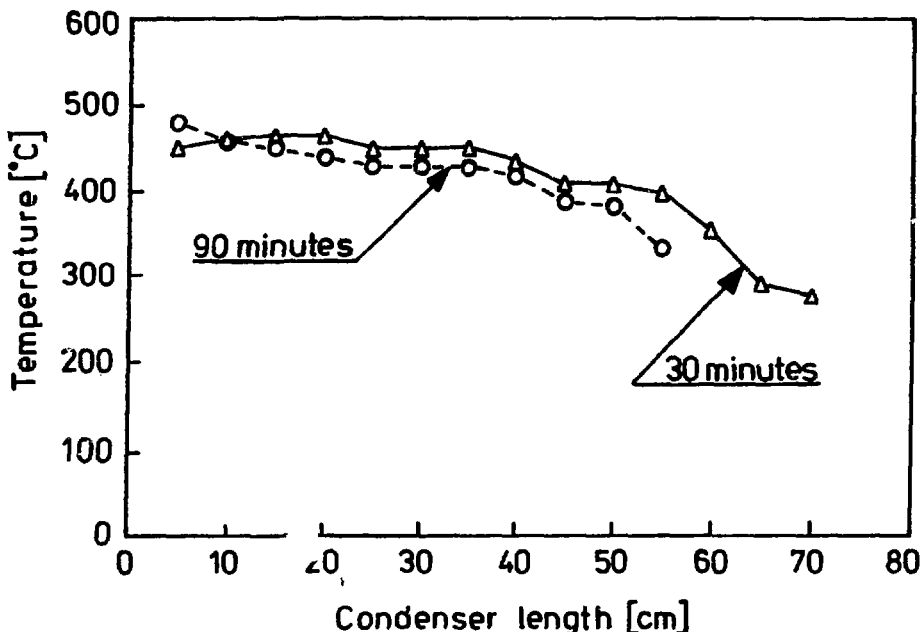


Fig.6. Temperature distribution along the condenser after heating for 30 and 90 minutes

The furnace was heated up for about 1 h before the distillate began to deposit on the walls of the condenser. That moment was assumed to be the beginning of the distillation. The experiment was carried out using the heated condenser. The distribution of temperature along the condenser is presented in (Fig.6). The measurements of radiation intensity were carried out every 100 s. The results obtained (corrected for the decay) are shown in (Fig.7). The increments in the radiation intensity are shown in (Fig.8).

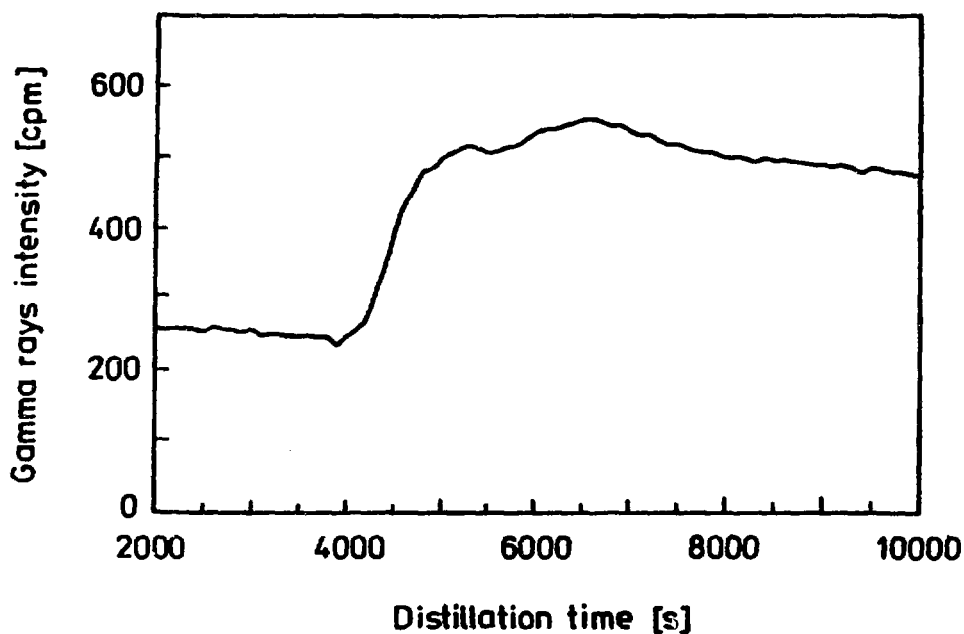


Fig.7. The dependence of radiation intensity of  $^{76}\text{As}$  on the time of antimony distillation

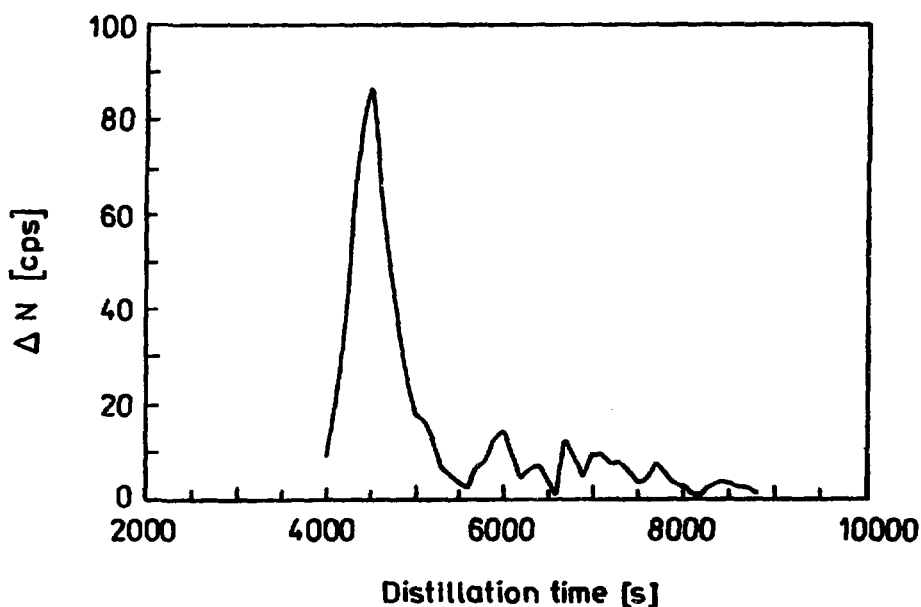


Fig.8. Increments of radiation intensity of  $^{76}\text{As}$  with the time of distillation of antimony

## 5. EXAMINATION OF THE ZONE MELTING PROCESS

The final purification of antimony was carried out by the zone melting process in typical apparatus supplied with high frequency current (about 0.4 MHz). The width of the heating zone was approx. 80 mm. The shift rate of the heating zone was approx. 40 mm/h. The run time of the process was about 9 h. Argon atmosphere was used. The mass of antimony subjected to the purification was 4800 g, the mass of arsenic with radioactive tracer - 0.501 g. The initial concentration of arsenic in antimony was 104 ppm. Arsenic was added to antimony as described in § 2. The antimony sample labelled with  $^{76}\text{As}$  was used as the standard of arsenic concentration of 104 ppm.

Table 3. Distribution of As concentration in antimony after eight passages of the purification zone

Condenser length [cm]	Distribution of As concentration in the ingot after successive passages of the zone [ppm]							
	1	2	3	4	5	6	7	8
1	88	45	55	36	47	39	38	36
3	82	64	55	46	46	42	42	34
5	80	67	59	50	51	45	45	38
7	80	71	61	57	53	48	49	43
9	83	72	61	57	51	51	47	40
11	84	72	62	58	57	55	51	45
13	86	73	67	61	62	55	50	47
15	91	75	73	63	63	61	57	55
17	89	82	75	66	67	59	58	53
19	95	80	80	67	66	65	60	61
21	94	83	81	75	67	69	67	62
23	94	86	84	76	72	72	67	63
25	94	87	88	80	73	75	72	71
27	98	87	90	83	77	80	76	73
29	96	90	92	85	83	84	84	79
31	99	94	100	90	87	96	89	84
33	96	96	103	93	91	101	98	89
35	96	109	105	100	95	106	107	101
37	97	116	113	101	107	115	107	109
39	101	117	118	111	116	125	125	128
41	111	112	116	113	118	132	121	134
43	126	121	128	122	135	142	127	134
45	130	140	152	131	153	155	142	141
47	127	133	142	119	157	141	159	136
49	125	134	136	129	163	146	166	134
51	127	137	146	146	173	161	176	149
53	129	145	160	165	176	183	178	168
55	124	141	159	165	169	180	180	167
57	131	152	165	176	181	198	196	176
59	86	180	104	596	185	169	93	190

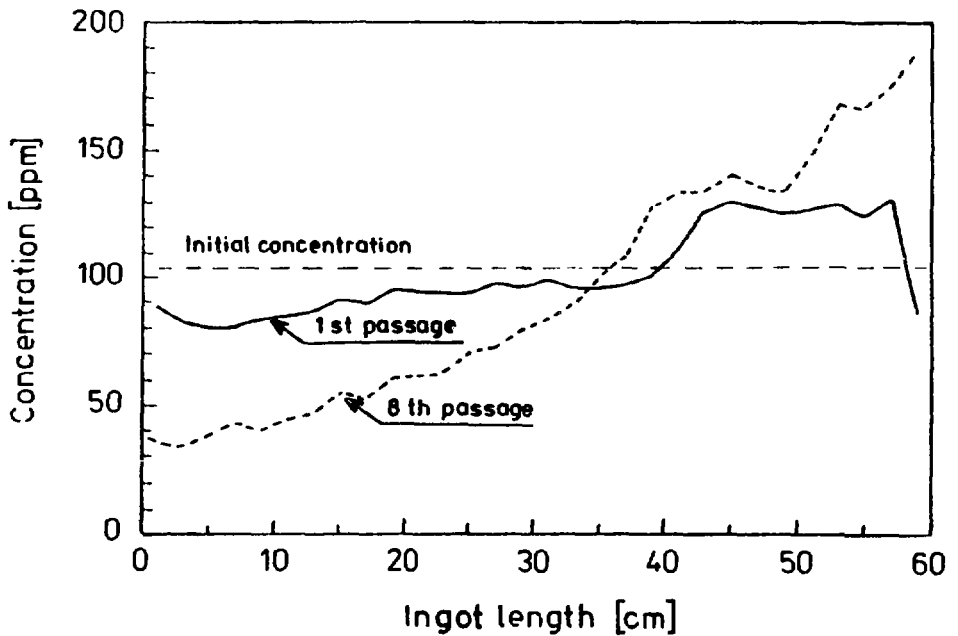


Fig.9. Distribution of As in antimony after the passage of purification zone

Table 4. Purification of antimony from arsenic in the zone melting process

Number of zone passages	As concentration in antimony [ppm]		Purification degree [%]	
	Length of purified ingot [cm]		with respect to:	
	21	total-59	initial values	former values
1	87	101	16	16
2	71	112	32	18
3	66	101	37	7
4	58	111	44	12
5	57	101	46	2
6	54	102	48	5
7	51	98	51	6
8	47	95	55	8
mean=103				
Initial concentration		104.4		



The radiation intensity of  $^{76}\text{As}$  was measured along the whole ingot every 20 mm. The measurements were carried out using the set-up described earlier [7]. The detector was shielded from all sides with a lead shield 60 mm thick. The NaI(Tl) crystal was provided with an interchangeable collimator 5 mm wide and 20 mm long. The ingot was positioned perpendicularly to the collimator window and moved above it.

The concentrations of As in antimony along the whole length of the ingot after each zone passage are shown in Table 3. The distributions of arsenic concentration in antimony after the 1st and 8th passages of the purification zone, compared with the concentration of arsenic in the starting antimony are shown in (Fig.9). The efficiency of the purification of antimony from arsenic in this process after eight zone passages is presented in Table 4.

## 6. EXAMINATION OF THE PROCESS OF ZONE REMELTING UNDER ARTIFICIAL SLAGS

The process of zone remelting under artificial slags was carried out in the apparatus designed for zone melting of metals. The conditions of the process, the way of introduction of  $^{76}\text{As}$  to the antimony and the method of the measurement of the radiation intensity were the same as described above. The initial concentration of arsenic in antimony was 47 ppm. The masses of the slags after the 1st and 2nd melts were 450 and 350 g, respectively, i.e. about one tenth of the charge mass.

The composition of the slag was as follows: 50%  $\text{NaNO}_3$ , 30%  $\text{NaOH}$  and 20%  $\text{K}_2\text{CO}_3$ . The slag was loaded on to the ingot and the remelting process was carried out. After the process the slag was sampled. The upper surface of the ingot covered with the slag was divided into sections 30 mm long. The slag was then removed from the surface. After the process the slag was distributed irregularly. After the 1st zone passage the slag was shifted to the end of the ingot. Therefore the sample No 15 was collected from a surface 150 mm long. Further samples were collected from surfaces 30 mm long (Table 5, column 1). After the second passage the slag was shifted towards the beginning of the ingot. The sample 46-58 was collected from the end section of the ingot, 130 mm long (Table 5, column 3). After the 1st zone passage the radiation intensity of  $^{76}\text{As}$  contained in the antimony was

Table 5. As concentration in the slag after 1st and 2nd passage of purification zone

Number of zone passage			
1		2	
Ingot length [cm]	As concentration [ppm]	Ingot length [cm]	As concentration [ppm]
15	38.9	3	15.9
18	93.0	6	16.7
21	60.8	9	15.7
25	49.4	12	13.7
28	41.9	15	15.7
31	56.3	18	20.3
33	68.8	21	17.9
36	33.8	24	16.5
42	32.1	27	14.8
45	35.8	30	30.7
48	58.6	33	27.0
51	38.0	36	27.4
54	39.9	39	25.7
57	58.4	42	27.6
-	-	45	27.8
-	-	46-58	20.4

mean x 50.4 20.9  
 standard dev.s 16.9 5.8  
 V=100 s/x 34 % 28 %

Table 6. Arsenic concentration in the ingot after the 1st zone passage (ingot under slags)

Ingot length [cm]	As conc. in antimony [ppm]	Mean conc. [ppm]	Ingot length [cm]	As conc. in antimony [ppm]	Mean conc. [ppm]
1	3.8		35	22	
3	4.3		37	21	
5	2.0		39	25	
7	1.6		41	28	
9	1.0		43	42	
11	0.8		45	42	
13	0.6		47	28	
15	0.2		49	30	
17	3.5		51	24	
19	7.6		53	32	
21	8.6		55	32	16
23	16				
25	12				
27	13				
29	12				
31	11				
33	15	6.2			

measured as described above. The results are shown in Table 6 and (Fig.10) illustrate arsenic concentration in antimony after the 1st zone passage. The slag was homogenized and the radiation intensity of  $^{76}\text{As}$  in the slag was measured with a NaI(Tl) detector (3"x 3").

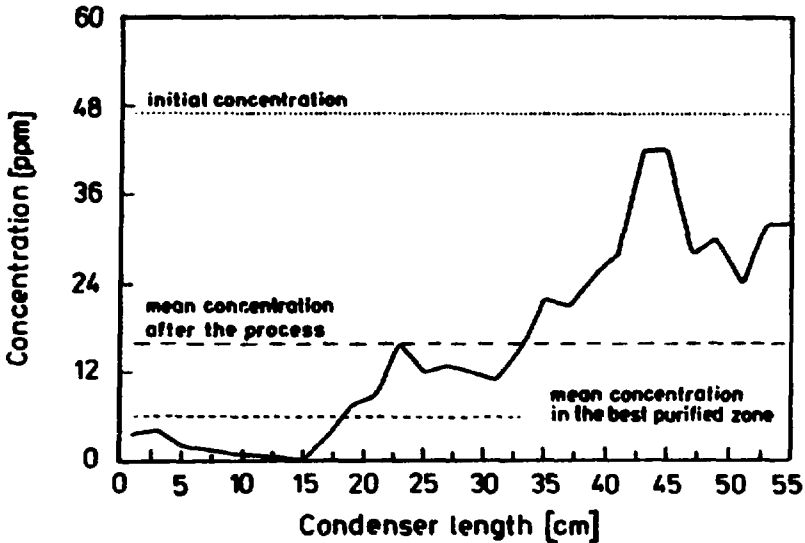


Fig.10. Distribution of As in antimony ingot after 1st passage of the purification zone (artificial slag)

The mass of the slag was about 2 g. The arsenic concentration in the slag, calculated on the basis of the above measurements, is shown in Table 5 (columns 2 and 4). The concentration of arsenic after the second zone passage was measured with the aid of a detection system of higher

Table 7. As concentration in antimony after 2nd zone passage (remelting under slags)

Sampling point along the ingot length [cm]	As concentration in antimony [ppm]
5	0.12
15	0.22
20	0.28
30	0.35
45	0.28

mean = 0.25

efficiency. That was necessary because the efficiency of the purification process was unexpectedly high; the radiation intensity of  $^{76}\text{As}$  after two passages of the zone fell below the detection limit equal to 3.5 ppm As under the applied conditions of irradiation and measurement (thermal neutron flux  $5 \times 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$ , counting time 100 s). In order to determine the arsenic concentration after the 2nd zone passage, five samples were collected from various points of the ingot. The sampling points and the corresponding arsenic concentrations in the samples are shown in Table 7. On the basis of the obtained data, the balance of arsenic in the products of the zone remelting process of antimony under artificial slags was made (Fig. 11).

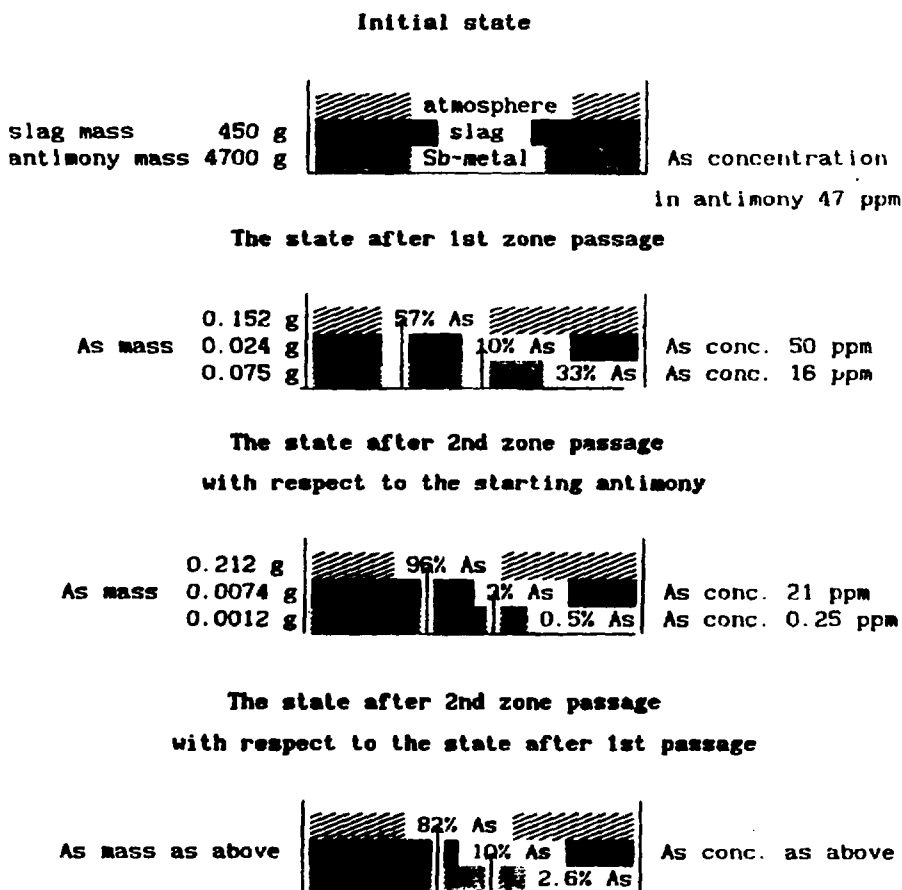


Fig. 11. Arsenic balance in the products of zone remelting of antimony under slags

## 7. DISCUSSION

The application of radiotracers in the study of processes of the production of antimony of semiconductor grade purity, makes it possible to determine the distribution of arsenic within each part of the purification system. That permits to obtain the results in a short time and in a wide concentration range (in the present case from  $10^{-2}$  to  $10^{-7}$  g/g).

In addition, on the basis of these studies interesting technological conclusions can be drawn.

The distribution of arsenic concentration in the antimony condensate obtained by vacuum distillation using unheated condenser is shown in (Fig.2 curve b). The mean concentration of arsenic in the condensate up to 7 cm from the beginning of the condenser - 151 ppm is insignificantly lower than the initial concentration - 154 ppm. The obtained data indicate that under the applied conditions purification does not practically occur.

The above distribution is the reference point for the results obtained using the heated condenser. The distribution of As in the condensate is shown in Table 1. For the heated condenser the condensate was divided into three sections 10 cm long. The mean arsenic concentrations in these sections of the ingot were 9, 20 and 46 ppm respectively. The masses of antimony were 334, 341, and 254 g, respectively (Table 2).

The degree of purification of antimony from arsenic in the zone melting process after single zone passage ranges from several to about twenty percent. After eight zone passages the purification degree is 55% for the section of antimony 210 mm long i.e. for 50% of the charge. (Table 3 and 4, Fig.9).

The attempts to determine the kinetics of the distillation process were successful. Both experiments confirm that the most intensive evaporation of arsenic in the vacuum distillation process takes place in the first few minutes of the process. It follows from these experiments that the distillation should be carried out in two stages; after a definite time the receiver should be replaced by another one and the distillation continued. However, in vacuum systems the change of the receiver can pose problems. We propose therefore to increase the temperature in the condenser, which will result in the transfer of the contaminated antimony into the space of the con-

denser that will not be used in the further distillation. The temperature of the condenser should be then decreased and the distillation continued.

The contaminated antimony can be removed from the ingot surface by local etching. All the operations proposed above should result in decreasing the arsenic content in the antimony by about 50%, because as follows from (Fig. 4) the layer of a thickness 500  $\mu\text{m}$  contains twice of arsenic than the deeper layers.

After a single passage of the zone with the ingot under slags the purification degree of antimony from arsenic was 87% for the ingot length 330 mm. The mean As concentration in antimony in this section of the ingot was 6.2 ppm (Table 6), while the mean concentration in the whole ingot was 16 ppm (Table 6, Fig. 10). It follows then that the As concentration was reduced by three times. It was found that after the 1st zone passage the mean concentration of arsenic in the slag was 50.4 ppm (Table 5, column 2). Had the total mass of arsenic (0.2 g) passed to the slag, the arsenic concentration would have been  $0.2 \text{ g}/450 \text{ g} = 488 \text{ ppm}$ . It follows then that only about 10% of the arsenic was retained in the slag. The 2nd passage results in further purification of antimony from arsenic. The As concentration in antimony after two zone passages was 0.25 ppm (Table 7). The purification degrees with respect to the starting material and to the material after the first zone passage were 99.5% (Fig. 11). The As concentration in the slag after the 2nd zone passage was 21 ppm (Table 5, column 4 and Fig. 11). The amount of arsenic retained in antimony after the first zone passage was approx. 0.075 g. Assuming that the mass of the slag does not change and that the total mass of arsenic passes to the slag the concentration of arsenic in the slag should be  $0.075 \text{ g}/350 \text{ g} = 215 \text{ ppm}$ . Hence the percentage of the retained arsenic is about 10% as well. In the above case the purification of antimony from arsenic is due not only to the fact that arsenic moves before the crystallization front, but also to the fact that volatile arsenic compounds pass to the atmosphere.

In the zone melting process without the slag about 0.2% of arsenic initially present in antimony passed to the atmosphere [4], while in the process of zone remelting under slags after the 1st and 2nd zone passages the corresponding amounts are equal to 57% and 82%, respectively.

The process of remelting under artificial slags is highly efficient. purification of antimony from arsenic takes place mainly in the proces remelting under slags and not in the zone melting process. However remelting under slags should be followed by vacuum distillation or zone melting in order to remove impurities introduced with the slag.

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