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MEASUREMENT OF DESORPTION FROM THE POLYCRYSTALLINE LAYER OF TiO_2 IN INCANDESCENT LAMPS

VIERA DUBRAVCOVÁ and JOZEF IVAN*

Dept. of Microelectronics, FEI, Slovak Technical University in Bratislava, Slovak Republic
**TESLA Nové Zámky Co., Nové Zámky, Slovak Republic*

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The pressure increase due to the desorption was measured in closed vacuum systems of incandescent lamps with tungsten filament. The inside surface of the glass bulbs was covered with light diffusing layer of TiO_2. Pressure differences were measured in clear lamps and lamps frosted with TiO_2. Desorption was stimulated by increasing the temperature for fixed intervals of time. Pressure was determined from the filament $I - V$ characteristics that were transformed to a linear plot. At 210 °C, desorption was approximately four times higher in TiO_2 coated lamps than in clear lamps.

1. Introduction

We introduce a simple method of measurement of changes of pressure in incandescent lamps. The pressure was changed by stimulated desorption due to the increased temperature for fixed time intervals.

The pressure determination method is based on the measurement of $I-V$ characteristics of lamp's filaments. The characteristics were transformed into straight line equations. Their absolute terms are dependent on the pressure.

2. The pressure determination

The classical incandescent lamp is similar to a Pirani vacuum gauge head. When its filament is heated by electrical current, thermal losses have three components: losses to electrodes and supports, losses to the gas and losses by radiation.

If constant temperature along the filament's length is presumed, the basic equation is:

$$RI^2 = k_e T + k_{vac} T + W_r \qquad (1),$$

where R is resistance, I - current, k_e - coefficient of losses to the electrodes and supports, $T = T_f - T_0$ where T_f is the temperature of the filament and T_0 is the ambient temperature, k_{vac} – coefficient of heat losses to the gas, W_r - energy emitted by radiation.

For radiation from metal, the formula found in Ref. 3 may be accepted:

$$W_r = \sigma A \left[T_f^4 (1 - e^{-\epsilon T_f}) - T_0^4 (1 - e^{-\epsilon T_0}) \right] \qquad (2),$$

where σ is the Stefan-Boltzmann constant, A is the filament surface area and ϵ is a constant, dependent on filament's surface properties and its shape (for tungsten wire $\epsilon \approx 1.47 \times 10^{-4}$ K^{-1}). After simplifying Eq. (2), the equation of straight line ($y = q + mx$) is obtained by dividing Eq. (1) by temperature T:

$$\frac{RI^2}{T} = (k_e + k_{vac}) + \epsilon \sigma A \frac{(T + T_0)^5 - T_0^5}{T}. \qquad (3)$$

Equation (3) conforms well with the experiment. Its absolute term q and coefficient m are:

$$q = k_e + k_{vac}, \quad m = \epsilon \sigma A \qquad (4)$$

and

$$y = \frac{RI^2}{T}, \quad x = \frac{(T + T_0)^5 - T_0^5}{T}.$$

The coefficient k_e may be neglected for a long thin wire and then q is proportional to k_{vac} :

$$q \simeq k_{vac} = A \lambda' \alpha p \qquad (5)$$

where λ' is thermal conductivity of the gas at 1 Pa in Wm^{-2} K^{-1} Pa^{-1} (e.g., under molecular conditions for air $\lambda' \approx 1.1$), α is accommodation coefficient (in our case, $\alpha = 0.4$ for tungsten), p is pressure in Pa.

It is possible to obtain the temperature T in (3) by numerical calculation using the temperature dependence of resistance R, which was approximated by the formula:

$$R = \frac{I}{U} = R_0 (1 + \beta_1 T + \beta_2 T^2 + \beta_3 T^3 + \beta_4 T^4) \qquad (6)$$

where R_0 is the resistance of the filament at ambient temperature T_0, measured by a weak current, and β_1, ..., β_4 are material constants.

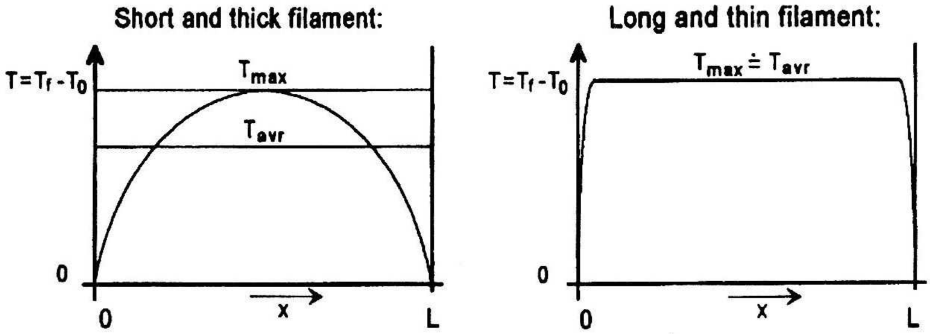

Fig. 1. The temperature profile along a heated filament in vacuum.

Hitherto, constant temperature was assumed along the lenght of the filament. Fortunately, this is acceptable to a high accuracy for long and thin tungsten filaments of incandescent lamps designed to operate at 220 V. Figure 1 shows two examples of possible solutions of the generally valid differential equation [1]

$$k_e^{'} \frac{\mathrm{d}^2 T}{\mathrm{d}x^2} - k_{vac}^{'} T - k_r^{'}[(T+T_0)^5 - T_0^5] + R^{'} I^2 = 0$$

$$T_{x=0} = T_{x=L} = 0. \tag{7}$$

3. Experimental

For our test, four lamps were randomly selected. Lamps have spherical glass bulbs of the diameter of 45 mm and tungsten filament for 225 V/40 W operation of a diameter of 25.18 μm. Inner pressure after 2 minutes of ageing was approximately 0.1 Pa. Two of these samples had bulbs frosted by new technology of TiO_2 layer deposition, and two samples were clear. Lamps were produced by the usual technology, with the exception of gas filling.

First, the value of R_0 was determined and after that five points $[U_i, I_i]$ of $I-V$ characteristics were measured (i=1,2...5). As a rule, last value of $R_i = U_i/I_i$ was approximately equal to $3R_0$.

After the first series of measurements, the samples were heated in an oven at 210 °C for 5 minutes and then, after cooling down, the second series of $I-V$ characteristics measurements were made. The third measurement was made after next 35 minutes of heating at the same temperature.

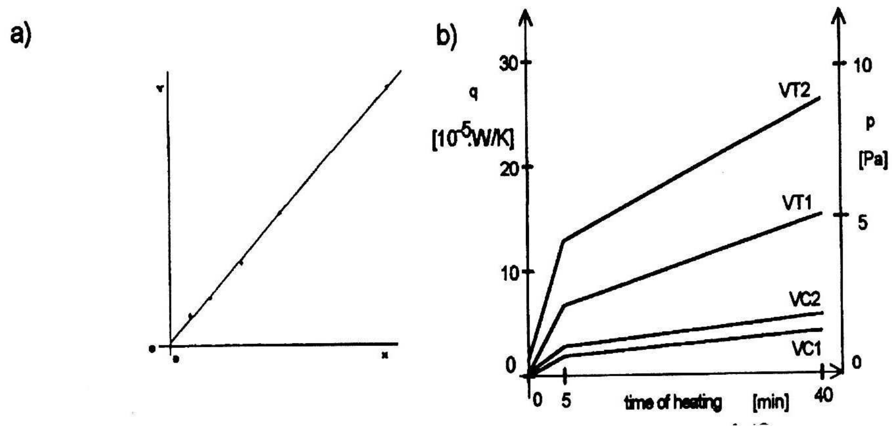

Fig. 2. a) Example of the sample VT1H0 data computing at PC (PrintScreen). Linearized characterisctics of the filament, at ambient temperature of 18 °C, resistance $R_0 = 98.204\Omega, x_1 = 5.5 \cdot 10^{10}$, $y_1 = 3.29 \cdot 10^{-5}$, $x_2 = 1.1 \cdot 10^{11}$, $y_2 = 5.13 \cdot 10^{-5}$, $x_3 = 1.9 \cdot 10^{11}, y_3 = 8.73 \cdot 10^{-5}$, $x_4 = 3.0 \cdot 10^{11}$, $y_4 = 1.39 \cdot 10^{-4}$, $x_5 = 6.0 \cdot 10^{11}$, $y_5 = 2.73 \cdot 10^{-4}$. If $y = q + mx$, where q depends on the pressure, $q = 3.76 \cdot 10^{-6}, m = 4.45 \cdot 10^{-16}$, and the correlation coefficient is 0.9987. b) Dependence of the coefficient q and the pressure p on heating time of the incandescent lamps. Heating temperature was 210 °C. Desorption was considerably higher in the samples with TiO_2 layer (VT1 and VT2) than in the clear ones (VC1 and VC2).

All data have been processed by a PC computer program. An example of the data is shown in Fig. 2. Results of numerical calculations are given in Table 1. The following constants were used to calculate the temperatures T_i: $\beta_1 = 4.5 \times 10^{-3}$ K^{-1}; $\beta_2 = 1.15 \times 10^{-6}$ K^{-2}; $\beta_3 = -3.1 \times 10^{-10}$ K^{-3}; $\beta_4 = 3.91 \times 10^{-14}$ K^{-4}.

TABLE 1.

Results of numerical calculations. Coefficient q is proportional to the pressure in bulbs ($p \simeq q/(2.95 \times 10^{-5}$ Pa)). Desorption was considerably higher in samples VT1 and VT2. Notation: VC1, VC2 - clear samples; VT1, VT2 - coated with TiO_2.

Sam.	Heated 0 minutes (H0)			Heated 5 minutes (H5)			Heated 40 minutes (H40)		
	q [10^{-5} W/K]	m [10^{-16} W/K^5]	press. p [Pa]	q [10^{-5} W/K]	m [10^{-16} W/K^5]	press. p [Pa]	q [10^{-5} W/K]	m [10^{-16} W/K^5]	press. p [Pa]
VC1	-0.17*	4.73	-0.057*	1.81	5.12	0.614	4.24	4.14	1.437
VC2	0.51	4.64	0.173	2.87	4.63	0.973	5.74	3.94	1.946
VT1	0.38	4.45	0.129	6.66	5.12	2.258	14.85	3.4	5.034
VT2	1.67	4.42	0.566	13.03	4.1	4.417	26.23	4.94	8.892
Note: *value is negative due to uncertainties in the measurement and to imperfection of the linear model. This case is usual at low pressure.									

Electrical values were determined with an accuracy of ±2%. Correlation coefficients of linear regression were in the range of 0.9464 to 0.99958.

Results in Table 1 are shown in graphical form in Fig. 2.

4. *Conclusions*

The results of measurements agree with expectation. Desorption of the surface covered by the polycrystalline layer of TiO_2 was approximately 4-times higher than desorption of the clear one.

The method of pressure determination was very simple, non-destructive, cheap and relatively fast. It was used with success in many other cases, i.e. for getter efficiency measurements in lamps.

However, it has some disadvantages:

– it does not give information on gas composition;

– pressure range is limited (approximately 0.1 to 100 Pa, depending on filament dimensions);

– there are uncertainties due to the imperfect linear model when heat losses of filament and emissivity of its surface are dependent on its average temperature.

References

1) A. Bláha, *Vákuové merania*, (Vacuum Measurements, SVTL publisher, Bratislava) (1960) p. 153;

2) K. Rektorys et al., *Prehled uzite matematiky*, (Overview of the Applied Mathematics, published by SNTL Prague) (1988);

3) A. P. Ivanov, P. *Elektricheskije istochniki sveta I.- Lampi nakalivania*, (Electric Light Sources I. - Incandescent Lamps, GONTI publisher, Moscow) (1938) p. 95;

4) M. Ruschitzka and W. Jitschin, *Physikalische Grundlagen des Wärmeleitungs - Vakuummeters*, Vakuum in der Praxis, Nr.1 (1993) p.37;

5) V Dubravcová, *Vákuová a ultravákuová technika*, (Vacuum and Ultravacuum Technics, published by ALFA Bratislava) (1992).

MJERENJE DESORPCIJE IZ POLIKRISTALNOG SLOJA TiO_2 U NITNIM ŽARULJAMA

Povećanje tlaka zbog desorpcije mjereno je u zatvorenim vakuumskim žarulja s volframovom niti. Unutarnja strana stijenki bila je prekrivena svjetlo-difuznim slojem TiO_2. Uspoređivale su se prozirne i 'bijele' žarulje. Desorpcija je stimulirana zagrijavanjem tijekom odredenog intervala vremena. Tlak se mjerio odredivanjem $I-V$ karakteristika žarne niti. Pri 210 °C ustanovljeno je da je desorpcija iz žarulja s TiO_2 oko četiri puta veća nego u prozirnim žaruljama.