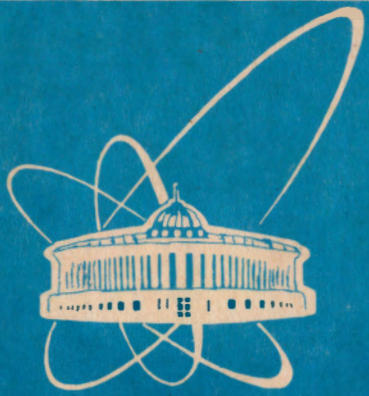

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VACUUM SCALING RELATIONSHIPS AND OUTGASSING RATES OF SOME MATERIALS USED IN CYCLOTRON VACUUM TECHNOLOGY

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Расчет вакуумных систем и газовыделение некоторых материалов, используемых при конструкции циклотронов

В работе рассмотрен принцип расчета вакуумных систем циклотронов и ионопроводов. Приведены данные газовыделения нержавеющей стали без прогрева и с прогревом до температуры 300°С. Определены экспериментальные данные удельных величин газоотделения конструкционных материалов, элементов уплотнений, применяемых в ускорительных и экспериментальных установках (сталь, текстит — текстолит, епокси «К4», тефлон, витон, и «О» ринг — ПЕРБУНАН-Н). Представлены спектры остаточных газов анализируемых материалов.

Работа выполнена в Лаборатории ядерных реакций им.Г.Н.Флерова ОИЯИ.

Сообщение Объединенного института ядерных исследований. Дубна, 1994

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Vacuum Scaling Relationships and Outgassing Rates of Some Materials Used in Cyclotron Vacuum Technology

A review of vacuum scaling relationships and outgassing rates of some materials used in the cyclotron vacuum technology in given. Scaling relationships are presented as a basis for understanding the vacuum configuration of the cyclotron beam lines. Typical, total specific outgassing rates for unbaked and baked beam pipe made of stainless steel together with specific outgassing rates of other materials used in construction of the cyclotron beam lines (steel «3», textit, epoxy «K4», teflon, viton, currently used «O» ring — PERBUNAN-N from Roumania) and its mass spectra and background of the tested chamber are shown.

The investigation has been performed at the Flerov Laboratory of Nuclear Reactions, JINR.

Communication of the Joint Institute for Nuclear Research. Dubna, 1994

1. Introduction

In the design and selection of suitable pumping systems for use in the cyclotron beam lines constructions, scaling relationships and outgassing characteristics of materials exposed to vacuum are utilized [1 - 2]. The scaling relationships allow us to determine the pressure profile along the beam pipe, the maximum pressure between pumps and conductances for different flows. The outgassing data are obviously based upon the throughtput measurements controlled by monopole (quadrupole) or other residual gas spectrometers. A variety of materials have been tested at room temperature. Test results are presented for materials as teflon, viton, currently used 'O' ring (Perbunan - N from Roumania) at our laboratory, textile glue epoxy resin (textolit - textit), and steel samples.

2. Scaling Relationships

The highest obtainable average pumping speed of the cyclotron beam line vacuum system strongly depends on the beam pump conductance. To illustrate this important limitation caused by the finite conductance, let us consider the system shown in Fig. 1. In the molecular flow regime the flow of molecules along the vacuum pipe to the nearest pump is expressed by the equation [3]

$$Q(x) = -w\frac{dp}{dx} \qquad \frac{dQ}{dx} = Aq \ , \tag{1}$$

where Q is the gas flow [Pa m^3 s^{-1}], w is the specific molecular conductance [m^4 s^{-1}] (w = LC), C is the conductivity [m^3 s^{-1}], p is the pressure inside the pipe [Pa], A is the specific surface area [m] (A = F/L), F is the surface area [m^2] and q is the specific outgassing rate (uniform) [Pa m s^{-1}]. These equations can be combined to give

$$w\frac{d^2p}{dx^2} = -Aq \tag{2}$$

together with the boundary conditions of this simple problem

$$\frac{dp}{dx}\Big|_{x=L/2} = 0 \quad \text{and} \quad p|_{x=0} = \frac{AqL}{S} \tag{3}$$

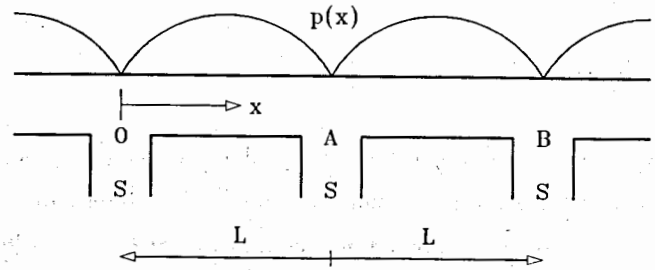

Fig. 1. Outline of the cyclotron beam line pumping system. S is the pumping speed of the pump in points "O", "A", "B" [m^3 s^{-1}], L is the distance between pumps [m] and x is the distance measured from the reference point "O" [m].

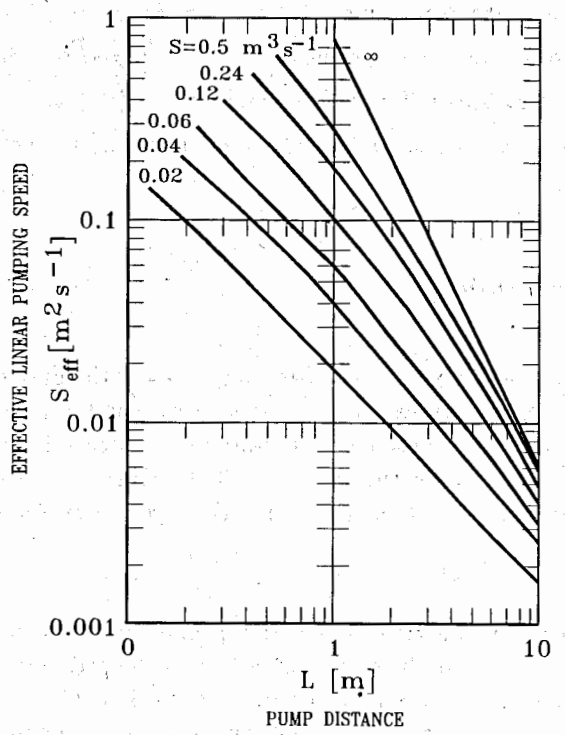

Fig. 2. Effective linear pumping speed S_{eff} as a function of pump distance L for various sizes of pumps, a vacuum beam pipe conductance w = 0.07 m^4 s^{-1} and different pumping speeds S = 0.02, 0.04, 0.06, 0.12, 0.24, 0.5 and ∞ m^3 s^{-1}, respectively.

which follow from evident symmetry considerations. As the solution we can find a well known parabolic pressure profile along the beam pipe

$$p(x) = Aq\Big(\frac{Lx - x^2}{2w} + \frac{L}{S}\Big). \tag{4}$$

The maximum pressure occurs at the midpoint between pumps

$$p_{max} = Aq\Big(\frac{L^2}{8w} + \frac{L}{S}\Big). \tag{5}$$

For the beam pipe the average pressure is more relevant

$$p_{av} = \frac{1}{L}\int_0^L p(x)dx = Aq\Big(\frac{L^2}{12w} + \frac{L}{S}\Big). \tag{6}$$

It is convenient to define an "effective linear pumping speed"

$$S_{eff} = \Big(\frac{L^2}{12w} + \frac{L}{S}\Big)^{-1} \tag{7}$$

so that $p_{av} = Aq/S_{eff}$. It is evident that S_{eff} cannot exceed $12w/L^2$ irrespective of how large pumps are used.

Equally, the lowest achievable average pressure is limited to $AqL^2/12w$. Since the conductance is generally determined by the appertures in electrodes and screens (screening of the high frequency), the only parameter is the interpump distance L. Obviously, many small pumps at short distance are preferable to a few and large pumps. Fig. 2 illustrates this effect for a vacuum beam pipe conductance of 0.07 m^4 s^{-1} and different pumping speeds S (0.02, 0.04, 0.06, 0.12, 0.24, 0.5, ∞ m^3 s^{-1}), respectively. It is shown that the effective pumping speed is practically the 'linear' function of the pump distance for various sizes of pumps. Such a pumping structure may be installed along the vacuum system and hence the analysis provides a 'linear pumping speed' inside the beam pipe.

The real pressure distribution and therefore the average pressure also follow from the equation similar to the previous one

$$-w\frac{d^2p}{dx^2} = Aq - sp, \tag{8}$$

where s is the specific pumping speed defined as $s = S/L$ [m^2 s^{-1}]. By the solution of differential equation (8) using the boundary conditions (3) we find other pressure profiles along the beam pipe. It holds that

$$p(x) = C_1e^{rx} + C_2e^{-rx} + \frac{Aq}{s}, \tag{9}$$

where

$$C_1 = \frac{AqL}{S} e^{-rL}(1+e^{-rL})^{-1}, \quad r = (s/w)^{0.5}$$

and

$$C_2 = \frac{AqL}{S}(1+e^{-rL})^{-1}.$$

Usually, the vacuum system is designed with respect to s≪w. In this configuration one can obtain

$$C_1 \cong C_2 \cong C_o = \frac{Aq}{2}\frac{L}{S}. \tag{10}$$

The equation (9) is reduced to

$$p(x) = C_o(e^{rx} + e^{-rx}) + \frac{Aq}{s} \tag{11}$$

and the average pressure is determined by

$$p_{av} = \frac{C_o}{rL}(e^{rL} - e^{-rL}) + \frac{Aq}{s} \cong \frac{2AqL}{S}. \tag{12}$$

It is evident that the lowest achievable pressure depends only on the ratio of 2AqL/S. The average pressure versus the distance between pumps L for different pumping speeds S (0.02, 0.04, 0.06, 0.12, 0.24, 0.5 and ∞ $m^3\ s^{-1}$) is shown in Fig. 3.

3. Molecular Conductance

In high vacuum the flow of molecules is limited by wall collisions, i.e. the mean free path λ of the gas molecules is greater than the characteristic dimensions of the vacuum system ($D/\lambda < 1$; D is the diameter of the beam pipe). In this molecular flow regime, the conductance C [$m^3\ s^{-1}$] is independent of the pressure, therefore the flux of gas is proportional to the pressure gradient $\triangle$p. It holds that

$$Q = C \triangle p. \tag{13}$$

Some useful expressions for conductance are [1]:

a) Orifice of area F [m^2];

$$C = 36.4F\sqrt{\frac{T}{M}} \quad m^3\ s^{-1}, \tag{14}$$

Table 1. Total specific outgassing rate q for unbaked and baked beam pipe made of stainless steel [2]

Process	Temperature [K]	q [Pa m s^{-1}]
unbaked 100 h **pumpdown**	293	10^{-6}
baked 30 - 150 h at 300 °C	573	10^{-9}

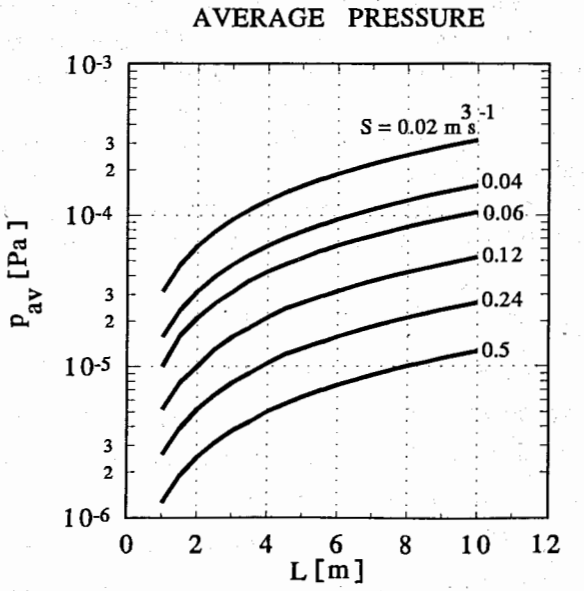

Fig. 3. Plot of the average pressure p_{av} versus distance L between pumps for different pumping speeds S = 0.02, 0.04, 0.06, 0.12, 0.24, 0.5 and ∞ $m^3\ s^{-1}$, respectively.

b) long cylindrical tube, radius r [m], length L [m];

$$C = 305 \frac{r^3}{L} \sqrt{\frac{T}{M}} \qquad \mathrm{m^3\ s^{-1}} \tag{15}$$

c) long tube with elliptic section (semi-axes a, b), a [m], b [m];

$$C = 431 \frac{a^2 b^2}{L(a^2+b^2)^{1/2}} \sqrt{\frac{T}{M}} \qquad \mathrm{m^3\ s^{-1}}, \tag{16}$$

where T is the absolute temperature [K] and M are the molecular weight.

The molecular flow through complex systems can be computed with Monte Carlo programs adapted to the specific geometry.

4. Outgassing of the Beam Pipe

For every vacuum system the size of the required pumps is directly related to the outgassing. The first important source is the static and thermal outgassing of weakly adsorbed molecules and diffusion of H_2 from the bulk of the material. The standard procedure to reduce the thermal outgassing is the well known bakeout of the beam pipe. The pressure inside an unbaked system is mainly determined by water vapours. In a clean and well baked system H_2 will be the dominant residual gas constituent. Typically, total specific outgassing rate q for unbaked and baked beam pipe made of stainless steel is given in Table 1. Therefore, the thermal outgassing rate dQ_T/dt of the surface area F = 0.1 m^2 is given by

$$\frac{dQ_T}{dt} = Fq = 10^{-7}\ \mathrm{Pa\ m^3\ s^{-1}}. \tag{17}$$

The second important source of gas in the beam line is the so - called 'dynamic' outgassing in presence of the beam. Here, strongly bound molecules can be desorbed which could not otherwise contribute to the thermal desorption.

The outgassing characteristics of several materials used in vacuum technology are shown in Fig. 4 [4]. The quantity of the outgassing rate very strongly depends on the finished treatment with materials (untreated; degreased: solvent, vapour; polished: mechanical, chemical, blast, electro; baked: non - metals at 80 - 100 °C up to 24 h, metals at 300 - 400 °C up to 100 h). It is also shown

that the best metals used in the vacuum technology are Al, Cu and stainless steel. Then, in order to obtain the specific outgassing rate less than 10^{-7} Pa m s^{-1} the vacuum systems are not using RUBBER, POLYAMIDS, EPOXY and PTFE - TEFLON materials, respectively.

The outgassing characteristics of materials exposed to vacuum are also published in papers [5-7].

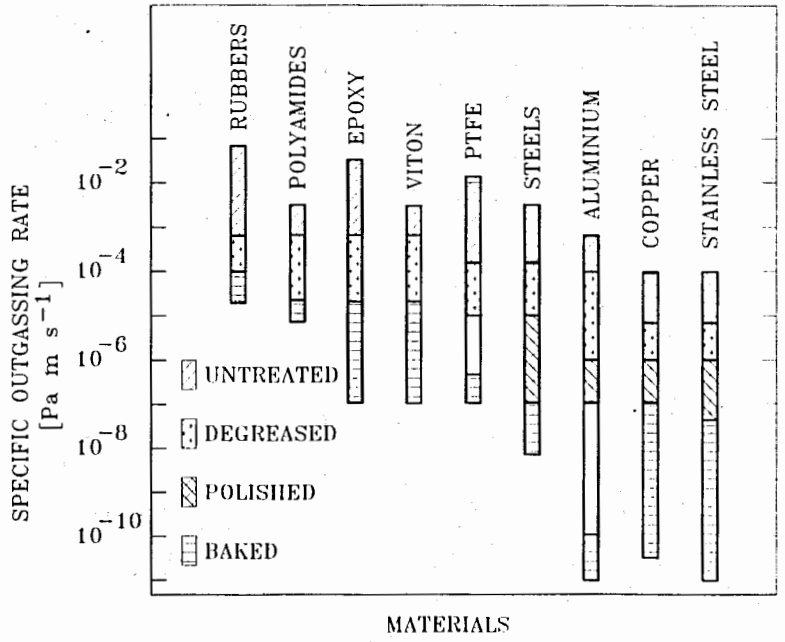

Fig. 4. Outgassing characteristics of several materials used in vacuum technology [4].

5. Residual Gas Species Results

Outgassing characteristics of several materials were studied in vacuum at room temperature for periods as long as 48 h. Samples were placed in a stainless steel vacuum chamber which was evacuated by a 0.5 $m^3\ s^{-1}$ turbomolecular pump. The vacuum chamber volume and surface area were approximately 0.0025 m^3 and 0.12 m^2, respectively. Pressures were measured from 0.1 MPa to 10^{-6} Pa by using a combination of thermocouple, Pirani, Bayard -Alpert and Penning gauges.

Typically, the test chamber and connecting lines were conditioned between test runs using a vacuum bakeout at about 150 °C at a base pressure of about 10^{-4} Pa. Concurrently, the RGA (Residual Gas Analyser) gauge was baked out at 150 °C. A minimum of 10 h was allowed for the chamber to cool to room temperature prior to loading the next test samples.

The residual gases which were evolved from the samples under test were determined using the zero order method of peak height summation and by subtracting the spectra measured of the empty test chamber from the spectra measured with the samples.

5.1 Vacuum Stand

Scheme of the stand is shown in Fig. 5. It is designed to pump the vacuum chamber with diagnostic elements and gauges to the pressure of 10^{-6} Pa. The entire system including the holder of the samples is constructed of stainless steel, combined with copper and steel materials, respectively. One rotary pump is used in order to produce the forvacuum (BL 90) [8].

The rotary and turbomolecular pumps continuously pump the whole system. The rotary pump is also used for slow - acting evacuation of the tested chamber before connection of the turbomolecular pump.

Proceeding from the dimensions of the vacuum system an effective speed S_{eff} is calculated for the center of the stand chamber. There was found to be: S^{ch}_{eff} = 0.1 $m^3\ s^{-1}$ for air at 10^{-5} Pa. The reduced pumping speed of the turbomolecular pump is mainly due to the small diameter of the used pipes and trap, respectively.

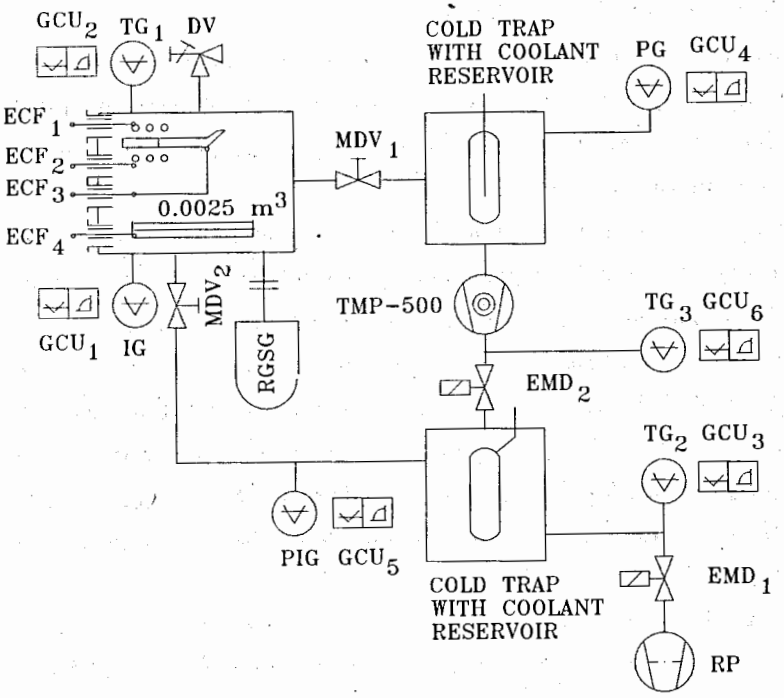

Fig. 5. Scheme of the stand used for measuring of the RGA spectra. RP is rotary pump, TMP is turbomolecular pump, ECF is electrical current feedthrough, DV is dosing valve, RGSG is residual gas spectrometer gauge, MDV is manual drive valve, EMD is electro - magnetic drive valve, PG is Penning gauge, IG is ionization gauge, TG is thermocouple gauge, PIG is Pirani gauge, and GCU is gauge control unit.

Outgassing rates are summarized in Table 2. It is seen that the VITON 'O' ring is better than PERBUNAN - N by a factor of 40 times and TEFLON is worse than STEEL by a factor of 50 times. The worst materials are the PERBUNAN-N, TEXTIT and TEFLON.

The outgassing rates presented for the VITON are the same as those reported in [9]. Similarly, our results again indicate not to recommend the use of the PERBUNAN-N, TEFLON and TEXTIT for clean interior surfaces of the cyclotron beam line vacuum systems.

The RGA spectra are shown in Figs. 6 - 10. The spectra are measured by the RGA spectrometers MX 7304 (monopole) [10] and IPDO - 2A (omegatron) [11], respectively. The spectrometer gauges are placed directly on the measuring chamber of the stand (Fig. 5). By spectra analysis one can see the peak sets as 27, 29; 39, 41, 43; 55, 57, 58; 69; 77, 78; 85, 87; 101, 103, 105; 130, 132, 134, 136; 149, 151, 153, 155; 198; corresponding to the characteristic clusters of the n - C_x H_y hydrocarbons, or m - C_r Cl_w F_z freons. These fragments arise also during the ionization processes from oils used in the backing pumps, some diffusion pumps and turbomolecular pumps.

In addition to the spectra the expressive water vapor contamination is illustrated,but there are also the peaks as 135, 137, 138, 140, 148 and 220 which can give rise to some difficulties in interpretation, mainly if the vacuum chamber is made of the stainless steel and if it is devided from the vacuum pumps by two liquid nitrogen traps. Nevertheless, by means of the spectral catalogs, where the spectra of the pure substances are standardized, it is possible to find the appropriate substances in the majority of cases.

Table 2. Specific outgassing rates q for some materials used in the cyclotron vacuum technology; PERBUNAN - N is produced in Roumania and other ones are produced in Russia

Sample	Exposure [h]	Pressure [Pa]	q [Pa m s^{-1}]
STEEL "3"	24	8×10^{-5}	3.6×10^{-6}
TEFLON	20	3.5×10^{-6}	2×10^{-4}
TEXTOLIT - TEXTIT	20	3.5×10^{-5}	1×10^{-3}
EPOXY	48	4×10^{-6}	8×10^{-6}
'O' RING - PERBUNAN -N	24	2.5×10^{-4}	2×10^{-3}
'O' RING - VITON	24	7×10^{-6}	6.5×10^{-5}

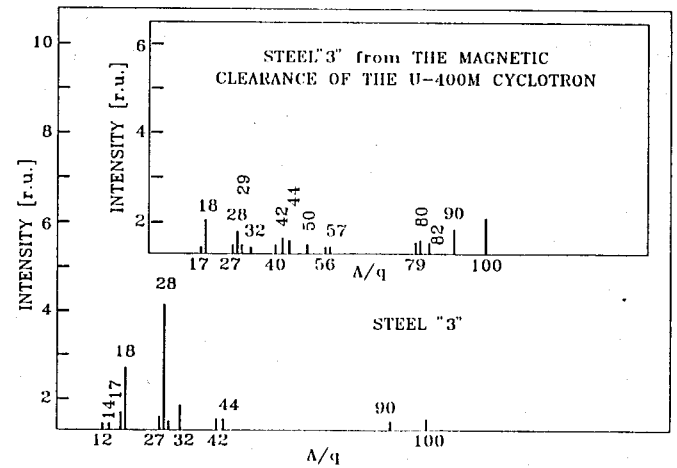

Fig. 6. Spectrum of STEEL "3" samples from the plasma of the RGA spectrometer IPDO - 2A (omegatron). Measuring conditions were: STEEL "3" - pumping time - 24 h; temperature - 20 °C; pressure - 8×10^{-5} Pa and desorption rate - 3.6×10^{-6} Pa m s^{-1}. STEEL "3" from the magnetic clearance of the U-400M cyclotron - pumping time - 24 h; temperature - 20 °C: pressure - 1×10^{-6} Pa and desorption rate - 1×10^{-6} Pa m s^{-1}.

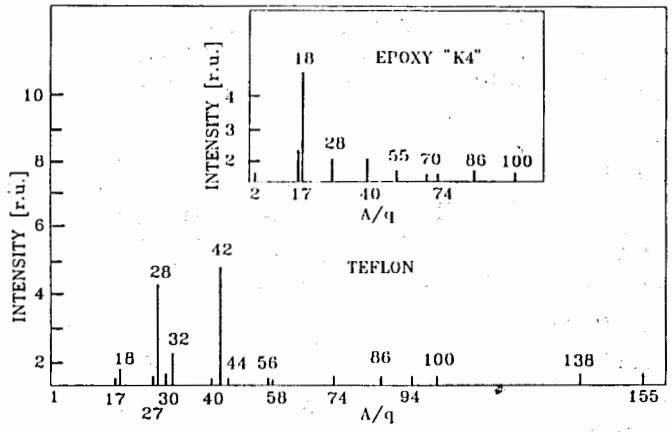

Fig. 7. Spectrum of TEFLON and EPOXY "K4" samples from the plasma of the RGA spectrometer IPDO - 2A (omegatron). Measuring conditions were: TEFLON - pumping time - 20 h; temperature - 20 °C; pressure - 3.5×10^{-6} Pa and desorption rate - 2×10^{-4} Pa m s^{-1}. EPOXY "K4" - pumping time - 48 h; temperature - 20 °C; pressure - 4×10^{-6} Pa and desorption rate - 8×10^{-6} Pa m s^{-1}.

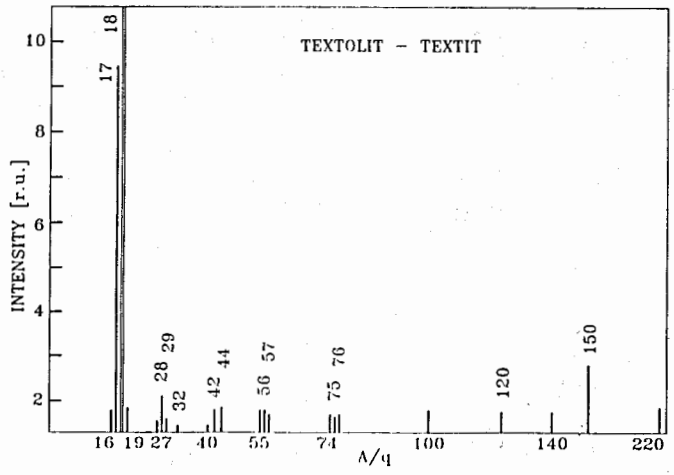

Fig. 8. Spectrum of TEXTIT from the plasma of the RGA spectrometer IPDO - 2A (omegatron). Measuring conditions were: Pumping time - 20 h; temperature - 20 °C; pressure - 3.5×10^{-5} Pa and desorption rate - 1×10^{-3} Pa m s^{-1}.

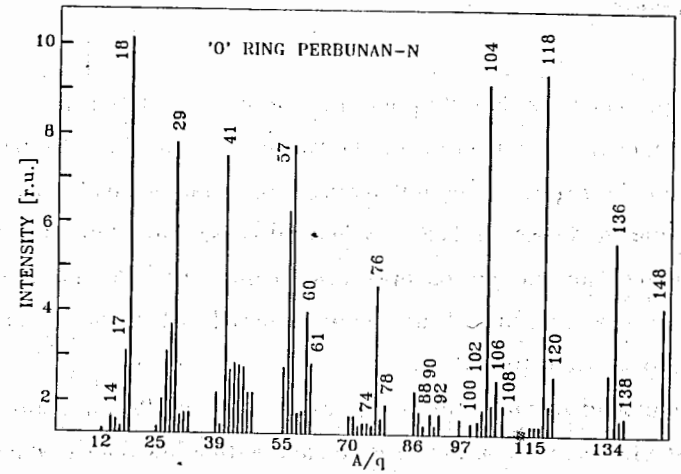

Fig. 9. Spectrum of 'O' RING PERBUNAN -N from the plasma of the RGA spectrometer MX 7304 (monopole). Measuring conditions were: Pumping time - 24 h; temperature - 20 °C; pressure - 2.5×10^{-4} Pa and desorption rate - 2×10^{-3} Pa m s^{-1}.

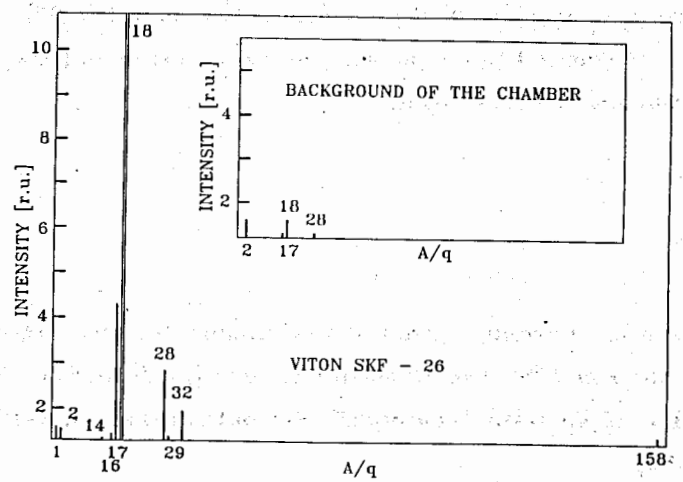

Fig. 10. Spectrum of 'O' RING VITON SKF - 26 and background of the chamber from the plasma of the RGA spectrometer MX 7304 (monopole). Measuring conditions were: 'O' RING VITON - pumping time - 24 h; temperature - 20 °C; pressure - 7×10^{-6} Pa and desorption rate - 6.5×10^{-5} Pa m s^{-1}. BACKGROUND of the chamber - pumping time - 72 h; temperature - 20 °C; pressure - 8×10^{-7} Pa and leakage - 1.3×10^{-8} Pa m^3 s^{-1}.

6. Conclusion

The present work describes not only the scaling relationships used at the construction of the cyclotron beam line vacuum systems but also provides the new data in the region of the outgassing rates and the RGA spectra for the different solid materials. Taken together, all these data show that it may be impossible to obtain the suitable operation vacuum (10^{-5} Pa) and the necessary cleanliness of the beam lines if the vacuum exposed surfaces have higher specific outgassing rates than 10^{-4} Pa m s^{-1}.

We recommend to make the beam tubes of the cyclotron beam lines with stainless steel, steel with stable structure and with a low relative magnetic permeability. The aluminium and copper can also be successfully used because of good mechanical properties, availability and very low desorption rates. It is practically impossible to separate the effects of outgassing, diffusion, and permeation which are manifold higher in polymers than in metals [12].

We don't recommend to use polymers for clean interior surfaces of the cyclotron beam line vacuum systems.

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