PROPERTIES OF PETP TRACK MEMBRANES OF DIFFERENT THICKNESSES

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Abstract. The basic properties of polyethylene terephthalate (PETP) track membranes (TM) made of the films with the thicknesses of 10 and 20 μm are investigated. The membranes with the pore diameters of 0.2, 0.4 and 1 μm were chosen for comparative study. The porous structure of the membranes was characterized by the following set of parameters: pore density, pore size, bubble point, pore radius distribution measured by the Coulter porometer. The tensile strength, water and gas flow rate were determined for both types of the TMs. The filtration processes of liquid and gaseous media were investigated. Advantages and disadvantages of "thin" and "thick" TM are discussed.

1. INTRODUCTION

For many years track membranes were produced of polymer films with the thickness of about 10 μm [1,2]. Several reasons caused the use of such thin material. First, fission fragments and accelerated heavy ions with the energy of 1 MeV/nucleon have rather short range. Heavy fraction of the fission fragments does not penetrate PET layer of 15 μm thickness. Similarly, the range of 1 MeV/nucleon xenon ions (the ions of this kind were accelerated on the U-300 cyclotron at FLNR JINR) is ca. 17 μm which sets an upper limit on the thickness of the track membrane. Second, according to theory the thicker is the membrane, the lower are the permeability and the rate of filtration process. That is why many attempts were done at the FLNR to manufacture TM with the thickness of 3 or 5 μm. The samples of such membranes showed very high initial flow rate but very short life time due to rapid plugging by particles to be separated.

Recently the beams of accelerated heavy ions with the energy higher than 1 Mev/nucleon have become available at U-400 in Dubna and at some other accelerators. It gave us a possibility to produce membranes with the thickness of 20 or even 30 μm. Study of the properties of these "thick" track membranes is of great practical interest.

2.EXPERIMENTAL

The samples of TM's were produced of biaxially oriented PET films with nominal thicknesses of 10 and 20 μm. The foil of 10 μm thickness was irradiated by xenon ions with the energy of 1 MeV/nucleon on U-300 cyclotron. The irradiation of the films with the thickness of 20 μm was carried out by 3 MeV/nucleon krypton ions on the U-400 cyclotron of FLNR. Since the mechanical strength of the thicker film is generally higher than that of the thinner one the irradiation conditions were chosen so as the track densities in the films of 20 μm thickness were higher than in the films of 10 μm thickness (in the following we denote them by TM20 and TM10, respectively). Both films were subjected to ultraviolet sensitization and subsequent chemical etching by alkali solutions in a usual way.

The porous stucture characteristics of the TM samples were measured by a set of different methods. The pore density and the size of openings on the surface

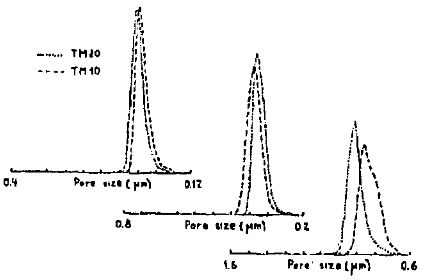

Fig. 1. Differential flow distributions (arbitrary units) as functions of pore diameter for the pairs of track membranes with nominal pore size of 0.2, 0.4 and 1 μm (from left to right)

were estimated by means of a scanning electron microscope (JSM-840, JEOL). The bubble point was measured with the use of ethanol as a wetting agent. The Coulter porometer II was used to determine pore size distribution and the values of mean flow pore size in each sample. The size factor was set equal to 0.48 (wetting liquid: Porofil). The burst strength was determined as a differential pressure which breaks 1 cm^2 unsupported membrane sample. The air flow rate was measured by means of the Gilmont flowmeters (pressure drop of 0.1 bar, sample area of 1 cm^2). The flow rate of destilled prefiltered water was measured for the samples of 50 mm in diameter (at the differential pressure P of 0.2 up to 0.7 bar). The filtration performance of the TM's was tested with tap water under dead end conditions. Retention efficiency in the process of air filtration was measured using laser particle counter MET-ONE type A2120.

For comparative study we have selected from a number of TM20's the samples with the bubble points and mean flow pore (MFP) sizes as close as possible to the corresponding parameters of the TM10's. The differential flow distributions measured by the Coulter porometer for three pairs of TM's are shown in Fig. 1.

3. RESULTS AND DISCUSSION

The parameters of the membranes are summarized in Tables 1-3. At least three measurements were performed to find the value of each parameter. In order to calculate pore diameters from the measured water flow rates given in the tables, Poiseuille flow through the pores was assumed [3]. The values of effective pore diameters were also calculated from the gas flow data using an appropriate computer program. The close agreement of pore sizes obtained by these two means demonstrates the validity of the assumptions used for calculations.

It is clearly seen from the data presented in Tables 1-3 that there is a difference in pore shape of TM10 and TM20. TM10's have cylindrical pore channels whereas pores in TM20's are tapered. It shows that etch rate ratio in the process of TM20 production is smaller because the average energy loss rate of 3 MeV/n Kr ions in the PET film is lower than that of 1 MeV/n Xe ions. Another possible reason may be a lower efficiency of oxidation process in tracks in thicker film because of longer time of oxygen diffusion into the film matrix. In the SEM picture (Fig. 2a) one can see the difference between the pore size on the surface and in the depth of the membrane of 20 μm thickness with the nominal pore diameter of 0.4 μm . In contrast to TM20, there is almost no similar effect in case of TM10 (see Fig. 2b).

TM10's and TM20's have different pore channel shapes and lengths but nevertheless they are characterized by almost identical pore size distributions measured by the porometer (Fig. 1). It allows us to assume that the diameter of cylindrical pores in the TM10 is very close to the diameter at the interior of tapered channels in the TM20 with the same nominal pore size.

In spite of higher porosity the thicker membranes show tensile strength about 10 per cent higher. It gives a significant advantage to TM20's.

Water and air flow rates for TM20's exceed those for TM10's although increasing thickness must have led to decreasing throughput. Obviously, the conical shape of pore channels and higher pore density of the TM20's compensate the influence of membrane thickness.

Filtration throughput of tap water for

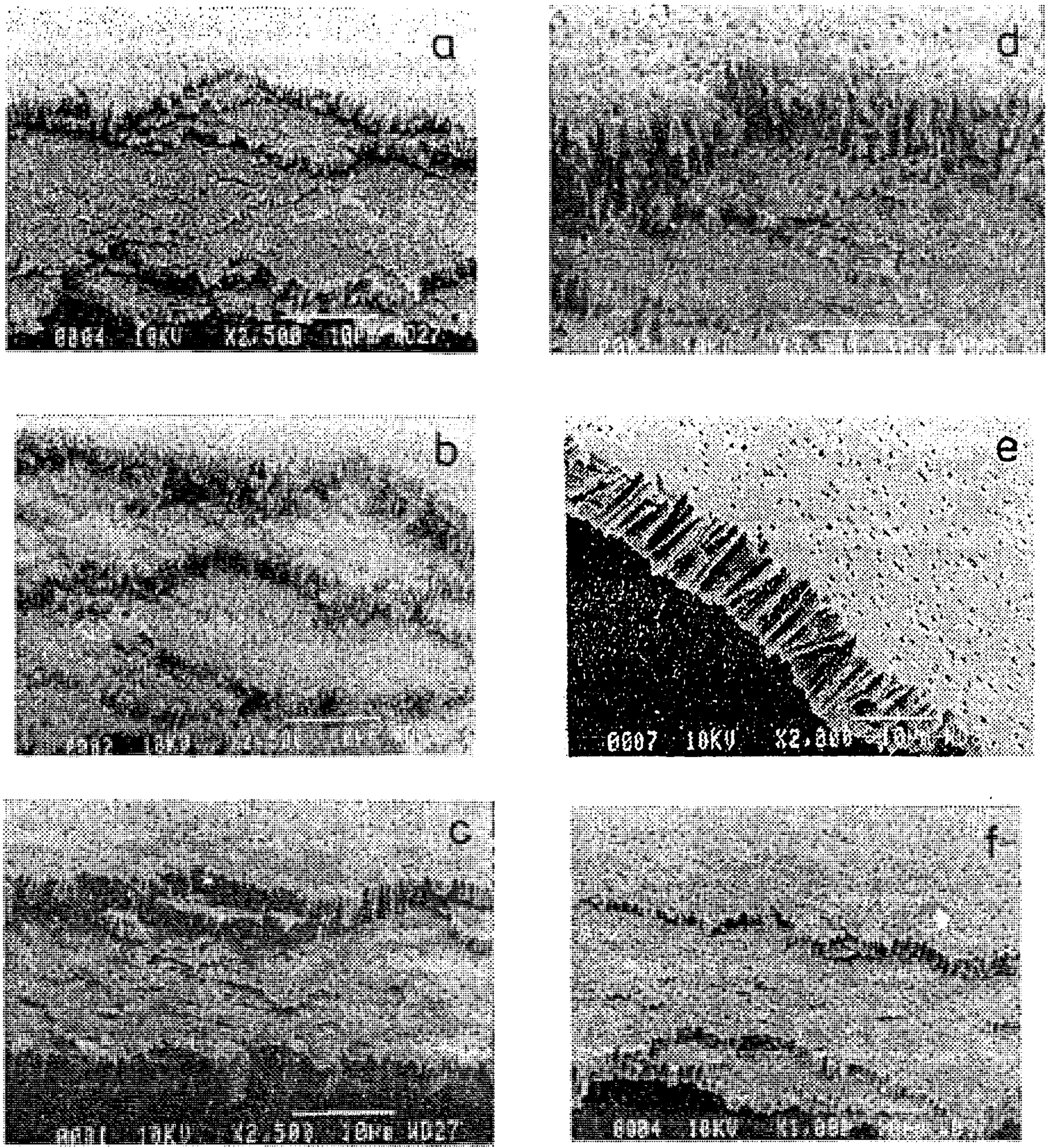

Fig. 2. SEM photographs of the surfaces and the edges of TM's broken in liquid nitrogen. a: thickness l = 10 μm, nominal pore size d=0.2 μm; b: l =20 μm; d=0.2μm; c: l =10 μm, d=0.4 μm; d: l =20 μm, d=0.4 μm; e: l =10 μm, d=1 mm; f: l =20 μm, d=1 μm

each pair of TM10's and TM20's as a function of time is shown in Fig. 3. In all three cases both initial filtration rate and dirt loading capacity of the TM20's are higher. In our opinion, the higher performance of the thick membranes can be explained by the higher pore density and intersections of pore channels in the membrane matrix. The latter can be explained as follows. As it is known, the tracks created in the PETP film at the stage of irradiation on the cyclotron have an angle distribution in a plane perpendicular to the film surface. Due to different angles of incidence the neighbouring pores can intersect each other in the bulk of the matrix (see photos in Fig. 2). The number of

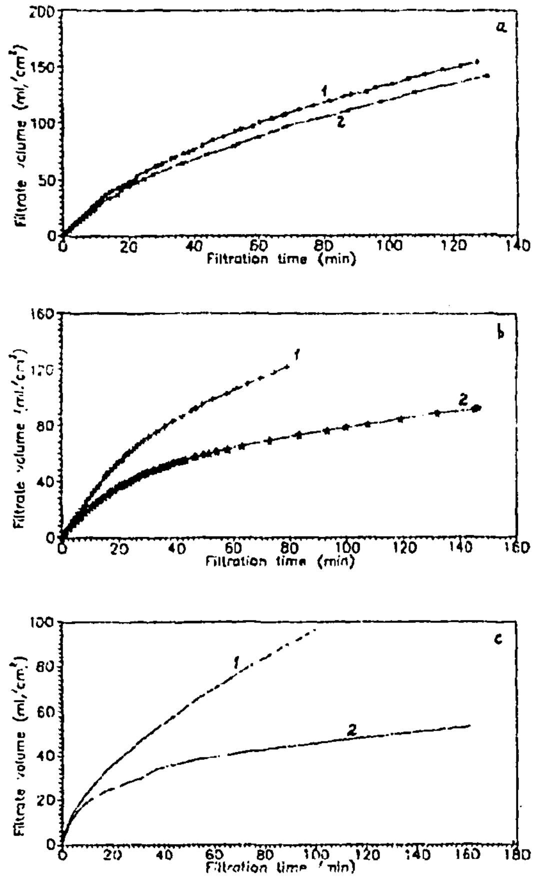

Fig. 3. The filtration throughput of tap water for the membranes with nominal pore sizes of 0.2 μm (a)., 0.4 μm (b) and 1 μm (c). The membrane thicknesses are 20 μm (1) and 10 μm (2). The inlet pressure is 0.8 bar

intersections increases with increasing thickness of the membrane. Fig. 4 illustrates the real porous structure of two membranes having 0.2 μm pores and the thicknesses of 10 and 20 μm. In the process of filtration some pores can be plugged by particles. In Fig. 4 is shown the situation when only one pore left open. The plugged pores which intersect open one participate in the total flux of the liquid through the membrane. The thicker the membrane, the greater number of pores are involved in the filtration process. This could be the reason why increasing thickness does not reduce the filtration rate as it could be expected from the Poiseuille's law. If the proposed model is valid one can draw a conclusion that inclined pore channels are very important for improvement of TM's properties as they provide many

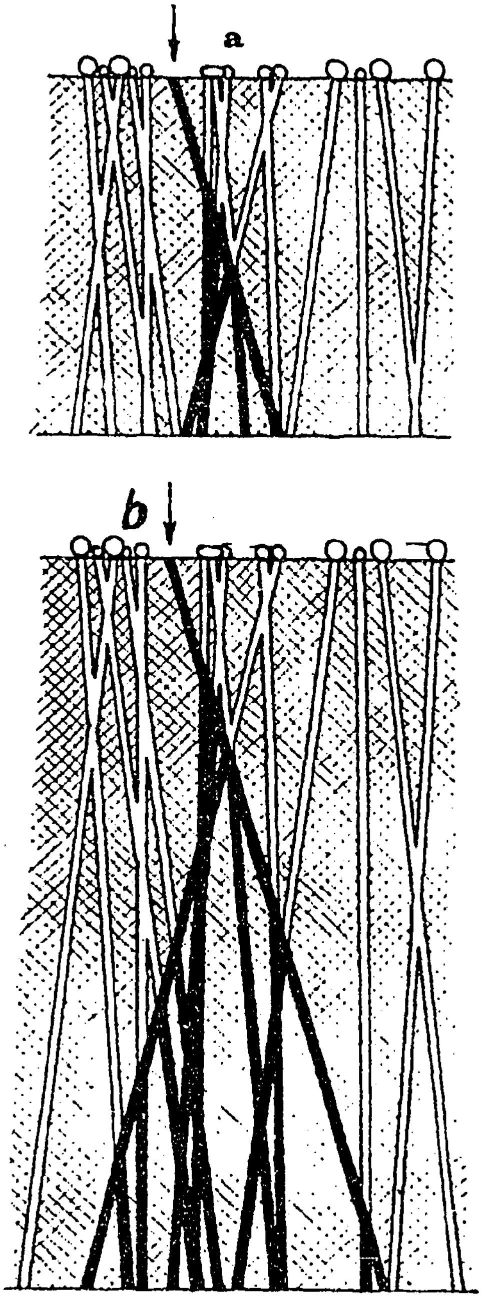

Fig. 4. The cross sections of TM10 and TM20 with the pore diameter of 0.2μm and pore density of ca. 3×10^8 cm^{-2}. All but one of the pores are plugged by particles in the filtration process. The water stream is shown in black. (a) TM10: 21 per cent of the total pore lenght and 6 pores share in the water flow. (b) TM20: 43 per cent of the total pore lenght and 12 pores share in th water flow

intersections in the membrane matrix. It should be noted that effects of pore intersections appear only if the porosity is high enough. In general, the angle distribution, the foil thickness to pore size ratio and number of pores define, to say,

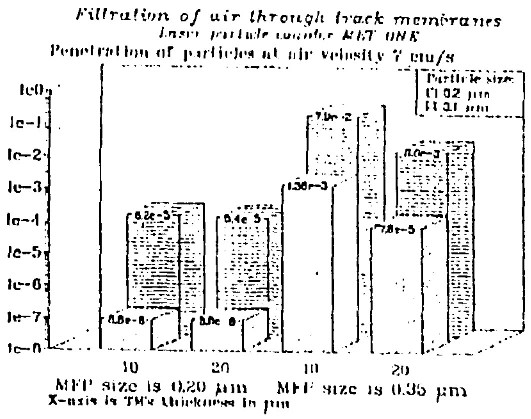

Fig. 5. Penetration of particles as a function of particle size, pore diameter and membrane thickness for ambient air filtration

Table 1. Parameters of track membranes with the nominal pore size of 0.2 µm

	TM10	TM20
Thickness, µm	9.9	20.0
Pore diameter obtained by SEM, µm	0.25	0.30
Pore density, cm^{-2}	$3.2x10^8$	$3.8x10^8$
Mean flow pore diameter, µm	0.198	0.205
Bubble point, bar	2.8	3.0
Burst strenght, bar	2.0	2.2
Air flow rate, 1/h cm^2	20	22
Air flow effective pore diameter, µm	0.23	0.27
Water flow rate, ml/min cm^2 (P=0.7 bar)	8	10.5
Poiseuille pore diameter, µm	0.22	0.27

topological properties of a membrane. These properties should be chosen properly so that the highest performance of the membranes will be achieved. In fact, the system of pores in the TM20 with the pore diameter of 0.2 µm is rather a unified whole (see photo in Fig. 2b) whereas Fig. 2f shows the membrane having a multitude single pores.

The results presented in Fig. 3 showed that the membranes with the pore size of 1 µm (especially TM10) are plugged the most rapidly. This phenomenon can be conditioned by low (in comparison with 0.2 or 0.4 µm TM's) pore density in these membranes and the reasons discussed above.

The results of filtration of ambient air are presented in Fig. 5. The retention efficiencies of TM10's and TM20's with the pore size of 0.2 µm do not differ for 0.1 and 0.2 µm particles i.e. the pore length does not play a marked role in the process of trapping of these particulates at a chosen air velocity. In contrast, the TM20 with 0.4 µm pores has better retention characteristics for small particles than TM10. In this case the particle sizes are sufficiently smaller than the pore diameter and, in addition, the velocity of air in pores of the TM20 is lower than in pores of the thinner membrane because of different porosities of the membranes compared. Therefore, the trapping can occur by the diffusion and inertial impaction mechanisms [4].

Table 2. Parameters of track membranes with the nominal pore size of 0.4 μm

	TM10	TM20
Thickness, μm	9.0	18.5
Pore diameter obtained by SEM, μm	0.40	0.65
Pore density, cm^{-2}	$8.5x10^7$	$1.2x10^8$
Mean flow pore diameter, μm	0.35	0.33
Bubble point, bar	1.8	1.7
Burst strenght, bar	2.3	2.6
Air flow rate, 1/h cm^2	28	40
Air flow effective pore diameter, mm	0.36	0.44
Water flow rate, ml/min cm^2 (P=0.45 bar)	9.6	16
Poiseuille pore diameter, μm	0.35	0.44

Table 3. Parameters of track membranes with the nominal pore of 1 μm

	TM10	TM20
Thickness, μm	9.0	18.0
Pore diameter obtained by SEM, μm	0.85	1.0
Pore density, cm^{-2}	$1.2x10^7$	$2.0x10^7$
Mean flow pore diameter, μm	0.83	0.91
Bubble point, bar	0.72	0.72
Burst strenght, bar	not measured	not measured
Air flow rate, 1/h cm^2	75	95
Air flow effective pore diameter, μm	0.81	0.83
Water flow rate, ml/min cm^2 (P=0.2 bar)	19	20
Poiseuille pore diameter, μm	0.92	0.89

Probably, under such conditions not only different pore length but different pore shape can also contribute to higher retention efficiency of thicker membranes.

4. CONCLUSIONS

The track membranes of two different thicknesses (10 and 20 μm) were compared using different tests. Contrary to expectation, the initial flow rate characteristics of thicker TM's are found to be not worse than those of thin membranes. The membranes with the thickness of 20 μm show higher tensile strength even at higher porosities. Moreover, performing tap water filtration by the thicker membranes of high porosity we observed the improved throughput which is assumed to be caused by the formation of the unified system of pores. It enables us to conclude that TM's with the thickness of 20 μm are a very promising product.

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