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IR Scattering by Optically Inhomogeneous Nanoporous Anodic Alumina Films

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Abstract—This paper presents results of thermographic studies of nanoporous alumina films having different geometric parameters of their porous layer (thickness and average pore diameter), which were exposed to thermal radiation. The films have been shown to shield thermal radiation. The present results suggest that nanoporous alumina membranes can be used as heat shields for reducing the thermal contrast of an object and the surrounding background in the IR spectral region.

Keywords: nanoporous films, alumina, thermal radiation, heat shield

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INTRODUCTION

The study of the optical properties of nanomaterials is an important issue in modern solid-state physics. The reason for this is that nanocomposites are a basis for the development and production of new materials with tailored physical and optical properties, whose parameters are determined by the size, shape, and density of nanosystems.

It is also important to study the properties of nanoporous dielectric materials, for example, of nanoporous anodic alumina, whose structure is formed by producing a network of evenly distributed nanochannels in a bulk material. The physical and optical properties of nanoporous materials may differ drastically from those of a pore-free material. In the case of nanoporous anodic alumina, the simple preparation process and the possibility of varying the properties of its pore structure by adjusting the process conditions [1, 2] make this material a very convenient system for gaining insight into physical effects in nanostructures. Of special note is that porous anodic alumina has a smaller refractive index and lower dielectric permittivity in comparison with bulk alumina, and its nanopores have an ordered structure.

Recently, Matyushkin et al. [3, 4] and Vrublevsky et al. [5] reported studies of transmission spectra of anodic alumina membranes in a wide wavelength range and analyzed particular spectral ranges corresponding to certain properties and parameters of the membranes. Porous alumina membranes have been shown to considerably reduce the IR transmission in the wavelength range from 8 to 14 μm. This spectral range includes the peak thermal radiation wavelength, $\lambda \approx 10$ μm, of biological systems.

The purpose of this work was to study heat flow shielding by nanoporous anodic alumina films using thermal imaging measurements.

EXPERIMENTAL

We studied nanoporous alumina films of various thicknesses (2, 5, 10, and 60 μm) prepared by the electrolytic anodization of 100-μm-thick aluminum foil (99.99%). Nanoporous anodic alumina with a highly ordered pore structure was produced by two-step anodization. In the first step, aluminum was anodized in an aqueous 0.3 M oxalic acid solution in a two-electrode cell with a Pt cathode, maintained at 15.0 ± 0.1°C. The anodization voltage was first raised from 0 to 40 V at a constant rate of 0.5 V/s, following which the anodization process was run in potentiostatic mode. Next, the oxide layer was chemically etched in solutions of phosphoric and chromic acids. In the second step, the aluminum was anodized at 40 V. To obtain films of various thicknesses (2–60 μm), we varied the process duration. After the anodization, the films were cleansed and dried in flowing hot air. In the final film preparation step, the aluminum on the backside of the porous anodic oxide samples was removed by selective etching. After complete metal dissolution, we obtained an impermeable porous anodic alumina membrane. In this manner, we produced anodic alumina membranes with an ordered pore structure.

The pore morphology and pore size in the films were assessed by scanning electron microscopy (SEM)

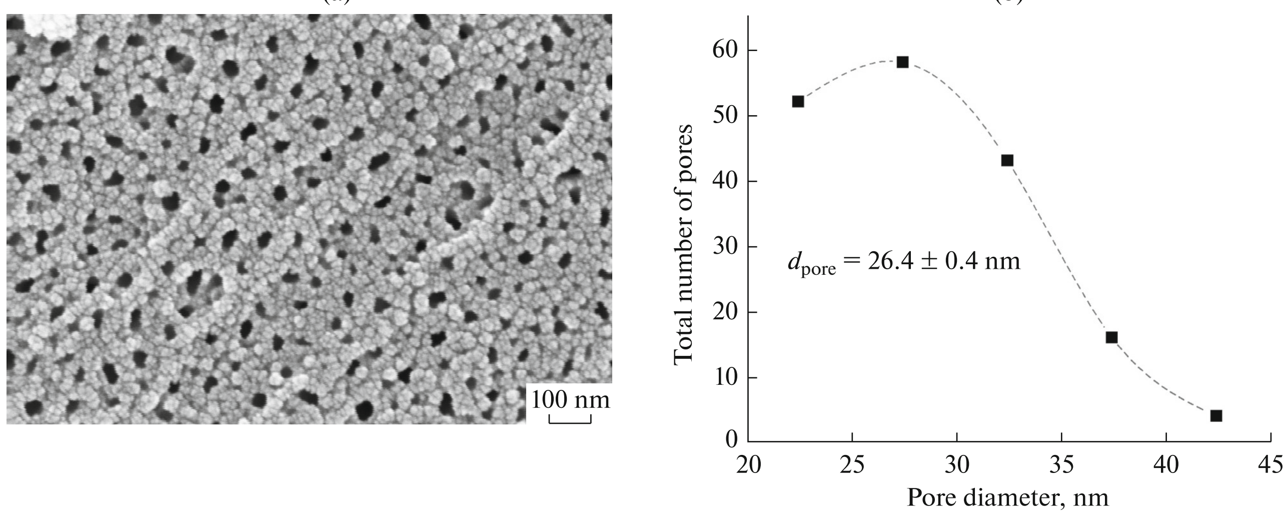

Fig. 1. (a) SEM image of the surface of the porous alumina film prepared in an oxalic acid solution and (b) results of image processing with the ImageJ program.

on a LEO 1550 VP electron microscope (Germany). In statistical image analysis, we used the ImageJ program.

Using SEM images of the surface of the porous anodic alumina (Fig. 1a), we determined the main pore diameter (d_{pore}) by fitting the pore size distribution with a Gaussian (Fig. 1b). In analyzing the data, the initial pore size distribution was assumed to contain both the starting, small-diameter pores and main, larger diameter pores. Since only data on the main pores are of practical importance, the small-diameter pores were left out of consideration in the fitting procedure. The maximum in the Gaussian was taken to correspond to d_{pore}.

To visualize a thermal image of objects, two types of thermal imaging cameras are used: cooled, operating in the near-IR spectral region (3–5 μm) and uncooled, operating in the mid-IR spectral region (8–14 μm). To detect and identify thermal objects, for example, a man, whose thermal radiation has a spectral wavelength of 9.3 μm, uncooled thermal imaging cameras are used. In this work, two thermal imaging cameras were used in thermographic studies of membranes: RGK TL-160 (temperature range −20 to +350°C) and MobIR M4 (temperature range −20 to +250°C).

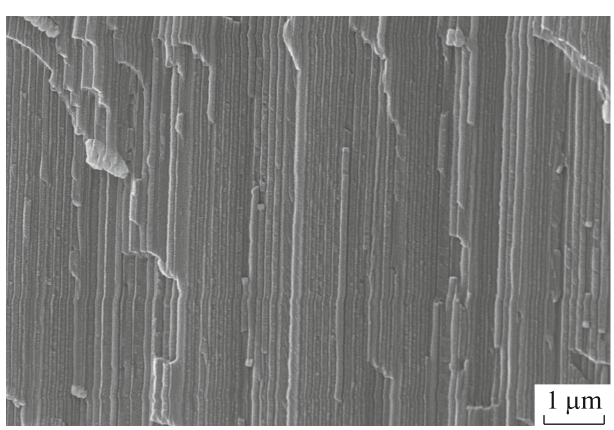

Fig. 2. Cross-sectional SEM image of the porous alumina film prepared in an oxalic acid solution.

The thermal radiation source used was a human palm. As a heat shield, we used a 5-mm-thick Teflon plate with two 14-mm-diameter holes: one was intended for samples, and the other, through which thermal radiation passed unimpeded, was used to assess heat shielding results. The Teflon plate (heat shield) was placed over the human palm (heat source), blocking the main thermal radiation and transmitting it only through the holes. In our experiments, the Teflon plate was located 3–5 cm from the thermal radiation source.

RESULTS AND DISCUSSION

According to SEM data, the pore size in the nanoporous anodic alumina films was approximately 26 nm and the center-to-center pore spacing was 84 nm. The pore had identical cylindrical shapes, were oriented parallel to each other, and were perpendicular to the film surface (Fig. 2).

We obtained thermograms of the films exposed to a thermal radiation source (Fig. 3) and constructed their temperature profile (Fig. 4).

In addition, we determined the heat source temperature as a function of nanoporous alumina film thickness (Fig. 5). In the case of the 10-μm-thick anodic alumina film, the degree of thermal shielding

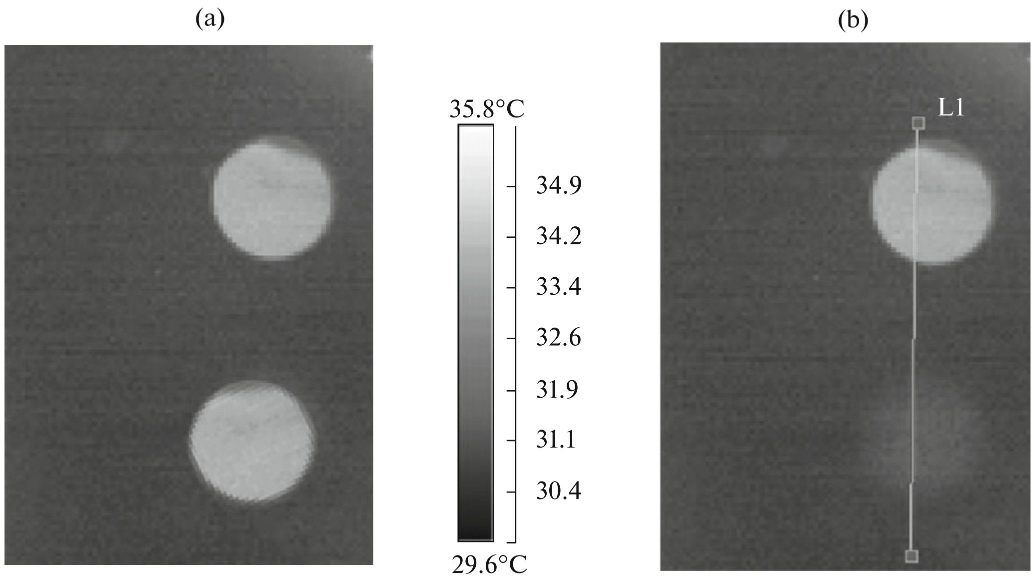

Fig. 3. Thermographic data: view of a biological object through the objective of a thermal imaging camera (a) when the heat flow passes through both the control and working holes and (b) when the working hole is closed by nanoporous anodic alumina.

(*D*) reached 56%. The degree of shielding was calculated using the following equation:

$$D = \frac{T_s - T_m}{T_s - T_{sh}} \times 100\%,$$

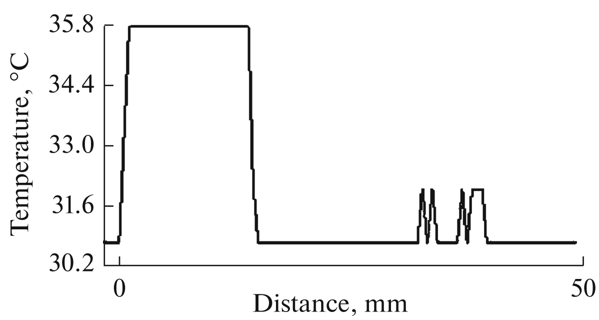

Fig. 4. Temperature profile along the line passing through the control and working holes.

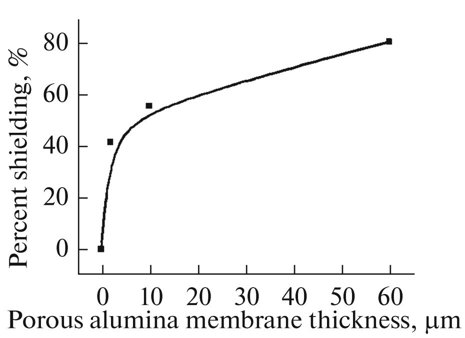

Fig. 5. Thermal radiation shielding as a function of nanoporous anodic alumina film thickness.

where T_s is the heat source temperature, T_m is the membrane temperature, and T_{sh} is the heat shield temperature.

The present results can be understood in terms of the diffusion scattering of thermal radiation by an optically inhomogeneous medium [1], where inhomogeneity sources are both small-diameter pores and various defects.

The use of the porous anodic alumina films considerably reduced the intensity of the thermal radiation transmitted from the heat sources: it reduced the temperature of the thermal spot emitted by a biological object (human palm) from 37.1 to 32.0°C (Figs. 3, 4). Our studies allowed us to obtain temperature profiles of the biological object with no mask and in the presence of a porous anodic alumina film as a mask.

CONCLUSIONS

Nanoporous anodic alumina films have been shown to considerably reduce the transmission of IR radiation in the wavelength range 8–14 μm. Their properties in the IR spectral region can be controlled by adjusting appropriate geometric parameters of the film. The present results suggest that such membranes can be used as heat shields for reducing the thermal contrast of an object and the surrounding background in the IR spectral region.

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