Measurements of porous aluminum oxide thermal properties

A D Kurilov\textsuperscript{1,2}, V V Belyaev\textsuperscript{1,3}, E E Alyasova\textsuperscript{4}, A V Osin\textsuperscript{4},
O A Tokareva\textsuperscript{4}, K D Nessemov\textsuperscript{3}, A A Belyaev\textsuperscript{1}, A Kh Abduev\textsuperscript{3}

\textsuperscript{1}Moscow Region State University, 24 Very Voloshino St., Mytishi, 141014, Russia
\textsuperscript{2}MIREA - Russian Technological University, 78 Vernadskogo Ave., Moscow, 119454, Russia
\textsuperscript{3}RUDN University (People’s Friendship University of Russia), 6 Miklukho-Maklay St.,
Moscow, 117198, Russia
\textsuperscript{4}JSC RUSALOX, Vladimir, Russia
E-mail: ad.kurilov@gmail.com

Abstract. A method of measurement of the thermal performances of composite materials
on the base of the porous aluminium oxide is described. The method takes into account the
heat inhomogeneity, the material inhomogeneity and anisotropy as well as specimen’s surface
radiation. Investigations of the thermophysics properties of the porous substrate fabricated
with using of the electrochemical aluminium oxide technology vs. temperature and long staying
in a climate chamber were fulfilled. The tests demonstrated that the climatic impact does
not influence on the high thermophysics properties of the aluminium oxide material that have
extremely high thermal conductivity $\geq 120$ W/(m·K).

1. Introduction
Efficient thermal conductivity of composite materials an important parameter for many practical
applications. Its theoretical simulation is a significant problem for basic investigations \cite{1-4}.

Both theoretical and experimental researches of thermal properties of porous construction
materials and their application were made in \cite{5-9}. The porous materials are used for thermal
isolation, catalytic converters in cars, chemical catalysts, sound absorption and thermal energy
storage applications \cite{10-14}. Recently attention is paid to thermal conductivity of such media
to develop new functional materials, MEMS cooling, textile and food industries, medical
technologies \cite{15-16}. Besides, the materials with special surface structure align anisodiametric
molecules of liquid crystals \cite{17-23}.

One of most perspective components with these materials became a universal heat sink
plate of JSC RUSALOX (www.rusalox.ru) with improved thermal performances and using of
electrochemical aluminium oxide technology to cool any heat dissipation electronic components.
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LED lighting device is described.

In our recent papers a design and application of such nanoporous plate for heat sink from an
LED lighting device is described \cite{24-26}. Useful effect is provided by high heat conductivity
of the material and high value of specific surface of the porous material. It can achieve a few tens
of square meters for the plate of size of 10×10 cm.
The goal of the work is the investigation of the thermal properties of the porous substrate fabricated with the electrochemical aluminium oxide technology vs. temperature in the range from 20°C to 50°C and long staying in a climate chamber.

2. Materials and Methods
The investigation objects were specimens of the aluminium oxide ceramics (Figure 1). They comprised a bilayer structure with one layer of aluminium and another one of aluminium oxide. The second layer thickness was of 0.75 mm.

At the initial phase the experiments were fulfilled at ambient temperature 26–30°C and then extrapolated to 20°C. After each test series the specimens were positioned into the climate chamber for 7.5 days. Then the thermophysical performances were measured.

Values of the thermal conductivity coefficients in wide temperature range were fulfilled by using LFA 457 MicroFlash®. The measurements are based on a method of laser flash. A short pulse of the light radiation is absorbed in a thin layer of the specimen frontal plane. A generated temperature disturbance is recorded on the rear specimen surface. These data provide calculation of the thermal conductivity coefficient (thermal diffusion) of the specimen in accordance with a formula:

\[ a = 1.388 \frac{l^2}{\pi \tau_{0.5}} \]  

where \( l \) is the specimen thickness; \( a \) is the thermal conductivity coefficient, m\(^2\)/s; \( \tau_{0.5} \) is time of obtaining of half of the difference between the maximum temperature and initial temperature on the rear specimen surface.

To discuss the results obtained the Parker method [27] was used. In the ideal case it works with assumptions as follows:

- adiabatic, homogeneous, isotropic specimen;
- homogeneous pulse heating;
- the pulse duration goes to zero (the pulse should be described by the Dirac distribution).

The temperature of the specimen rear surface rises to a definite value and then keeps constant under irradiation of the homogeneous specimen at perfect conditions. In the experiment the
perfect conditions are difficult to realize. Therefore the experimental dependences have a maximum with further temperature reduction.

Different methods and models were developed to apply the Parker method for the real conditions and take into account the experiment non-perfectness:

• heat losses and radiation of the specimen surface,
• non-zero duration of the laser pulse,
• inhomogeneity of the pulse heating,
• the material is non-homogeneous and non-isotropic.

To take into account the losses of the heating as well the surface radiation the specimens are tested in comparison with the reference specimen or their surface is processed with special materials, e.g., graphite.

The non-zero deviation of the pulse duration is compensated by using a finite correction. Other deviations from the perfect model are taken into account with the aid of a special software.

A device IT-S-400 was used to measure the specific thermal conductivity. It is determined in the wide temperature range from $-150^\circ$C to $175^\circ$C. A comparative method of the dynamic calorimeter with a heat meter and adiabatic shell. The accuracy of the specific thermal conductivity measurement is 4%.

3. Results and discussion

The thermophysics performances of the aluminium oxide materials were determined in the range from the room temperature to 50$^\circ$C. In the experiment the temperature conductivity coefficient was measured firstly. Then the thermal conductivity coefficient was calculated by using the formula:

$$\lambda = aC_{p}\rho,$$

where $\rho$ is the specimen density, kg/m$^3$; $a$ is the temperature conductivity coefficient, m$^2$/s; $C_{p}$ is the specimen specific heat capacity, J/(kg·K); $\lambda$ is the thermal conductivity coefficient, W/(m·K).

This value can be easily calculated for the known valued of the sample mass and size. The mass was measured by weighing with a balance RADWAG AS 220/C/2 with accuracy 0.001 g. The size was measured by a KRAFT WERKEZEUGE calipers with accuracy 0.01 mm. The specimens’ mass and size were measured after each climate test to check their moisture saturation.

The specimens’ surfaces were coated with the graphite. All the specimens were positioned with their oxide surface in relation to the radiation source.

Seven specimens of the aluminium oxide were tested. Average value of the specific heat capacity in the range $25$–$50^\circ$C was as high as 0.871 kJ/(kg·K) after the first climatic test and 0.861 kJ/(kg·K) after the last climatic test. The change (1.5%) does not change the measurement accuracy. These values are close to the specific heat capacity of the aluminium (0.886 kJ/(kg·K)) in this range.

Data on both thermal conductivity coefficient and temperature conductivity coefficient are presented in Tables 1 and 2.

Average value of the thermal conductivity coefficient is equal to 125 W/(m·K) at 25$^\circ$C. Maximum deviation from the average value (7%) is obtained for the specimen 2. The random error is as high as (3.6%) at the confidence probability 0.95.

These specimens were tested in the climate chamber. Then repeated measurements of the thermophysics properties were fulfilled. These data are presented in Tables 3 and 4.
Table 1. The temperature conductivity coefficient of the specimens investigated, \( a \) mm\(^2\)/s

| No. | 25  | 30  | 35  | 40  | 45  | 50  |
|-----|-----|-----|-----|-----|-----|-----|
| 1   | 53.17 | 53.02 | 52.87 | 52.73 | 52.58 | 52.43 |
| 2   | 56.95 | 56.65 | 56.34 | 56.04 | 55.73 | 55.43 |
| 3   | 55.27 | 55.02 | 54.51 | 54.23 | 54.00 | 54.00 |
| 4   | 50.75 | 50.51 | 50.31 | 50.09 | 49.87 | 49.65 |
| 5   | 51.00 | 50.78 | 50.56 | 50.33 | 50.11 | 49.89 |
| 6   | 51.17 | 50.97 | 50.76 | 50.56 | 50.36 | 50.16 |
| 7   | 51.72 | 51.46 | 51.20 | 50.94 | 50.68 | 50.42 |
| 8   | 51.22 | 50.98 | 50.74 | 50.50 | 50.26 | 50.02 |
| 9   | 53.00 | 52.73 | 52.47 | 52.20 | 51.94 | 51.67 |
| 10  | 50.74 | 50.62 | 50.50 | 50.38 | 50.25 | 50.13 |

Table 2. The thermal conductivity coefficient of the specimens investigated, \( \lambda \) W/(m·K)

| No. | 25  | 30  | 35  | 40  | 45  | 50  |
|-----|-----|-----|-----|-----|-----|-----|
| 1   | 125.91 | 125.81 | 125.72 | 125.62 | 125.52 | 125.42 |
| 2   | 133.40 | 133.00 | 132.51 | 132.07 | 131.63 | 131.18 |
| 3   | 130.76 | 130.41 | 130.06 | 129.71 | 129.36 | 129.01 |
| 4   | 120.37 | 120.10 | 119.83 | 119.55 | 119.28 | 119.01 |
| 5   | 120.71 | 120.43 | 120.15 | 119.88 | 119.60 | 119.32 |
| 6   | 121.41 | 121.18 | 120.96 | 120.73 | 120.51 | 120.28 |
| 7   | 123.10 | 122.73 | 122.37 | 122.00 | 121.63 | 121.26 |
| 8   | 122.54 | 122.22 | 121.90 | 121.58 | 121.25 | 120.93 |
| 9   | 127.16 | 126.78 | 126.41 | 126.03 | 125.66 | 125.29 |
| 10  | 121.59 | 121.55 | 121.50 | 121.45 | 121.41 | 121.36 |

Table 3. The temperature conductivity coefficient of the specimens investigated after 4 climatic tests, \( a \) mm\(^2\)/s

| No. | 20  | 25  | 30  | 35  | 40  | 45  | 50  |
|-----|-----|-----|-----|-----|-----|-----|-----|
| 1   | 54.70 | 54.37 | 54.04 | 53.71 | 53.39 | 53.06 | 52.73 |
| 2   | 55.71 | 55.45 | 55.19 | 54.92 | 54.66 | 54.40 | 54.14 |
| 3   | 55.97 | 55.66 | 55.35 | 55.04 | 54.73 | 54.42 | 54.12 |
| 4   | 52.99 | 52.73 | 52.48 | 52.23 | 51.98 | 51.73 | 51.48 |
| 5   | 52.91 | 52.64 | 52.38 | 52.11 | 51.84 | 51.57 | 51.30 |
| 6   | 52.18 | 51.96 | 51.75 | 51.53 | 51.31 | 51.09 | 50.88 |
| 7   | 53.35 | 53.13 | 52.91 | 52.70 | 52.48 | 52.26 | 52.04 |
| 8   | 53.14 | 52.88 | 52.62 | 52.36 | 52.10 | 51.84 | 51.58 |
| 9   | 53.24 | 53.00 | 52.76 | 52.52 | 52.29 | 52.05 | 51.81 |
| 10  | 50.89 | 50.63 | 50.37 | 50.11 | 49.85 | 49.59 | 49.33 |
Table 4. The thermal conductivity coefficient of the specimens investigated after 4 climatic tests, $\lambda$ W/(m·K)

| No. | $t, ^\circ$C | 20 | 25 | 30 | 35 | 40 | 45 | 50 |
|-----|--------------|----|----|----|----|----|----|----|
| 1   | 129.95       | 129.42 | 128.88 | 128.34 | 127.81 | 127.27 | 126.74 |
| 2   | 131.57       | 131.20 | 130.83 | 130.46 | 130.09 | 129.72 | 129.35 |
| 3   | 131.94       | 131.46 | 131.08 | 130.70 | 130.34 | 129.93 | 129.56 |
| 4   | 125.51       | 125.15 | 124.79 | 124.43 | 124.07 | 123.71 | 123.36 |
| 5   | 124.78       | 124.38 | 123.98 | 123.58 | 123.18 | 122.78 | 122.38 |
| 6   | 124.61       | 124.33 | 123.99 | 123.59 | 123.19 | 123.70 | 123.29 |
| 7   | 126.65       | 126.38 | 126.05 | 125.65 | 125.25 | 124.87 | 124.48 |
| 8   | 125.74       | 125.36 | 124.98 | 124.59 | 124.20 | 123.82 | 123.43 |
| 9   | 126.90       | 126.58 | 126.25 | 125.92 | 125.59 | 125.26 | 124.94 |
| 10  | 121.25       | 120.86 | 120.57 | 120.28 | 119.99 | 119.70 | 119.31 |

Conclusions
A method of measurement of the thermal performances of the porous aluminium oxide substrates is described. It is based on methods of the laser flash and the dynamic calorimeter. As the heat capacity is determined firstly by the material composition the $C_p$ value of the porous oxide does not almost depend on the temperature, it is close to this parameter value for the pure aluminium.

Comparative results of the measurements of both thermal conductivity and temperature conductivity coefficients of the new nanoporous material on the base of the aluminium oxide. The tests demonstrated that the climatic impact does not influence on the high thermophysics properties of the aluminium oxide material.

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