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EFFECT OF TEMPERATURE, PRESSURE, AND COMPOSITION OF ATMOSPHERE ON
THERMAL CONDUCTIVITY OF HEAPS OF POWDERED TITANIUM, ZIRCONIUM, AND
SILICON

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The thermal conductivity of titanium, zirconium, and silicon in a vacuum and in helium was experimentally investigated. The obtained experimental thermal-conductivity data are compared with calculated values for titanium and zirconium.

For optimization of the conditions of the sintering process we measured the thermal conductivity of finely dispersed titanium and zirconium powders at 300-1000°C in a vacuum ($P = 5 \cdot 10^{-3}$ mm Hg) and in a helium atmosphere ($P = 220$ mm Hg). The main characteristics of the investigated powders are given in Table 1.

The thermal conductivity was measured by the steady radial heat-flux method with indirect heating of the specimen (tube method). The investigated powder was poured into the annular gap formed by a porcelain tube of diameter 20 mm and a graphite cylindrical container of diameter 80 mm and wall thickness 1.5 mm. The length of the tube and container was 300 mm. The porcelain tube contained a heater of diameter 12 mm, made of carbon with low thermal conductivity (8-10 W/m·deg). In view of the low thermal conductivity of the heater material, the leakage of heat in the axial direction was slight. This loss was calculated from the known thermal conductivity and temperature drop along the heater axis. The test section in the center of the specimen was 50 mm long. Measurements showed that the gradient of the temperature field in the test section was slight and, hence, the axial heat loss in the specimen could be neglected. The sample temperature was measured with four Chromel-Alumel thermocouples, two of which were mounted on the outer surface of the porcelain tube along its axis, and the other two were mounted on the inside surface of the graphite container parallel to its axis. Four thermocouples were required for averaging of the radial temperature field

TABLE 1. Main Characteristics of Dispersed Titanium, Zirconium, and Silicon

Material	Particle diameter, μ	Bulk density, g/cm ³	Density, g/cm ³	Porosity
Titanium	70	1,685	4,5	0,625
Zirconium	70	2,525	6,49	0,61
Silicon	50	0,805	2,33	0,655

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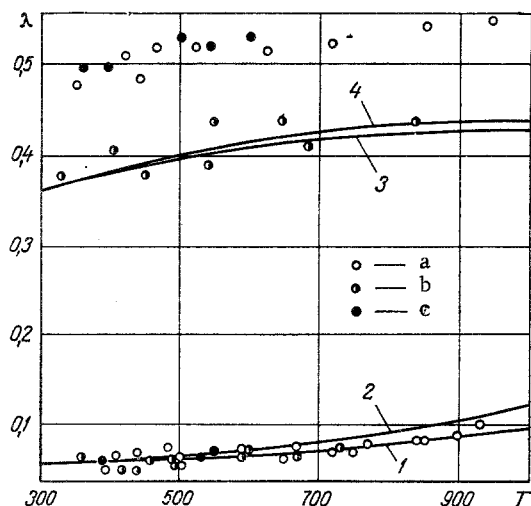


Fig. 1. Temperature dependences of thermal conductivity of titanium, zirconium, and silicon powders. Experimental points: a) silicon, b) zirconium, c) titanium; theoretical curves: 1) zirconium in a vacuum, 2) titanium in a vacuum, 3) titanium in helium, 4) zirconium in helium. λ , W/m·deg; T, °K.

over the test section of the sample. The thermocouples, contained in a porcelain sheath, were mounted in quartz tubes with the smallest possible gap, and the thermocouple junction projected beyond the end of the tube. The quartz tubes were attached and, hence, fixed on the surfaces of the porcelain tube and graphite container. This ensured a fixed position of the thermocouples in the sample of dispersed material.

The heater was powered by an OSU-40 power transformer, and the voltage was regulated by an RNO-250-10 autotransformer. The heat flux passing through the sample was calculated from the electrical parameters: the current flowing through the heater and the voltage drop on the working section of the heater. The current in the heater circuit was measured through a UTT-6M current transformer with a D-57 ammeter, and the voltage drop was measured with an R-56 ac potentiometer.

The thermal conductivity was calculated from the equation

$$\lambda = \frac{IU \ln r_1/r_2}{2\pi l \Delta T}, \quad (1)$$

where I is the current, A; U is the voltage drop on the working section of the heater, V; l is the length of the working section of the heater, m; r_1 and r_2 are the radii of the points at which the thermocouple junctions are situated; ΔT is the radial temperature drop.

The maximum theoretical experimental error was $\pm 10\%$.

Figure 1 shows the temperature dependences of the thermal conductivity of titanium, zirconium, and silicon powders. The thermal conductivity, measured in a vacuum, was the same (within the limits of error) for the three materials. The thermal conductivity of dispersed zirconium, measured in a helium atmosphere, was approximately 20% lower than the thermal conductivity of titanium and silicon. The mean temperature of dispersed zirconium rose to 840°K and that of silicon to 950°K (in helium). Titanium powder could also be heated to a high temperature. Above 600°K, however, titanium particles began to sinter, and the sample acquired the properties of a solid, which led to a sharp increase in thermal conductivity of the system.

The obtained experimental thermal conductivity data were compared with calculated values for titanium and zirconium.

For the calculation we used the geometric model of a "bound" material, which was thoroughly investigated in [1]. The structure of a bound material is shown schematically in Fig. 2a. The structure consists of a "matrix" formed by randomly arranged, but fairly densely packed, contacting granules ("first-order structure") and a spatial network of larger cavities penetrating the matrix and forming along with it the "second-order structure."

The degree of sintering (degree of binding) of the material can be characterized by the relative size of the actual contact spot y_2 , equal to the radius r_2 of the actual contact spot to the granule radius r ; i.e., $y_2 = r_2/r$ (Fig. 2b).

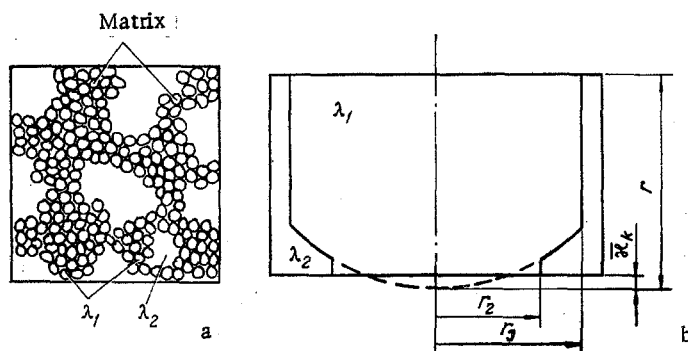


Fig. 2. Model of granular system: a) real structure; b) deformation of spherical particle.

In the initial relation for the free heap (before sintering) the actual area of contact was seven to eight orders less than the cross-sectional area of the granules ($y_2 \ll 1$). In the pressing and sintering process the porosity of the material decreases; i.e., $m_2 \rightarrow 0$ (a monolith), and the relative area of contact increases, approaching the ultimate value $y_2 \rightarrow 1$.

It was shown in [1] that for the majority of important practical cases, where the thermal conductivity of the granules λ_1 is much higher than that of the component in the pores λ_2 ($\nu = \lambda_2/\lambda_1 \ll 1$), the thermal conductivity of the sintered material is almost predetermined by the actual area of contact of the sintered particles. The problem of analytical determination of the thermal conductivity of sintered material is solved approximately by consideration of the heat transfer through the contacts of the sintered granules and in the pores between them.

Using the approximate methods of solution described in [1], we selected areas of contact y_2 at which the theoretical and experimental values of the thermal conductivity of the above materials in a vacuum were the same.

Calculations made for titanium and zirconium showed that the investigated materials were slightly sintered materials with a relative radius of actual contact $y_2 = 1 \cdot 10^{-2}$ for titanium and $y_2 = 0.7 \cdot 10^{-2}$ for zirconium.

We observed good agreement between the results of calculation and experiment in a vacuum (Fig. 1). There was some divergence (up to 25%) of the calculated and experimental values for titanium in a helium atmosphere. The calculated thermal-conductivity values for titanium powder agreed with the calculated and experimental values for zirconium powder, since the thermal conductivities of monolithic titanium and zirconium are close in value (in the temperature range 300-1000°K the thermal conductivity of titanium is 15.5-19.4 W/m°K, and that of zirconium is 21.5-19.9 W/m°K), and the granule sizes and porosities (in titanium $m_2 = 62.5\%$, and in zirconium $m_2 = 61\%$) are practically the same. Calculation revealed that the effective thermal conductivity of porous materials in a helium atmosphere is appreciably affected by the accommodation coefficient, which characterizes the degree of completeness of energy transfer when gas molecules collide with the surface of the particles.

We could not find any experimental data on the accommodation coefficient for titanium and zirconium and, hence, for an approximate calculation we used the values of the accommodation coefficient for tungsten [1], whose value varies from $\alpha = 0.4$ at $T = 300^\circ\text{K}$ to $\alpha = 0.1$ at $T = 1000^\circ\text{K}$.

The thermal conductivity of silicon powder was not calculated, since there are no reliable thermal-conductivity values for monolithic silicon.

The theoretical relations proposed in [1] can be used to check and correlate the results of measurements, and also to predict the thermal conductivity of slightly sintered porous powders.

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