

PRESENTATION OF AN INSTATIONARY MEASUREMENT METHOD FOR
DETERMINATION OF THE MATERIAL PROPERTIES THERMAL DIFFUSIVITY
AND SPECIFIC HEAT

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ABSTRACT

In order to estimate or to compute the thermal load of materials the corresponding thermophysical properties have to be known:

- thermal diffusivity
- thermal conductivity
- density
- specific heat

Numeric computations are often done by usage of finite element or finite differences computer programs. These programs require the knowledge of the temperature dependence of the material parameters thermal diffusivity and specific heat, which can be measured by the flash diffusivity method.

INTRODUCTION

Heat protective coatings on thermally loaded materials should have good temperature stability and low thermal diffusivity. The aim is a decrease of heat transfer into the uncoated or coated material. In this case it is possible to use higher operating temperatures resulting in higher efficiency. On the other hand, cheaper substrates can be used for coated materials or the service life is extended, if the temperatures within the material are decreased, and the same external temperature load is applied.

As the substrate shall be protected, the maximum temperatures in the interface between substrate and coating must not exceed the melting temperature of the substrate or that of one of the coatings. The material strain caused by temperature gradients and phase transitions together with the external mechanical loads can give information, why the coating system failed. The optimization of

coating systems with respect to temperature loads is possible, if the temperature dependence of the thermal diffusivity and the temperature load of the material are known. The resulting temperature fields are determined by finite element- or finite differences computations under variation of the thickness of the coatings and the system of layers.

The measurement equipment and the solution of the heat transfer differential equation

Fig. 1 shows the experimental equipment for the measurement of thermal diffusivity. It consists of water cooled heatable vacuum chamber, xenon flash lamp with reflector (or laser), data recording electronics and minicomputer.

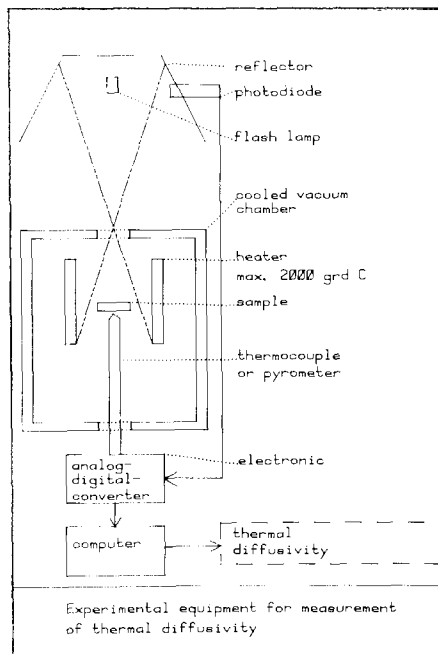


Fig. 1. Experimental equipment

The sample is heated to the chosen temperature, at which measurements shall be performed. The samples are flat disks with a diameter of 10 - 13 mm and a height of 1 - 2 mm. On one surface a small temperature pulse is generated by the flashlamp or by the laser. This temperature pulse penetrates into the sample and rises

its temperature. At the other side of the sample the temperature increase is measured and recorded by a thermocouple or a pyrometer.

The time dependence of the temperature rise is used to determine the thermal diffusivity. The experimental equipment must be adapted to the solution of the instationary heat transfer differential equation:

$$\partial T / \partial t = \gamma \cdot \partial^2 T / \partial x^2$$

T : temperature, t : time, x : displacement,

γ : thermal diffusivity

The velocity of temperature equilibrium is described by the thermal diffusivity γ . The relation to the thermal conductivity is

$$\gamma = k / c_p \cdot \rho$$

k : thermal conductivity, c_p : specific heat, ρ : density

The following boundary conditions are assumed for the solution of the differential equation and the experimental equipment:

- the heat flux in the sample from one side to the other is in axial direction
- the entire sample surface is heated homogeneously
- the temperature rise time on the back surface of the sample has to be long compared to the time duration of the temperature pulse on the surface
- the sample is in the adiabatic state
- the heat emission from the surfaces of the sample can be neglected
- the sample material is homogeneous.

Measurements

The thermal diffusivities of pure nickel, armco iron, pure copper and electrolytic copper have been investigated. Fig. 2 shows the thermal diffusivity of pure nickel (purity 99,99 %) from room temperature up to about 1000 °C. The crosses (+) are measured while heating, the stars (*) while cooling. The measurements are in good agreement. The thermal diffusivity at room temperature has a value of about 22 mm²/sec, then it decreases

and at about 350 °C a distinct minimum appears. This minimum is due to the Curietemperature, at which a magnetic transition from the ferromagnetic to paramagnetic phase occurs. From 400 °C to 1000 °C only a small increase in the value of thermal diffusivity was measured.

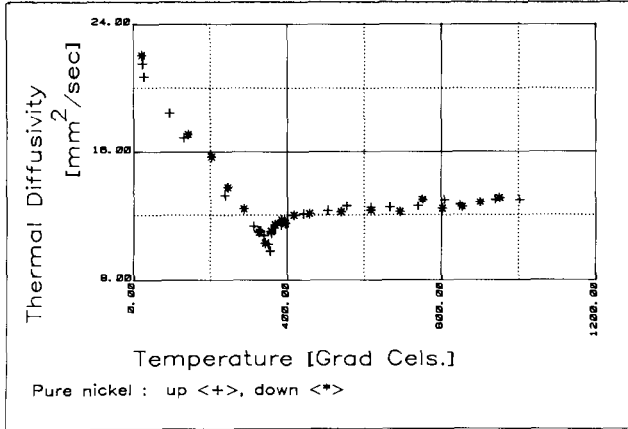


Fig. 2. Temperature dependence of the thermal diffusivity of pure nickel

Fig. 3 shows the thermal diffusivity of armco iron up to about 1000 °C. The thermal diffusivity at room temperature has a value of about 20 mm²/sec. When the temperature increases the thermal

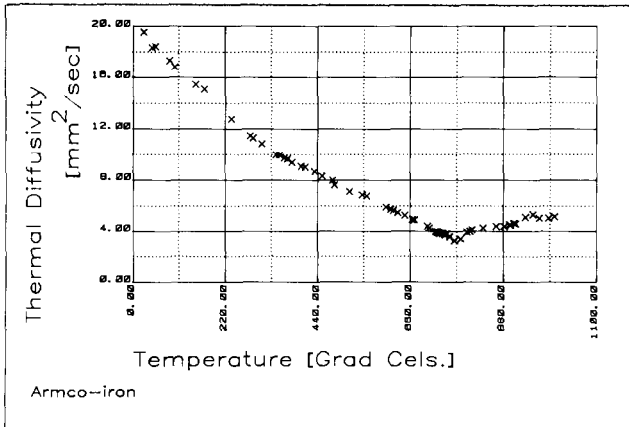


Fig. 3. Temperature dependence of thermal diffusivity of armco iron

diffusivity decreases significantly (as in the case of nickel) and at about 730 °C a minimum appears, which is again caused by the magnetic transition. At about 900 °C a slight transient occurs. This transient is due to the phase transition (α/γ) from ferrite to austenite.

Fig. 4 compares two measurements of thermal diffusivity of pure copper (purity 99.998 %) and electrolytic copper (purity 99.98 %). The temperature dependence of pure copper is shown in the upper curve. The lower curve describes the temperature dependence of electrolytic copper.

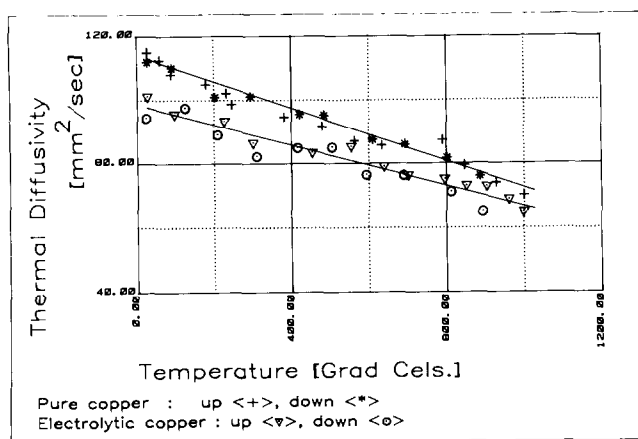


Fig. 4. Temperature dependence of thermal diffusivity of pure and electrolytic copper

The values for pure copper (+,*) and electrolytic copper (triangle, circle) coincide very well while heating and cooling.

Though the impurity of electrolytic copper compared to pure copper is only insignificantly higher, the value of thermal diffusivity at 20 °C decreases by 13 %. The difference between both materials decreases at higher temperatures, as the defect generation is increased.

Specific heat

Especially at high temperatures the specific heat is one of the basic properties of ceramic or metallic materials, the knowledge of which is necessary for high temperature applications. The flash diffusivity method can be used to determine the specific heat.

Originating from the unnormalized solution of the heat transfer differential equation,

$$T(t) = (Q/\rho \cdot c_p \cdot L) \cdot (1 + \sum (-1)^n \exp(-n^2 \cdot \pi^2 \cdot \gamma \cdot t/L^2))$$

it is obtained as the result for long times, when the entire heat of the temperature pulse has been absorbed, and temperature equilibrium exists:

$$T_{\max} = Q/\rho \cdot c_p \cdot L$$

T_{\max} : maximum of temperature rise

Q : absorbed heat, ρ : density,

c_p : specific heat, L : sample thickness.

T_{\max} is the maximum temperature rise of the sample. The absorbed heat Q can be determined by comparison measurement with a standard material, the specific heat of which is known. The surface of both materials must have the same reflectivity.

In this case the following equation is valid:

$$\begin{aligned} Q &= (c_p \cdot \rho \cdot T_{\max})_{\text{standard}} \\ &= (c_p \cdot \rho \cdot T_{\max})_{\text{unknown}} \end{aligned}$$

The unknown specific heat can be evaluated by this equation without knowledge of the absolute temperature and the amount of heat.

The disadvantages of this method are:

- The precision at low temperature is not as high as with adiabatic calorimetric measurements.
- Difficulties of measurement in the range of phase transition.
- Measurements only on solids, not possible on powders or fluids.

The advantages are:

- Small sample mass (about 1 g)
- short measurement duration (about 100 ms)
- large temperature range
- simultaneous measurements of specific heat and thermal diffusivity.

The temperature dependent values of thermal diffusivity and specific heat can be used to compute the temperature rise in coated or uncoated materials, in order to estimate if an application under given external temperature load is possible.