THERMAL DIFFUSIVITY OF MATERIALS USING FLASH METHOD APPLIED ON CoCr ALLOYS

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Metoda "flash" a devenit metoda standard de testare pentru măsurarea difuzivităţii termice α, în cazul materialelor solide, omogene, izotrope şi opace. Am măsurat influenţa puterii lămpii flash asupra rezultatelor în cazul unui aliaj CoCr ASTM F75 în intervalul de temperatură 20 - 800°C. Incertutudinea relativă a determinărilor de difuzivitate temică a fost estimată între ±1 şi ±3%, în funcţie de temperatură.

The flash method is a well-established nonsteady-state measurement technique for measuring the thermal diffusivity, α, of solid homogeneous isotropic opaque materials. We measured the influence of the flash lamp power on the results in the case of a CoCr ASTM F75 type alloy in the temperature range from 20 to 800°C. The relative uncertainty of the thermal diffusivity determination is estimated to be from ±1 to ±3%, depending on the temperature.

Keywords: thermal diffusivity, Flash Method, CoCr alloy

1. Introduction

Thermal diffusivity is an important property for materials being used to determine the optimal work temperature in design applications made under transient heat flow, for processes control and quality assurance [1,2].

The thermal diffusivity (α, of a medium) is the thermophysical property that determines the speed of heat propagation by conduction during changes of temperature with time [3]. The heat propagation is faster for materials with high thermal diffusivity. The thermal diffusivity is related to the thermal conductivity (λ), specific heat capacity (C_p) and density (ρ: $\alpha = \lambda/\rho C_p$) [4,5].

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The thermal diffusivity, having dimension length²/time, is expressed in the unit m²/s, and affects any conductive transient heat transfer process within the medium, according to this definition [6,13]. The main goal of this paper was to characterize the thermal diffusivity of CoCr ASTM F75 type alloy.

2. Methods

The development, specification, and quality control of materials often require the measurement of thermophysical properties. This data can be critical to a successful design, especially with the rapidly increasing cooling requirements that result from the packaging of higher performance devices [7]. A variety of methods, involving both steady state and transient techniques, are available for measuring thermal diffusivity, specific heat, thermal conductivity and thermal resistance. In recent years the flash diffusivity method (ASTM E1461), due to its unique advantages, has seen increased use for characterizing many of the materials [8,9,10]. Other methods commonly employed include the guarded heat flow meter, ASTM E1530, guarded comparative, ASTM E1225 and its modification ASTM D5470, various "probe" methods based on the transient hot wire technique and the transient plane source technique [11].

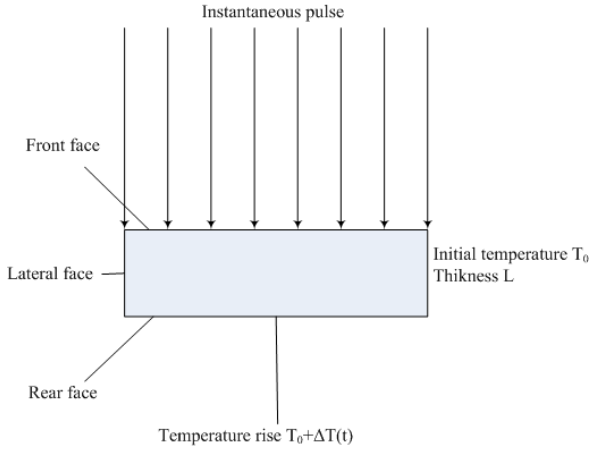


Fig. 1 Schematic of the flash method [15]

The laser flash method is based upon the measurement of the temperature rise on the back face of a thin disk sample resulting from a short energy pulse on the front surface. The sample (12,5mm in diameter and about 1–5mm thick) is placed in a vacuum furnace and isothermally heated at a uniform temperature. Then, a short (450μs) flash pulse of 1.06μm wavelength irradiates one side of the sample. The temperature rise on the opposite sample face is measured by an IR detector (InSb). A high-speed recorder collects data representing the temperature rise. The diffusivity is calculated from the shape of the temperature-time curve

(thermogram) and the thickness, *e*, of the sample. The absolute values of the energy absorbed, the temperature rise, and the emissivity of the back face of the sample are not necessary [12,14].

After the sample has been stabilized at a desired temperature T_0, a nearly instantaneous pulse of energy is deposited on its front face, and the temperature increase $\Delta T(t)$ on the rear face of the sample is recorded as a function of time (Fig. 1). The thermal diffusivity is then determined from this thermogram [15,16].

The characteristic shapes of the temperature increase curves are depicted in Fig. 2. If no heat loss is involved, the temperature of the rear face will rise to a maximum and remain at that level indefinitely (curve A) [7]. However, with increasing heat losses, the temperature on the rear face decreases after reaching a maximal value (curves B and C).

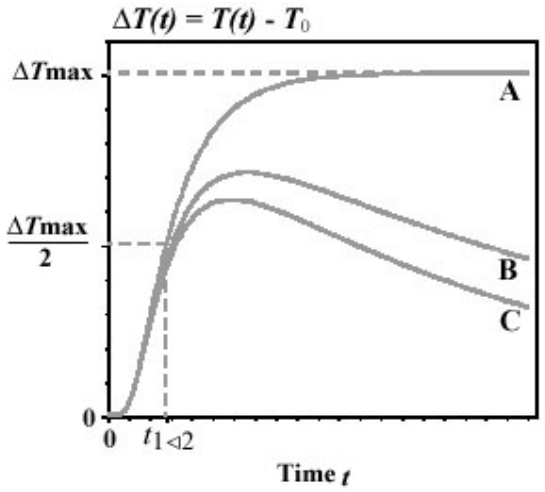


Fig. 2 Temperature increase for various experimental conditions

The original method proposed by Parker assumes an isotropic and adiabatic sample (no heat loss). The thermal diffusivity is determined from the thickness, *L*, of the sample and the time, $t_{½}$, the thermogram takes to reach half of the maximal temperature increase:

In deriving the mathematical expression from which the thermal diffusivity is calculated, Parker [1] starts from the equation of the temperature distribution within a thermally insulated solid of uniform thickness L, as given by Carslaw and Jaeger [12].

$$T(x,t)=\frac{1}{L}\int_0^1 T(x,0)dx+\frac{2}{L}\sum_{n=1}^{\infty}\exp\left(\frac{-n^2\pi^2\alpha t}{L^2}\right)\cdot\cos\frac{n\pi x}{L}\int_0^1 T(x,0)\cos\frac{n\pi x}{L}dx \quad (1)$$

where α is the thermal diffusivity of the material. If a pulse of radiant energy Q is instantaneously and uniformly absorbed in the small depth g at the front surface $x = 0$, the temperature distribution at that instant is given by:

$$T(x,0)=\frac{Q}{\rho\cdot C\cdot g} \text{ for } 0<x<g \quad (2)$$

$$T(x,0)=0 \text{ for } g<x<L \quad (3)$$

With this initial condition, 1 can be written as:

$$T(x,0)=\frac{Q}{\rho\cdot C\cdot g}\left[1+2\sum_{n=1}^{\infty}\cos\frac{n\pi x}{L}\frac{\sin\frac{n\pi g}{L}}{\frac{n\pi g}{L}}\cdot\exp\left(\frac{-n^2\pi^2}{L^2}\alpha t\right)\right] \quad (4)$$

where ρ is the density and C is the specific heat capacity of the material. In this application only a few terms will be needed, and since g is a very small number for opaque materials :

$$\sin\frac{n\pi g}{L}\approx\frac{n\pi g}{L} \quad (5)$$

At the rear surface, where $x = L$, the temperature history can be expressed by:

$$T(L,t)=\frac{Q}{\rho\cdot C\cdot g}\left[1+2\sum_{n=1}^{\infty}(-1)^n\cdot\exp\left(\frac{-n^2\pi^2}{L^2}\alpha t\right)\right] \quad (6)$$

Two dimensionless parameters, V and ω can be defined:

$$V(L,t)=\frac{T(L,t)}{T_M} \quad (7)$$

$$\omega=\frac{\pi^2\alpha t}{L^2} \quad (8)$$

$\boldsymbol{T_M}$ represents the maximum temperature at the rear surface. The combination of (6), (7) and (8) yields:

$$V = 1 + 2\sum_{n=1}^{\infty}(-1)^n \exp(-n^2\omega) \tag{9}$$

When $V = 0.5$ and $\omega = 1.38$:

$$\alpha = \frac{1.38 \cdot L^2}{\pi^2 t_{1/2}} \tag{10}$$

or:

$$\alpha = 1.38\frac{L^2}{t_{1/2}} \tag{11}$$

where $t_{1/2}$ is the time required for the back surface to reach half of the maximum temperature rise [1].

The inadequacy of the Parker solution became obvious almost immediately after its introduction, as nearly every one of these assumptions is violated to some extent during an experiment. So, gradually, investigators introduced various theories to describe the real process, and solutions describing corrections to counter the violation of each of the boundary conditions.

The ideal correction would encompass all factors present, but to date, no such general correction has been developed. Instead, individual or paired corrections accounting for deviations were introduced. The result is that one can end up with an array of numbers that may vary substantially after using these corrections. This is understandable, as historically, each investigator has focused on one or another deviation from the ideal model, while assuming ideality and constancy of the others. This by itself is a substantial violation of principles, as in reality all parameters vary concurrently, in an extent dictated by the particular conditions of the experiment.

Some situations may aggravate one condition, for example having a long pulse, others may introduce other deviations, such as excessive heat losses from the front face due to using very powerful pulses, etc. It is therefore incumbent upon the investigator to choose the most proper correction in harmony with the conditions of the experiment analyzed.

The finite pulse width effect, for example, occurs strongly when thin samples of high thermal diffusivity are tested [2, 7, 8, 9, 10], while the radiative heat losses become dominant at high temperatures [14], when testing thick samples. In contrast, nonuniform heating can occur during any thermal diffusivity

experiment [16]. This can occur when a circular surface smaller than the sample itself is irradiated, or the flux density of the pulse varies from point-to-point over the sample's surface.

For the same amount of absorbed energy, the dimensionless half-max time of the resulting thermogram at the center of the rear face of the specimen differs considerable from the one obtained with uniform irradiation. This effect can be reduced by increasing the ratio between the sample's thickness and its radius.

The same result can be achieved by using a temperature measurement system, which automatically integrates the signal obtained from the rear face of the specimen.

3. Results

ASTM F-75 Cobalt Chromium alloys are widely used in orthopaedic implants such as cemented total hip or knee arthroplasty as well as in metal-on-metal bearings in total hip arthroplasty. The alloys feature medium strength and stiffness combined with high corrosion resistance and good biocompatibility.

Chemical analysis shows that the alloy compositions match well with the ASTM F75 standard specifications and exhibit extremely low levels of impurities, see Table below. Note that high purity is important in orthopaedic implants; for example, it has been reported that Ni possesses allergic potential, and Si may cause embrittlement.

Chemical Composition of CoCr ASTM F75

Cobalt,	Co	Balance
Chromium,	Cr	28,5%
Molybdenum,	Mo	6%
Carbon,	C	0,3%
Iron,	Fe	0,2%
Nickel,	Ni	0,25%
Manganese,	Mn	0,5%
Silicone,	Si	0,7%
Nitrogen,	N	0,15%
Tungsten,	W	0,01%
Phosphorus,	P	0,01%
Sulphur,	S	0,005%
Aluminium,	Al	0,05%
Titanium,	Ti	0,01%

Initial micrographs were taken from the sample surface in order to evaluate the material homogenity and also to see its dendritical structure.

The thermal diffusivity was performed using a FlashLine 3000 device, using a 3 cartidge carrousel. For each sample 3 tests were made using the same

program type having a long equilibrium type, but modifying each time the flash lamp discharging power. We used for each test a Stainless steel standard sample in order to validate the obtained results.

The experimental results will show the thermal diffusivity values for the mentioned alloys and their variation with temperature (Fig. 4) and testing parameters (xenon lamp discharge power - Fig. 5). The sample were discs of 12,5 mm diameter and various height, the measurements being made on 3 sample for each alloy type, from room temperature to 800°C with a 50°C increment.

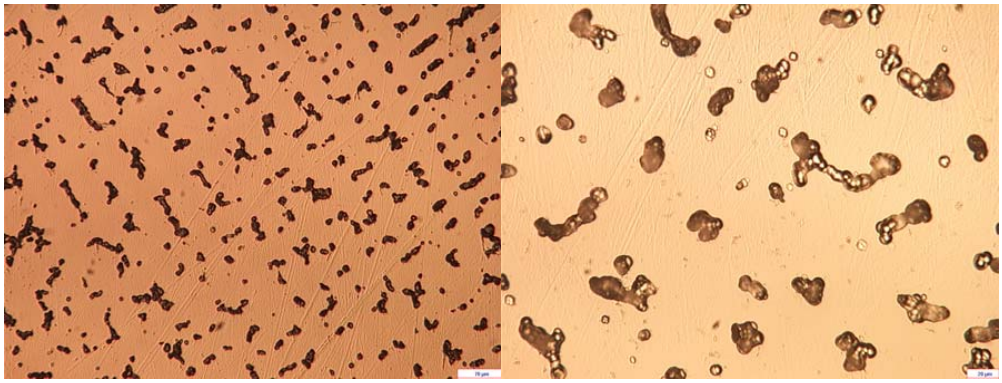


Fig. 3 Optical microscopy image on the Co-Cr-Mo alloy sample (left 200x, right 500x)

Table 1

Temperature	Diffusivity values (cm^2/sec) (±0,0003)		
	Flash lamp power		
	400	600	700
59	0.0344	0.0351	0.0356
107	0.0367	0.0376	0.0374
157	0.0390	0.0392	0.0392
206	0.0406	0.0408	0.0404
255	0.0422	0.0418	0.0420
304	0.0438	0.0442	0.0440
354	0.0458	0.0457	0.0453
404	0.0467	0.0467	0.0463
454	0.0476	0.0472	0.0473

50 6	0.048 6	0.048 4	0.048 2
55 7	0.049 5	0.049 3	0.049 3
60 9	0.050 3	0.050 7	0.050 6

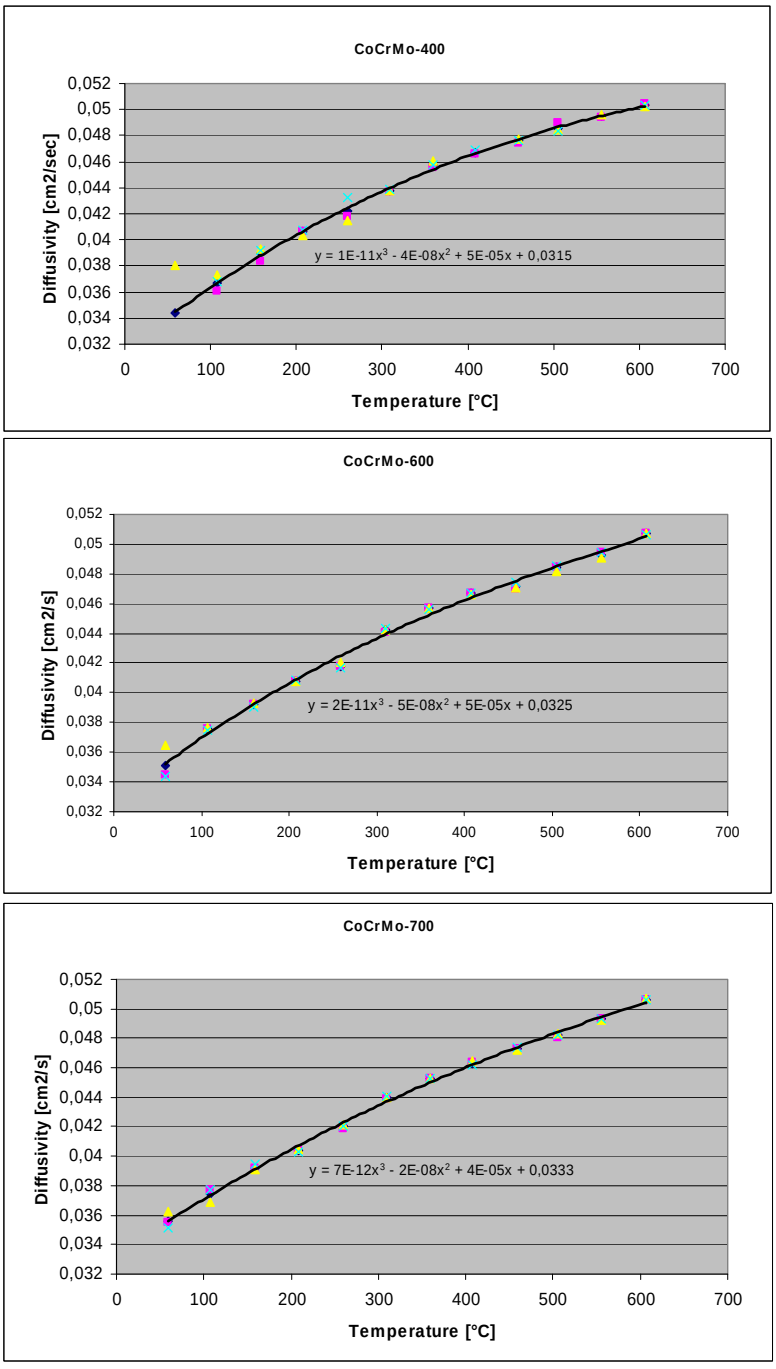
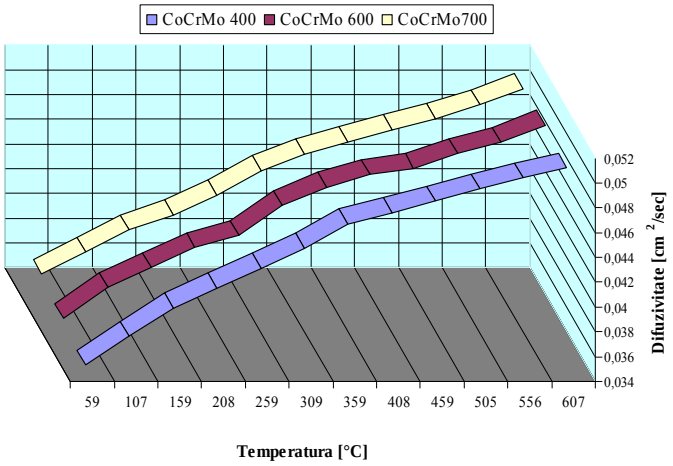


Fig. 4 Diffusivity results on CoCrMo alloy type sample for 400, 600 and 700 Flash lamp power

4. Discussion

It is a very difficult task to choose the best correction, and often not enough information is known about the equipment and the testing parameters to do it prudently. In principle, one must return to the original premise: the accuracy of the data depends on the agreement between the mathematical and experimental models. The purpose of applying corrections to the experimental data is to bring it in closer agreement with the ideal solution by accounting for the aberrations. In consequence, one can perform a series of corrections according to the various schemes developed over the years, and then study the results in relationship to the ideal solution.

The new obtained values on the tested alloys gave as the solution to choose the best diffusivity measurement parameters. As an example, for the F75 alloy type, choosing a low energy pulse for the xenon lamp leads to a reduced dispersion and uniform results only at low temperature (max. 300°C), for higher temperatures the energy pulse requesting to be increased.



5. Conclusion

We measured the influence of the flash lamp power on the results in the case of a CoCr ASTM F75 type alloy in the temperature range from 20 to 800°C. The relative uncertainty of the thermal diffusivity determination is estimated to be from ±1 to ±3%, depending on the temperature.

For metallic alloys having comparable conductivity values with F75 CoCr alloy, the flash power lamp should be set at minimum value for low temperatures diffusivity measurement, and at maximum value for high temperatures.

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