SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage: https://semarakilmu.com.my/journals/index.php/fluid_mechanics_thermal_sciences/index
ISSN: 2289-7879

Determination of Al_2O_3 Powder Thermal Conductivity Using DSC at NIS-Egypt

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ARTICLE INFO

Article history:
Received 19 March 2022
Received in revised form 26 May 2022
Accepted 5 June 2022
Available online 1 July 2022

Keywords:
Thermal conductivity; powder; DSC; thermometry

ABSTRACT

The mission of the thermal metrology laboratory is to maintain, disseminate, develop and realize the International Temperature Scale 1990 (ITS-90). One of the services that the laboratory has introduced is the routine calibration to all industrial sectors that covers almost all fields inside and outside Egypt. This paper explains how to use Differential Scanning Calorimeter (DSC) to determine the thermal conductivity of unknown powders. The paper is based on the ability to determine the thermal conductivity (k) of powders from the melting peak of the reference metal. This technique assists for the first time to establish a new activity at the National Institute for Standardization to test the thermal conductivity of powders, which had been requested by many clients. Experiments were carried out using alumina powder Al_2O_3 and metal reference beads of Sn and Zn which have diameter ranges from 1.9 mm up to 4.12 mm in an atmosphere of nitrogen during Sn and Zn melting. The results show good performance. The results showed confidence that will enable to do more studies at NIS to see if this technology can be applied to a wider spectrum of materials.

1. Introduction

Thermal Metrology Laboratory (ThML) at the National Institute of Standards (NIS) is one of the leading laboratories that satisfy the metrological traceability of temperature to all medical, industrial, agriculture, and other sectors inside and outside Egypt. Our mission is to maintain, disseminate and develop the unit of thermodynamic temperature that was defined by the Boltzmann constant in the advanced research branch and the approximated thermodynamic temperature that was realized by the International Temperature Scale 1990 (ITS-90). Furthermore, there are another mission for ThML is to introduce the industrial routine calibration, thermal exposure, viscosity, humidity, thermal conductivity, and more services that are traceable to ITS-90 [1-8]. Thermal conductivity is a physical quantity that must be taken into consideration when evaluating building materials, as it is one of the most important requirements, especially in extreme areas in high or low temperatures in a thermal climate. Heat transfer equations and theories are used to determine the thermal conductivity (k) of

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https://doi.org/10.37934/arfmts.97.1.149156

an unknown substance [9]. There are different methods of measuring thermal conductivity, and each one of them is suitable for a narrow and specific group of materials. One of the methods is using Differential Scanning Calorimetry (DSC).

Materials with low thermal conductivity have a slower rate of heat transfer such as insulators, while for example metals have higher thermal conductivity, unlike insulators. Heat sink applications use materials with high thermal conductivity, while thermal insulation typically uses materials with low thermal conductivity. The effect of temperature on the thermal conductivity of metals and non-metals varies. The ability of a material to conduct heat is measured by designating its coefficient of thermal conductivity. The letter k is widely used to represent it [8].

Differential Scanning Calorimetry (DSC) is a type of thermal analysis equipment that monitors the flow of heat in and out of a substance as a function of temperature and time. DSC is generally used to determine the transition temperatures and the relevant heat for a reaction.

In the last years, there has been an associate interest of makers and laboratories in measuring the thermal conductivity of materials. This measurement has become a major concern. This study aims to determine the thermal conductivity coefficient of an unknown powder sample as one of the applications that can be used as a differential thermal analysis device (DSC) to meet the customers' requests, as there is a wide range of applications approved in several scientific and technical sectors to determine the thermal conductivity of powdered materials [10-12].

2. Experiment and Results

The experimental work begins by using the Differential scanning calorimeter instrument which is Shematzu model DSC-50 to determine the thermal conductivity of powders [13-17].

By using this technique, the maximum thermal contact between the metal used and the crucible is applied. A spherical metal Sn and Zn as a reference was put at the bottom of the crucible during the first Run of DSC measurements. DSC schematic diagram is shown in Figure 1. The melting peak's slope (Se) is calculated using the formula described below

$$S_e = \frac{1}{R+R_c} \quad (1)$$

where Se is the melting peak's slope, R is the thermal resistance of the crucible; Rc is the thermal resistance of the metal

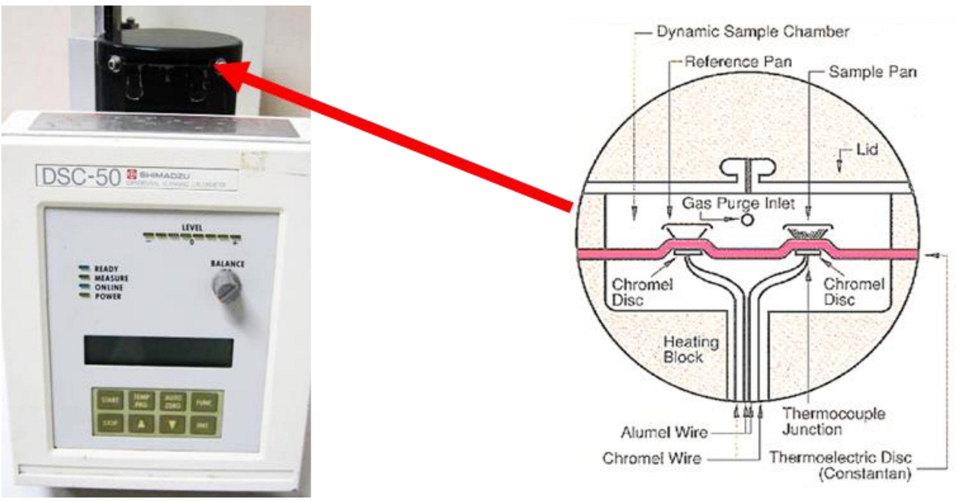

Fig. 1. DSC schematic diagram

Figure 2 shows an example of the reference metal DSC melting curve.

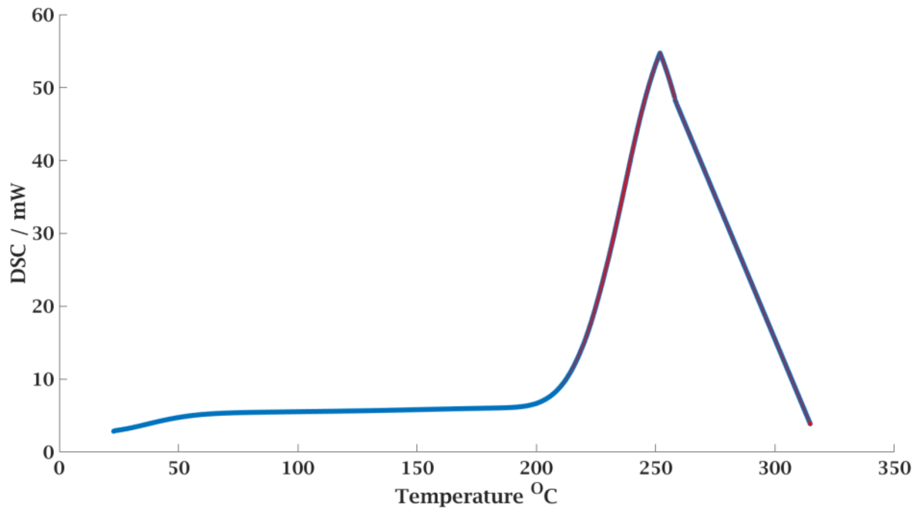

Fig. 2. DSC melting curve

The second run was done by the DSC crucible filled with the unknown powder and partially sinking a spherical metal reference inside it as indicated in Figure 3. In this work, alumina powder with a purity of more than 99% was used. A spherical reference metal bead was put on top of the powder filling the crucible and sinking into the powder until its center was concentric to the crucible. In the DSC device, the filled crucible was heated at a continuous and constant rate of 10 °C/min until the metal is completely melted. The melting peak's slope is determined by the powder's resistance.

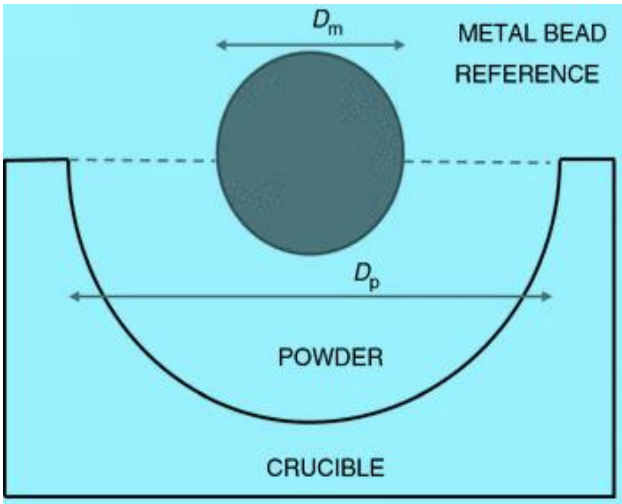

Fig. 3. A schematic diagram of spherical reference metal bead is placed on top of the powder filling the crucible

The slope of the melting peak was calculated using the formula described below

$$S_p = \frac{1}{R+R_c+R_p} \quad (2)$$

where S_p is the melting peak's slope; R is the thermal resistance of the crucible; R_p is the thermal resistance of the powder; R_c is the thermal resistance of the metal.

The thermal resistance of the powder R_p can be obtained and calculated using Eq. (1) and Eq. (2). Then, using Eq. (3), we can calculate the thermal conductivity coefficient of the powder.

$$K = \frac{1}{2\pi R_p}\left(\frac{1}{r_i} - \frac{1}{r_e}\right) \tag{3}$$

where r_i is the reference metal sphere radius, r_e is the crucible radius and R_p is the thermal resistance of the powder

The experimental approach has been carried out with two different techniques to check the validity of the results

i. Fixed heating rate and variable Sn and Zn metal diameters reference
ii. Fixed Sn reference metal diameters and variable heating rate

2.1 Fixed Heating Rate and Variable Sn and Zn Metal Diameters Reference

Several runs were carried out to assure the quality, repeatability, and reproducibility of the results. A variety of Sn and Zn metal reference beads with sizes ranging from 1.9 to 4.12 mm were used. The aluminum crucibles were lightly tapped after being filled with alumina powder.

Table 1, Table 2, Figure 4, and Figure 5 show the results obtained from experiments carried out with powders in N_2 at the melting point of Sn and Zn at a heating rate of 10°C min^{-1} respectively.

Table 1
results of experiments carried out with powders in N_2 at the melting point of Sn

Powder	Metal sphere diameter/mm	Heating rate °C min^{-1}	Thermal conductivity W °C^{-1} m^{-1}
Al_2O_3	2.1	10	0.11
	2.88	10	0.11
	3.17	10	0.12
	3.43	10	0.12
	3.80	10	0.123
	3.99	10	0.124
	4.12	10	0.125

Table 2
Results of experiments carried out with powders in N_2 at the melting point of Zn

Powder	Metal sphere diameter/mm	Heating rate °C min^{-1}	Thermal conductivity W °C^{-1} m^{-1}
Al_2O_3	1.9	10	0.115
	2.9	10	0.12
	3.35	10	0.122
	3.9	10	0.125

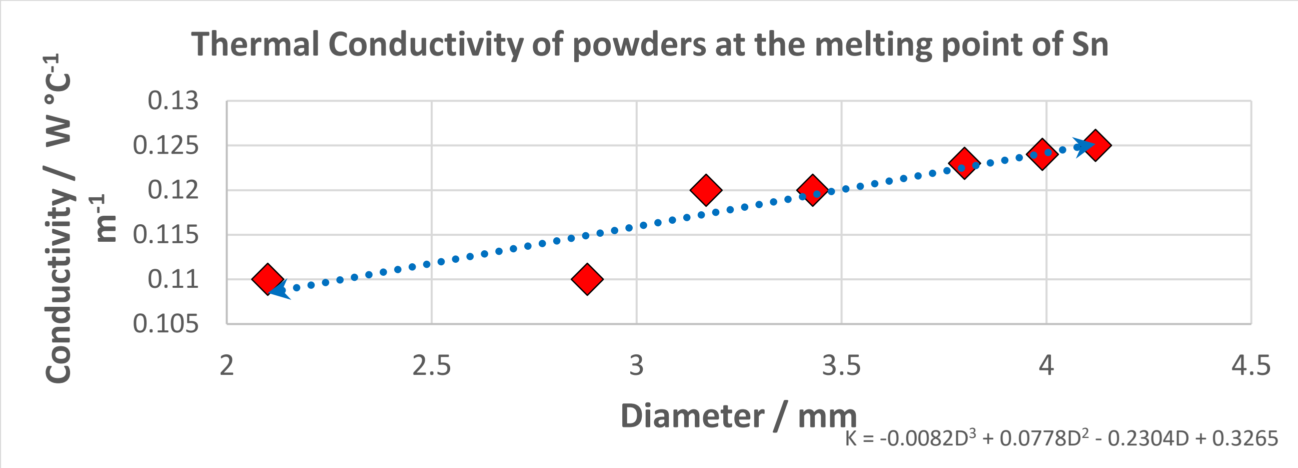

Fig. 4. The relation between the thermal conductivity of powder at the melting point of Sn

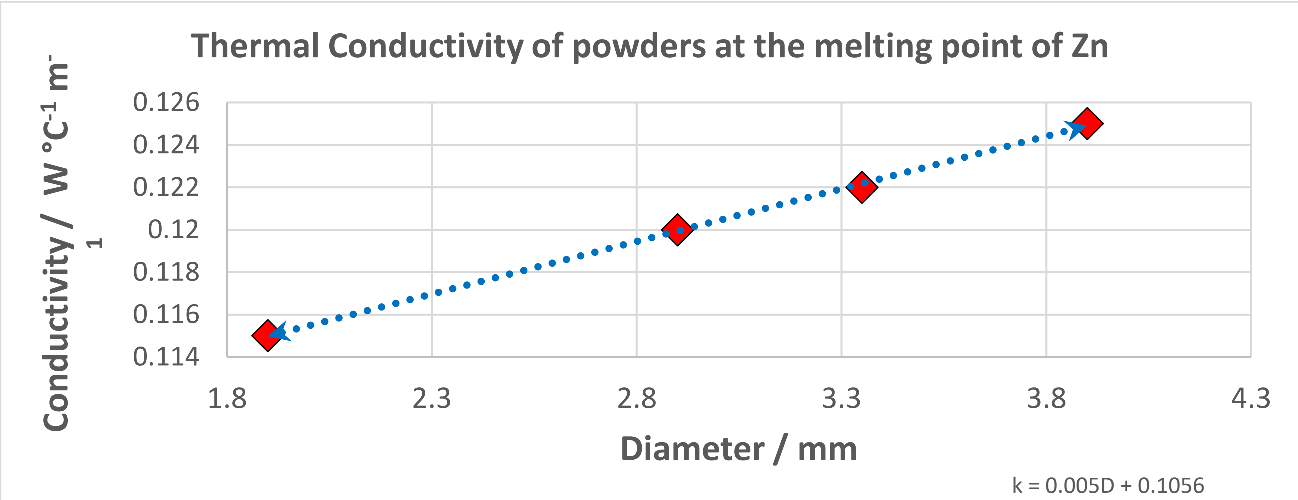

Fig. 5. The relation between the thermal conductivity of powder at the melting point of Zn

The differences between the calculated thermal conductivity values at different diameters are very small. Differences found for different reference metals at changing the diameters from 1.9 to 4.12 mm did not show any observable effects on the thermal conductivity values. These show that the system is suitable for measuring the thermal conductivity of powders.

2.2 Fixed Sn Reference Metal Diameters and Variable Heating Rate

A second run with a set of trials was conducted at different heating rates (5°C, 10°C, 15°C, and 20°C min $^{-1}$). The slope exactly represents the reciprocal of the powder resistance plus the DSC sensor resistance. Table 3 and Figure 6 summarize the results obtained at different heating rates utilized to verify the method on alumina powder, as well as the results of thermal conductivity of alumina powder. More studies are currently being done at NIS to check if this technology can be applied to a wider spectrum of materials or not. Low heating rates cause the slope to rise.

Table 3
Parameters and results of experiments carried out with powders in N_2 at the melting point of Sn at different heating rates

Powder	Metal sphere diameter/mm	Heating rate °C min^{-1}	Melting slope /mWºC^{-1}	Thermal conductivity W °C^{-1} m^{-1}
Al_2O_3	3.69	5	2.15	0.129
		10	1.7	0.121
		15	3.5	0.121
		20	3.3	0.11

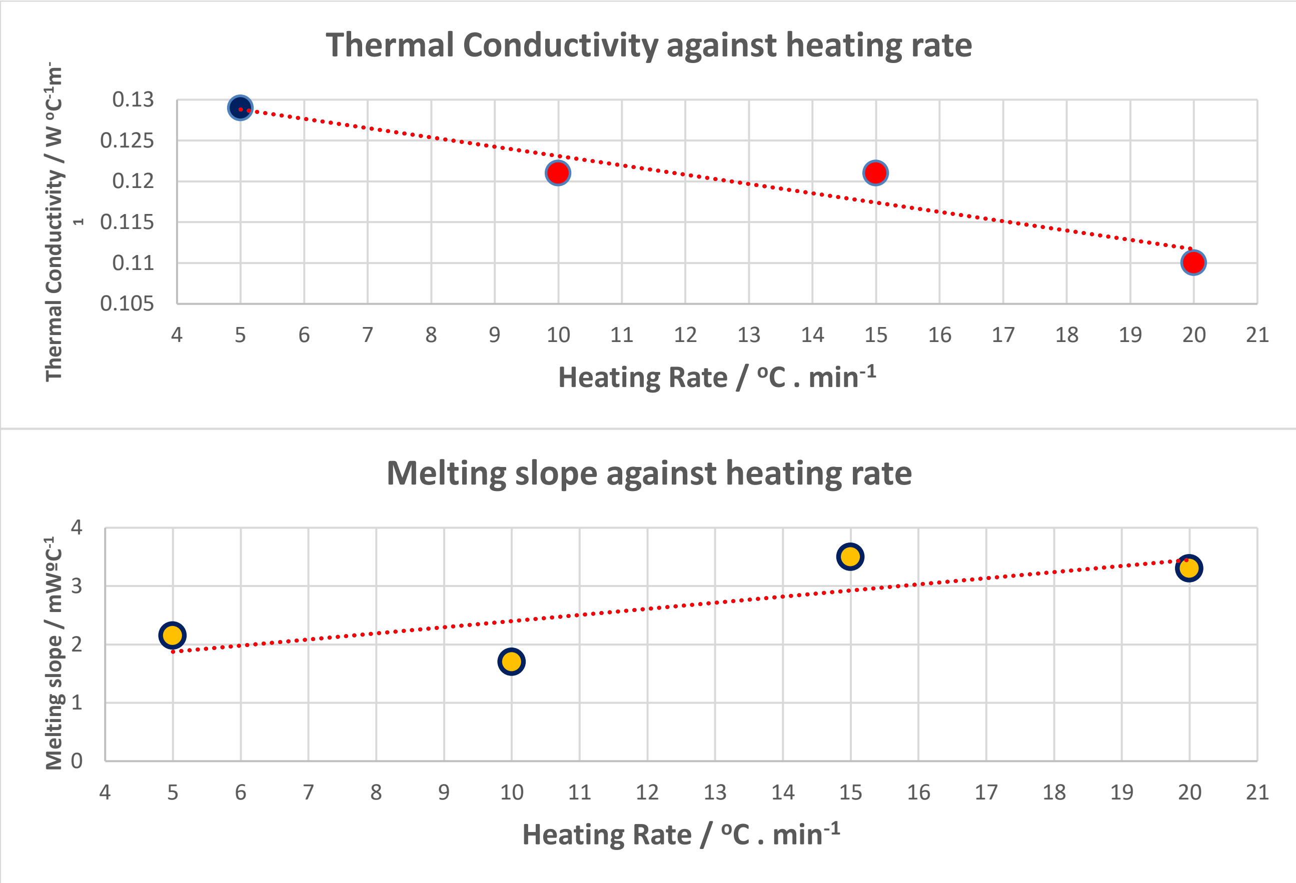

Fig. 6. The effect of heating rate on melting curve slope and the value of k

The differences between the calculated thermal conductivity values of Al_2O_3 powder at different heating rates are very small. Differences found for different reference metals at changing the heating rates from 5 to 20 °C min^{-1} did not show any observable effects on the thermal conductivity values. These show that the system is suitable for measuring the thermal conductivity of powders.

3. Conclusion

The Thermal Metrology laboratory at the National Institute of Standards has started a new challenge to be able to measure the thermal conductivity of powders with Differential Scanning Calorimetry DSC. Experiments were carried out using alumina powder Al_2O_3 and metal reference beads of Sn and Zn which have diameter ranges from 1.9 mm up to 4.12 mm in an atmosphere of nitrogen during Sn and Zn melting at heating rates from 5 to 20 °C min-1. This technique assists for the first time to establish a new activity at the National Institute for Standard to test the thermal conductivity of powders, which had been requested by many clients. The results show good performance and give confidence that will enable to do more studies at NIS to see if this technology can be applied to a wider spectrum of materials or not.

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