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# DEVELOPMENT OF A THERMAL CONDUCTIVITY CELL WITH NANOLAYER COATING FOR THERMAL CONDUCTIVITY MEASUREMENT OF FLUIDS

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Various techniques and methodologies of thermal conductivity measurement have been based on the determination of the rate of directional heat flow through a material having a unit temperature differential between its opposing faces. The constancy of the rate depends on the material density, its thermal resistance and the heat flow path itself. The last of these variables contributes most significantly to the true value of steady-state axial and radial heat dissipation depending on the magnitude of transient thermal diffusivity along these directions. The transient hot-wire technique is broadly used for absolute measurements of the thermal conductivity of fluids. Refinement of this method has resulted in a capability for accurate and simultaneous measurement of both thermal conductivity and thermal diffusivity together with the determination of the specific heat. However, these measurements, especially those for the thermal diffusivity, may be significantly influenced by fluid radiation. Recently developed corrections have been used to examine this assumption and rectify the influence of even weak fluid radiation. A thermal conductivity cell for measurement of the thermal properties of electrically conducting fluids has been developed and discussed.

***Keywords:*** *nanolayer coating, thermal conductivity, thermal conductivity cell, thermal diffusivity, thermal radiation, transient hot wire technique*

## Introduction

Measurement of thermal conductivity involves several parameters that are common to different techniques and methodologies. In addition to the variations due to the nature and type of samples, all methodologies require determination of the actual amount of heat transferred through the sample along and perpendicular to the heat flow path in a given thermal environment. The calculated value is expressed in the same unit as that provided for a standard of the same material. Conductivity, as opposed to conductance, provides dimensional attributes to the calculated value. Thus, thermal conductivity is related to a material property that denotes a rate process of heat transfer. Thermophysical properties such as thermal conductivity, thermal diffusivity, specific heat and other thermal parameters of materials obtained by various measurement and/or thermo-analytical techniques are important for industrial applications as well as for materials characterization. Therefore, it is not surprising that many authors [1–8] have investigated the thermal properties including thermal conductivity, thermal diffusivity and thermal decomposition together with other studies to characterize various materials. The thermal conductivity of fluids, in particular, has direct use in the design of heat exchangers. Therefore, it is very essential to develop suitable instruments as well as techniques for its accurate experimental measurement. The transient hot-wire instrument provides rapid and accurate measurements of the thermal conductivity of fluids [9–11]. Various corrections [10] are necessary to account for the differences between the actual and ideal heat transfer models. This includes thermal radiation effects which depends on the surrounding fluid if it is transparent to or absorbs thermal radiation. Earlier transient line-source measurements have considered propane to be transparent to thermal radiation. Radiation-corrected measurements for propane show significant deviations from NIST [12] which does not include these corrections.

## Significance of thermal properties measurement

Accurate and reliable thermal conductivity data of fluids are essential to evaluate convective heat transfer coefficient for safe and economical design of heat

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exchangers involving heat transfer across a fluid-solid interface. In such heat transfer processes, convective heat transfer coefficient is a very important parameter and can be evaluated from the following dimensionless equation [13a]:

$$\overline{\mathrm{Nu}} = C\mathrm{Re}^{\mathrm{m}}\,\mathrm{Pr}^{\mathrm{n}} \tag{1}$$

where $C$, $m$ and $n$ are empirical parameters. For heat transfer in a tubular geometry, these parameters, $C$ and the exponent $m$ and $n$, are either constant or expressed by empirical relations [13b]. In Eq. (1), the dimensionless quantities Nusselt number (Nu), Reynold number (Re) and Prandtl number (Pr) are defined by:

$$\overline{\mathrm{Nu}} = \frac{\overline{h}_{\mathrm{c}} D}{k};\ \mathrm{Re} = \frac{\rho V D}{\mu};\ \mathrm{Pr} = \frac{c_{\mathrm{p}}\mu}{k} \tag{2}$$

where $\overline{\mathrm{Nu}}$, Re and Pr are expressed in terms of the fluid properties and fluid flow and the geometrical characteristics of the physical system. Accurate thermal conductivity measurements can also be utilized for other applications such as detection of impurities and quality control in mixtures as well as to develop and verify theoretical models for thermophysical properties of dense fluids. However, in the absence of any accurate theoretical model for dense fluids, experimental measurement is the only reliable method for determination of the thermal conductivity of fluids. In addition, one of the experimental techniques provides simultaneous measurement of the thermal diffusivity and determination of the specific heat of fluids.

## Experimental

### *Electrically non-conducting fluids*

The experimental techniques for accurate measurement of thermal conductivity of fluids fall into the following two broad categories, steady state method and transient technique which are described below.

### *Steady state method*

This method employs a thin layer of fluid between two smooth isothermal surfaces maintained at two different temperatures. Guarded hot-plate apparatus, concentric cylinder apparatus and concentric shell apparatus fall in this category. Guard heaters are used in the first two methods to maintain the desired direction of heat flow. The heat flux and temperature difference across the fluid layer is accurately measured and the thermal conductivity ($k$) of the fluid is calculated from the Fourier's heat conduction relation [13c]:

$$q = -kA\frac{\mathrm{d}T}{\mathrm{d}x} \tag{3}$$

where $q$ is the heat transferred by conduction through area A and $\mathrm{d}T/\mathrm{d}x$ is the temperature gradient in the direction ($x$) of heat transfer and normal to the surface area A. Steady state methods are inherently time consuming and slow as thermal systems take long time to reach steady state conditions. The influence of parasitic heat losses and the radiation effects, if present, can significantly influence the measurements [14].

### *Transient method*

The most widely used transient technique utilizes a very fine wire stretched vertically and surrounded by the test fluid. A constant current through the wire results heat dissipation from the wire into the surrounding fluid. As a result of this process, the fine wire undergoes a transient temperature rise which depends on the thermal conductivity of the surrounding fluid. Therefore, the thermal conductivity of the fluid is obtained from the transient response ($\Delta T_{\mathrm{w}}$~time) of the wire. This technique was first proposed by Stalhane and Pyk [15]. A complete solution by van der Held [16, 17] led to a mathematical formulation of an ideal model of the transient line-source technique and an absolute measurement of the thermal conductivity ($k$) of the fluid from the following equation:

$$\Delta T_{\mathrm{w}} = \frac{q_{\ell}}{4\pi k}\ln\left(\frac{t_2}{t_1}\right) \tag{4}$$

where $\Delta T_{\mathrm{w}}$ is the temperature rise of the wire during the time period $t_1$ to $t_2$ and $q_{\ell}$ the heat dissipation rate per unit length of the wire. There are two variations of this apparatus – potential-lead type [18–20] and compensated-wire type [21, 22] which have been used to closely model the ideal line-source.

### *Anodizing procedure*

The cell was cleaned and rinsed, and the wires were chemically polished by mixing acid solutions of $H_2SO_4$, $HNO_3$ and HF in recommended proportions to remove the residual oil and dirt prior to anodizing. After anodizing, a tantalum oxide film having a high dielectric constant in amorphous state which includes moisture and impurities such as organic substances needed a heat treatment at a temperature which is higher than the formation temperature of the tantalum oxide film. As a result of the heat treatment, the moisture and the impurities existing in the film were removed. All the procedures were performed in the fume hood in the chemistry laboratory as described below.

1) Preparing polishing solution: The solution for polishing the wires is a mixture of 98% $H_2SO_4$, 70% $HNO_3$, and 40% HF solution in the ratio of 10:4:3 by volume.

- 530 mL 98% $H_2SO_4$ 212 mL 70% $HNO_3$, and 159 mL 40% HF were prepared. As HF acid is a highly toxic and corrosive solution which dissolves glass, it was stored in a high density polyethylene container which is generally marked as HDPE (high density polyethylene) on the bottom.
- The glass container with the $HNO_3$ acid surrounded by ice in the tray was placed on the stirring machine. It was pre-cooled to minimize the temperature rise, expected during mixing procedure to follow.
- $H_2SO_4$ solution was poured into $HNO_3$ solution slowly and carefully because of the heat arising from the acid solution.
- The mixture of $HNO_3$ and $H_2SO_4$ solution was decanted into the HDPE container surrounded by ice in the tray on the stirring machine.
- HF solution was poured into the mixture solution very slowly and carefully.

2) The cell was placed into the mixture solution to polish only for 3 s.

3) The cell was pulled out and placed into the hot water at 60–70°C next to the mixture solution. The reason for not placing the cell into boiling water directly is to avoid breaking the wires.

4) The cell was removed after the water had been boiled for 15 min, and placed into the distilled water and rinsed three times.

5) Anodizing the hot wire cell: The cell was placed into the 0.2 M $H_2SO_4$ solution in demineralized water to be anodized. As shown in Fig. 1, the cell is set up to be anode by connecting it to the multimeter to monitor the current flow, which is connected to the positive of the DC power. The platinum wire in spiral shape in order to increase the area of coating becomes cathode as it is connected to the negative of DC power.

6) The voltage was turned up to 50 V, and held constant until the current flow fell to about 0.015 mA (up to 10 mA of noise may be seen on the 300 mA range in HP3478A). The value of current through the circuit is shown in Table 1. Under these conditions, a layer of tantalum pentoxide ($Ta_2O_5$) is formed on the surface of the wires with a beautiful color. Utmost care was taken so that tantalum wires did not touch the platinum wire during the anodizing process, otherwise, 50 V DC power will burn the wires.

7) The cell was rinsed three times in the distilled water.

8) The cell was placed in an oven and heated for 6 h at the 180°C.

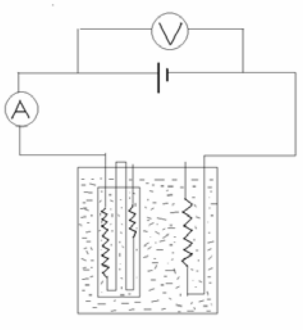

**Fig. 1** Schematic of tantalum wire anodizing circuit

**Table 1** Current of electrical circuit during anodization

| Time | DC current/mA |
|---|---|
| 12:13PM | 120 |
| 12:13:30 | 80 |
| 12:14 | 3.5 |
| 12:15 | 1.69 |
| 12:16 | 1.25 |
| 12:17 | 0.975 |
| 12:21 | 0.590 |
| 12:26 | 0.380 |
| 15:07 | 0.033 |
| 15:14 | 0.032 |
| 18:13 | 0.020 |
| 21:11 | 0.017 |
| 9:36AM | 0.014 |

*SEM-EDS measurement*

JEOL JSM6400 Digital SEM (scanning electron microscope) which is equipped with PGT-EDS system was utilized to perform experimental analysis on the sample of anodized hot wire after the anodizing work was finished.

## Results and discussion

*Electrically non-conducting fluids*

The ideal line source model employs a fine wire of infinitely small radius $a \to 0$ and infinite length $\ell \to \infty$. In the actual model, however, the wire has a finite diameter and a finite length which is attached to thick wires at the ends in a hot-wire cell. The use of potential leads as well as the compensating wire allows the ideal line source to be realized in a hot-wire cell. At small radial distance $r=a$, the temperature rise at time $t$ is approximated by [23]

$$\Delta T_{\mathrm{w}}(a,t)=\frac{q_\ell}{4\pi k}\left(\ln\frac{4\alpha t}{a^2C}\right)+\frac{a^2}{4\alpha t}+\ldots \quad (5)$$

If the wire radius is very small, the above solution can be further simplified to [22]:

$$\Delta T_{\mathrm{w}}(a,t)=\frac{q_\ell}{4\pi k}\left(\ln\frac{4\alpha t}{a^2C}\right)=G\ln(t)+A \quad (6a)$$

where

$$G=\frac{q_\ell}{4\pi k}$$

and

$$A=\frac{q_\ell}{4\pi k}\ln\frac{4\alpha}{a^2C} \quad (6b)$$

Equation (6a) shows a straight line relationship of $\Delta T_{\mathrm{w}}(a,t)\sim\ln(t)$ with slope G and intercept A [24]. The slope $G$ and the intercept A both can be determined experimentally from accurate and precise measurement of the transient response of the wire. The transient hot-wire technique, therefore, provides an experimental method of simultaneous measurement of the thermal conductivity and the thermal diffusivity of fluids from the following equations obtained from Eq. (6b):

$$k=\frac{q_\ell}{4\pi G}$$

and

$$\alpha=\frac{a^2C}{4t_0}\mathrm{e}^{A/G} \quad (7)$$

In addition, the specific heat of fluids can also be determined from the following equation:

$$\alpha=\frac{k}{\rho c_{\mathrm{p}}} \quad (8)$$

if the density ($\rho$) of the fluid is known either directly or from its Equation of State.

The hot-wire instrument, measurement technique and data processing have to ensure to account for various deviations of the actual line-source model from the ideal model. Convection and radiation heat transfer, if present, can result in significant error. Proper experimental procedure [22, 24–28] has to be followed in order to avoid convection effects and correct for radiation effects.

Figure 2 shows the schematic diagram of the hot-wire cell. The transient line source technique provides an absolute, rapid, accurate and reliable measurement of the thermal conductivity of fluids. It also provides a simultaneous measurement of the thermal diffusivity of fluids and specific heat determination. This technique permits convection-free measurements. In addition, the influence of radiation effect, if present, is less pronounced in comparison to the steady-state methods as the surface area of the hot wire is very small compared to the isothermal surfaces in the steady-state techniques.

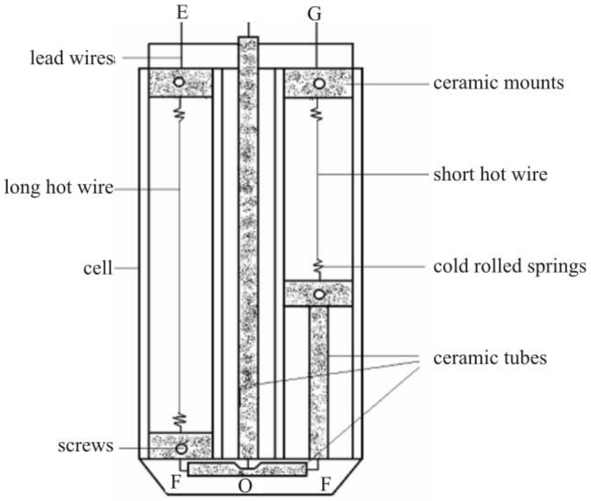

**Fig. 2** Schematic diagram of the hot-wire cell – compensating wire type

*Electrically conducting fluids*

The above system works well with electrically non-conducting fluid as there is no (or infinitely small) current leakage into the fluid and all the current passes through the wire. However, in case of electrically conducting fluids, there is a significant current leakage into the surrounding fluid. Therefore, the mathematical model and the solutions described above are not applicable. Measurement for such fluids are have been attempted earlier [29–31] using a very thin insulating layer on the wire.

To overcome these problems, Nagasaka and Nagashima [28] proposed the use of a polymeric coating on the platinum wire of 40 μm in order to insulate the wire from the fluid. The thickness of insulation layer in this case was 7.5 μm which is the 18.75% of the diameter of platinum (40 μm) resulting in large corrections. To avoid this problem, Alloush *et al.* [30] used a tantalum wire cell with a wire diameter of 25 μm. This wire was anodized and a thin insulating layer of tantalum pentoxide was deposited on the surface of this wire. The oxide layer is resistant to chemical attack and mechanically stable. Also, the insulating layer is estimated to be less than 100 nm thick and the associated corrections to the measurement based on the transient technique are estimated to be minimal.

The earlier work by Wang, Prasad and Venart [31] from the University of New Brunswick research group employed 25 μm diameter tantalum wire, which was anodized resulting in a very thin layer of tantalum pentoxide ($Ta_2O_5$) on the wire surface. Measurements of the thermal conductivity of sodium hydroxide and lithium hydroxide solutions of varying concentrations were reported [31].

A modified cell has been developed in our laboratory for measurement of the thermal properties of electrically conducting fluids. The anodizing procedure is outlined here in details.

*Anodizing tantalum wire*

In this work, the 0.2 M sulfuric acid solution was used to anodize the tantalum wires by an electrochemical process. The tantalum wire is used as the anode by connecting it to the positive terminal of a 50 VDC power supply and the cathode is connected to the negative terminal of the supply. In this work, the cathode is a platinum wire. When the circuit is closed, electrons are withdrawn from the metal at the positive terminal allowing ions at the tantalum surface to react with water to form an oxide layer on the metal. The electrons return to the cathode where they react with hydrogen ions to make hydrogen gas. It has been known that the weak organic acid solutions could produce uniform oxide films on tantalum with good dielectric properties without many holes and imperfections.

The chemical equations describing the anodization process are as follows:

Anode:

$$2Ta \rightarrow 2Ta^{5+} + 10e$$

$$2Ta^{5+} + 10OH^{-} \rightarrow Ta_2O_5 + 5H_2O$$

Cathode:

$$10H_2O + 10e \rightarrow 5H_{2+}10OH^{-}$$

This coating layer is dielectric of tantalum pentoxide. The voltage and current are controlled to form the pentoxide layer. Tantalum is a valve metal, and the amorphous pentoxide grows stable to form a uniform, closely coupled layer over the surface. The thickness of coating layer depends on the magnitude of the anodization voltage. The thickness of coating is estimated to be in the range of 70 to 120 nm [32].

In this work, the process of anodizing was continued until there was no appreciable current flow. Actually very small current at relative high voltage indicates that a good electrical insulation layer has been formed that is free from significant imperfections. At a fixed anodization voltage there is an exponential decay in current flow with elapsed time as the thickness of the oxide layer increases to its final value. The tantalum pentoxide layers are optically transparent and behave as interference filters. The layer thickness could be indicated from the color of the anodized film. This will progress from a black to green to yellow to orange to red and then repeat as the layer gets thicker. In this work, the golden color was obtained with an anodization voltage of about 50 V and by waiting until the current cannot be measured any more with the employed multimeter set in mA range.

EDS (Energy disperse X-ray spectroscopy) is an analytical tool predominantly used for chemical characterization based on the fundamental principle that each element listed in the Chemical Periodic Table has a unique response to electromagnetic waves. It is deduced that the tantalum pentoxide film has been formed on the tantalum wire based on the indication that the oxygen content in the coated wire is higher than that in the bare wire.

Tantalum is dark, dense, ductile, hard and highly conductive of heat and electricity. This metal is well-known for its resistance to corrosion by acids. Actually it is almost immune to attack by normally aggressive aqua regia below 150°C. The melting point of tantalum is 3290 K (3017°C). $Ta_2O_5$ is a high refractive, low absorption material useful for coating in the near UV to IR regions and decomposes only at the temperature above 1470°C. Tantalum pentoxide is not soluble in water and its melting point is about 1872°C. The thermophysical properties of Ta and $Ta_2O_5$ are shown in Table 2.

With the attainable 10 nm resolution and available magnifications range from 10 to 300000×, the pictures of bare wire and anodized wire, as well as the cross sections were obtained, as shown in Figs 3–5.

**Table 2** Thermophysical properties of the wire and coating

| | Tantalum | Tantalum pentoxide |
|---|---|---|
| Symbol | Ta | $Ta_2O_5$ |
| Molar mass/g mol$^{-1}$ | 180.948 | 441.893 |
| Thermal conductivity/W m$^{-1}$ K$^{-1}$ | 57.5 | 0.4 |
| Specific heat/J kg$^{-1}$ K$^{-1}$ | 140 | 306 |
| Density/kg m$^{-3}$ | 16400 | 8240 |

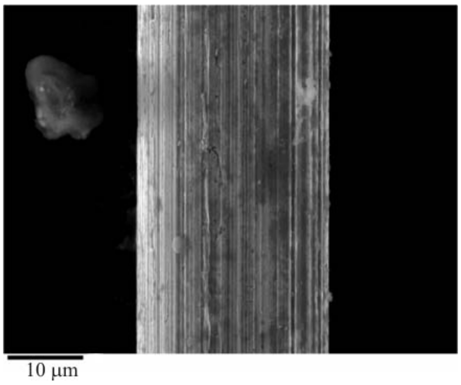

**Fig. 3** Picture of the bare tantalum hot wire

Figure 6 shows a photograph of the compensating wire cell designed and built at UNB. Two tantalum wires, a long wire and a short wire, are assembled in a cylindrical cavity machined in a Teflon rod. The wires are attached to thick Ta wires mounted in ceramic pieces which are, in turn, secured to the Teflon body with Teflon screws. This cell is currently in use for measurement of the thermal conductivity and thermal diffusivity of poly (acrylic) acid solutions which will be discussed in a future work. The hot-wire instrument with anodized Ta-wire cell provides a suitable means of measurement of the thermal properties of electrically conducting fluids. The measurement technique and data processing system can account for various deviations of the actual line-source model from the ideal model. The measurements are corrected for radiation effects, if present, and the influence of the insulating layer.

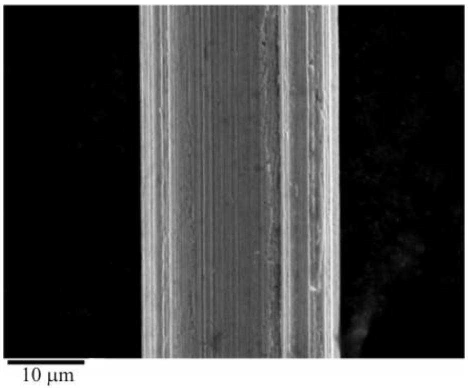

**Fig. 4** Picture of the anodized tantalum hot wire

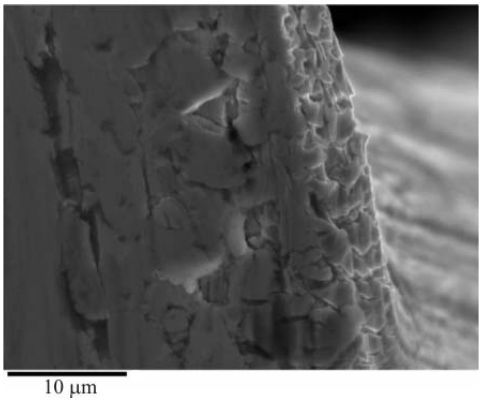

**Fig. 5** Picture of the cross section of the anodized tantalum wire

**Fig. 6** Thermal conductivity cell for measurement of the thermal properties of electrically conducting fluids

## Nomenclature

| | |
|---|---|
| $a$ | radius of the wire |
| $A$ | parameter in Eq. (6) |
| $C$ | a constant (exponential of Euler's constant) |
| $c_p$ | specific heat of fluid |
| $D$ | characteristic dimension of the pipe/tube |
| $G$ | parameter in Eq. (6) |
| $\bar{h}_c$ | convective heat transfer coefficient |
| $K$ | thermal conductivity of fluid |
| $\overline{\mathrm{Nu}}$ | Nusselt number |
| Pr | Prandtl number |
| $q_\ell$ | heat dissipation rate per unit length of wire |
| Re | Reynolds number |
| $V$ | velocity of fluid |
| $t$ | time |
| $t_0$ | reference time (=1 s) |
| $T$ | temperature |

### *Greek symbols*

| | |
|---|---|
| $\alpha$ | thermal diffusivity of fluid |
| $\Delta T$ | temperature difference |
| $\mu$ | viscosity of fluid |
| $\rho$ | density of fluid |

### *Subscripts*

| | |
|---|---|
| $\ell$ | per unit length |
| $w$ | wire |
| 0 | reference |
| 1 | initial |
| 2 | final |

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