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MEASUREMENT OF THERMAL CONDUCTIVITY OF THE OXIDE COATING ON AUTOCLAVED MONEL - 400

by

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Chemistry Division

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BHABHA ATOMIC RESEARCH CENTRE

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INTRODUCTION

Monel, a nickel-base alloy, has wide industrial applications and is used in heat exchangers of the primary coolant of some of the nuclear power reactors. Under operating conditions of the reactor a thin oxide coating gets formed on it which is expected to affect the heat transfer characteristics. It is thus desirable to measure thermal conductivity of these oxide coatings.

The direct measurement of thermal conductivity of thin coating is rather difficult. According to Fourier law of steady state heat transfer i.e.

$$q = -\lambda \frac{dT}{dx}$$

where q is heat flux, $\frac{dT}{dx}$ is temperature gradient and λ is the thermal conductivity.

It thus becomes imperative to measure temperature difference across the film thickness for a known heat flux. To overcome this difficulty some indirect (1) and some transient methods (2) have been employed. Herein the measurement of thermal conductivity of the oxide coating on monel-400 has been reported. The method used is simple, direct and reliable and was applied for the first time by the authors for making such measurements on thermally insulteing metal oxide coatings (3).

PRINCIPLE OF THE TECHNIQUE

The oxide coating is applied on an electrically conducting wire having stable characteristics. This coated wire is placed in a constant temperature bath and a constant direct current is passed through it. It will thus get heated and lose heat to the surrounding. The amount of heat lost will depend on λ_c, thermal conductivity of the oxide coating which in turn will determine the effective wire temperature. Temperature is determined by considering it as a resistance thermometer. The analytical expression (4) for λ_c involves a convection heat transfer coefficient which is difficult to determine experimentally. To eliminate it, a second uncoated wire of a noble metal having similar surface finish as that of the coated wire is connected in series with it.

EXPERIMENTAL

The schematic set up is shown in Fig.1. Herein, a bare platinum wire (dia. $\sim 2.6 \times 10^{-4}$m) and oxidised wire of monel-400 * (dia. $\sim 4.0 \times 10^{-4}$m) were used. Monel wire was autoclaved at 673K in steam for 20 days at a pressure of 10.35 M Pa resulting in the formation of oxide coating of thickness $\sim 9 \times 10^{-6}$m. Double distilled water of pH 6.8 was used in the autoclave. The constant temperature bath contained demineralized water having an electrical conductivity of $< 0.3 \times 10^{-4} \mho m^{-1}$ and its temperature was maintained better than 0.05 K using Melab's proportional temperature controller. The contstant direct current source used in the system was of Transpack type SS 100/5 (Universal Instruments Co., Bangalore, India) which had a stability of 0.01%. Current was varied from 0.8 to 3.8 amps. and was measured accurately

* Its composition ′ ˢ .

Element	Ni	Cu	Fe	Mn	Si	C	S
Weight%	64.3	32.4	1.4	0.9	0.5	0.3	0.02

....3

by the voltage drop across a standard 0.05 ohm, 10W, DALE resistor. Cambridge Vernier Potentiometer has been used to measure voltage difference. The thickness of the oxide coating was measured using standard metallographic technique.

After attaining steady state, the voltage drop across both the coated and uncoated wire as well as the current passing through them was measured. The procedure was repeated for different values of current and a plot of resistance versus power for each of the wires, gave a straight line (Fig.2).

Resistance of the wires at 273 K and their temperature coefficient of resistance around the temperature of the experiment has been determined experimentally. The oxide coating was characterised using X-ray powder technique and showed the presence of oxides of all the major constituents.

ANALYTICAL EXPRESSION

Under the conditions of the experiment, the thermal conductivity is given by [4,3]

$$\lambda_c = \frac{2.303A \log (r_o/r_f)}{S-(1/r_o)\ B\ (\alpha/\alpha')} \qquad (1)$$

where $A = \frac{R_o \alpha}{2\pi L}$, $B = \frac{AS' r_f'}{A'}$,

R_o is the resistance of the oxidised wire at 273K, α is the temperature coefficient of resistance of its filament around the temperature of the experiment, r_o is its radius, r_f is the radius of its filament, L is the length of the wire between the voltage leads and S is the slope of the resistance versus power plot for the wire. Quantities with primes refer to the platinum wire used herein.

RESULTS AND DISCUSSION

Reproducibility of the technique has been established using different sets of wires. The slope of the straight line in the plot of 'Resistance vs Power' has been determined using least square fit.

Using relation (1) and the measured parameters, thermal conductivity of the oxide coating has been evaluated to be 0.17 $Wm^{-1}K^{-1}$ for a coating thickness of $\sim 9 \times 10^{-6}$ m at 308 K. No other published data is available to make the comparison. However, this value is appreciably different from that of the bulk monel-400 value of 26 $Wm^{-1}K^{-1}$ and is thus expected to affect the heat transfer. It is important to bear in mind that only the effective thermal conductivity has been measured and it is expected to depend not only on the respective amount of the oxides present but also on the porosity of the coating.

The accuracy of the method is expected to be nearly six percent. The errors involved in the determination of this value may be due to:

1. the determination of exact length of the coated portion of the wire between the leads; (a practical difficulty arises in spotwelding the lead at exact separation junction of the coated and uncoated portion);

2. the non-uniformity of coating thickness and non-circularity of wire, (the error due to this source is minimised by taking several measurements at different portions along the length of the wire);

3. the assumption of convection heat transfer coefficient being the same for both the coated and uncoated wire, (this is justifiable only if the surface finish of both the wires is approximately the same);

4. radial temperature gradient in the wire, and

5. interface resistance, (the contribution due to this factor will be negligible if the coating on the wire is firmly adherent).

CONCLUSIONS

It may thus be concluded that this method for measurement of thermal conductivity of thermally insulating metal oxide coatings is direct and reliable. It has advantages over the transient methods in its simplicity and cost, however it has limitations. It is not easily applicable for very thin (thickness $\leq 1\,\mu$), highly porous coatings and of materials having relatively large thermal conductivity.

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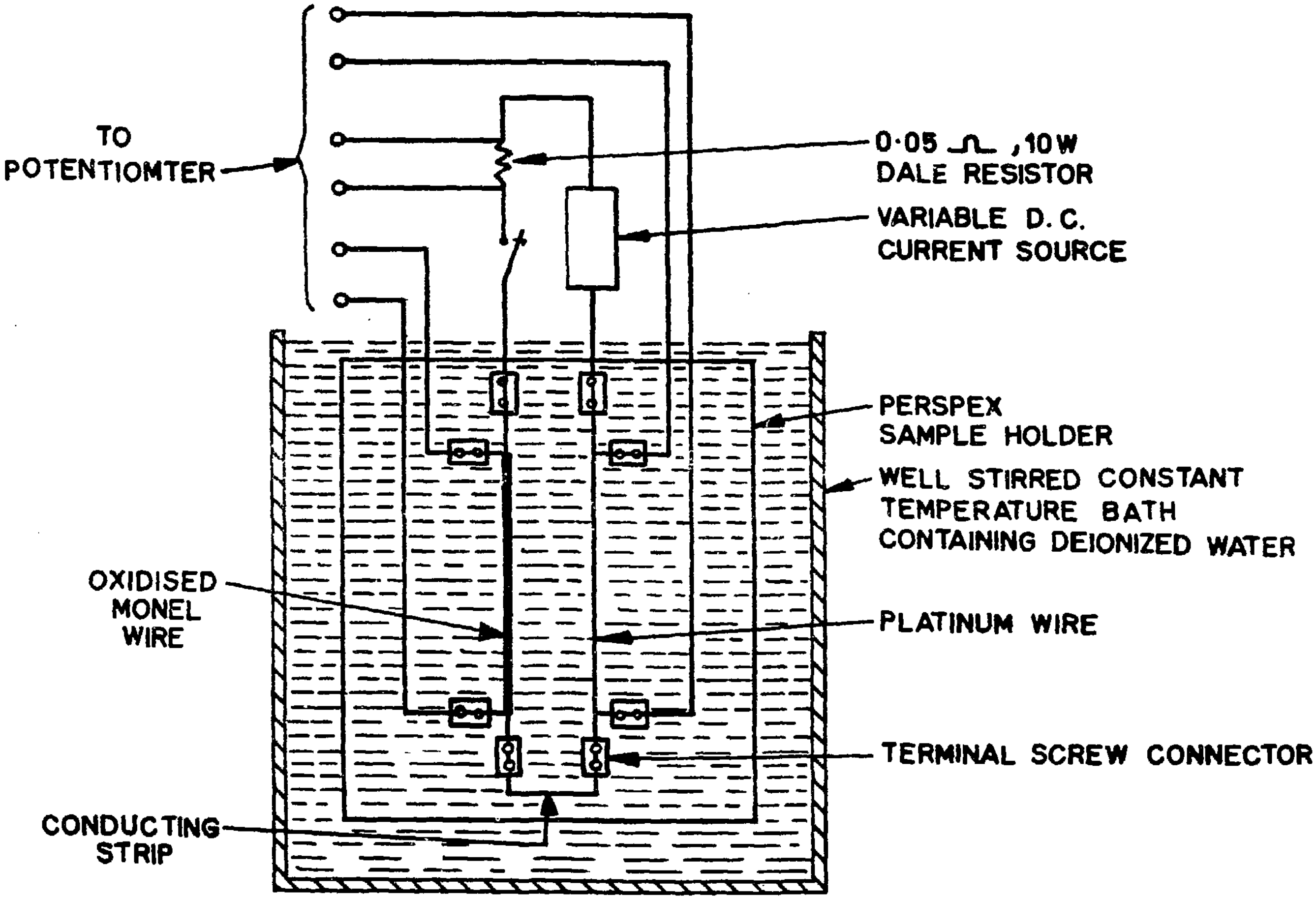

Fig. 1: Schematic diagram of the apparatus for determination of the thermal conductivity of electrically insulating coatin

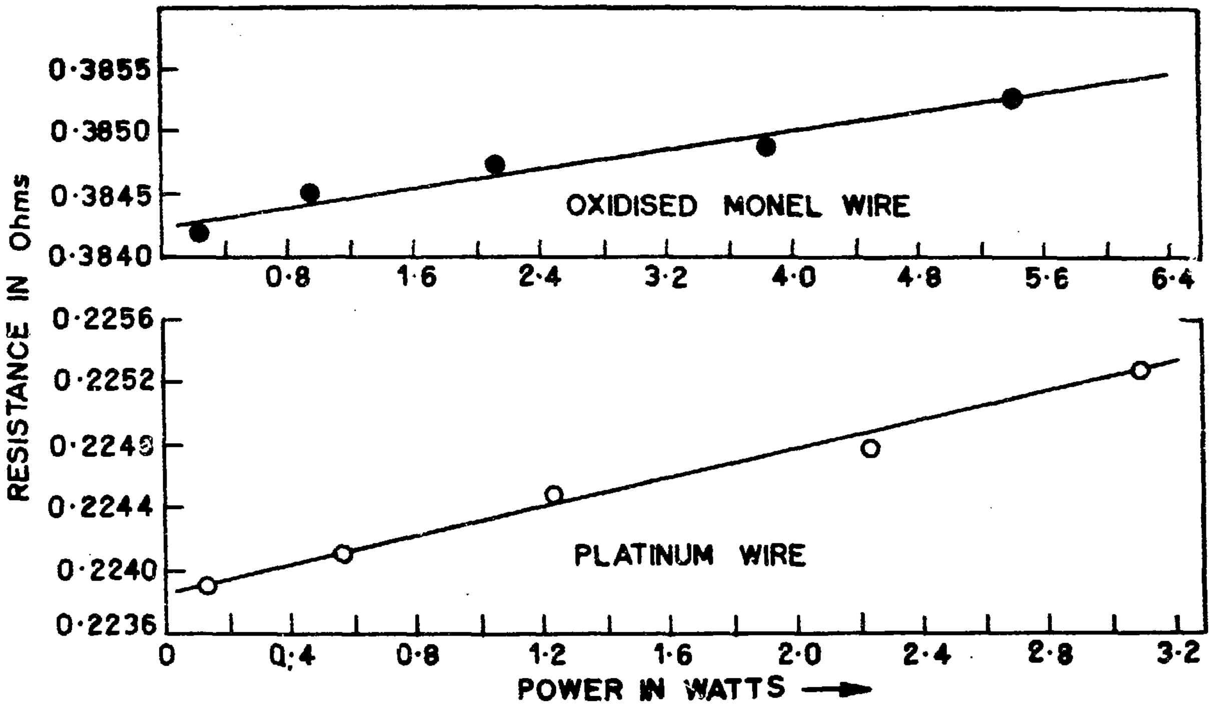

Fig. 2: Resistance of oxidised Monel - 400 wire and platinum wire vs. power.