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DESIGN, FABRICATION AND SENSITIVITY ANALYSIS OF THE RESISTANCE TEMPERATURE DETECTOR THIN FILM SENSORS

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Abstract - Thin film heat transfer sensors are most cost effective resistance temperature detector (RTD) sensors for dynamic temperature measurements mainly because of very fast response time (milliseconds or less). These sensors are prepared by deposited high conducting very sensitive gauge material (platinum/nickel/silver) on the insulating surface (pyrex/macor/quartz). The purpose of this work is to fabricate different types of thin film sensors by using high conducting platinum and nanomaterials. After fabrication all these sensors are statically calibrated by oil bath type methods and the typical value of sensitivity for each sensor are calculated and then compared the results between them.

Keywords - *Thin film sensors, oil bath calibration technique, sensitivity analysis.*

I. INTRODUCTION

The measurement of heat transfer rates to the aerodynamic surfaces and high-speed flow environments are very important in the design of hypervelocity aerodynamic vehicles. When flows are generated in short duration impulse facilities, the total time of flow is restricted to only a few milliseconds or less. In such cases the technique used for surface heat fluxes or transient temperature measurement must be suited for transient conditions and should have a response time fast enough to trace variations caused by rapidly changing flow conditions. The RTD sensors are used to measure the transient surface temperatures by mounting the sensors embedded inside the heated material surface. The surface heat fluxes are then estimated from the transient temperature history and analytically one dimensional heat transfers modeling for semi-infinite body. Thin film sensors are RTD and suitable for measuring surface temperatures history in very short duration tests has been well established by [1]. These sensors have very high response time (~1μs) and can be fabricated in house [2]. Normally, these sensors have a highly sensing gauge material (Platinum/Nickel/Quartz) mounted on insulating substrate (Pyrex/Macor/Quartz). Platinum is in the form of paste can be used to fabricate a thin film on the substrate material and has extremely sensitive resistance that varies linearly with temperature [3] and radiation based calibration technique indicate that these sensors are suitable for accurate heat fluxes measurements [4].

Most of the literatures do highlight the importance of thin film sensors for prediction of transient heat flux measurement. However, when it is desired to have measurement of small order of magnitude of heat flux (~1 W/m^2), the accuracy becomes an issue. It is mainly because of the limitations in thermal properties of gauge material and its sensitivity. With recent advances in nano-technology, it is possible to enhance the thermal properties by mixing nano-materials into the gauge material. The nano-materials have shown the improvement in thermal conductivity and heat transfer considerably. Dispersion or suspension of nano materials of high thermal conductivities into base fluids gives rise to higher thermal conductivity of the mixtures [5], thereby increasing the heat transfer coefficient. Carbon nano tubes (CNTs) and Graphene nano-materials are a new form of materials and are found to be most effective nano materials because of their unique thermal conductivity (5000W/m-K), chemical stability, excellent electrical conductivity, high surface area and strong mechanical strength associated with high aspect ratio [6-13], because of their very high thermal conductivity and very large aspect ratio, nano-materials are expected to be preparing thermal property enhancement. CNT based electrodes are prepared by mechanical abrasion onto graphite surface as paste [14] and in the form of composite [15]. The electrodes modified with mixture of (platinum chemicals and CNT) and (platinum chemicals and graphenes) have recently received much interest for the purpose of designing sensors [16, 17] and [18, 19]. However, severe aggregation always takes place in the as prepared CNTs because of the non-reactive surfaces, intrinsic Vander Waal forces, and very large specific surface areas and aspect ratios [20]. Most of the graphene studies have focused on its physical properties such as its electronic properties and these studies have demonstrated some applications in gas sensors [21, 22]. Such properties indicate that graphene may be a good support for electro catalysts compared to other nano-materials. Another advantage of graphene is its potential low manufacturing cost as compared to other nano structured carbon materials such as CNT. Noble nanoparticles such as platinum exhibit electro catalytic behavior to hydrogen peroxide and have been widely used for sensing applications [23, 24]. It will be attractive to prepare nano particle functionalized means platinum nano particles nanocomposite because such a functionalized may generate synergy on electro catalytic activity and thus enhance the sensitivity of the biosensor.

The platinum based thin film RTD sensors are well established in engineering and scientific application but no open literature has been observed in view of fabrication of platinum thin film temperature sensors with high conducting nano-materials (CNT and Graphene). So, an attempt has been made here to fabricate platinum and nano-materials based sensors. Therefore, the purpose of this work is to prepare the mixture of platinum and nano-materials (CNT and Graphene) in a mass fraction of 35% of nano-materials and fabricate the nano-materials based sensors in the laboratory. Three types of thin film sensors are fabricated by depositing the platinum paste, (platinum/CNT) and (platinum/Graphene) over a substrate material (pyrex). After fabrication these thin film sensors are statically calibrated by oil bath type method. In the oil bath based experiment the typical value of temperature coefficient of resistance (TCR) and sensitivity are calculated and it is found that these handmade nanomaterials based sensors are also very sensitive.

II. DESIGN AND FABRICATION OF THIN FILM SENSORS

2.1 Preparation of platinum and nanomaterials mixtures

Platinum chemical binders are commonly used in the thin film sensors because of their very high reactivity and chemical inertness. To fully utilize the catalytic activity of platinum, it is critical to anchor the platinum strongly on the supporting material and to prepare platinum based working sensors with a large surface area on which electro chemical reactions can take place. Nanomaterials such as black carbon, carbon nano fibers, CNTs and Graphenes are widely used as a supporting matrix. Recently, CNTs and Graphenes have attracted much interest as the metal catalyst support for electro catalytic and sensing applications due to their unique electrical properties, high surface to volume ratio and low cost. Considering these aspects platinum, CNT and Graphene form a nano composite and becomes an ideal material for thin film sensors. The CNTs and Graphenes are oxidative activated so as to generate functional groups such as COOH, OH and CO for better adhesion of the platinum to the CNT and Graphene surface [5]. The platinum chemical and nanomaterials are as shown in Fig. 1. The platinum chemicals mixed with nanomaterials (CNT and Graphene) in a beaker with a mass fraction of 35% nanomaterials. Platinum/CNT and Platinum/Graphene based mixtures are formed separately and these solutions are then continuously stirred for 45mins in the instrument called, tip-sonicator. Tip sonicator are used to convert ultrasonic power supply into high frequency mechanical vibrations. The ultrasonic vibrations are intensified by the probe and focused at the tip where the atomization takes place. Probes mixed the mixture uniformaly in a beaker with vibrating action. The liquid travels through the probe, and spreads out as a thin film on the atomizing surface. During stirring process the temperature of the mixtures also increases, thus it is further cooled to ambient temperature, naturally. Once the reaction was complete, the Pt/CNT and Pt/Graphene products were dispersed through ultrasonic agitation for one hour.

2.2. Determination of thermal property of the mixtures

The thermal conductivity enhancement ratio has been defined as the ratio of effective thermal conductivity of the mixtures $\left(K_{eff}\right)$ to the thermal conductivity of the base material (K_1). There exist some empirical correlations to calculate effective thermal conductivity of two-phase mixture based on thermal conductivity model for a two-phase mixture consisting of a continuous and discontinuous phase [25]. The effective thermal conductivity of the mixtures is given by,

$$K_{eff} = \frac{2K_2 + K_1 + \beta(K_2 - K_1)}{2K_2 + K_1 - 2\beta(K_2 - K_1)} K_1 \qquad (1)$$

In a similar manner, the effective thermal diffusivity $\left(\alpha_{eff}\right)$ is given by,

$$\alpha_{eff} = \frac{K_{eff}}{(\rho c)_{eff}} = \frac{K_{eff}}{(1-\beta)(\rho c)_1 + \beta(\rho c)_2} \qquad (2)$$

Where, K, ρ and c are thermal conductivity, density and specific heat and suffices 1 and 2 indicates platinum and nanomaterials, respectively. The effective values of the thermal properties are plotted against the mass fraction of nanomaterialsl and are shown in Fig. 2 and Fig. 3. The result shows that all of the models predict increasing thermal conductivity and diffusivity with increasing particle mass fraction of the nanomaterials in the platinum chemical. The results also show that platinum/CNT and platinum/Graphene suspensions have noticeably higher thermal conductivities and diffusivity compared to that of platinum only. With nanomaterials (CNT and Graphene) composition of 35%, it is noticed that thermal conductivity is enhanced by 78% while thermal diffusivity is found to increase by 102%. However, with further increase in nanomaterials composition, the surface adhesive property of the mixture decreases. Thus, the film formation on the pyrex substrate could not be achieved due to improper surface contact.

2.3. Thin film sensor fabrication from the mixtures

Thin film sensors consist of sensing gauge element mounted on insulating substrate. In the present work, high purity platinum paste, platinum/CNT and platinum/Graphene mixtures are used for film deposition on highly polished pyrex substrate (6mm diameter and 10mm thickness). Platinum, Platinum/CNT and platinum/Graphene mixtures are applied on such adequately polished substrate surface in mass fraction $\left(\beta = 35\%\right)$ of nanomaterials (CNT and Graphene). These mixtures are necessarily the liquid containing fine platinum particles in suspension of chemical agents which attack the surface of the substrate material to provide highly adherent film. Evaporation of these chemical binders has been ensured by drying the film at around 200°C in the temperature controlled oven. These platinum chemical, platinum/CNT and platinum/Graphene based films are then naturally cooled to the atmospheric room temperature. Since this film does not make appropriate contact with connecting wires, silver paste is applied on either sides of the sensor to achieve necessary electrical connections. After applying these films, they are dried by gradual heating till 200°C in the oven and then cooled naturally to room temperature. Three types of thin film sensors are fabricated (Platinum, Platinum/CNT and Platinum/Graphene based) as shown in Fig. 4.

2.4. Determination of TCR and Sensitivity

Thin film sensors are passive device therefore before experiment it is energized by constant current power source by flow constant current through the sensors (7mA-12mA). The resistance of a metallic thin film sensor is very sensitive to temperature and increases with the temperature during the flow over it. This results in the change in the voltage of the circuitry. For a 'ΔT' change in the gauge temperature, change in voltage across the gauge 'ΔV' is given by the following expression:

$$\alpha_0 = \frac{\Delta V}{V_o(\Delta T)} \tag{3}$$

Where, α_0 is TCR of the sensor and it can be measured by using oil bath technique and V_0 is the initial voltage.

Sensitivity analysis is the study of how the variation in the output of a statistical model can be attributed to different variations in the inputs of the model or in another way; it is a technique for systematically changing variables in a model to determine the effects of such changes. Sensitivity analysis investigates the robustness of a study when the study includes some form of statistical modeling. Sensitivity analysis can be useful to sensor modelers for a range of purposes including support decision making or the development of recommendations for decision makers. Sometimes a sensitivity analysis may reveal surprising insights about the subject of interest. For instance, the field of multi-criteria decision making studies the problem of how to select the best alternative among a number of competing alternatives. This is an important task in decision making. In such a setting each alternative is described in terms of a set of evaluative criteria. These criteria are associated with weights of importance. Intuitively, one may think that the larger the weight for a criterion is, the more critical that criterion should be. However, this may not be the case. It is important to distinguish here the notion of criticality with that of importance. By critical, we mean that a criterion with small change in its weight may cause a significant change of the final solution. It is possible criteria with rather small weights of importance to be much more critical in a given situation than ones with larger weights. That is a sensitivity analysis may shed light into issues not anticipated at the beginning of a study. This in turn may dramatically improve the effectiveness of the initial study and assist in the successful implementation of the final solution.

The sensitivity of a material is represented as $\left(S\right)$, depends on the material's temperature and crystal structure. Typically metals have small sensitivity because most have half-filled bands. Electrons (negative charges) and holes (positive charges) both contribute to the induced thermoelectric voltage thus canceling each other's contribution to that voltage and making it small. In contrast, semiconductors can be doped with an excess amount of electrons or holes and thus can have large positive or negative values of the thermo power depending on the charge of the excess carriers. The sign of the sensitivity can determine which charged carriers dominate the electric transport in both metals and semiconductors. If ΔT is the temperature difference and ΔV is thermoelectric voltage between the two ends of a material, then the sensitivity of a material is defined as,

$$S = \frac{\Delta V}{\Delta T} \tag{4}$$

2.5 Oil bath based calibration technique

Oil bath type experiment is used to calculate TCR and sensitivity of each thin film sensors. The TCR of a thin film sensor essentially determines its sensitivity with respect to temperature change. The useful linear variation of voltage and temperature mainly depends on factors such as, uniformity of the metallic layer, purity of the film material, size of the deposits and bonding with the substrate material. The oil bath arrangement provides gradual step variation in the temperature can be fed to the sensors and the corresponding changes in voltage can be monitored from any type of output signal measuring instruments as shown in Fig. 5. Here, a beaker containing all sensors is kept inside an oil-bath, which is heated by a constant heating source. Thus, all these sensors experience convective heating though the hot air inside the beaker. A thermometer mounted in the beaker along with thin film sensors is also used to monitor the temperature of the hot air. Since, the sensors are passive device, so they are energized by a constant current source (~10mA). The same instrument also used to record the change in output voltage signals across the sensors for corresponding change in temperatures. In the present experiments, the change in voltage readings are taken when temperature of air is raised from 45°C to 85°C at the interval of 10°C. Voltage variation from its initial value with change in temperature is the outcome of this experiment. Similar readings are taken during natural cooling process. For both heating and cooling process, these variations are shown in Fig. 6, Fig. 7 and Fig. 8. During heating and cooling process the values of output voltage signals are not exactly same due to some resistance of extension wires but nearly much closed. The value of TCR and sensitivity are calculated for each sensors and it is found that the value of TCR for platinum/CNT (Pl/CNT) and platinum/Graphene (Pl/Gra) mixtures based sensor has to be enhanced (from its base value) 42% and 46% when for compositions corresponding to 35%, and the variation in the sensitivity as shown in the Fig. 9 and its value is found to be enhanced by 34% and 37% respectively.

III. CONCLUSIONS

This work present development in the thin film heat transfer sensors using high conducting nano materials. Thin film sensors based on platinum, platinum/CNT and platinum/Graphene particles was successfully design and fabricated in a 35% mass fraction of the nanomaterials. The fabrication method indicates these sensors are rigid in construction, uncomplicated manufacturing method and reusable. In case a sensor is damaged, it only has to be equipped with a new film. The entangled nano porous platinum and nanomaterials structures could provide a suitable heat transfer rate and a large three phase boundary area. Such properties are critical when preparing good sensors with the high catalytic reactivity of platinum. The thermal conductivity and diffusivity of the mixtures are calculated from empirical equations and the result show that an increase in mass fraction of the nano particles in the mixtures then thermal property of the mixtures also increases. The oil bath techniques are used to get a good linear response between temperature and voltage signals. TCR and sensitivity also increases when fraction of nanomaterials mixed in the platinum based material. Furthermore, this thin film sensors exhibited better sensitivity and selectivity compared to those of binary supported materials. The temperature sensitivity of the manufactured sensors is checked regularly then it is found that for research purposes platinum/nanomaterials based thin film sensors have been used to measure surface heat fluxes. Therefore, the well dispersed and very small amount of platinum supported nano particles provided the ability to fabricate a high sensitive thin film sensors, this is important for mass production processes due to the high price of platinum chemicals. These results suggest that nano structure controlled novel platinum, platinum/CNT and platinum/graphene sensors allow for the fabrication of a high performance with minimal expensive platinum sensors.

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FIGURES

(a.) (b.)

Fig. 1 : High conducting (a.) Platinum (b.) Nanomaterial

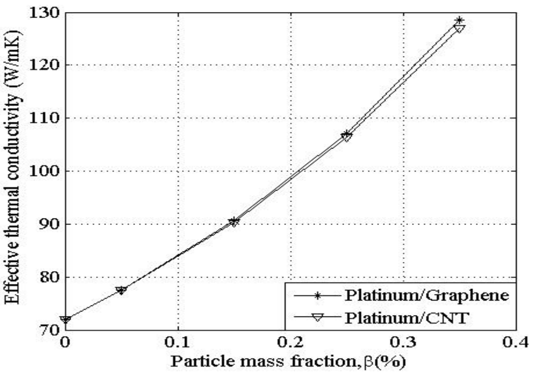

Fig. 2 : Variation of effective thermal conductivity with different mass fraction of nanomaterials with platinum.

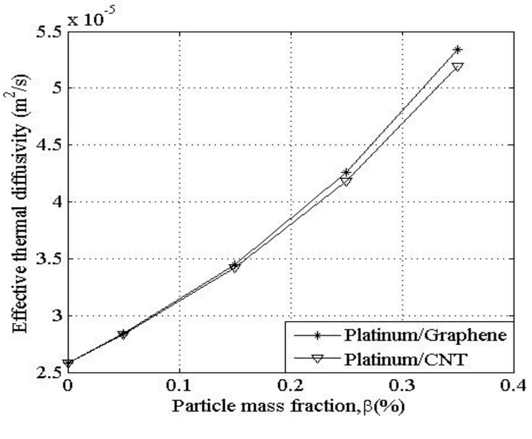

Fig. 3 : Variation of effective thermal diffusivity with different mass fraction of nanomaterials with platinum.

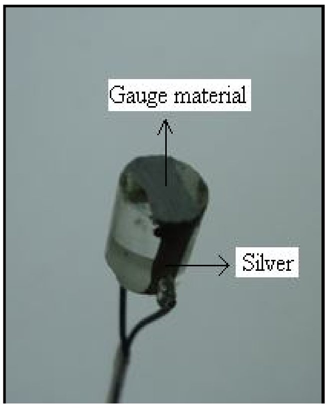

Fig. 4 : Platinum/nanomaterials based thin film sensor.

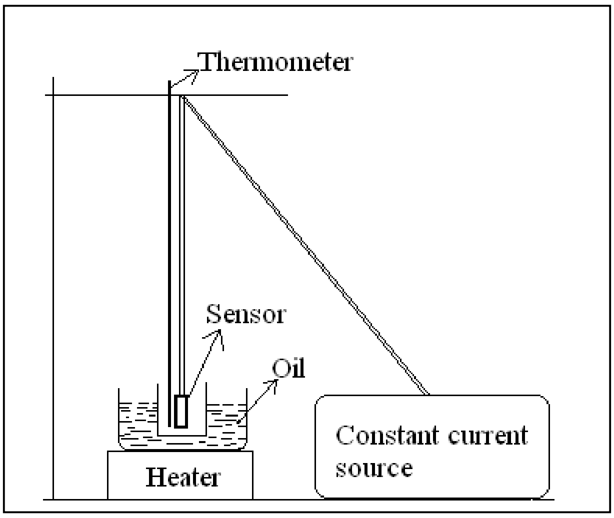

Fig. 5 : Schematic diagram of oil bath type calibration.

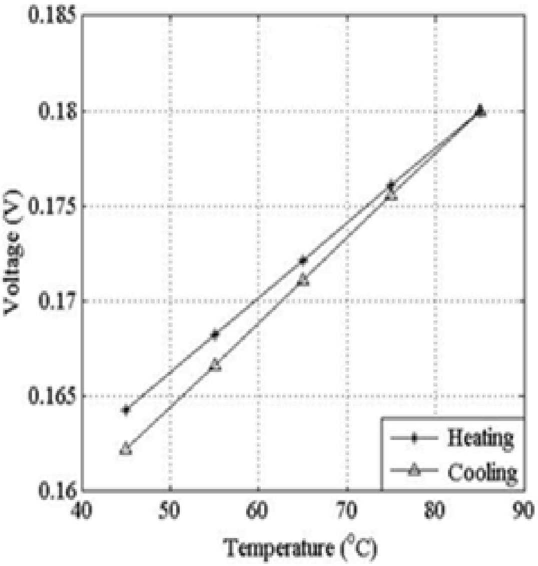

Fig. 6 : Variation of voltage with temperature for platinum based sensor.

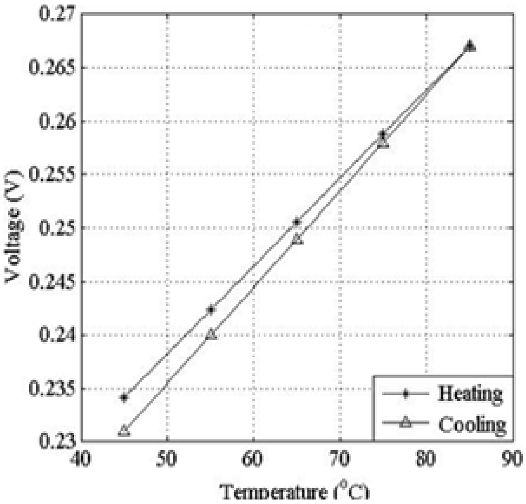

Fig. 7 : Variation of voltage with temperature for platinum/CNT based sensor.

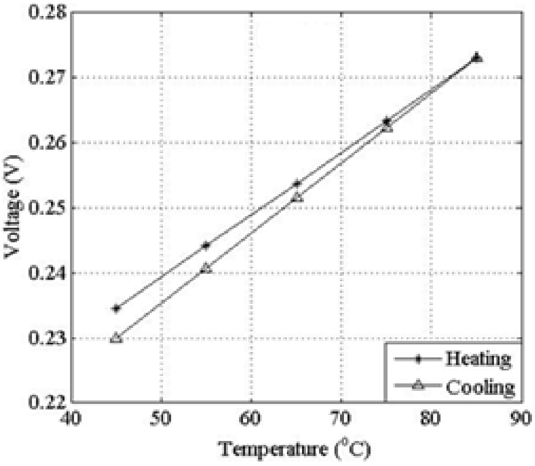

Fig. 8 : Variation of voltage with temperature for platinum/graphene based sensor.

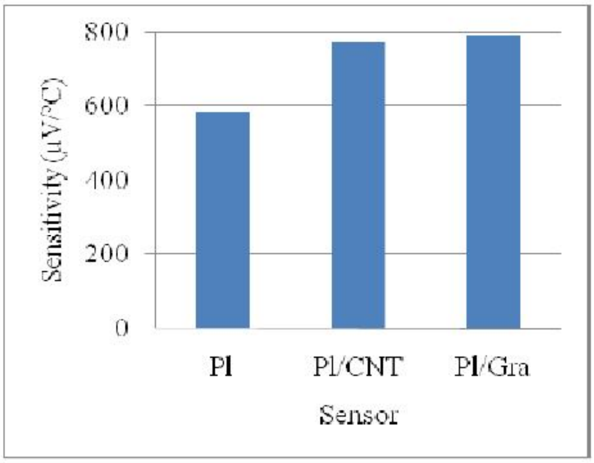

Fig. 9 : Comparison between sensitivity for different types of thin film sensors