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A DIAMOND THIN FILM FLOW SENSOR

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Abstract

We present the results of theoretical modeling and experimental testing of a diamond thin film sensor for flow studies. It is shown that the high thermal conductivity of a diamond film can enhance the frequency response of the flow sensor. One-dimensional heat diffusion equation was solved using the finite difference method for determining the frequency response. Two different sensor structures were analyzed: a Ni film on a quartz substrate (Ni/Q) and an intermediate layer of diamond film between the Ni film and quartz substrate (Ni/D/Q). The theoretical model predicts a frequency response for the Ni/D/Q sensor higher than that of the Ni/Q sensor. Diamond films for the Ni/D/Q sensor were deposited onto the quartz substrate by microwave plasma-enhanced chemical vapor deposition (MPECVD). The conditions for a high nucleation density were established for obtaining a continuous diamond thin film. A subsequent nickel film patterned and deposited serves as the sensing arm in the bridge circuit of an anemometer. The measured frequency response of the Ni/D/Q sensor combination is greater than 220 kHz, as compared to the Ni/Quartz sensor response of 120 kHz.

1. Introduction

Diamond thin films possess a unique combination of properties among solid state materials including high thermal conductivity, chemical inertness, high resistivity and large breakdown field. Applications utilizing these properties have recently been reported in hydrogen sensing [1], temperature sensing [2], and flow sensing [3]. High velocity, high temperature flow sensing capabilities are critical for detecting transitions in the hypersonic and supersonic regime. A hot-film anemometer consisting of a conducting thin film deposited on an insulating substrate detects transition regions in fluid flow. The high frequency components, a function of the Mach and Reynold's number, need to be determined for analyzing the flow characteristics. Conventional metal film sensors are limited by the properties of the thin film to measuring instabilities around 60 kHz. Researchers at NASA Langley have developed thin metal film sensors of Cr and Ni for boundary layer transitions at both ambient and cryogenic temperatures [4]. However, a need arises for identifying appropriate materials for measuring high speed flow transitions in the 200 - 400 kHz range at temperatures above 500 ^{o}C.

Frequency response analyses of thin film sensors were carried out by Vidal [5] and Lowell and Patton [6]. Decoupling the thermal effects of the film from the substrate allows the film temperature to change faster than the substrate temperature. This corresponds to maintaining a large ratio of film thermal conductivity to substrate thermal conductivity (k_f / k_s). The maximum frequency response is related to ($k_f/\rho_f c_f d_f^2$ or α_f/d_f^2) where ρ_f, c_f, α_f, and d_f are the density, heat capacity, diffusivity and thickness of the film. Therefore, achieving a high frequency response requires the combination of a thin film material with a high k_f and a substrate material with a low k_s. Table 1 lists the thermo-physical properties of the chosen thin film and substrate materials which meet the above discussed criteria. Diamond is chosen as the film material due to the following properties: highest value of thermal conductivity, large negative temperature coefficient of resistance and chemical inertness at high temperatures. Also, doping diamond has yielded resistivity values in the range 0.1-100 Ω-cm [7]. Quartz has a low value of thermal conductivity among other possible substrate materials as Si and Al_2O_3. As mentioned earlier, a low value of k_s is important for high frequency analysis. Therefore, the (D/Q) combination, which has a very high k_f/k_s ratio, should exhibit a high frequency response.

Frequency response simulations were carried out to predict the sensitivity of Ni and diamond films deposited on quartz substrates. The results of such simulations should indicate the effect the interlayer of diamond has on the frequency response. The finite difference method was used to solve the one-dimensional heat diffusion equation for determining the frequency response of the sensor. The details of the

Table I. Thermo-physical properties of materials at 300 K

Material Parameter	Ni	Diamond	Quartz
ρ (kg/m^3)	8900	3500	2650
k (W/m^{o}K)	90.7	2300	10.4
c (J/kg^{o}K)	444	509	745

analysis are explained elsewhere[8]. Fig.1 illustrates a schematic of the model used for analyzing the frequency response. The frequency response values were determined using a sinusoidal forcing function. The result is shown in Fig.2. The 3dB response of the normalized temperature amplitude variation for the Ni/D/Q sensor is around $2X10^5$ Hz. The response of the Ni/Q sensor is about an order of magnitude lower. Hence, the diamond film interlayer will enhance the frequency response of the proposed flow sensor.

2. Sensor Fabrication

Two different types of sensors were fabricated for frequency responses measurement. Fabrication of the Ni/Q sensor was carried out by patterning and depositing nickel films on fused quartz substrates by electron-beam evaporation to a thickness of 0.5 μm. For the Ni/D/Q sensor, diamond films were grown on quartz substrates using a microwave plasma-enhanced chemical vapor deposition system. The deposition chamber consist of a 6" vertical cavity with a 4" diameter graphite susceptor connected to an induction heater. The temperature of the susceptor is monitored by a thermocouple inserted at the base of the susceptor. For uniform thin film growth, a considerable nucleation density is necessary. Diamond nucleation and growth processes on quartz substrates have been investigated by several researchers[9-12]. Previous investigations of diamond nucleation on various forms of silica by MPECVD has produced different results. Lauten et. al. demonstrated the formation of diamond on SiO_2 without substrate abrasion prior to deposition [9]. Instead, it is shown that a gas phase seeding process consisting of increasing the CH_4 concentration, decreasing substrate temperature and lowering microwave power is sufficient for increasing the nucleation density. For this present work, we define nucleation enhancement of diamond films on quartz substrates as a two-step mechanism. The initial step is abrasion with 1μm diamond paste prepared with a Minimet abrasion apparatus. This prescratching procedure causes an immediate means of nucleation by seeding stable fragments on which diamond growth can occur. The second step, similar to Lauten's proposal, is called carbon saturation of the substrate. Carbon species impinge on the substrate from the gas phase and diffuse into the bulk of the substrate. When the substrate becomes incapable of being penetrated by diffusion, the surface carbon concentration increases and eventually stabilization of the nucleus is formed. Creation of a large density of these stable nuclei is imperative for assuring uniform thin film growth. The abraded samples were placed upon the graphite susceptor during deposition. The chamber is first evacuated to a desired pressure with a turbomolecular pump. Table II lists the deposition parameters used for nucleation and growth of the diamond thin films. The deposition parameters given under the nucleation step are initialized. Maintaining a 14% CH_4 in H_2 gas mixture during a two hour seeding time was found to be sufficient for enhancing the nucleation density. After gas phase seeding, modifications of the deposition parameters were necessary for uniform thin film growth. A 3% CO in H_2 was used during the growth step. This process promoted the formation of OH radicals within the gas phase species. The combination of OH radicals and atomic H should effectively promote diamond growth by preferentially etching non-diamond carbon faster than diamond.

Table II. Nucleation and Growth Process Parameters

Parameters	Nucleation Step	Growth Step
Hydrogen Flow (sccm)	430	900
Methane Flow (sccm)	70	9
CO Flow (sccm)	0	27
Substrate Temperature (^{o}C)	600	750
Total Pressure (Torr)	20	35
Microwave Power (W)	723	1000
Time (hours)	2	6

Diamond film growth was confirmed by Raman spectroscopy and SEM. The SEM pictures shown in Fig. 3 reveal a continuous thin film, and verify a high nucleation density achieved using the two step process. A nickel film was subsequently deposited upon the diamond film. Due to the surface roughness of the diamond films, a nickel film thickness of 1.6 μm was necessary. After depositing the nickel films, both the Ni/Q and Ni/D/Q sensors were connected to a testboard.

3. Frequency Response Measurement

The frequency response of each sensor was determined using a Dantec Constant Temperature Anemometer (CTA) system. The main unit of the CTA model consists of a servo amplifier, filter decade resistance, square wave generator, and other auxiliary components. An associated CTA Standard Bridge and 5 meter Cable Compensation Unit are connected to the main unit of the CTA. Other equipment utilized for testing the sensors include: 100 MHz Yokogawa digital oscilloscope, multimeter, and 5 meter coaxial cable. A constant temperature anemometer is one method of implementing hot wire or hot-film anemometry. Hot film anemometry operates on the principle that the electrical resistance of a metallic conductor is a function of its temperature. In a constant temperature anemometer, the sensing film is placed in a variable current feedback loop which maintains a constant temperature. Any resistance change is detected as a voltage difference between the arms of a Wheatstone bridge. This voltage difference is electronically conditioned in stages and fed back as an input to the bridge. Therefore, the varying film resistance and compensating current created will be proportional to the flow velocity. However, a technique exists for determining the frequency response of a sensor using an anemometer in the absence of a flow. The "cold resistance" of the sensor, which is the resistance value without an applied signal, is measured. It is then multiplied by a constant known as the overheat ratio, to get a resistance value termed as "hot resistance." The variable resistance of the bridge circuit is initially set to the hot resistance value. A 3 kHz square wave signal is applied to the sensor input and the output is displayed on the digital oscilloscope. Fig.4 shows the frequency response of both the Ni/Q and Ni/D/Q sensors. The measured frequency response of the Ni/D/Q sensor is better than 220 kHz, as compared to the Ni/Quartz sensor response of 120 kHz. This suggest that an intermediate layer of diamond has enhanced the frequency response of the Ni/Q sensor.

4. Conclusions

The results of theoretical modeling predicted a high frequency response for a sensor which has an interlayer of diamond film. Though the theoretical values were not achieved, the experiments clearly demonstrated that the (Ni/D/Q) sensor has a significantly higher frequency response than that of the (Ni/Q) sensor. The high thermal conductivity of a diamond film is a factor for enhancing the frequency response of the Ni/D/Q flow sensor. Such sensors will be useful for high speed flow studies.

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6. References

1. W. P. Kang, et. al., J. Electrochem. Soc., **141**, 2231, 1994.
2. M. Aslam, et. al., in 'Diamond Film Semiconductors', SPIE **2151**, 145, 1994.
3. S. Albin, et. al., in 'Diamond Film Semiconductors', SPIE **2151**, 152, 1994.
4. C. B. Johnson, et. al., Proc. ICAF'87 Record, 141, 1987.
5. R. J. Vidal, WADC Tech Note 56-315, **AD-97238**, 1956.
6. H. H. Lowell and N. Patton, NACA TN **3514**, 1955.
7. B. V. Spitsyn, Proc. MRS In. Conf. **1991**, 909, 1991.
8. S. Albin, et. al., 'Thermal Anemometry', ASME, FED-vol.**167**, 181, 1987.
9. F. S. Lauten, Y. Shigesato, and B. W. Stoner, Appl. Phys. Lett. **65**, 210, 1994.
10. D. J. Pickrell, et. al., J. Mater. Research, **6**, 1264, 1991.
11. S. Prawer, et. al., J. Appl. Phys. **69**, 6625, 1991.
12. J. Rankin, et. al., J. Mater. Research, **9**, 2164, 1994.

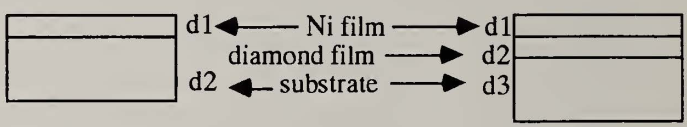

Fig.1. Two sensor structures analyzed by finite difference method.

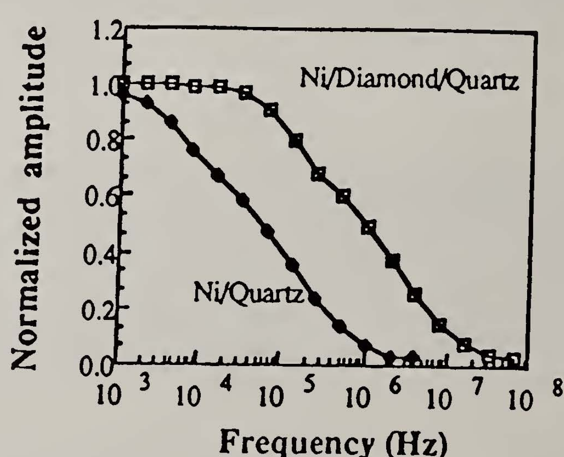

Fig. 2. Theoretical frequency response of the two sensors.

Fig. 3a. Diamond grown on an untreated quartz substrate showing a low nucleation density. Well-defined facets are clearly visible indicating the good quality of the crystals.

Fig. 3b. Continuous diamond film growth on a surface-treated quartz substrate using gas phase seeding.

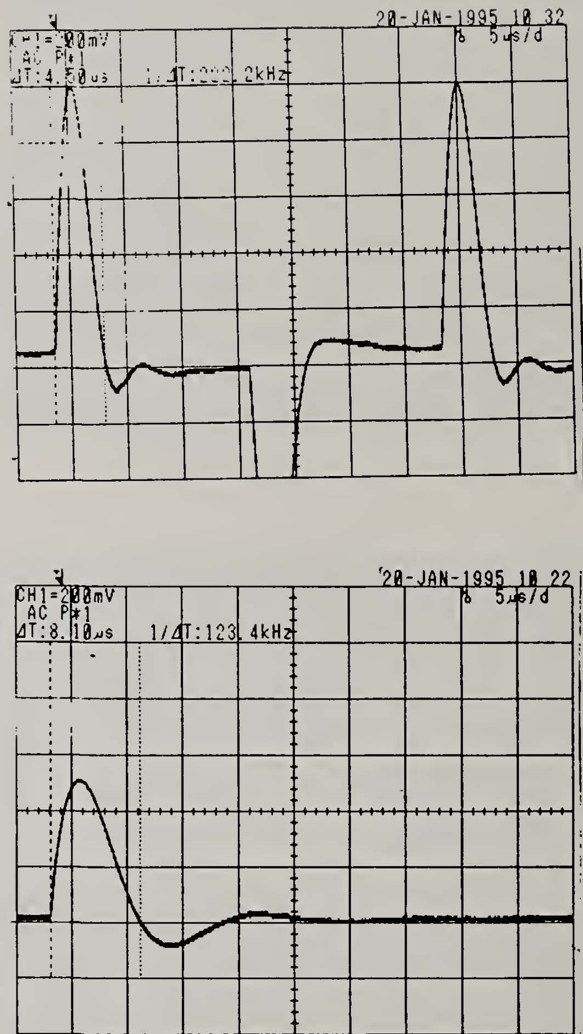

Fig.4. Frequency response of Ni/Diamond/Quartz sensor (top) and Ni/Quartz sensor (bottom), respectively. The measured frequency response of the Ni/D/Q sensor is >220 KHz, as compared to the Ni/Q sensor response of 120 KHz. This is a clear demonstration of the suitability of diamond film for flow sensing applications.