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SiC MICROSENSOR WITH PIEZORESISTIVE DIAMOND SENSING ELEMENTS

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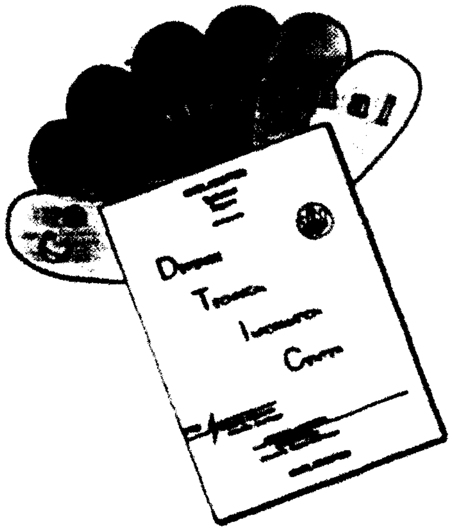

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13. ABSTRACT *(Maximum 200 words)*

Advanced microfabrication processes have been developed for novel high temperature pressure sensors utilizing diamond and SiC materials. Such sensors represent a new generation of high temperature piezoresistive sensors. The accomplishments of Phase I include the following:
a) Growth of polycrystalline diamond on β-SiC substrates.
b) The first isolated p-type diamond resistive elements grown on intrinsic diamond.
c) Establishment of microfabrication processes for sensor manufacture.
d) Demonstration of a large piezoresistive effect in poly-diamond.
e) Demonstration of a new field-assisted-bonding process which allows dielectrically isolated SiC elements to be formed.

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INTRODUCTION

The two most common measurements of operating parameters in aeronautical propulsion systems, wind tunnels, materials processing, and biomedical applications involve temperature and pressure. As these applications become more advanced, increasing need is developing to accurately measure these parameters in hostile environments and at high temperatures. For example, in the wake of the current drive to reduce energy costs, airlines are attempting to develop more fuel efficient engines while maintaining or even upgrading the safety factor. This need requires computer control of injection valves, fuel/air ratios and other operating parameters within the engines. In order to achieve this, precise monitoring of pressures in different segments of the engines during flight is required. Since efficiency in aeronautical propulsion systems is substantially improved at higher temperatures of the combustion gas, the emphasis has been to increase the operating temperature of the systems, which can be as high as 2000°F in some sections of the engines. To assure proper working conditions highly reliable, durable high temperature sensors are required. Furthermore, in order to develop such engines, test sensors are required to verify existing design codes. Wind tunnel testing and instrumentation has similar demands on the measurement of pressure and temperature.

SCIENTIFIC AND TECHNOLOGICAL BACKGROUND

Silicon-based piezoresistive sensors fulfill many of the requirements of pressure sensor applications. The Wheatstone bridge design [1] allows the simultaneous measurement of both temperature and pressure with a high degree of accuracy. Wafer thinning techniques have been developed for Si, which allow the fabrication of integral force collector-piezoresistor networks [2]. However, there are fundamental problems with using silicon as a sensor at elevated temperatures, e.g. silicon pn-junctions are not reliable above 300°C, due to junction leakage. This has motivated the use of silicon-on-insulator technology for piezoresistor fabrication [3]. However, silicon undergoes plastic deformation under minimal loads at temperatures exceeding 600°C [4], rendering it useless as a sensing element. In order to make a sensor which can be applied at temperatures above 600°C, a more temperature resistant semiconductor must be used. Several novel materials are currently being considered for high temperature sensor applications, among them diamond and SiC [5-7]. Both of these exhibit excellent thermal and mechanical properties at elevated temperatures [6-8]. Furthermore, diamond has the largest gauge factor reported thus far for any high temperature semiconductor [6,7]. In fact, the gauge factor of monocrystalline diamond exceeds that of silicon [7]. This makes it a potentially useful sensor material, especially in high temperature applications where sensitivity is critial.

In this study, a sensor was proposed which utilizes diamond sensing elements grown on a single crystal SiC diaphragm force collector. While it may be possible to form a monolithic membrane/sensing element structure from either poly-diamond or SiC, that structure would not have the mechanical benefits of a single crystal diaphragm coupled with the high strain sensitivity of diamond. Furthermore, SiC is almost three times as compliant as diamond, thus increasing the device's sensitivity for a given diaphragm thickness when SiC is used as the force collector.

During the course of the program technology was developed for diamond-on-SiC pressure sensors. All of the necessary processes for the fabrication of such sensors have been developed. This includes advances in the areas of growth and patterning of diamond resistors, metallization, cupping, and dicing processes. Electrically isolated diamond piezoresistors have been fabricated on SiC substrates. However, because of lack of time, the authors were unable to test the piezoresistive sensors which had been fabricated.

The gauge factor of poly-diamond was also investigatigated by the bending beam method. The results indicate that the gauge factor of diamond is significantly higher than that of SiC or poly-silicon.

An electrostatic bonding process was also developed by which single crystal ß-SiC can be bonded to an insulator, such as pyrex glass. This process can be utilized to package diamond/SiC sensor chips or to fabricate dielectrically isolated SiC sensing elements on a SiC force collector. SiC-on-insulator elements could be utilized in a high temperature microsensor.

SECTION 0: FABRICATION PROCESS OF THE PROPOSED SENSOR

This section describes the process design for the proposed diamond-on-SiC microsensor. Firstly, a single crystal ß-SiC epilayer is grown on <100> Si to a thickness ranging from 0.2 to 20 μm (Fig. 1a). The Si substrate backside is then oxidized to a thickness of 1.5 μm (Fig. 1b). This oxide will serve as the mask for the Si etching which produces the flexible membrane structure. The SiC surface is then prepared for diamond deposition and a 1-3 μm intrinsic diamond (i-diamond) film is grown on the surface (Fig. 1c). SiO_2 is sputtered onto the i-diamond to serve as a mask for the selective depostition/lift off of doped diamond. Windows are etched in the sputtered SiO_2 to form piezoresistor structures (Fig. 1d). Doped diamond is then grown on the sample, such that it nucleates mostly on the exposed diamond surface. The SiO_2 on the front surface is removed in HF along with any sporadic diamond grains that nucleated on it. As will be described later, it is necessary to etch the diamond surface for a short time in hydrogen plasma to prevent surface current leakage (Fig. 1e). The piezoresistors are subsequently metallized with Pt (Fig. 1f). The thermal SiO_2 on the Si substrate backside is then patterned and the Si is etched down to the SiC epilayer to form diaphragm structures (Fig. 1g). This sensor chip can be packaged for low temperature (<500°C) applications using standard Si tranducer packaging technology. The subsequent sections of the report describe each of the sensor fabrication processes individually.

SECTION 1: CVD GROWTH OF POLY-DIAMOND ON SiC SUBSTRATES

Polycrystalline diamond growth has been performed by high pressure microwave plasma-assited chemical vapor deposition on SiC substrates. In order to form an isolated diamond sensor network on the SiC substrate it was necessary to grow an intrinsic diamond layer on the SiC followed by a patterned doped layer. It may also be possible to pattern both layers. However, a major concern in the latter approach is that there might be a current leakage pathway along the sidewalls; therefore the former was taken.

The substrates used were single crystal epitaxial β-SiC layers, typically 10 μm thick, grown on (100) Si. These were supplied by J.A. Powell of NASA Lewis. In preparation for diamond growth, the SiC surfaces were mechanically abraded by polishing them with a 0.1 μm diamond grit. This procedure increases the nucleation density of the diamond on the surface and allows the formation of a more uniform deposition across the layer than that achieved on an "as grown" SiC surface.

Using established diamond deposition conditions for Si (T=850°C, Power=1.5 kW, Pressure=40 T, 500 sccm hydrogen, 5 sccm methane, and t=20 hrs) a contiguous, pinhole-free poly-diamond layer, nominally 5-10 μm thick, was grown over ≈ 80% of the SiC substrate. Figure 1.1 is the Raman spectra of the diamond-on-SiC film. This spectra indicates that the film produced is indeed poly-diamond and is almost identical to the spectra observed on diamond films grown on (100) Si in the same CVD system [6].

In order to maximize the sensor output, it is desirable to keep the diamond films (both intrinsic and doped) as thin as possible. This is because diamond has a much higher Young's modulus than SiC, and thus a thick diamond film could inhibit the flexure of the diaphragm. To accomplish this, a process was developed during the program which resulted in the deposition of diamond films with a finer grain size. These "smooth" films typically had a grain size of ≈ 0.5 μm and could be grown to a thickness of 1.5 μm without loosing their continuity. The process parameters for both the larger grain, rough films and the fine grain, smooth films are shown in Fig. 1.2. The deposition rate of the fine grain films was measured to be ≈ 0.425 μm/hr.

Many of the SiC substrates exhibited some warpage which caused the surface abrasion process to be somewhat non-uniform. This resulted in some pinholes forming at times in the diamond films. The pinhole density can be reduced by growing the diamond layer thicker, albeit with a loss of device sensitivity. Another possible solution would be to use SiC wafers with a thicker Si substrate. Thicker SiC-on-Si wafers typically are much flatter since the lattice and thermal mismatches cause less warping to occur. Prior to diamond depositions performed on SiC, the Si substrate was usually thinned from 16 mils to 8 mils. This was done in order to allow the diaphragms to be formed in the Si with Kulite masks which were designed for a 5-8 mil wafer. It should be noted that it is rather trivial to design a diaphragm mask for thicker wafer. This was not done in Phase I to avoid the cost and time involved in developing a new mask set.

Doping of the diamond films was accomplished by placing the sample substrate on top of a boron dopant wafer. During the diamond deposition, the boron outgasses from the wafer and is incorporated into the poly-diamond film. Because of the geometry, more doping occurs at the edge of the diamond wafer than in the center. There are also variations between "old" boron dopant wafers and "new" wafers. However, despite these constraints, doping can be accomplished, albeit with deviations in conductivity. It is recognized that gaseous doping sources (e.g. diborane) could be used in dedicated systems and uniformity improved.

SECTION 2: PATTERNING OF DIAMOND FILMS AND FORMATION OF PIEZORESISTOR NETWORKS

In order to achieve patterned resistor structures, a selective deposition/lift off process was developed for SiC. Sputtered SiO_2 was chosen as the sacrificial lift-off layer. Diamond tends to nucleate sporadically on SiO_2, if at all, under the deposition conditions used, leaving a film which is not contiguous and can be removed by an HF dip. The SiO_2 is deposited on the intrinsic diamond, and then the SiO_2 is patterned into a reverse-field mask of the desired structure. Subsequently, a doped diamond layer is deposited, but nucleates almost completely on the bare diamond and not on the SiO_2 layer. The SiO_2 may then be removed in HF.

Figure 2.1 are SEM micrographs of patterned doped layers grown on intrinsic diamond-on-SiC. The layers were grown using the fine grained process. At the point at which these pictures were taken, the SiO_2 sacrificial layer had not been removed. The pattern definition of the SiO_2 is very good and well defined features were achieved in the doped diamond. Furthermore, isolated piezoresistor networks have been fabricated in the diamond-on-SiC structure. However, several unexpected problems occurred during the course of the research which caused delays in achieving these results. These delays caused the authors to run out of time before the fabricated piezoresistors could be tested.

Some of the obstacles and achievements in developing patterned, isolated resistor structures are described below:

a) Durability of the SiO_2 layer: SiO_2 is etched in the hydrogen plasma which accompanies diamond growth at varying rates which depend on the properties of the SiO_2 layer. For an SiO_2 layer thickness of 1 μm, it could survive the diamond deposition for between 1 and 5 hrs. It is believed that this large variation is caused mostly by the density of the SiO_2. Future research could utilize CVD deposited SiO_2 rather than sputtered films to allow for thicker patterned features to be fabricated. Diamond deposition could be performed with 1 μm of SiO_2 for at least 1 hr with the SiO_2 remaining intact. This would result in a film ≈ 0.4 μm thick, which is acceptable in most applications.

b) Pattern Definition: Initially, it was attempted to pattern both the intrinsic and doped diamond layers on the SiC. However, because of the scratched SiC surface, caused by the abrasion process, the SiO_2 deposited on the bare SiC surface could not be delineated well in a buffer HF etch. Tracking of the scratches by the HF etch resulted in ragged feature edges in the SiO_2. Using reactive ion etching, it was possible to achieve smooth, well defined features. However, a smoother surface existed when a blanket layer of i-diamond was deposited on the SiC. As a result, patterning of the SiO_2 in HF was not a problem, as shown in Fig 2.1.

c) Leakage: Several of the patterned doped diamond films exhibited very large leakage currents between adjacent devices, rendering the piezoresistive elements useless. There was no conductivity observed across the surface of the SiO_2, thus ruling out surface leakage on the SiO_2. Initially, it was believed that the leakage was due to pinholes in the i-diamond film. To eliminate this possibility, the i-diamond films were grown thicker, to a thickness of 2-3 μms. This proceedure did not reduce the leakage. It was then speculated that the boron had penetrated the SiO_2 and left a shallow conductive surface layer on the i-diamond. To test this hypothesis, a sample with a patterned doped layer was dipped in HF to remove the SiO_2, and then probed. Electrical conduction was then observed at *all* points on the surface of the i-diamond. The substrate was subjected to thirty minutes of H_2 plasma etching, which removes diamond at a slow rate. The sample was visually inspected and the presesce of the patterned diamond layer confirmed. Probing with tungsten probes determined that electrical isolation was achieved. It is apparent that the leakage problems were probably caused by a conductive surface layer on the i-diamond. Since probing of the i-diamond before doped diamond deposition resulted in no current flow, the conductive layer most probably formed during the doped diamond deposition. It is reasonable to assume that boron penetrated the SiO_2 layer, since borosilicate glass, B_2O_3 can form within the SiO_2. Pure B_2O_3 has a melting point below 500°C, indicating that boron is probably highly volatile within the SiO_2 matrix at the temperatures of diamond deposition.

One of the best diamond-on-SiC pieces was over 1 cm x 1 cm and contained over 120 complete devices. The sample had 1 μm of patterned SiO_2 on top of the i-diamond and doped diamond deposition took place for 1 hr. The SiO_2 was removed in HF and the sample was then etched in the H_2 plasma for 3 min. Probe measurements were taken at points as indicated in fig. 2.2, and the following results were obtained at numerous sites of different devices on the piece: a) $R_{AB} \approx$ 7 to 20 Mohms, b) $R_{AC} = R_{AD} =$ **OPEN**. Open means that there was no current flow, the same reading that occurred if the probes were suspended in air. Figure 2.3 is an i-V plot of a typically representative device. Based on the approximate dimensions of the piezoresistors, the resistivity of the film is calculated to be $\approx$ 10-20 Ω-cm. To the authors' best knowledge, this plot represents the first report of isolated doped poly-diamond structures on intrinsic poly-diamond films. Although isolated diamond resistors on non-diamond insulating substrates [7,9,10], and an FET PCD device with doped on undoped diamond layer have previously been reported [11], these and other efforts [12] do not show the direct measurement of device isolation reported here.

SECTION 3: OTHER SENSOR PROCESSES

In addition to growth and patterning of devices, several other procedures were developed to facilitate diamond/SiC sensor manufacture. These include diaphragm formation, metallization and dicing. The details of each of these processes are described below:

1) Sensor Cavity Formation (Cupping): In order to form a diaphragm structure, the wafer must be selectively thinned by chemical etching. In the case of a SiC-on-Si wafer this can be done by patterning the Si by means of any of the known Si etches, such as KOH, $HF:HNO_3$, Iodine or EDP. SiC is not attacked at room temperature by any wet chemical etch and thus acts as an etch-stop. This allows diaphragms in SiC/Si to be etched very easily and precisely. Figure 3.1 shows diaphragms formed in ß-SiC/Si. The Si backside was masked with SiO_2, and the Si was etched in iodine. As this is an isotropic etch, round cavities with tapered walls were formed (fig. 3.1). The surface roughness of the Si backside (fig. 3.1) was present before any processing of the wafer took place. The bright dots visible on the back of the SiC membrane and the hillocks and orange peel which appear on the frontside are products of the SiC CVD growth. Cavities were also formed effectively using square masks and a KOH etch.

2) Metallization: Platinum was utilized as the ohmic contact to the doped poly-diamond films. These contacts, as deposited, were rectifying, but after a 40 min. anneal at 480°C, they exhibited a linear i-V dependence between I = .01 and 10 mA. The contact pads were rectangular with dimensions of 8 mils x 6 mils. This size of contact is typical for semiconductor piezoresistive sensors. Because of the large contact dimension, forming ohmic contacts should not be considered a significant problem. However, the durability of metallizations at the elevated temperatures requires further study.

3) Dicing: Microcracks usually form in epitaixial ß-SiC-on-Si during the cutting or scribing processes because of the large density of stacking faults in the material. This problem was eliminated by waxing a thin cover glass on top of the wafer before it was cut. This procedure prevented any cracks from forming in the material.

All of the processes necessary for the fabrication of a low-temperature prototype diamond-on-SiC sensor have been developed. These include the fabrication of patterned, isolated piezoresistive elements on a ß-SiC substrate, metallization of doped diamond, cupping and dicing. Once the sensor chip is fabricated, it can be packaged by epoxying, glassing or bonding the diaphragm to a standard header and placing the header into a silicon transducer housing.

SECTION 4: PIEZORESISTANCE OF POLY-DIAMOND

Preliminary measurements were conducted in parallel to the diamond-on-SiC depositions to examine the gauge factor of diamond. Doped diamond films were grown on Si at various conditions including those described in section 2. The silicon substrate was then etched away, creating free standing films which were cut into strips, attached to an alumina substrate, and metallized. These substrates were subsequently placed into a calibrated strain fixure.

Figure 4.1 shows the gauge factor plotted as a function of the resistance. The gauge factor ranges from 150 to 250 for resistances above 1 kΩ, and 10 to 20 at resistances below 100 Ω. Although the sizes of the diamond strips were not uniform, most of the variation in the resistance is due to the film resistivity. The resistivity is influenced by the growth parameters as well as the distance from the dopant wafer.

Figure 4.1 indicates that the piezoresistance of diamond is a strong function of doping level. This effect has been observed in many piezoresistive semiconductors [13,14]. At the low doping levels the gauge factor of poly-diamond is much larger than that of polysilicon (GF = 10-50) [7] or single crystal SiC (GF = 10-40) [14]. This indicates that diamond may be significantly more sensitive at the high temperatures of interest. However, in order to ascertain this conclusively, it is necessary to study the piezoresistive effect in diamond as a function of growth parameters, resistivity, and temperature.

SECTION 5: FIELD-ASSISTED-BONDING OF SiC

The proposed diamond-on-SiC sensor requires application of advanced technology that could also be used to develop monolithic β-SiC sensors. The major difficulty with β-SiC is the high defect density of the heteroepitaxial material [15] which results in large junction leakage currents. As an alternative to diamond-on-SiC sensors, an attempt was made to develop technology for dielectrically isolated β-SiC sensors which utilize β-SiC/insulator/β-SiC structures.

One process that could be used to form SiC-on-insulator elements is field-assisted-bonding (FAB) [3]. The mechanism of this process relies on placing the semiconductor into contact with an insulator which has a high density of mobile ions, such as Na^+ or K^+. The insulator is then heated up to a temperature at which it becomes somewhat conducting, typically 400-700°C. A large anodic bias (≈50-1000 V) is placed between the semiconductor and the insulator. This bias causes the positively charged mobile ions to move away from the insulator surface interface, generating a current typically ≈ 1-50 μA. The internal electric field produced in the structure forces the semiconductor and the insulator to come into intimate contact. The presence of O^- or OH^- ions at the surface, due to the migration of the positive ions, causes a surface oxidation, and hence bonding between the semiconductor and the insulator. Silicon on insulator structures fabricated by anodic bonding are very durable and provide excellent electrical isolation. Furthermore, because the bonding is of a chemical nature, the structures produced are thermal shock resistant.

As grown β-SiC epilayers were polished to a mirror-like finish using a 0.1 μm diamond grit. The polished surface was placed against an optically smooth pyrex wafer, and the two wafers were heated to 500°C. A potential of 750 V was applied for 10 min. between the SiC and the pyrex which generated a current of 10-50 μA and caused bonding to occur. After the samples were cooled down, an attempt was made to separate the two wafers manually, but it was unsuccessful. The entire epilayer surface bonded to the pyrex, with the exception of some small bubbles which were caused by particle contamination.

It should be noted that the SiC surface preparation for diamond growth is very similar to the polishing steps required for FAB. Therefore, it is possible that this process could be useful for packaging a diamond-on-SiC sensor chip. Furthermore, it could be useful to isolate SiC piezoresistors on a SiC diaphragm.

CONCLUSION

During the course of this Phase I, novel processing techniques were developed for high temperatures diamond-on-SiC pressure sensors. Isolated, doped diamond on i-diamond resistors were reported for the first time. Contiguous polycrystalline diamond multilayer films were grown on a new substrate, SiC. A novel dielectric isolation process for SiC has been demonstrated which can be utilized to fabricate high temperature microsensors based on a SiC-insulator-SiC piezoresistor/diaphragm structure. High gauge factors were found in poly-diamond which demonstrates the usefulness of the material for microsensors.

There are currently no piezoresistive devices which operate at temperatures above 500°C. The proposed diamond and SiC based microsensors have the potential to extend the range of pressure measurement by several hundred degrees. If successful, this will result in commercially available high temperature sensors for rocket engines, advanced aeronautical propulsion systems, materials processing and space systems. For diamond as an electronic material, this represents a large commercial market where its properties may exceed those of both Si and SiC. However, it is necessary to study the gauge factor and TCR (temperature coefficient of resistivity) of diamond at elevated temperatures and develop appropriate temperature compensation circuitry. More detailed characterization and process development has the potential to produce a new generation of high temperature microsensors which could fulfill the present and future needs in the field of high temperature technology.

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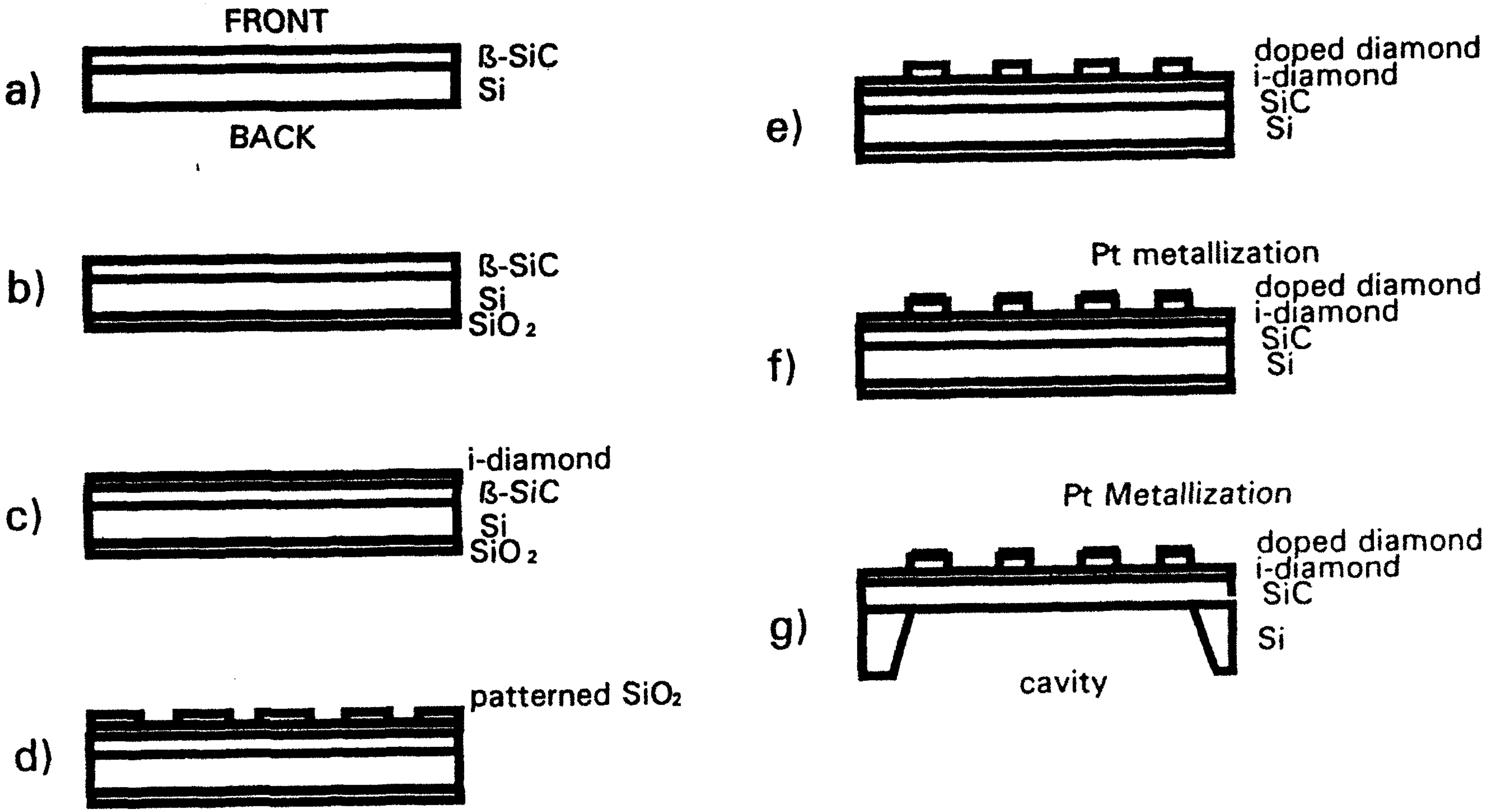
FIG 1: FABRICATION PROCESS FOR THE PROPOSED MICROSENSOR



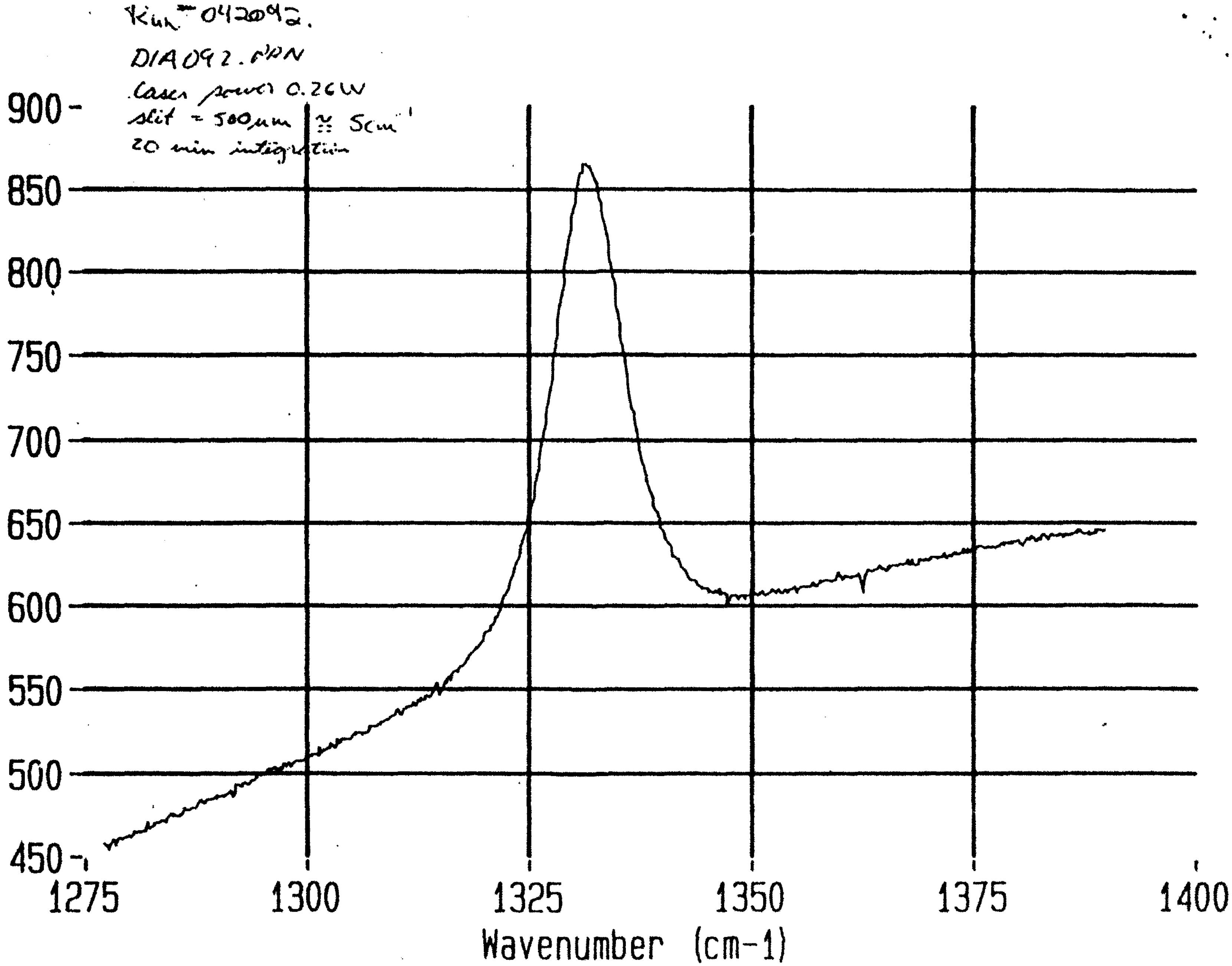


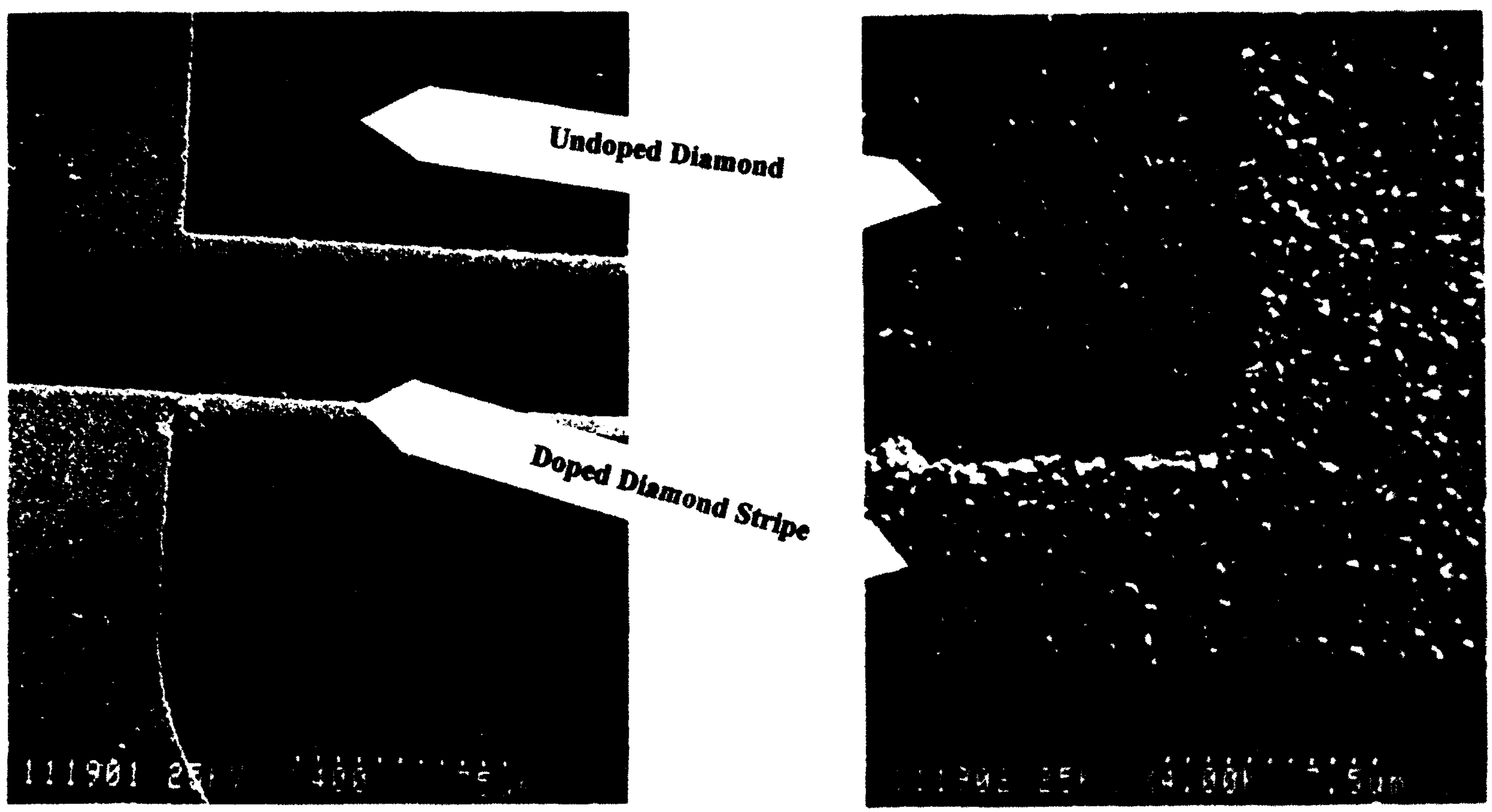
Figure 1.1: RAMAN Spectra of poly-crystalline Diamond grown on SiC

Deposition Parameters for Large Grain (rough) and Fine Grain (smooth) Poly-Diamond Films

Parameter	Rough Film	Smooth Film
Base Pressure	0.1 Torr	0.1 Torr
Substrate Temperature	850 C	860 C
Hydrogen Flow Rate	50 %	39.6 %
Methane Flow Rate	4 %	4 %
Oxygen Flow Rate	0 %	1 %
Forward Power	1500 W	644 W
Reflected Power	0 W	0 W

Fig. 1.2: Diamond Film Deposition Conditions

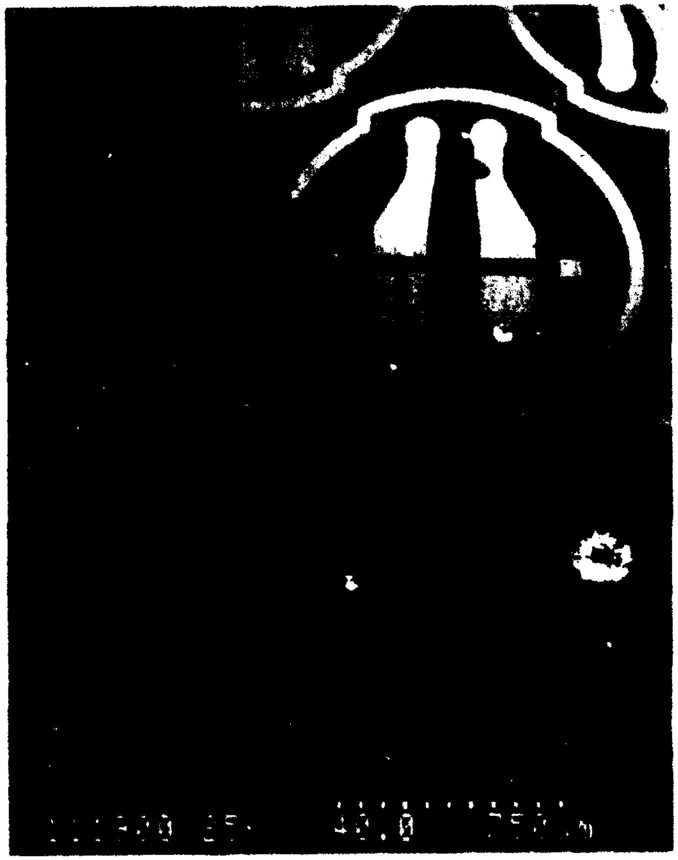
(a) (b)



Patterned doped diamond on an undoped diamond layer.

Figure 2.1 SEM of PDFPZR Microsensor Structures on SiC

(c)



(d)

Figure 2.1 :SEM of PDFPZR Microsensor Structures on SiC

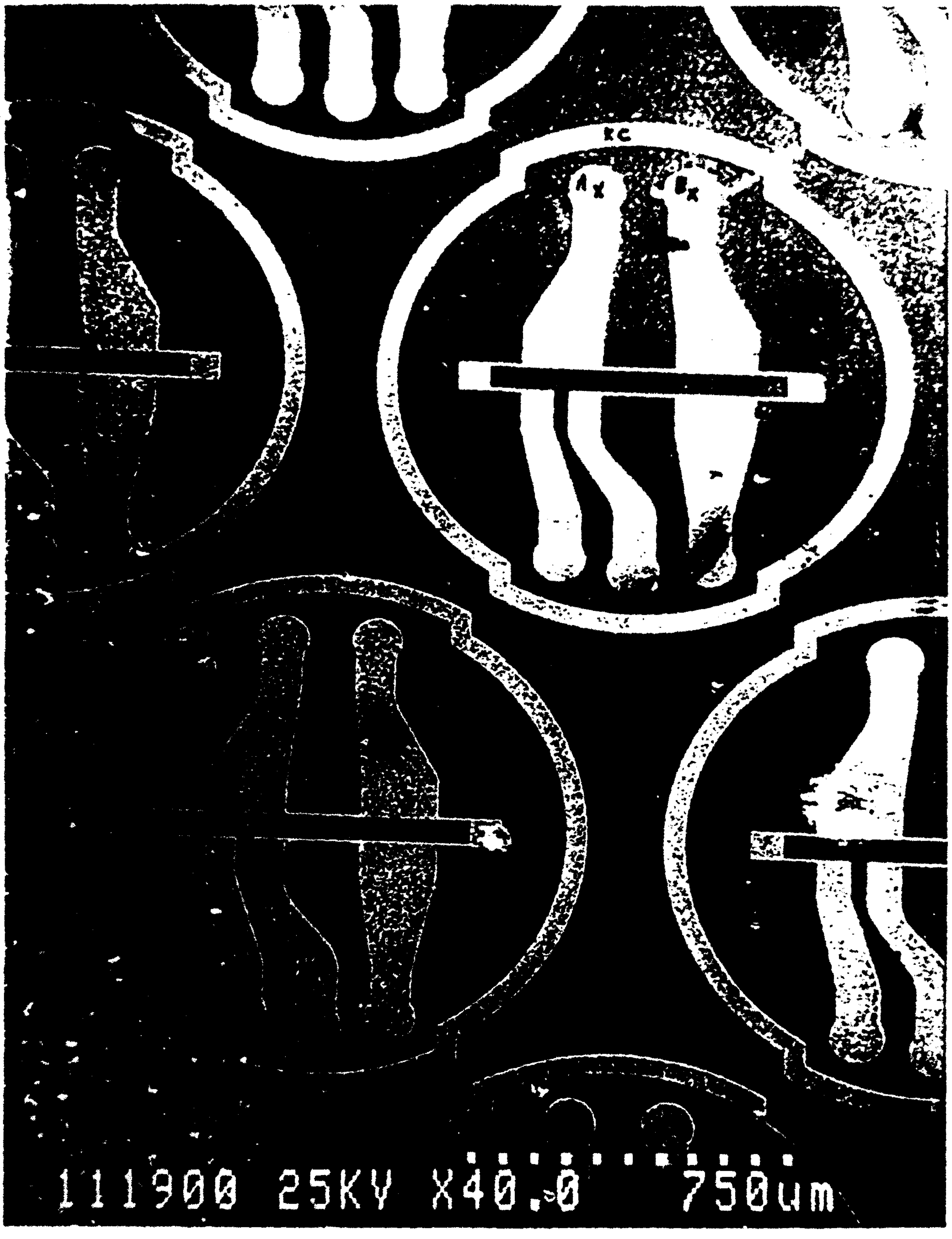


Figure 2.2 Probe placement points for I-V measurement

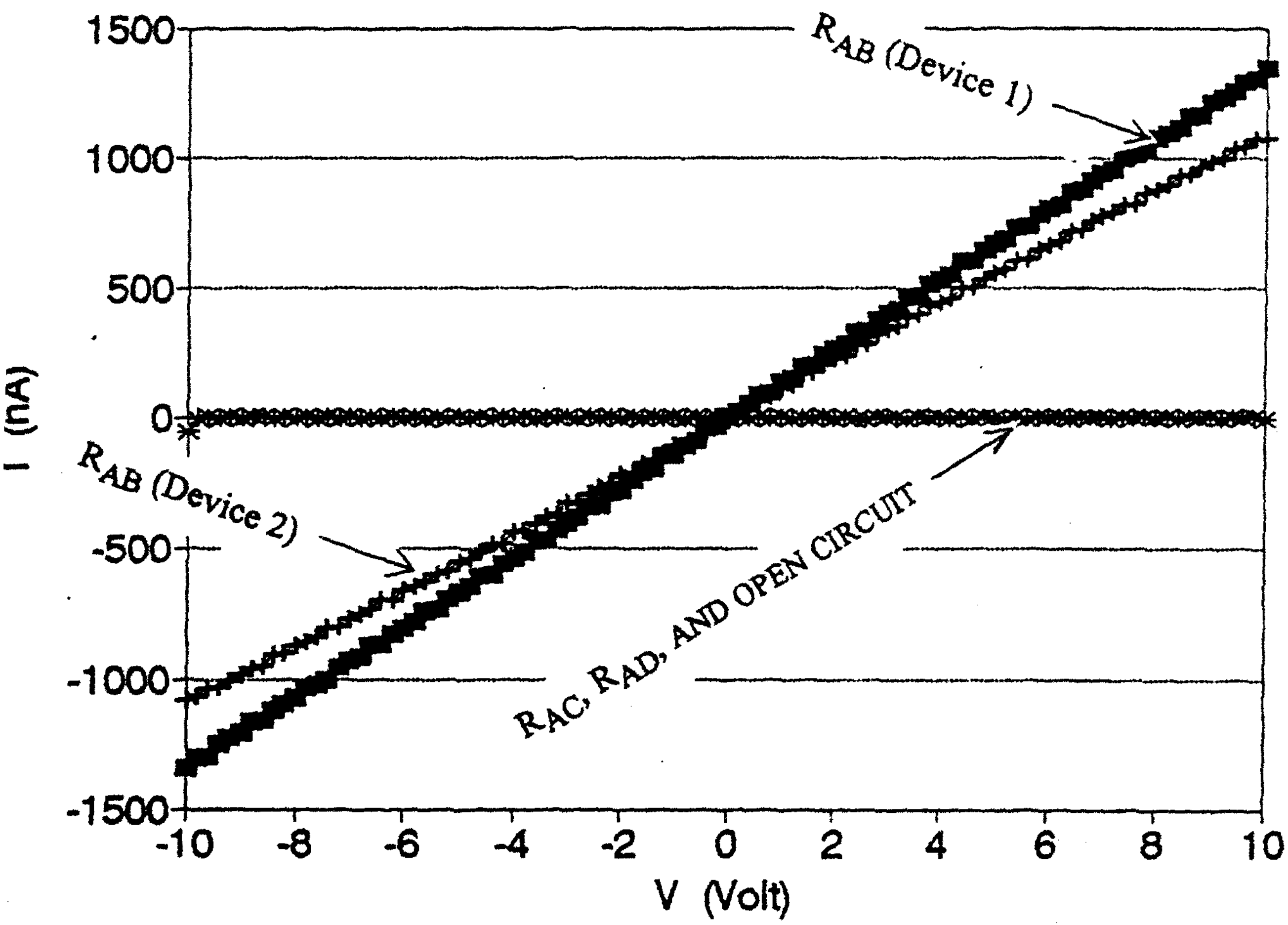
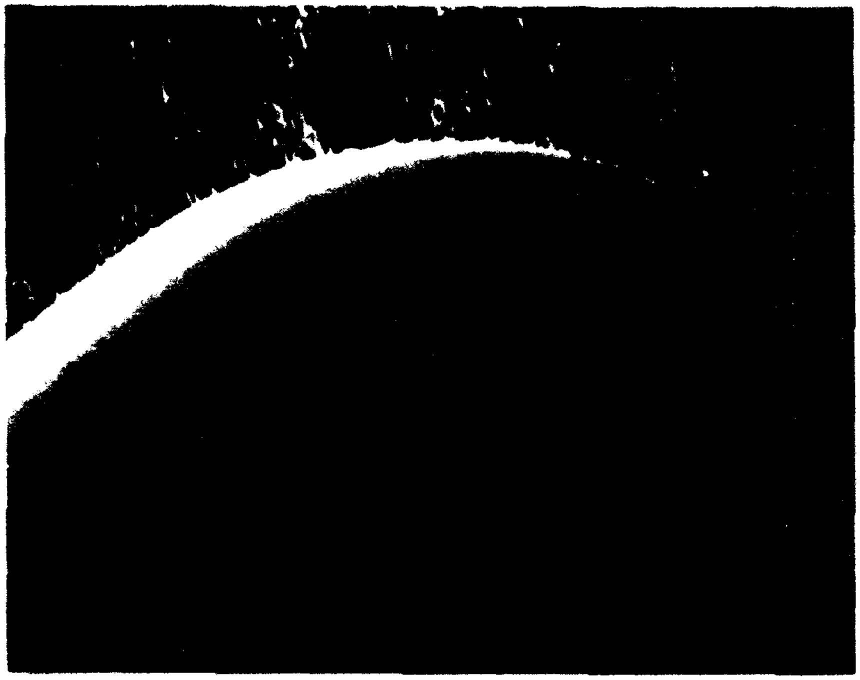


Figure 2.3 Typical I-V plot for p-D on i-D pressure sensor devices

Fig. 3.1:

SEM OF SILICON CARBIDE MEMBRANE ON SILCON SUBSTRATE
BACKSIDE VIEW MAGNIFICATION INDICATED



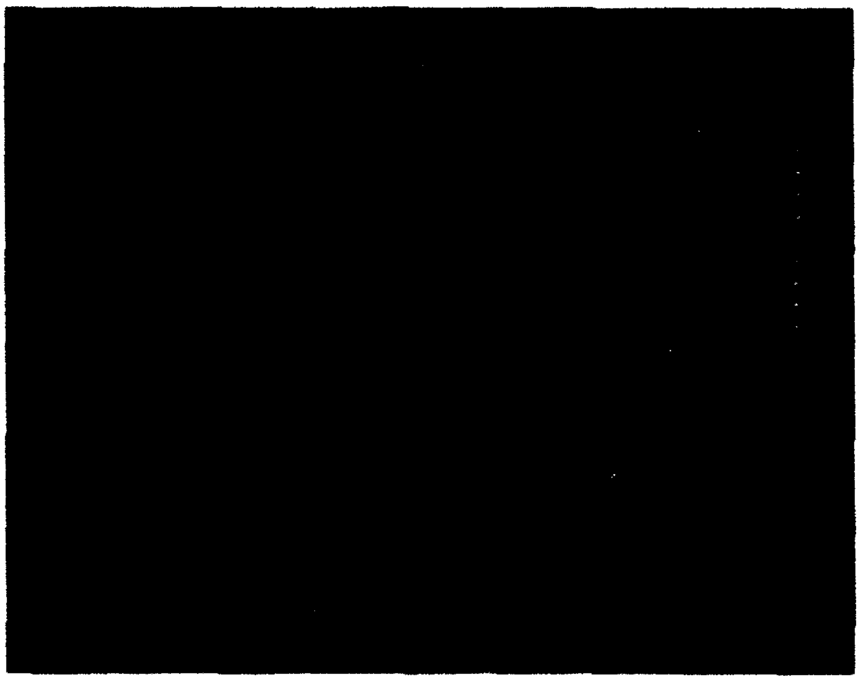
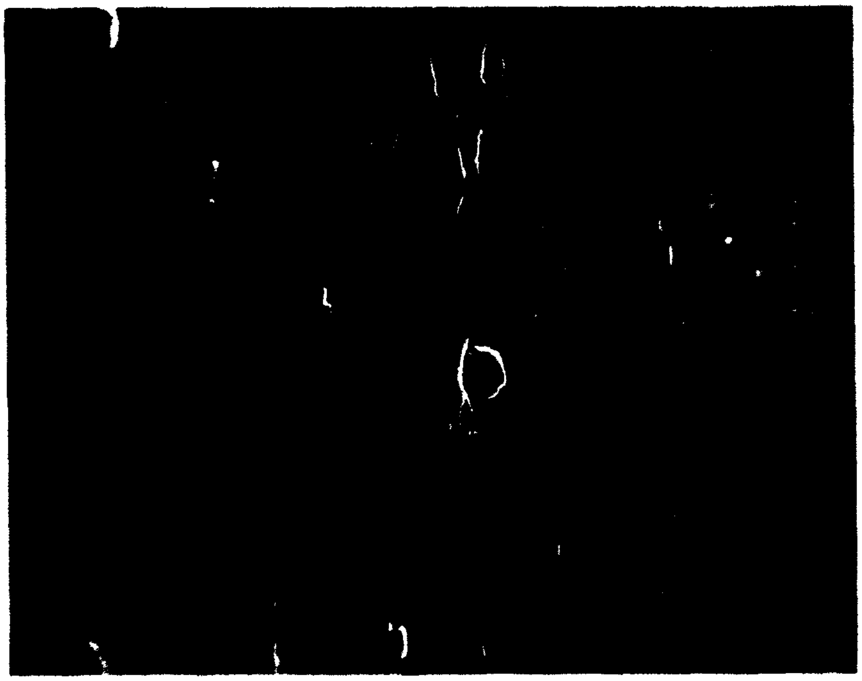


Fig. 3.1:

SEM OF SILICON CARBIDE MEMBRANE ON SILCON SUBSTRATE
BACKSIDE VIEW MAGNIFICATION INDICATED



SEM OF SILICON CARBIDE MEMBRANE ON SILCON SUBSTRATE
FRONT VIEW MAGNIFICATION INDICATED

Fig. 3.1:

Figure 4.1

Gauge Factor vs. Resistance

Polycrystalline Diamond