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REVIEW PAPER

Wide bandgap semiconductor thin films for piezoelectric and piezoresistive MEMS sensors applied at high temperatures: an overview

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Abstract The use of wide bandgap semiconductor thin films as sensing materials for micro-electrical–mechanical systems (MEMS) sensors has been the subject of much discussion in the academic and industrial communities. The motivation is that such materials are recognized as being suitable for extreme environment applications, namely: high temperatures, intense radiation and corrosive atmospheres. Among the wide bandgap semiconductor materials, aluminum nitride (AlN), zinc oxide (ZnO), diamond-like carbon (DLC) and silicon carbide (SiC) are highlighted due to their inherent sensing properties and compatibility with MEMS fabrication processes. Here we show an overview on the development technologies and applications of AlN, ZnO, DLC and SiC thin films in piezoelectric and piezoresistive MEMS sensors. Emphasis is placed on the influence of the temperature on the piezoelectric and piezoresistive properties of these films.

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1 Introduction

Harsh environments can be described as involving high or excessive exposure to heat, pressure, voltage, corrosive gases, radiation, vibration, shock, moisture, contamination, or extreme fluctuations in operating temperature range (Clatterbaugh et al. 2013). Developing devices capable of making measurements in harsh environments is especially problematic (Horsfall and Wright 2006). Selecting appropriate materials for use in such environments can be a determining factor in whether the device will operate as designed over its required service life. Material properties that are important, when designing for harsh environmental conditions include elastic modulus, coefficient of thermal expansion, yield strength, thermal conductivity, and resistance to creep and fatigue. All these properties are temperature dependent.

Micro-electro-mechanical systems (MEMS) have led to significant advances in actuator and sensor technology in fields from automotive to medicine because they are small, light and low-cost. In the use of MEMS sensors in harsh environments, some problems associated to degradation of their structural materials have been observed. Degradation of mechanical properties such as loses of elasticity and plastic deformations have been reported for MEMS materials exposed to extreme conditions. From the point of view of electrical properties, there is a reduction of semiconductors bandgap and redistribution of dopants (Sohi 2013).

The major difficulty with MEMS for harsh environments is that most of the sensors are based on silicon platform. Silicon is a semiconductor material ill-suited to extreme conditions of temperature, power, radiation, as well as, corrosive atmospheres. Apart from other extreme conditions, the effect of temperature on silicon properties, and consequently on the performance of silicon-based devices, has been the focus of many studies in recent years. Another point to be considered is how temperature affects the different physical principles of sensing and actuation used in MEMS devices. The temperature limit at which silicon sensors work properly has been well discussed in the literature (Yang 2013).

Piezoresistive elements have been used in many applications to provide analog signals for a variety of physical phenomena. For many years, silicon has been the material most employed as piezoresistive element in pressure sensors and accelerometers. The temperature dependence of the piezoresistive effect in silicon is well documented in the literature. It is known how the piezoresistive coefficients change with the temperature. The temperature limit has been verified up to 175 °C for silicon sensors and up to 500 °C for silicon-on-insulator (SOI) technology (Fraga et al. 2011a).

A number of materials have been investigated as potential candidates to replace the silicon in electronic and MEMS devices for high temperature applications. Wide bandgap semiconductor materials are preferred due to their inherent material advantages like high breakdown field, high thermal conductivity and high-saturated electron velocity. In addition, they exhibit chemical inertness and good mechanical properties (Hariz 1999).

Among the wide bandgap semiconductor materials, diamond, silicon carbide (SiC) and DLC (diamond-like carbon) have emerged as piezoresistive materials to use in extreme environmental conditions where conventional silicon sensors do not work properly (Werner et al. 1998).

In relation to piezoelectric sensors, aluminum nitride (AlN) and zinc oxide (ZnO) thin films are shown as wide bandgap semiconductor materials to replace ceramics like lead zirconate titanate (PZT) and lithium niobate ($LiNbO_3$). The properties that make AlN and ZnO films suited for applications as sensing materials include good piezoelectric properties, easier deposition and compatibility with standard MEMS fabrication techniques. Moreover, their advantages also include the compatibility with silicon electronics, possibility of manufacturing mechanical structures with stable motion control high-frequency responses and large force generation over small displacements (Krupa 2009).

In this work, we present an overview on development technologies and applications of wide band gap thin film materials, AlN, ZnO, DLC and SiC, in piezoelectric and piezoresistive sensors for high temperature applications. The deposition conditions of these films that affect their sensing properties are summarized. The influence of the temperature on piezoelectric and piezoresistive properties is also evaluated. Furthermore, some sensors based on AlN, ZnO, DLC and SiC films are reported.

2 Wide bandgap materials selection and their properties

The appropriate selection of materials plays a fundamental role in the development of devices with outstanding performance. A comparison among the properties of silicon and wide bandgap materials (diamond, SiC, ZnO, AlN and DLC) is shown in Table 1 (Krupa 2009; Oon and Cheong 2013; Coleman and Jagadish 2006). SiC polytypes, AlN, ZnO and DLC are good candidates for high temperature applications because they have large band gap at least two times higher than the silicon.

Note that diamond exhibits the most ideal properties for devices and sensors: low dielectric constant, which results in low capacitance and low noise; low leakage current due to its high intrinsic resistivity; high speed (fast signal processing) because of its high electron mobility; and heat spreader due to its high thermal conductivity.

Diamond, as well as silicon and germanium, is a non-piezoelectric material due to their inherent symmetry,

Table 1 Comparison among the properties of silicon (Si) with some wide bandgap materials

Property	Si	4H-SiC	6H-SiC	3C-SiC	Diamond	ZnO	AlN	DLC
Bandgap (eV)	1.1	3.2	3.0	2.3	5.45	3.37	6.2	1.0–4.0
Dielectric constant	11.8	10	9.7	9.6	5.5	8.66	8.5	3.0–6.0
Breakdown field (MV/cm)	0.3	2.0	2.4	1.2	5.6	4.0	1.8	>2.5
Intrinsic electrical resistivity (Ω cm)	10^3–10^5	$>10^{12}$	10^4–10^8	150	10^{13}–10^{16}	1×10^7	$>10^{11}$	10^7–10^{12}
Thermal conductivity (W/cm-K)	1.5	4.5	4.5	4.5	20	0.6–1.16	2.0	3.6
Saturated electron velocity (10^7 cm/s)	1.0	2.0	2.0	2.0	2.7	3.0	1.5	–
Electron mobility (cm^2/Vs)	1,350	720[a] 650[b]	370[a] 50[b]	900	1,900	210	300	10^{-5}
Intrinsic carrier concentration (cm^{-3})	1.5×10^{10}	8.2×10^{-9}	2.3×10^{-6}	6.9	1.6×10^{-7}	$<10^6$	9.4×10^{-34}	–

[a] Mobility along a-axis

[b] Mobility along c-axis

meanwhile, binary semiconductors, as for example SiC, have asymmetry and generally are piezoelectric materials. Although the SiC exhibits good piezoelectric properties and some piezoelectric MEMS sensors based on it have been shown in the literature, in this review, the SiC will be discussed only as a piezoresistive material given that AlN and ZnO thin films have been highlighted as one of the most important piezoelectric semiconductors.

The properties of wide bandgap semiconductor materials in thin film form have been extensively investigated in the last years. It has been shown that the properties of such films significantly change with different preparation parameters and different synthesis methods, thus their applications in sensors are far from being a mature technology like silicon.

In general, thin film materials are selected according to the properties required for a specific application. Different properties of each material used such as chemical composition, structure and morphology, as well as, thermal and electrical compatibility between the thin film and substrate and their ability to withstand processing conditions without degradation, are factors that influence the choice. Furthermore, the advantages of synthesis method and the easy material processing should be also considered. Thus one of the goals of this review is to discuss the influence of film characteristics on the piezoelectric response of AlN and ZnO thin films and on the piezoresistive response of DLC and SiC thin films.

3 Piezoelectric materials

Piezoelectric materials are energy transduction materials, i.e., they can generate an electric potential in response to an applied mechanical stress. This phenomenon is known as direct piezoelectric effect. In addition, they can also exhibit the converse piezoelectric effect, i.e., when subjected to the electrical field a mechanical stress is produced.

The piezoelectric effect is found in materials with a specific electrical crystalline structure. A piezoelectric material cannot be isotropic, or identical in all directions. If there was symmetry in the material, there would be no electric polarization yield (Trolier-Mckinstry and Muralt 2004; Vatansever et al. 2012). The Fig. 1 shows three materials. The material in Fig. 1a is isotropic and yields no resultant electric polarization when a force is applied. The materials in Fig. 1b, c yield parallel and perpendicular polarizations, respectively, when a force is applied.

Therefore, if pressure is exerted on certain crystals, the molecules will re-align and produce a charge across the crystal. A charge can be read as a voltage. A piezoelectric crystal is like a capacitor that is pressure-sensitive.

Materials of different groups can exhibit piezoelectric properties: ceramics, semiconductors and polymers.

In the last three decades, piezoelectric materials in thin film form, mainly semiconductors and ceramics, have been the focus of intensive research due to their potential both for surface acoustic wave (SAW) and for bulk acoustic wave (BAW) devices. The use of piezoelectric thin films in MEMS applications offers advantages such as efficient voltage-deflection conversion, high energy densities, low noise, high frequency operation and low power requirements. Studies have shown that the degree of piezoelectricity depends on the quality of the piezoelectric film. As mentioned, piezoelectric materials are naturally anisotropic, i.e. their properties vary depending on the direction in which they are measured. Electromechanical response of piezoelectric materials depends on their structure and configuration of defects (Momeni et al. 2012).

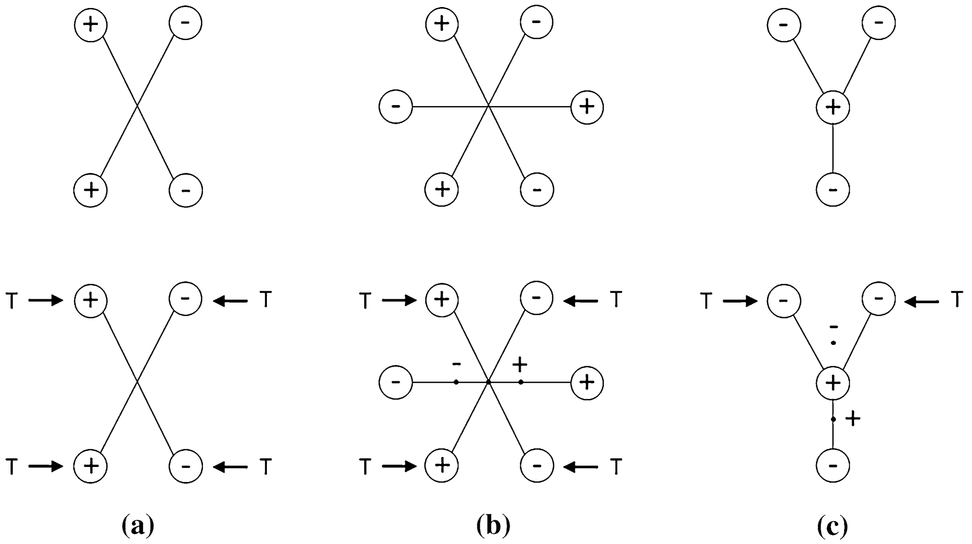

Fig. 1 Illustration of material polarizations with stress (adapted from course material, University of Dayton 2013)

Table 2 Properties of piezoelectric materials: PZT, $LiNbO_3$, AlN and ZnO

Property	PZT	$LiNbO_3$	AlN	ZnO
Piezoelectric constant (C/m^2)	$e_{31} = -6.5$ $e_{33} = 23.3$	$e_{31} = 0.23$ $e_{33} = 1.33$	$e_{31} = -0.58$ $e_{33} = 1.55$	$e_{31} = -0.57$ $e_{33} = 1.32$
Piezoelectric coefficient (pm/V)	$d_{31} = -120$ $d_{31} = -170$ $d_{33} = 60–130$	$d_{31} = -7.4$	$d_{31} = -2.0$ $d_{33} = 3.9$	$d_{31} = -5.0$ $d_{33} = 5.9$
Electromechanical coupling coefficient k^2	0.57–0.69	5.5	0.24	0.33
Elastic modulus (GPa)	68	203	308	201
Hardness (GPa)	8.0	–	17	5.0
Resistivity (Ω cm)	1×10^9	2×10^{10}	1×10^{11}	1×10^7
Thermal expansion α (/ºC)	2×10^{-6}	1.5×10^{-5}	4.3×10^{-6}	6.5×10^{-6}
Acoustic velocity (m/s)	3,900	3,980	10,127	5,700
Dielectric loss angle tan δ (10^5 V/m)	0.01–0.03	–	0.003	0.01–0.1

The wide bandgap semiconductor thin films, AlN and ZnO, due to low dielectric losses and high breakdown field, are recognized as piezoelectric semiconductor for high temperature applications. These characteristics together with other chemical and structural properties make AlN and ZnO potential piezoelectric materials for harsh environments. Both materials exhibit hexagonal wurtzite (WZ) structure with piezoelectric response along [0001]. ZnO and AlN thin films for piezoelectric sensor applications are generally deposited by sputtering. Reports show that ZnO films, with better piezoelectric properties, are sputter deposited at room temperature whereas temperatures from 100 to 900 °C are used for AlN sputter deposition (Trolier-Mckinstry and Muralt 2004). The oriented growth and crystal quality are requirements for thin film piezoelectric devices in order to take the best properties for a given application.

In Table 2 are summarized the piezoelectric properties of AlN, ZnO, PZT and $LiNbO_3$. The choice of the piezoelectric materials depends on the targeted application. The PZT has the highest piezoelectric coefficients, whereas $LiNbO_3$ the most electromechanical coupling coefficient. However, the deposition of PZT thin films usually requires processing at over 600 °C, which is not compatible with some microfabrication processes (Wang et al. 2012a). As can be observed, the AlN exhibits the higher acoustic velocity, which makes it preferred for SAW sensors.

On the other hand, the lower electromechanical coefficient limits its application in piezoelectric filters due this parameter determining the bandwidth of the filter (Muralt et al. 2005). It can be also observed that AlN is a hard material with hardness of 17 GPa whereas ZnO is soft with hardness of 5 GPa.

Additionally, in recent years, AlN and ZnO have emerged as piezoelectric materials for bio-applications due to toxicity of PZT ceramic that containing lead compounds harmful for health (Krupa 2009). They have also play an important role in MEMS energy harvesting due to their low power requirement and high available energy densities. AlN and ZnO are also considered promising materials for spintronics since it can possess ferromagnetic properties (Meng et al. 2013a).

3.1 AlN Thin films as Piezoelectric Material and their Sensor Applications

The piezoelectric properties of AlN thin films are frequently reported in the literature. AlN thin films, deposited in monocrystalline and polycrystalline forms on different substrates (semiconductor, metal and dielectric), are being used in a variety of MEMS and MOEMS (Micro-Opto-Mechanical Systems) devices due to their compatibility with CMOS (complimentary metal-oxide semiconductor) fabrication technology (Muralt 2008). The advantages associated with the use of AlN material include high hardness, high melting point and chemical inertness, which ensure that it will not be degraded during processing. Before fabricating the piezoelectric device, knowledge of the properties of AlN thin films is fundamental to carry out appropriate design consideration. Film characteristics such as degree orientation, film composition, orientation, grain size, stress, and surface roughness depend on deposition parameters. These characteristics influence piezoelectric properties as for example electromechanical coupling factor and acoustic velocity.

3.1.1 Influence of film characteristics on piezoelectric properties

Several studies on the synthesis of AlN thin films by sputtering processes have reported a common problem: the oxygen incorporation due to residual gases in deposition chamber. The affinity of Al with oxygen is much higher than with nitrogen, thus all oxygen in the chamber is incorporated into the AlN film, which results in the formation of aluminum oxide (Al_2O_3). In view of this, some authors have discussed the influence of oxygen content on the piezoelectric response of sputter-deposited AlN films. The following correlations have been observed:

- The electromechanical coupling factor (fundamental parameter for SAW devices) decreases significantly with oxygen content. An explanation is that the oxygen incorporated into the AlN lattice affects the grain size due to the formation of extended defects (Vergara et al. 2004). Moreover, the formation of Al_2O_3 retards the single grain columnar growth and leads to a poor preferred orientation as well as a poor surface topography (Liaw and Hickernell 1995). On the other hand, the presence of oxygen does not contribute significantly to the residual stress of the AlN film.
- The (002) X-ray diffraction intensity decreases with oxygen increases content in the AlN film. The best piezoelectric properties were found for oxygen content less than 0.5 %. In this oxygen concentration range, the AlN film exhibited strong SAW response, whereas that for oxygen content greater than 4 % was observed a degradation of the acoustic properties (velocity, piezoelectric constants and propagation loss (Liaw and Hickernell 1995);

From these studies, it is clear that significant oxygen concentrations into AlN films result in poor film quality and consequently at low piezoelectric properties. Hence, one of the goals of the optimization of sputtering deposition parameters should be to eliminate (or control) oxygen incorporation as much as possible. In order to control oxygen incorporation, the most common procedure is reducing the residual pressure in the chamber before sputtering deposition process.

The electroacoustic properties of AlN films with 0.5 % oxygen content were investigated by Engelmark et al. Test structures consisting of Al interdigital transducers formed on $AlN/SiO_2/Si$ structures were fabricated. The test structures exhibited high acoustic velocity of 4,900 m/s, low propagation losses of 4.7 dB/cm and good electromechanical coupling of 0.37 % (Engelmark et al. 2000).

Although less reported, argon (Ar) is another contaminant that affects the piezoelectric properties of AlN films. The presence of argon in sputter-deposited AlN films is due to the ionic bombardment induced by negative bias voltage applied to the substrate. Vergara et al. observed that the electromechanical coupling is independent of Ar content. However, the compressive stress and crystal quality of the AlN films depend on argon content; therefore, the piezoelectric constants are also influenced.

In spite of many studies on piezoelectricity in AlN thin films reported that the high piezoelectric response is observed on (002) preferred orientation, Sanz-Hervás et al. obtained good values of piezoelectric coefficients in AlN films with weak (002) peak and rocking curve as wide as 8°. They conclude that this anomalous behavior can be related to defects (probably, inversion domains) (Sanz-Hervás et al. 2006).

The correlations between piezoelectric coefficients (d_{33}) of AlN thin films and substrate roughness were investigated by Artieda et al. It was observed that a high d_{33} coefficient is achieved at low substrate roughness and low AlN film mechanical stress. In addition, it was also observed that increasing substrate roughness and stress there is a deterioration of d_{33} which is correlated with a higher density of opposite polarity grains (Artieda et al. 2010).

A more detailed study of the stress on AlN thin films for application in piezoelectric MEMS was reported by Sah et al. The following structures were analyzed: thin films (1), thin membranes (2) and resonant microstructures (3). The residual stress of the AlN films and membranes is 300–450 MPa, while lower values were obtained for suspended microstructures. The good stability of stress with aging, the stability of microstructures and the piezoelectric properties indicated that the AlN devices can be highly reliable without any deterioration in their performance (Sah et al. 2010). It is noteworthy that AlN films, depending on their deposition conditions, can achieve stress values in the order of GPa, which make them unsuitable for the fabrication of suspended structures for MEMS applications.

The effect of post-deposition rapid thermal annealing (RTA) on the crystal quality and the piezoelectric response of sputtered polycrystalline AlN thin films has also been reported. The RTA processing improves the crystal quality, which was not accompanied by a significant improvement on the piezoelectric response. This behavior can be attributed to the presence of grains with opposite polarities that could not be rearranged through the RTA treatment (Vergara et al. 2006).

Another issue frequently discussed in the literature is the temperature effects on piezoelectric properties of sputtered AlN films: (1) the piezoelectric coefficient d_{33} exhibits a constant value at temperatures ranging from 20 to 300 °C (Kano et al. 2006) and (2) when a pressure is applied, the piezoelectric response hardly changes in the temperature range from 25 to 700 °C, which confirm their potential for wide-band pressure sensor under high temperatures (Ooishi et al. 2006).

Most of the studies have reported the piezoelectric properties of AlN thin films grown on silicon or SiO_2/Si substrates due to MEMS fabrication techniques are based on silicon processing. However, piezoelectric properties of sputter-deposited AlN on metal substrates, especially titanium and platinum, have been investigated (Doll et al. 2010).

Recent works have shown that AlN thin films on diamond, sapphire and SiC substrates are promising for

SAW devices operating at high frequency. Assouar et al. observed that AlN/Si structure has a phase velocity of 5,055 m/s, whereas the same AlN film on sapphire substrate has a phase velocity of 5,536 m/s, which indicates that substrate used influences to SAW characteristics (Assouar et al. 2002). Aubert et al. discussed the potential of AlN/sapphire structure as an alternative piezoelectric material to langasite (LGS) for high-temperature SAW applications. They observed that while in the LGS SAW, the signal is completely lost after 8 h at 1,050 °C, the device based on AlN/sapphire stays alive for 60 h at this extreme temperature (Aubert et al. 2011).

3.1.2 *Piezoelectric sensors based on AlN thin films*

There is a variety of piezoelectric devices based on AlN thin films being used in different applications such as communication systems, energy harvesters, defense, and biomedical systems, among others. Some applications can be highlighted, such as:

- Defense and aerospace: (1) piezoelectric AlN MEMS resonators with high potential for gravimetric gas sensors (Khine et al. 2011) and (2) AlN micromachined piezoelectric microphone for aircraft fuselage arrays used to identify aircraft noise sources and/or assess the effectiveness of noise-reduction technologies (Williams et al. 2012).
- Biomedical: Piezoelectric AlN films, with underlying insulating ultra-nanocrystalline diamond (UNCD) and electrically conductive grain boundary nitrogen-incorporated UNCD (N-UNCD) and boron-doped UNCD (B-UNCD) layers, have been used as membranes for MEMS implantable drug delivery devices (Zalazar et al. 2013).

Research has been performed on the development of piezoelectric AlN MEMS sensors for high temperature applications. Studies on temperature behavior of mass sensitive dual-delay-line SAW-devices in gas sensors based on AlN piezoelectric layer were reported by Bender et al. They observed that higher sensitivity and stability could be achieved by controlling and changing temperature during the measurement compared to uncontrolled devices (Bender et al. 2003). Piezoelectric pressure sensors based on AlN thin film have been successfully tested at temperatures up to 973 K with the frequency of 1 and 10 Hz under the stress of 200 ± 40 N, respectively, in nitrogen atmosphere (Kishi et al. 2006).

In the development of AlN sensors, an issue that has been discussed is the influence of buffer layers, especially SiC and ZnO, on the performance of SAW sensors. Hoang and Chung compared SAW characteristics of the interdigital transducer (IDT)/AlN/3C-SiC structure with the IDT/AlN/Si structure at 160 MHz in the temperature range 30–150 °C. Improved temperature stability was observed for AlN/3C-SiC structure with temperature coefficient of frequency (TCF) measured of −18 ppm/°C and an insertion loss decrease of about 0.033 dB/°C (Hoang and Chung 2009). Meng et al. reported the preparation of AlN films on Si substrate with ZnO buffer layer. The purpose of this study was to show the advantages of the use of ZnO buffer layer given that AlN and ZnO have the same crystal structure besides small lattice mismatch. The current–voltage curves results indicated that the ZnO buffer layer highly improved the insulating properties of the AlN films (Meng et al. 2013b).

3.2 ZnO thin films as piezoelectric material and their sensor applications

ZnO has been considered to be an alternative to AlN for piezoelectric sensor applications due its the highest piezoelectric tensor among tetrahedrally-bonded semiconductors (Li et al. 2007). Furthermore, highly oriented ZnO thin films can be sputtered at a lower temperature than AlN, which also become the ZnO a good choice for piezoelectric sensor fabrication. Another characteristic of ZnO that is frequently highlighted, when it is compared the other wide bandgap semiconductors used in devices, is the high exciton binding energy of 60 meV (Coleman and Jagadish 2006).

Several studies show ZnO as a promising material for biomedical and energy harvesting applications. The development of ZnO piezoelectric biosensors has been motivated by the basic properties of the ZnO such as non-toxicity, biosafety and bio-compatibility (Fulati et al. 2009).

3.2.1 *Influence of film characteristics on piezoelectric properties*

Studies on the growth of piezoelectric ZnO films by sputtering processes are generally focused on the effects of the following deposition parameters: oxygen-to-argon gas ratios, substrate temperature, RF power and pressure. The main goal is the optimization of the deposition parameters in order to obtain ZnO films with high c-axis orientation along (002) plane, given that there is strong correlation between the degree of c-axis orientation and the piezoelectric activity.

Some general comments on the correlations between the sputter deposition parameters and the piezoelectric properties of ZnO films:

- The temperature substrate and gas pressure have a strong effect on the piezoelectric response and crystallinity of the ZnO thin films in terms of grain size, residual strain and surface roughness (Tiron et al. 2013). Increasing the substrate temperature, the (002) orientation increases due to increases of the mobility of sputtered particles associated to improves of the adhesion between particle and substrate. Piezoelectric response and crystallinity of ZnO films are improved considerably by decreasing the gas pressure and/or by increasing substrate temperature (Tiron et al. 2013);

- Oxygen-to-argon gas ratios: Increasing O_2 flow rate, the (002) peak intensity decreases because the (100) orientation is favored. An optimal deposition condition observed was for the gas ratio of $O_2/(Ar + O_2)$ of 25 % and the working pressure of 0.8 Pa. On these conditions, ZnO thin film has largest grain size, a very smooth and dense surface besides a good transverse piezoelectric constant d_{31} (Wang et al. 2012b);
- In highly oriented ZnO films, the lattice stress can be reduced by varying the sputtering pressure and substrate temperature (Kutepova and Hall 1998).
- The deposition rate of ZnO films increases with RF power increases (Sayago et al. 2005). Small changes in the RF power changed significantly the morphology of ZnO films.
- The ZnO film thickness responsible for piezoelectric activity is generally less than the physical thickness of the film and is adjacent to a layer having different acoustic impedance, which leads to an increase in the resonant frequency of the film (Wacogne et al. 1994). Theoretical and experimental works have shown the correlation between the piezoelectric strain constant and the c-axis orientation distribution obtained from the full width at half maximum (FWHM) of the x-ray rocking curves at the ZnO (002) diffraction. It has been observed that the experimental effective piezoelectric strain constants are, at most, 60 % of the value predicted by the theoretical calculations. This discrepancy can be associated to the cancellation of piezoelectric activity by ZnO grains of opposing polarity (Gardeniers et al. 1998).
- Besides silicon substrates, glass and stainless steel wafers have been used in the deposition highly oriented ZnO films with suitable properties for electromechanical devices (Barker et al. 1997).
- Intermediate layers, such as SiO_2 and Al, are used in the deposition of ZnO thin films onto (100) Si wafers for temperature compensation and for enhancement of the electromechanical coupling factor (Hall and Kutepova 1997);
- For high-frequency SAW devices, ZnO/diamond and ZnO/AlN/diamond structures are promising due to combining high velocity and high electromechanical coupling (Park et al. 1999; El Hakiki et al. 2005).

3.2.2 *Piezoelectric sensors based on ZnO thin films*

ZnO thin films have been commonly used for microcantilever applications. The ZnO piezoelectric layer is sandwiched between two electrodes (as for example Al/Au, Au/Cr, Au/Ti and Au/Cr, among others) on the beam. The most common materials used for beam is silicon, silicon dioxide (SiO_2), silicon nitride (Si_3N_4) and diamond.

One of the applications of ZnO films is in bulk acoustic resonator for the hydrogen sensor working at room temperature. Resonators working at 2.39 GHz consisting of a ZnO piezoelectric stack and an Al/W Bragg reflector using a palladium (Pd) film coated as an electrode have been developed. The resonance frequency of the resonator reduces progressively with the increase of hydrogen concentration due to the mass addition on the Pd layer after hydrogenation. These resonators are considered a promising and feasible platform for the hydrogen detection (Chen et al. 2011).

Another interesting application is for the intelligent food packages. ZnO SAW sensors directly built on the protein zein have been developed for intelligent food packaging platform. These sensors have an operating frequency of 687.38 MHz. The humidity detection is realized through the SAW frequency shifts with a sensitivity of 3,151.22 ppmV. Tests indicated that the combination of a ZnO SAW sensor with anti-microbial zein-coated flexible substrate enables wireless sensing of food freshness and achievement of food protection, which is promising for the intelligent food packages (Reyes et al. 2013).

SAW ultraviolet (UV) sensors fabricated from a sputter ZnO thin film using the third harmonic mode have also been reported. ZnO thin films, highly (002)-oriented and with good optical properties, are used as an active layer for UV detection. IDT/ZnO/Si structure were fabricated and exposed under UV light at a wavelength of 380 nm. As a result, under a UV intensity of 3 mW/cm^2, the SAW UV sensor was greatly shifted by 400 kHz at the third harmonic mode compared to a frequency shift of 10 kHz in the fundamental mode (Phan and Chung 2012).

Other applications include: (1) ZnO based film bulk acoustic resonators (FBAR) to monitor UV, RH, acetone and ozone in the environment (Qiu and Yu 2011) and (2) SAW sensors on ZnO/langasite substrates in order to apply them as harsh-environment oxygen sensors (Greve et al. 2011).

4 Piezoresistive materials

Another important group of MEMS sensors is based on the piezoresistive effect. Piezoresistive materials exhibit a change in electrical resistance when subjected to pressure (stress). The effect of applied stress is to change the number and the mobility of the charge carriers within a material, thus causing large changes in resistivity. This resultant change in resistivity characterizes the piezoresistive effect. Thus, in contrast to the piezoelectric effect, the piezoresistive effect only causes a change in electrical resistance, not in electric potential. Compared to piezoelectric materials, piezoresistive materials have very high sensitivity and better low frequency response.

The piezoresistive effect is widely used for pressure, force and acceleration sensors. Most of piezoresistive sensors commercially available are based on silicon platform. The main drawback of these sensors is represented by the fact that they cannot operate at high temperature because of the p-n insulation of the piezoresistors.

Although the piezoresistive effect is generally associated to crystalline materials, it has been observed in different amorphous films, especially diamond-like carbon (DLC) and SiC due to their high GF and resistance to harsh environments. From point of view of high temperature applications (>300 °C), SiC is more appropriate. More specifically, SiC remains stable in high temperature, oxidizing ambient, whereas diamond graphitizes (Senesky et al. 2009).

4.1 DLC films as piezoresistive material and their sensor applications

As highlighted previously, under some extreme conditions like very high temperature or high particle radiation, silicon may fail to sustain the required properties for MEMS sensors. To overcome some drawbacks of silicon materials, researchers are continuously trying to look for new materials for MEMS applications. Diamond and DLC can play important role for MEMS fabrication.

Among potential applications of DLC films are: (1) high frequency MEMS devices due to its high elasticity and tensile strength, biosensors for diagnostics and therapies, surface coatings for surgical instruments and prosthetic replacements due to its biocompatibility and (2) chemically modified surfaces that can act as sensing trace of gases to detect biomolecules (Santra et al. 2012).

The use of DLC films as sensing materials have received much attention due to their mechanical, optical and electrical properties associated to chemical inertness and biocompatibility. The growth temperature for amorphous DLC (<150 °C) is much lower than that of diamonds (typically at >900 °C) (Phan and Chung 2012). This makes it possible to integrate high temperature DLC piezoresistive sensors with electronic control circuits on the same substrate, thus opening up widespread applications. Many properties mechanical and chemical of DLC thin films make them excellent materials for MEMS applications in harsh environments which include piezoresistive sensors (e.g. pressure and acceleration). In Table 3 are shown properties of DLC films. As can be observed, DLC films have high elastic modulus, higher than the of Si (E = 180 GPa), which allows the development of piezoresistive microcantilevers that can be operated at high frequencies (Luo et al. 2007; Cao 2011).

On 1997, Chalker published a brief review on the diamond-like carbon technology, where reported the processing capabilities for the fabrication of micromechanical devices such as pressure transducers and accelerometers from piezoresistive effect in p-type diamond (Chalker 1997). Since then, some studies have discussed the piezoresistive properties of DLC films obtained by different techniques.

DLC films, grown by plasma-assisted chemical vapour deposition (PACVD), integrated in silicon boss membrane as strain gauge materials have been reported. High gauge factor (GF) values in the range of 16–36 were obtained, independent of longitudinal and transversal strain configurations and at temperatures in the range of 22–45 °C (Tibrewala et al. 2007).

The influence of deposition parameters, like substrate bias voltage, pressure and gas flow on the GF values of DLC films grown by PECVD (plasma enhanced chemical vapor deposition) have been reported (Petersen et al. 2012):

- For films prepared at a constant pressure (2.0 Pa) and constant gas flow (50 sccm): GF decreases (from 150 to 60) with substrate bias voltage increases (from −100 V to −400 V), whereas deposition rate increases from 7.5 to 25 nm/min;

Table 3 Properties of DLC films

DLC	Density (g/cm^3)	Hardness (GPa)	Elastic modulus (GPa)	sp^3 (%)	H (at.%)	Surface roughness (nm)	Residual stress (GPa)
a-C:H (hard)	1.6–2.2	10–20	300	30–60	10–40	1–30	1–2
a-C:H (soft)	0.9–1.6	<5		50–80	40–65		
ta-C: H	2.9	61		75	22–28	5–100	8–10
ta-C (evaporated)	1.9–2.0	2–5	757				
ta-C (MSIB)	3.0	30–130		90	<9	0.5–1.5	

- For films prepared at constant bias voltage (−200 V or −400 V) and constant gas flow (50 sccm): GF increases (from 25 to 90) when working pressure is varied from 0.5 to 2.0 Pa; for constant bias voltage (−200 V) and constant working pressure (2.0 Pa), GF increases (from 100 to 1,000) with gas flow increases (from 50 to 200 sccm).

DLC strain gauges integrated on micromachined silicon boss membrane force sensors were tested under vertical and horizontal connection as well as longitudinal and transversal orientation at temperature range from room temperature to 60 °C, revealing piezoresistive gauge factors typically in the range 20–30 (Peiner et al. 2006a).

The GFs of DLC containing tungsten (W) nickel (Ni) and silver (Ag) have also been measured:

- W-DLC films, deposited onto Si substrate by plasma enhanced chemical vapor deposition and DC magnetron co-sputtering of tungsten metal target, exhibit a linear strain dependence of electrical resistance under well controlled temperature condition and a GF of 6.1 (Takeno et al. 2008);
- Ni:a-C:H thin films, deposited on different substrates by reactive sputtering in a wide range of process parameters, have been shown as promising materials because combine a high GF approx. 12 and a very small temperature coefficient of resistance (TCR) (Koppert et al. 2009);
- DLC:Ag films, deposited by DC unbalanced magnetron sputtering, have been evaluated. There is a dependence of the GF of DLC:Ag films on both structure of the DLC matrix as well as on Ag atomic concentration and size of the Ag clusters, which can be verified due the GF of DLC:Ag films increases with the increase of the sp^3/sp^2 carbon bond ratio and decrease of the size of sp^2 bonded clusters. Moreover, the GF of films having the same DLC matrix structure can differ more than 1.5 times due to the different amount of Ag in the film. However, there is not correlation between the TCR and the structure of the DLC:Ag films (Tamulevičius et al. 2013).

The effects of temperature on the resistivity of DLC films have been investigated. There is dependence: the resistivity of the DLC films decreases with temperature increases (Meškinis et al. 2008).

Sputter-deposited amorphous carbon (a-C) film as a piezoresistive strain gauge into a silicon micro cantilever force sensor was developed. It was found linear characteristics of the strain gauge resistance versus the applied force in the range of 0 to ±600 μN revealing piezoresistive gauge factors from 36 to 46 (Peiner et al. 2006b).

A preliminary analysis of the applicability of sputter-deposited DLC thin films on thermally oxidized (100) substrates for high temperature piezoresistive sensors was reported. It was found GF around 70 and TCR smaller than 100 ppm/°C (Fraga et al. 2012).

Multifunctional sensors based on DLC thin films prepared at low temperatures (<150 °C) were tested. GF values from some 100 to 1,200 were obtained, proving the optimum properties of these films for force/pressure transducers (Lüthje et al. 2005).

4.2 SiC films as piezoresistive material and their sensor applications

SiC has long been recognized as a semiconductor material for device applications involving high temperatures. The physical and chemical characteristics of the SiC (wide bandgap, ability to operate at high temperature, mechanical strength, high chemical inertness and radiation resistance), particularly when compared to silicon, have shown its potential for harsh environment sensors. However, SiC polytypism requires a special attention given that although all SiC polytypes have the same atomic composition, the electrical properties are different. The most common SiC polytypes used in devices have hexagonal (6H-SiC, 4H-SiC and 2H-SiC) and cubic (3C-SiC) crystalline structures.

In recent years, SiC has emerged as a piezoresistive material. Several studies on piezoresistive properties of SiC in thin film and bulk forms have been published. Regarding the SiC thin films, the use of 3C-SiC and amorphous SiC (a-SiC) in MEMS sensors have been discussed. Table 4 compares the mechanical and electrical properties of these films (Reddy et al. 2008; Medeiros et al. 2012). As can be observed SiC films exhibit high elastic modulus and high hardness.

On 1997, one of the first studies on piezoresistive properties of SiC films was published by Shor et al. They characterized the n-type β-SiC (3C-SiC) as a piezoresistor and observed that the GF decreases at 450ºC to approximately half of the room-temperature value (GF = −31.8).

Table 4 Mechanical and electrical properties of SiC films

Material	Hardness (GPa)	Elastic modulus (GPa)	Resistivity (Ω cm)
Single crystal 3C-SiC on (100) Si	31.198 ± 3.7	433 ± 50	–
Polycrystalline 3C-SiC on (100) Si	33.54 ± 3.3	457 ± 50	30–33
Amorphous SiC (a-SiC) on (100) Si	16.7	245.90	0.45–59

However, TCR measurements of the piezoresistors on the range 25–800 °C showed a roughly constant TCR of 0.72 %/°C (Shor et al. 1999).

In general, the works on piezoresistive effect in SiC films have reported that their temperature coefficient of gauge factor (TCGF) remains unchanged at high temperatures.

Recently, the applicability of PECVD SiC thin films, deposited on oxidized silicon wafers at substrate temperature of 900 °C, for strain transduction in harsh environment MEMS sensors were verified at temperatures up to 600 °C (Jakovlev et al. 2013).

The effects of carbon content on the piezoresistive properties of non-stoichiometric silicon carbide (Si_xC_y) films, deposited on thermally oxidized (100) Si substrates by PECVD, have also been discussed. High GF and low TCR were obtained for the films. A correlation between film composition and the GF was observed: with the carbon content decreases the GF increases (Fraga and Koberstein 2013).

In addition, the effect of nitrogen doping on piezoresistive properties of Si_xC_y thin films prepared by PECVD was reported. It was observed that nitrogen doping increased the piezoresistive coefficient and the TCR of the films (Fraga et al. 2010a, b).

A comparison between piezoresistive properties of SiC films produced by PECVD and RF magnetron sputtering showed that the GF of PECVD SiC film is greater than of the sputtered film. Besides, the PECVD SiC film exhibited a smaller TCR up to 250 °C (Fraga 2011).

A high temperature pressure sensor with 3C–SiC piezoresistors on SOI substrate was developed. A GF of −18 at room temperature was measured. The GF decreased in the temperature range of −10 to 200 °C. Tests showed that the pressure sensor has sensitivity is 3.5 mV/V bar at room temperature decreasing to 2.1 mV/V bar at 200 °C for a 100-μm thick circular center boss diaphragm (Eickhoff et al. 1999).

A piezoresistive β-SiC-SOI pressure sensor with an on chip polycrystalline SiC thermistor for high operating temperatures was also developed. The fabricated pressure sensor chip was tested in the temperature range between room temperature and 573 K. The sensitivity at room temperature is 2.0 mV/V.bar. The temperature coefficient of the sensitivity (TCS) between room temperature and 573 K is −0.16 %/K (Ziermann et al. 1999).

Another piezoresistive β-SiC-on-SOI pressure sensor for measuring the cylinder pressure in engines of automobiles was reported. The sensor was characterized under static pressures of up to 200 bar in the temperature range from room temperature to 300 °C. The sensitivity of the sensor at room temperature is 0.19 mV/bar and decreased to 0.12 mV/bar at 300 °C (Berg et al. 1998).

Wu et al. developed a high-temperature pressure sensors based on polycrystalline and single-crystalline 3C-SiC piezoresistors and fabricated by bulk micromachining the underlying 100-mm diameter (100) Si substrate. For the sensor with phosphorus-doped atmospheric pressure chemical vapor deposition (APCVD) polycrystalline 3C-SiC (poly-SiC) piezoresistors on Si diaphragm with Si_3N_4, poly-SiC films were deposited at different temperatures and doping levels and exhibited −2.1 as the best gauge factor and a sensitivities up to 20.9-mV/Vpsi at room temperature. For the second sensor, epitaxially-grown unintentionally nitrogen-doped single-crystalline 3C-SiC piezoresistors were fabricated on Si/SiO_2 diaphragms and tested at temperatures up to 400 °C, exhibited a sensitivity of 177.6-mV/Vpsi at room temperature and 63.1-mV/Vpsi at 400 °C. The estimated longitudinal gauge factor of 3C-SiC piezoresistors along the [100] direction was estimated at about −18 at room temperature and −7 at 400 °C (Wu et al. 2006).

A poly-3C-SiC piezoresistive micro-pressure sensor for extreme environment applications prepared with a combination crystal growth technology using chemical vapor deposition (CVD) and micromachining techniques was fabricated and tested. The pressure sensitivities of the sensors were 0.1 mV/Vbar. The nonlinearity of the devices was ±0.44 % FS, and the hysteresis was 0.61 %·FS. The TCS of the pressure sensors was -1,867 ppm/°C, the TCR was −792 ppm/°C, and the TCGF to 5 bars was −1,042 ppm/°C, from 25 to 400 °C (Chung 2010).

The fabrication and characterization of a $SiC/SiO_2/Si$ piezoresistive pressure sensor, consisting of six PECVD SiC thin-film piezoresistors configured in Wheatstone bridge on a thermally oxidized micromachined silicon square diaphragm (1,800 × 1,800 μm), was reported by Fraga et al. The output voltage of the sensor was measured for applied pressure ranging from 0 to 12 psi and voltage supply of 12 V (Fraga et al. 2010a, b). A preliminary evaluation of the influence of the temperature on the sensitivity of this sensor was performed up to 250 °C indicating its potential for high temperature applications (Fraga et al. 2011a, b).

5 Conclusions

Piezoelectric and piezoresistive technologies offer a wide range of sensors, actuators and transducer possibilities. The excellent properties of wide bandgap semiconductor thin films, such as high saturation velocity, high breakdown field, high elastic modulus, high chemical stability and high corrosion resistance, make them potential candidates to be employed as piezoelectric or piezoresistive sensing element in harsh environment applications, especially at high temperatures. Recent advances in the synthesis and processing of wide bandgap semiconductor thin films have allowed the fabrication of devices with high reliability and performance, which has motivated their use in new emerging applications.

Wide bandgap semiconductor thin films, DLC and SiC, have shown to be promising for piezoresistive MEMS sensors; whereas AlN and ZnO thin films have been used in piezoelectric MEMS sensors. Besides the superior physical and chemical properties, these wide bandgap thin films are compatible with MEMS fabrication techniques.

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