Effects of annealing conditions on temperature coefficient of resistance of Pt/AlOx thin-film thermistors

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**Abstract**

Uncooled microbolometer arrays incorporating vertically-aligned carbon nanotube absorbers are increasingly being adopted in satellite instrumentation for monitoring the Earth’s radiation budget. A key requirement for such microbolometers is a thermistor having high temperature coefficient of resistance (TCR), low 1/*f* noise, while surviving high-temperature carbon nanotube growth at 800 °C. In the present work, Pt thin film thermistors are fabricated on SiNx/SiO2/Si substrates using DC magnetron sputtering. To achieve enhanced adhesion of the Pt thin film, an interlayer of AlOx is deposited on the substrate via reactive high power impulse magnetron sputtering. To maximize the positive TCR, the Pt/AlOx is subjected to different annealing conditions by varying temperature, time, and gaseous environment. With an increase in the annealing temperature and duration, Pt/AlOx thin films exhibit an improvement in TCR. Microstructural and morphological investigations suggest that improvement in TCR is related to the recrystallization of Pt and the resulting increase in grain size. A relatively high TCR of 0.308 %/°C (TCR of bulk Pt = 0.359 %/°C) was obtained at an operational range of 20 - 50 °C when Pt was annealed at 800 °C for 1 h and 3 h in air and Ar, respectively. Deposition of Pt without an AlOx interlayer resulted in thin film blistering and delamination when annealed at 800 °C for 1 h in air. A Pt/AlOx thermistor with a TCR of 0.308 %/°C, annealed at 800 °C for 3 h in Ar has the potential for use in microbolometers that must undergo high-temperature growth of vertically aligned carbon nanotube absorbers.

Keywords: thermistor, platinum; aluminum oxide; HiPIMS; annealing; temperature coefficient of resistance

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1. **Introduction**

 Earth orbiting satellites have provided the opportunity to monitor the Earth’s energy imbalance (EEI) with long term decadal continuity. The EEI is measured by using satellite-based radiometers to monitor top of atmosphere energy fluxes: incoming solar irradiance, outgoing shortwave radiation (OSR, 0.3 μm - 5 μm) and outgoing longwave radiation (OLR, 5 μm – 100 μm). The difference between the solar radiation absorbed by the Earth and re-emitted back to space is approximately 1 W/m2 [1]. Currently, satellite instrumentation is not sufficiently accurate to measure the relatively small EEI and the value is being confirmed with terrestrial measurements [2]. Amongst various other initiatives to accurately track Earth’s radiation budget, the Libera NASA mission has been designed to extend the climate record of NASA’s Clouds and the Earth’s Radiant Energy System (CERES) using new radiometer technology [3][4]. This new technology aims to introduce electrical substitution radiometers (ESR) using vertically aligned carbon nanotube (VACNT) absorbers for higher accuracy earth remote sensing of the OSR and OLR [5–7][8].

 Microbolometer arrays are the de facto standard for uncooled sensing of infrared radiation, yet are narrowband (typically 8 μm – 16 μm) and are not typically used as radiometers (unless calibrated) [9]. Bolometers with VACNT absorbers and electrical substitution heating have recently been demonstrated as high-accuracy, broadband (0.3 μm – 100 μm) radiometers compatible with silicon microfabrication techniques [10,11]. Developing these single-element radiometers into a microbolometer array is currently underway [6][12]. A key component of a microbolometer or radiometer is the thermometer, which measures the resulting rise in temperature of the detector element when the radiant flux is incident. Typically, the temperature change is measured with a thin-film thermistor as a change in electrical resistance. The responsivity of a detector is proportional to the temperature coefficient of resistance (TCR) of the thermistor [13]. In this regard, platinum is considered as a potential thermistor choice because of its low or absent 1/*f* noise despite a low TCR near 0.3 %/°C [14]. In addition, Pt displays other favorable properties such as chemical inertness and excellent high temperature stability. These qualities make Pt thermistors suitable for integration with VACNT bolometers (high-temperature growth) and satellite instrumentation (long-term stability) [15–18].

 Electrical properties such as TCR and resistivity of thin films strongly depend on the density, crystallinity, surface microstructure, and grain size of the film. Along with deposition process parameters, post-processing of the film such as annealing can strongly influence electrical performance [19–21]. Annealing a film can cause recrystallization, a process that involves the thermally activated motion of grains and subgrain boundaries. Due to the movement of boundaries, defects and/or dislocations in the material reduce significantly; resulting in a film with larger grains and lower dislocation density [22,23]. The prime factor determining the recrystallization process is the energy difference between the deformed grain state and recrystallized state [24]. As the recrystallization process induces several changes in the microstructure of the film, it is important to understand how annealing-induced recrystallization influences the TCR and resistivity of Pt thin film thermistors.

Along with controlling the microstructure of the thermistor material through annealing, the incorporation of a thin-film interlayer may influence the properties of the Pt film [25]. For example, the presence of an interlayer plays an important role in improving the adhesion of the film to the underlying substrate such as silicon oxide (SiOx) or silicon nitride (SiNx). At high annealing temperatures, maintaining film adhesion may be challenging due to microstructural changes such as hillock formation, grain coarsening, hole formation, and agglomeration. In addition, such changes in microstructure may result in degraded electrical properties [26–28]. With commonly used adhesion layers such as Ta or Ti, there are reports of interdiffusion of these elements into the Pt layer, resulting in an increase in resistivity and degradation of TCR [29,30]. This diffusion can also be correlated with reduced adhesion at the Pt/Ti or Pt/Ta interface. Olowolafe et al. demonstrated that when a Pt/Ti/SiO2 film was annealed in an O2 environment at 600 °C, several components like Pt3Si, PtTi, and TiO2 were formed [31]. Kang et al. elucidated that the annealing of Pt/Ti/SiNx/Si specimen at 600 °C for 30 min in O2 ambient lacked adhesion due to the formation of a brittle TiO2 phase, depletion of the Ti adhesion layer, and an increase in internal stress [32]. It was shown by Bernhardt et al., that a Zr layer can significantly improve the adhesion of a Pt film compared to a Ti interlayer. However, oxidation of the Zr layer is evident when annealed at 700 °C in air [33]. Concerning these issues, an oxidation-resistant interlayer to replace Ti, Ta, and Zr that can hinder interdiffusion is needed. In this case, one needs to consider a material that can withstand high thermal annealing temperatures (> 600 °C) for extended times in different atmospheres. Considering all these aspects, the use of metal oxides has been proposed as a substrate or interlayer. A number of metal oxides (e.g. AlOx, SiOx, MgO) have high dissociation temperatures (typically > 1000 °C) and are commonly used as diffusion barriers [34][35,36]. Aluminum oxide (AlOx) is a promising material due to its chemical stability and low diffusion coefficient at elevated temperatures [37–39]. AlOx can act as a diffusion barrier to protect the Pt layer from undesired interdiffusion at the interface between Pt and SiNx.

 The properties of a sputtered interlayer can be altered simply by the choice of the method used. Direct current (DC), radio frequency (RF), or high power impulse magnetron sputtering (HiPIMS) have each been shown to exhibit significant differences in the deposited microstructure of any film [40–42]. Compared to DC and RF sputtering processes, HiPIMS technology allows for high power densities not previously possible. Process control over very high power densities offers the generation of: intense pulses of sputtering power, a highly ionized plasma, and high energy adatoms [43,44]. These advantages can influence the thin film nucleation morphology, crystallinity, adhesion, density, and residual stress [45,46].

This work is focused on the investigation of lithographically patterned Pt/AlOx thermistors on SiNx/SiO2/Si substrates using lift-off lithography. HiPIMS was employed to deposit AlOx as an interlayer and the Pt film was deposited by DC magnetron sputtering. To get a clear understanding of the recrystallization process and its influence on the electrical properties of the film, Pt/AlOx thermistors were annealed at different temperatures, times, and gaseous environments. As-deposited and annealed Pt/AlOx were inspected for surface morphology and grain size. Resistivity and TCR of the Pt/AlOx were measured after exposure to different annealing conditions. The influence of the AlOx interlayer on adhesion and electrical film performance has been elucidated. This investigation allows us to investigate the feasibility of integrating Pt/AlOx thermistors in VACNT bolometers.

1. **Experimental procedure**
	1. **Fabrication process of the Pt thermistor**

AlOx and Pt films were deposited in a high vacuum sputter system in the Boulder Microfabrication Facility (BMF) at the National Institute of Standards and Technology (NIST). The system is capable of sputtering using a pulsed DC power supply (2000 W, 0-100 kHz) and a HiPIMS supply (6000 W, 1000 V peak, 600 A peak, 2.5 - 1000 μs pulse duration, 0 - 10 kHz). An RF source with a maximum power of 100 W is coupled to the substrate holder. **Table 1** summarizes the process parameters used for AlOx and Pt deposition by sputtering.

Pt/AlOx thermistors were fabricated using photolithography and lift-off techniques. **Fig. 1** is a flow chart of the Pt/AlOx fabrication process followed. First, prime double-side polished silicon substrates (76.2 mm diameter) with 150 nm of thermally grown silicon dioxide, were coated with 500 nm of low-pressure chemical vapor deposition (LPCVD) silicon-rich low-stress SiNx (**Fig. 1a**). Wafers were loaded in the sputter system and RF cleaned in an Ar plasma (100 W, 1.33 Pa, 1 min) before a 10 nm thick AlOx layer was deposited using reactive HiPIMS at room temperature (**Fig. 1b**). After sputtering, photoresist (1 µm thick) was spin-coated onto the AlOx film (**Fig. 1c**) followed by a 1 minute, 95 °C hot plate bake. The photoresist pattern was then exposed in a maskless aligner with a dose of 350 mJ/cm2 at 405 nm followed by a 1 minute, 110 °C post-exposure bake. The exposed photoresist was puddle developed (**Fig. 1d**), rinsed in deionized water, ashed in oxygen plasma (50 W, 53.33 Pa, 30 s), and reloaded into the sputter system. Wafers were RF cleaned in an Ar plasma (100 W, 1.33 Pa, 1 min) followed by DC sputtering of Pt (175 nm) at room temperature (**Fig. 1e**). Photoresist was removed using acetone and isopropanol in an ultrasonic bath to obtain the final Pt/AlOx pattern (**Fig. 1f**).

**Fig. 2** shows design and optical microscopy images of the lithographically patterned thermistor meanders. Wafers are patterned with a 3 x 3 matrix consisting of nine rectangles of area 11 mm x 13 mm as shown in **Fig. 2.a.1**. Three rectangles are unpatterned and six rectangles contain thermistors of two different sizes: meander style-1 (11 µm linewidth, 13 µm pitch) and meander style-2 (2 µm linewidth, 4 µm pitch). Meander style-1 is two interdigitated serpentine thermistors with square corners and is 8187 squares (length/width) (**Fig. 2b.1-2**). Meander style-2 is a serpentine pattern with a constant cross-sectional width wire and is 1877 squares (**Fig. 2b.3-4**). The magnified rectangle shown in **Fig. 2a.2** contains one of meander style-1 and two of meander style-2. The optical images in **Fig. 2b.1-2** and **Fig. 2b.3-4** refer to meander style-1 and meander style-2.

After the microfabrication of the Pt/AlOx thermistors, the wafers were annealed at atmospheric pressure in a 100 mm diameter quartz tube furnace. Different annealing conditions were tested to improve TCR of the Pt/AlOx thermistor. Annealing temperatures were varied from 500 °C - 900 °C. The heating rate was kept at 10 °C/min, and the annealing time was varied from 0.5 h to 3 h. The annealing process was performed either in an air environment (N2: 157 sccm, O2: 43 sccm) or Ar only (200 sccm).

 **2.2. Characterization of Pt thermistor**

The microstructure of the Pt/AlOx films was investigated using a scanning electron microscope (SEM). The grain size was measured from SEM images using commercial software. For each sample, the diameter of 30 grains was measured using the length measurement tool (setting the scale in accordance with the SEM image bar). The crystalline quality of the Pt Films was evaluated by X-ray diffraction (Cu K-alpha1). The diffractograms were collected at 40 kV/44 mA, using a Ge(220)x2 primary beam monochromator, in a parallel beam configuration. The 2q-ω scans were performed at 2q ranging from 20° to 90° with a step size of 0.005 °/step and at a scan speed of 0.156 s/step. AlOx interlayer and Pt film surfaces were analyzed by X-ray photoelectron spectroscopy (XPS) (K-alpha+ XPS, 1486.6 eV). Prior to the analysis, AlOx and Pt samples were Ar ion cleaned (3 keV), for 60 s and 180 s, respectively. For the AlOx film, Al2p and O1s peaks were collected in high resolution, to determine Al/O ratio. For both, as-deposited and annealed Pt films, Pt4f and O1s high resolution peaks were acquired. The peaks were analyzed using commercial software.

The electrical properties of Pt/AlOx thermistors (meander style-1 and meander style-2) were characterized through resistivity and TCR measurements. The resistivity (Ω·nm) was calculated using the resistance (Ω) measured from a 4-point resistance probe (tip spacing = 1.59 mm) and a contact profilometer measurement of the film thickness (nm).

For the meanders, the resistivity is given by:

 $ρ\_{meander}=\frac{R\_{meander}}{L/W}∙t\_{Pt}$ **Eq. 1**,

where ρmeanderis the resistivity of the meander*, R*meanderis the measured 4-point probe resistance*,* *tPt* is the thickness of the Pt film, and *L/W* is the number of squares in meander style-1 (8187) or meander style-2 (1877).

TCR values were measured using a custom apparatus capable of collecting the 4-point resistance in 27 different regions. The 4-point resistance measurements of lithographically defined meanders were read out using a multimeter programmatically switchable between the 27 locations. Electrical contact was achieved with printed circuit board (PCB) mounted pogo pins (four pins spaced 2 mm apart linearly) that make press-contact to the deposited thin film or to bond pads connected to the meanders. A thermoelectrically controlled temperature chamber with a range from 10 °C to 60°C was used with a maximum deviation of ±0.1 °C at a user-specified setpoint. TCR was measured at 20 °C and 50 °C with 60 min dwell times at each temperature.

The formula to determine TCR is:

 $TCR= \frac{\left(R\_{2}- R\_{1}\right)}{\left(T\_{2}- T\_{1}\right)∙R\_{1}} ∙ 100 [\%/°C]$ **Eq. 2,**

Where *R*1is the 4-point probe resistance at 20 °C, *R2* is the 4-point resistance at 50 °C, *T1* = 20 °C, *T*2 = 50 °C.

1. **Results and Discussion**
	1. **Microstructure of Pt/AlOx**

The surface morphology of as-deposited and annealed samples was investigated by SEM to understand the effect of annealing temperature on Pt/AlOx films. Annealing was performed at 500 °C, 600 °C, 700 °C, 800 °C, and 900 °C for 1 h in air. For as-deposited films, smooth surfaces, and grain sizes on the order of 20 nm are observed as shown in **Fig. 3a**. After annealing at 500 °C (**Fig. 3b**), minimal change in the grain size is observed. At 500 °C, the thermal energy is insufficient to significantly deform the initial grains [47]. When the sample was annealed at 600 °C in air, the grains become larger (**Fig. 3c)**. Above 600 °C, coalescence of smaller grains results in major grain growth, which is evident from the SEM images of the annealed samples from **Fig. 3c-e**. During the grain growth process, grains in the film start growing at the expense of neighboring grains. When a subgrain reaches a critical size, it transforms into a stable recrystallized grain [48]. In this way, the fine-grain matrix is completely consumed in the process of formation of larger grains. The grain size continuously increases when the annealing temperature was increased from 500 °C to 800 °C for 1 h in air (**Fig. 3b-e**). It is estimated that the recrystallization process of Pt initiates at temperatures above 600 °C [49,50]. At 800 °C for 1 h in air, the grain sizes reached a maximum of ≈ 120 nm and became densely packed which is expected to be advantageous for a Pt thermistor. An annealing temperature of 900 °C does not result in significantly increased grain size. Additionally, the adhesion of the film was reduced after annealing at 900 °C. It can be distinctly observed that the film annealed at 800 °C is continuous, uniform, dense, and pinhole-free. The recrystallization process is homogenous throughout the film. The change in microstructure indicates that post-deposition annealing is essential for improving Pt film microstructure.

The influence of annealing time on the microstructure of Pt/AlOx films was also investigated. **Fig. 4** shows the change in surface morphology of the films, annealed at 600 °C and 800 °C in air for 0.5 h, 1 h, and 3 h. From **Fig. 4a.1**, it can be observed that the sample annealed at 600 °C for 0.5 h exhibited a rough morphology. Grain-to-grain connectivity increases for the film annealed at 600 °C for 1 h, as shown in **Fig. 4a.2**. It is well known that with an increase in annealing time, grain size becomes larger. The important parameter for grain growth in a thin film is achieving low grain boundary energy resulting from the reduction in the total grain boundary area. Therefore, some grains continue extending to the neighboring matrix by consuming the fine-grained matrix until they meet with a grain of similar or bigger size [51]. When annealing the sample at 600 °C for 3 h, it leads to greater recrystallization and the formation of a more uniform surface morphology with a larger grain size, as depicted in **Fig. 4a.3**. The possible reason for this structural change for longer annealing times is there exists a characteristic/threshold time at the annealing temperature for complete recrystallization. Higher annealing temperatures can provide a larger driving force in the grain growth process. Therefore, the surface morphology of the film was studied after annealing Pt/AlOx at 800 °C in air with a hold time of 0.5 h to 3 h. **Fig. 4b.1** reveals a terraced microstructure with a combination of larger and smaller grains. In the case of the sample annealed at 800 °C for 0.5 h in air, the grains exhibited an uneven size distribution indicating that surface atoms had insufficient time to drive the coalescence process for homogeneous recrystallization. On the other hand, annealing for 3 h at 800 °C enhanced grain growth for some grains in the matrix resulting in abnormal grain growth (**Fig. 4b.3**) [52]. The film annealed at 800 °C for 1 h has a uniform and smooth surface with the desired film adhesion (**Fig. 4b.2**).

**Fig. 5.** represents the microstructural evaluation of the film when annealed in an oxygen-free, inert Ar environment. Unlike Pt/AlOx annealed at 800 °C for 1 h in air, recrystallization was incomplete when Pt/AlOx annealed at 800 °C for 1 h in Ar, as shown in **Fig. 5a.** Compared to air annealing, a completely inert Ar environment during annealing retarded the process of recrystallization. As a result, the formation of defect-free recrystallized regions was slower. However, **Fig. 5b** shows that grain size significantly improved, and recrystallization took place after annealing the film for 3 h in Ar at 800 °C. The morphology appeared well crystallized and dense. **Fig. 5c**  illustrates that when the sample was annealed at 1000 °C for 1 h in Ar, the microstructure shows a discontinuity with some abnormally grown large grains and some smaller matrix grains which may be due to inhomogeneous thermal activation effect at different locations; thus, restraining the normal grain growth process [52]. Under these conditions, some grains preferentially nucleate by taking a lead and grow in favorable sites. As a result, a huge difference in grain size is observed, resulting in an uneven nucleation process. This demonstrates that the recrystallization of Pt at a higher temperature can be uncontrolled in an inert atmosphere. Moreover, abnormal grain growth leads to the formation of larger hillocks causing rapid diffusion of atoms from the surrounding area. The large flux of Pt atoms towards hillocks results in a lack of materials in the vicinity giving rise to poor adhesion/failure of Pt film [53,54]. Such phenomena and the evolution in grain microstructure have a detrimental effect on practical applicability.

* 1. **Electrical properties**

TCR of a metallic thermistor film can be influenced by factors including grain size, compactness of the grains, entrapped sputtering gas, existing impurities, porosity, homogeneity, and roughness of the film [55,56]. Any of these factors are responsible for influencing the TCR of a thin film compared to the TCR of pure elemental bulk material. According to the Callendar-Van Dusen equation, the TCR for Pt resistance thermometers is 0.359 %/°C in the range of 20-50 °C from Eq. 2 [57]. TCR is strongly by the annealing time and temperature. Therefore, in the present work, the TCR of thefilms annealed at 500 °C to 900 °C for 1 h in air are evaluated. For as-deposited Pt/AlOx films the TCR is 0.228 %/°C. The lower value of TCR for the as-deposited film can be attributed to the small grain size of the film. **Fig. 6a** shows the variation of TCR and grain size as a function of annealing temperature. As the annealing temperature is increased, both TCR and grain size also increase. This may be explained by the evolution of film microstructure during the recrystallization process driven by annealing, as discussed previously (**Fig. 3**). When depositing Pt/AlOx film by sputtering, many lattice defects such as dislocations or grain boundaries are generated in as-deposited films. By annealing as-deposited films at high temperatures, thermal energy in the system generally removes the defects to reduce the free energy of the system by activating the mobility of the Pt atoms and initiating the recrystallization process, resulting in increased TCR. However, for films annealed at 900 °C, the grain size was not significantly different than that of films annealed at 800 °C. The insignificant change in TCR for films annealed at 900 °C can be correlated with the diminishing increase in grain size.

The resistivity for annealed Pt/AlOx films at different temperatures in air and Ar as a function of annealing time is presented in **Fig. 6b**. The as-deposited Pt/AlOx film has a resistivity of 156 Ω·nm. In the as-deposited film, the resistivity arises from structural imperfections such as impurities, intragranular defects, magnetic disorder, nano-grained microstructure, dislocations, grain boundary scattering, and surface scattering. [58]. Upon annealing samples at 600 °C and 800 °C, the resistivity decreased drastically with an increase in annealing duration. The reduction in resistivity between as-deposited and annealed films is directly correlated with enlarged grain size after annealing for a longer duration, evident from SEM micrographs (**Fig. 4**). It should be noted that the drop in resistivity is slower at 600 °C than 800 °C. This difference can be attributed to the rate of grain growth at 800 °C being higher than that at 600 °C, resulting in improved conductivity of the film 800 °C annealed film. Microstructural observation confirmed the grain growth process for Pt above 600 °C was fast and that grain-size enhancement resulted in a film with well-connected grains when annealed at 800 °C for 1 h in air (**Fig. 3c-e**). At 600 °C, when the sample was annealed for 1 h in air, the resistivity was 127 Ω·nm whereas after annealing for 3 h and 6 h at 600 °C, the resistivity remained constant at 122 Ω·nm. At 600 °C, most grain growth takes place rapidly (< 1 h), then the grain growth plateaus. The enlargement of grains beyond a certain time at a particular temperature is not energetically favorable and can be correlated with various factors such as slow grain boundary movements or pinning by thermal grooves [59,60]. It should also be noted that films annealed at 600 °C for 3 h in air had a resistivity of 122 Ω.nm whereas when films annealed at 800 °C for 1 h had a resistivity of 117 Ω nm. This observation suggests that at 600 °C, a larger grain size was not reached despite the longer annealing time (3 h vs 1 h at 800 °C). Low annealing temperatures (< 800 °C) have insufficient activation energy to reduce grain boundaries and dislocation density on practical time scales (< 3 hrs.). Therefore, at 600 °C, stagnation in grain growth is reflected in the resistivity values. The most significant change in resistivity was observed when the film was annealed at 800 °C for 3 h in Ar. The lowest resistivity of 112 Ω. nm was obtained from this film, very close to the bulk Pt resistivity, 106 Ω·nm [61]. The resistivity of a film strongly depends on electron scattering at defects in a thin film such as grain boundaries. Small grain size films have a high density of grain boundaries and high resistivity, evident in the as-deposited films. The recrystallization, nucleation, and growth process induces the formation of larger grains by reducing grain boundary density, leading to lower resistivity in the film [62]. In contrast to the previous observation, the resistivity of the film increased to 129 Ω·nm when annealed at 1000 °C for 1 h in Ar. Even though annealing was performed at a higher temperature, the resistivity increased drastically. A different mechanism must be responsible for such deteriorated performance of the film. Discontinuities in the film due to larger hillock formation at this temperature probably are one of the major reasons for such degraded electrical properties [63]**.** Additionally, plastic deformation at the top layer at a high temperature can be responsible for such increase in resistance [29].

The comparative graph of TCR values annealed at 600 °C, 800 °C, and 1000 °C in different gaseous conditions with increased annealing time is displayed in **Fig. 6c**. TCR of the films are strongly influenced by the annealing duration and recrystallization process. Bigger grain size and low resistivity are indicative of higher TCR. The Pt/AlOx film annealed at 800 °C shows a higher TCR than that of the film annealed at 600 °C because the grain growth kinetics are intensified at higher annealing temperatures, resulting in a larger grain size at 800 °C. The highest TCR value of 0.308 %/°C for Pt/AlOx is achieved after annealing in air at 800 °C for 1 h. TCR of the film annealed at 800 °C for 3 h in Ar was 0.308 %/°C, as well. A TCR of 0.320 %/°C was achieved for the film annealed at 1000 °C for 1 h in an Ar environment, but this film exhibited uneven grain growth, as illustrated in **Fig. 5c**. Hence, controlling the annealing temperature and duration provides a convenient way to control the proper growth mechanism, grain boundary movement, and configurations of recrystallized atoms. Therefore, choosing the correct balance between these interactions of annealing temperature and duration is essential to achieving the highest TCR and high-quality films.

* 1. **Structural and compositional analysis**

The crystalline structures of the as-deposited and annealed (800 °C in air for 1 h) Pt films were investigated by XRD measurements, as shown in **Fig. 7**. The diffractogram of **Fig. 7a** confirms the face-centered cubic structure of Pt crystal (JCPDS Card 04-0802). The peaks for as-deposited and annealed samples around 39° and 86° correspond to the preferential orientation of Pt film towards Pt (111) and Pt (222), belonging to the same family of planes. Pt (111) is the favorable state with minimum surface and interfacial energy [64]. **Fig. 7b** is the magnified XRD spectra for as-deposited and annealed films around Pt (111) peak region, showing a narrowing of the peak after annealing, as well as a slight shift in 2q to a higher range. The sharper peak corresponding to the annealed sample indicates better crystallinity compared to as-deposited samples whereas the shift in the peak can be correlated with a change in lattice strain during the annealing process due to the removal of micro stresses resulting from different points and lattice defects like dislocations, vacancies, voids or interstitial atoms [65]. Moreover, the XRD study of the as-deposited and annealed sample showed that the deposited Pt film was free of any oxygen contamination. No evidence of PtO peak is observed from the XRD data of as-deposited and annealed film indicating that annealing at 800 °C in air for 1 h did not cause oxidation of the Pt layer.

With the intention to analyze the electronic state of the elements in AlOx (10 nm) and Pt/AlOx film, XPS was performed. The as-deposited AlOx interlayer was evaluated, prior to Pt deposition. The high resolution XPS peaks of Al2p and O1s, corresponding to AlOx film is shown in **Fig. 8a-b.** In the Al2p spectrum (**Fig. 8a**), Al2p peak is located at ≈ 74 eV confirming the formation of AlOx [66,67]. In **Fig. 8b**, O1s peak is deconvoluted in two components, centered at ≈ 531 eV and 532.5 eV, indicating the presence of Al-O and Si-O bonds. Commercial software was used to estimate the atomic ratio between Al2p and O1s which came out to be ≈ 0.38 (for Al2O3 stoichiometric, the Al/O ratio is 0.67).

**Fig. 8c-d** shows Pt4f and O1s peaks obtained from Pt/AlOx. As shown in **Fig. 8c**, Pt4f peak has an asymmetric doublet with two well-separated spin-orbit components, Pt4f5/2and Pt4f7/2. Doublet spacing is ≈ 3.35 eV, for both as-deposited and annealed (800 °C in air for 1 h) samples [68]. This characteristic doublet indicates that Pt is in the metallic state. Additionally, no prominent O1s peak in **Fig. 8d** was observed, confirming the absence of oxygen in the Pt film before and after annealing. XPS analysis supports the XRD data that the Pt did not undergo oxidation during annealing at 800 °C for 1 h in air. It should be noted that the partial pressure of O2 during annealing was maintained at 17.6 kPa (atmospheric pressure = 83-86 kPa) which is not favorable to form solid PtO on the surface of the film at 800 °C, as validated by the Ellingham diagram of Pt/PtO [69,70].

* 1. **Inspections of meanders**

From the study, we found three samples to have the highest TCR values: films annealed at 800 °C for 1 h in air (0.308 %/°C), 800 °C for 3 h in Ar (0.308 %/°C), and 1000 °C for 1 h in Ar (0.320 %/°C). Given the similar TCR for all these samples, microscopic investigations of the lithographically patterned meanders are also important to investigate their state after high-temperature annealing. Optical microscope images of meander style-1 and meander style-2 thermistors for the aforementioned samples are shown in **Fig. 9a.1-c.1** and **Fig. 9a.2-c.2**, respectively. When the samples were annealed at a higher temperature, meander style-2 structures show fewer surface deformations whereas the meander style-1surfaces are relatively rough, which can be attributed to the narrower wire width of the meander style-2 (2 µm) compared to the meander style-1 (11 µm). Meander style-1 and meander style-2 structures, when annealed at 800 °C for 1 h and 3 h in air and vacuum, respectively, show relatively smooth appearance and continuity. Thermistors annealed at 1000 °C for 1 h in Ar showed a roughened surface with blistering.

Retaining a smooth film at a high temperature is very challenging for smaller structures as the annealing process generally induces significant changes in the morphology of a film. **Fig. 10** represents low and high magnification SEM images of meander style-2 structures corresponding to the samples annealed at 800 °C for 1 h in air (**Fig. 10a.1-2**), 800 °C for 3 h in Ar (**Fig. 10b.1-2**), and 1000 °C for 1 h in Ar (**Fig. 10c.1-2**). Formation of Pt crystallites (hillocks) was less for the sample annealed at 800 °C for 3 h in Ar than the sample annealed at 800 °C for 1 h in air. Large hillocks can affect the performance of thermistors more than many small hillocks. The release of thermal stress generated during annealing is considered the primary reason for generation of hillocks [71,72]. The hillock formation is suppressed significantly when the film was annealed in an Ar environment, which is evident in **Fig. 10b.1-2**. High surface diffusion of Pt atoms during annealing in presence of oxygen can be responsible for generating a greater number of hillocks compared to the sample annealed in an Ar environment [73,74]. It is interesting to observe that when annealed at 1000 °C for 1 h in Ar, meander style-2 shrunk and deformed, as shown in **Fig. 10c.1-2**. This film also detached from the substrate. At 1000 °C, thin films agglomerate into a collection of beads caused by the high surface-to-volume ratio of the thin films. Particularly, the surface area of Pt is reduced through a capillarity process as the surface diffusivity of Pt is very rapid at 900 °C ( ≈ 1.5 × 10-5 cm2/s) [16,75]. Fast diffusion of Pt atoms results in formation of hillocks in some places whereas discontinuity is created at the edges due to lack of material. The driving force of such discontinuity is the energy difference in chemical potential between atoms on a curved surface and a planar surface [74]. Similar observations concerning the degradation of the film due to agglomeration and hole formation have been reported for gold, silver, and other metallic thin films [73,75]. Although the Pt/AlOx film annealed at 1000 °C in Ar for 1 h exhibited a very good TCR, line edge roughening, microstructural instability, and poor adhesion to the substrate (**Fig. 10c.2**) is unacceptable for device use.

The microstructure of Pt films not only depends upon annealing conditions, but also on the presence of an interlayer between the film and the substrate. **Fig. 9d.1-2** represents optical microscope images of meander style-1 and meander style-2 structures consisting of Pt without an AlOx interlayer after annealing at 800 °C for 1 h in air. In **Fig.** **9.d.1**, formation of blistering is evidence of adhesion loss to the substrate whereas irregular and distorted edge of meander style-2 is observed in **Fig.** **9.d.2**. SEM images in **Fig. 10d.1-2** represents microstructure of meander style-2 without an AlOx interlayer. Outward bulging at the edges can be observed which caused the film to peel off from the substrate. By incorporating an AlOx interlayer, it was possible to preserve adhesion of the Pt film even after annealing at 800 °C in air or Ar, as represented in **Fig. 10a.1-2** and **Fig. 10b.1-2**.The adhesion loss of Pt film without interlayer may be due to formation of Pt2Si or PtSi at the interface as a result of Si diffusion from the SiNx [76,77].Additionally, the large mismatch in coefficient of thermal expansion (CTE) of Pt (8.8 x 10-6/°C) and Si (2.6-3.3 x 10-6/°C) is a source of stress during annealing [78]. To release the stress in the film, Pt atoms move towards the surface through grain boundaries generating a large number of hillocks which can eventually cause rapid failure of the film [79]. When annealed at 800 °C for 1 h in air, the TCR of a Pt film was 0.283 %/°C whereas the TCR of a Pt/AlOx film was 0.308 %/°C. The AlOx interlayer not only enhances adhesion and mechanical stability of Pt with the substrate, but it also improves the TCR of the structure. An interface component also plays a significant role in the grain growth process. With the presence of an intermediate AlOx layer, the grain size became larger compared to the film without an AlOx interlayer, as shown in **Fig.** **10a.2** and **Fig.** **10b.2** compared to **Fig. 10d.2**. Without the presence of AlOx as a diffusion barrier, Si atoms diffuse into the Pt layer resulting in the formation of undesired phases causing poor adhesion and affecting grain growth [80,81]. Moreover, the difference in CTEs between Pt and SiNx can possibly interrupt the recrystallization process leading to smaller grain size [82]. Hence, the appropriate selection of the intermediate layer as well as its deposition process promotes suitable grain growth processes and ensures the protection of the Pt layer against diffusion during annealing.

1. **Summary and conclusions**

Incorporation of Pt thermistors in uncooled microbolometer arrays utilizing VACNT absorbers is certainly a technological challenge, as the fabrication requires the growth of VACNTs at high temperatures. In the present work, Pt/AlOx thermistors were deposited on SiNx/SiO2/Si substrate using magnetron sputtering, followed by an extensive investigation to study the effect of annealing conditions on surface morphology and electrical properties of Pt/AlOx. It was demonstrated that grain size and TCR increased with an increase in annealing temperature and maximized when annealed at 800 °C for 1 h in air. Surface oxidation did not take place as confirmed by XPS. XRD analysis showed the presence of preferentially oriented Pt (111) in the annealed film. For finding optimal annealing condition, short annealing times at high temperatures (800 °C/1000 °C) were compared with longer annealing times at lower temperature (600 °C). A short annealing time at 800 °C for 1 h in air is sufficient to achieve desired electrical properties compared to prolonged annealing at 600 °C. However, for films annealed in an Ar environment at 800 °C for 1 h, the grain growth slowed down and grain size became larger only when annealed for 3 h at 800 °C. At a higher temperature (1000 °C for 1 h in Ar), deformation of meander style-2 was observed due to abnormal grain growth and high surface diffusivity of Pt. Though annealing at 800 °C for 1 h in air and 3 h in Ar resulted in similar resistivities (117 Ω.nm and 112 Ω.nm, respectively) and TCRs (0.308%/°C), surface roughening and hillock formation were not prominent when annealed at 800 °C for 3 h in Ar. Additionally, the AlOx interlayer between Pt and SiNx/SiO2/Si was found to be beneficial in improving film adhesion, grain growth, and TCR.

Based on the experimental results, Pt/AlOx thermistors can be successfully integrated into uncooled microbolometer arrays incorporating VACNT absorbers grown at elevated temperatures (600 °C to 800 °C). Comparing the TCR and operating temperature range of the Pt/AlOx structure to previously reported Pt-based thermistors, the performance of Pt/AlOx thin films explored in this work is comparable to or better than other thermistors [16,18,83,84].

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