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Microhotplates with TiN heaters

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ABSTRACT

Titanium nitride (TiN) has been investigated as a heater material for microhotplates and microreactors. TiN is available in many CMOS processes, unlike many other microheater materials. In addition, TiN has a very high melting point (2950 °C) meaning that it is stable up to higher temperatures than platinum (Pt) and polysilicon. For the first time, TiN is tested inside a conventional membrane of LPCVD silicon nitride (SiN). Two types of sputtered TiN are considered: high stress and low stress. Their performance is compared with that of e-beam evaporated Pt. The maximum average temperature of TiN heaters is 11% higher than those of Pt, and reaches over 700 °C. Failure of the TiN heaters is due to rupture of the membrane. Failure of the Pt heater is due to electro-stress migration. For high-stress TiN, the temperature coefficient of resistance is almost constant and close to that of Pt, making the material very suitable for temperature sensing. In the case of low-stress TiN the temperature coefficient of resistance (TCR) becomes nonlinear and changes sign. The large differences between the materials are explained by the grain structure. The different grain structures are related to the sputtering parameters according to the Thornton model.

1. Introduction

MEMS microhotplates generate high temperatures at low power consumption and exhibit a fast thermal response time. For this reason, they are often used for gas sensors [1,2], membrane-type microreactors [3–9], materials characterization [6–8], and infrared emitters [1,10]. A microhotplate generally consists of a thin film heater coil, wire, or meander which is suspended within a silicon rim for thermal isolation. Often, the heater is supported by a membrane containing low-stress silicon nitride (SiN). The average temperature of the heater is determined from the change of the electrical resistance of the heater or of an additional wire used as temperature sensor.

A popular material for hotplate heaters is platinum (Pt) [2,4,10–14]. This metal can handle large current densities, is highly resistant against oxidation, and can operate up to 550–600 °C without structural changes [4]. It has a melting point of 1768 °C. In addition, it is very suitable for temperature sensing because of its stable temperature coefficient of resistance (TCR). The disadvantages of Pt are its temperature limit, and the fact that it is nonstandard in CMOS fabrication technology.

For these reasons, hotplate heaters have been developed from various other materials to have a complete compatibility with CMOS processes or to increase the temperature of operation of hotplates for applications in micro-reactors, micro-SOFCs and micro-IR emitters for instance. Different materials available in CMOS processes have been evaluated until now as heating elements. The most obvious material that was first used as heater was polysilicon (poly-Si) [1,15,16]. Poly-Si is useful up to 550 °C, above which its resistivity becomes unstable [17,18]. Lately, heaters based on monocrystalline silicon were developed for CMOS hotplates but these MOSFET type heaters still suffer of a lack of stability at high temperature [19,20]. Heaters for higher temperatures, up to 1000 °C, have been made of tantalum silicide (Ta_5Si_3) [21]. Work has been also reported on the use of tungsten based heaters in a SOI technology with satisfying results for the operation of hotplates for metal-oxide gas sensors (300–500 °C) but limitations in their stability when operated at temperatures higher than 600 °C [22,23]. However, this material is not so widely available. It is possible to reach over 1000 °C by using tin oxide doped with antimony [24], and by poly-SiC [25]. Unfortunately, those materials are still nonstandard in CMOS technology.

As an alternative material, we have investigated titanium nitride (TiN) [26]. Thin layers of TiN are widely used in CMOS metallization processes as a diffusion barrier, so the tools for deposition are

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widely available. The element Ti has little or no influence on CMOS transistor operation, even when used in the frond end of the process [27]. In addition, TiN has the potential to reach high temperatures because of its very high melting point (2950 °C for bulk material). It can have a low electrical resistivity (20 μΩ cm [28]), depending on the conditions of deposition. Incidentally, TiN has been used as a heater before [29], but not in hotplates. For this application, TiN has the additional advantage that the residual stress can be tuned over a wide range. This increases the strength of the heater. It also has a very moderate heat conductivity (15 $W\,m^{-1}\,K^{-1}$ for bulk material). This promises low conductive heat losses through the connecting wires.

This paper presents the fabrication of hotplates with two types of TiN. Their performance is characterized with respect to resistivity, TCR, maximum heater temperature, and response time. The hotplates are compared to Pt hotplates of the same geometry. The observed properties of TiN are related to the deposition conditions and to its grain structure.

2. Design, fabrication, and characterization

The layout of our hotplates is quite classical and is shown in Fig. 1. It consists of a metallic heater coil of 0.33 mm wide and 210 nm thick, which is sandwiched in between a membrane of silicon nitride of 1 mm wide and 1 μm thick. This construction gives an electrical and chemical isolation from the atmosphere. Our reactor application requires a closed membrane and low thermal losses to the rim, so silicon bridges as in [1] are not incorporated. The heater is equipped with four contacts to enable accurate measurement of the resistance and power dissipation of the hot zone. Hotplates of such a design loose heat mainly by natural convection; radiation and conduction are much smaller [30]. This means that the thermal resistance towards the environment depends mainly on the geometry and is quite independent of the heater material.

The hotplates are fabricated on (1 0 0) silicon wafers of 100 mm wide and 525 μm thick. These are covered by 200 nm wet thermal oxide and 500 nm low-stress SiN deposited by LPCVD at 850 °C. The heaters are deposited and patterned, and covered by second layer of 500 nm low-stress LPCVD SiN. Contact holes to the bond pads are opened by plasma etching. On the back side, windows are opened in the nitride to form a mask for etching the silicon substrate. This etch is performed in a solution of potassium hydroxide (KOH, 33 wt.%, at 85 °C) to release the membranes. The result is shown in Fig. 2.

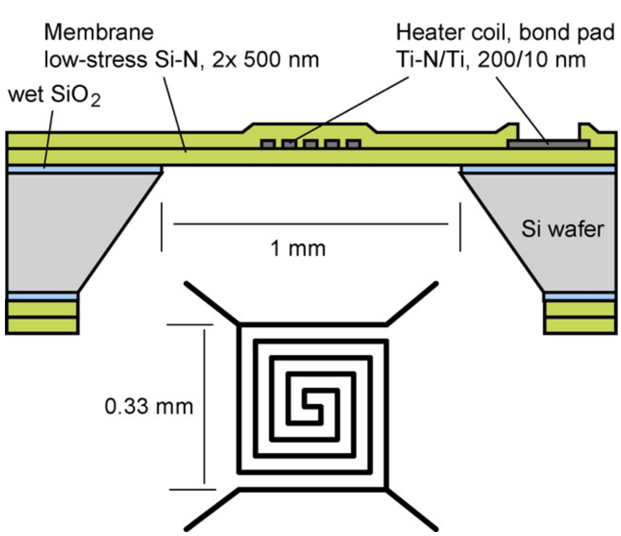

Fig. 1. Schematic cross-section of the hotplates and top view of the heater coil [26].

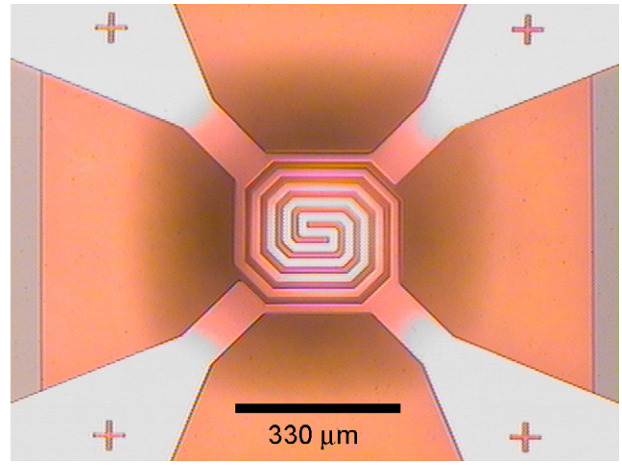

Fig. 2. Pt hotplate during operation under an optical microscope. The shadow is due to thermal buckling [26].

The heaters of TiN are made by reactive sputtering in a Trikon Sigma dc magnetron reactor. The TiN has a thickness of 200 nm and is deposited on 10 nm Ti for a better adhesion to the SiN. Two different types of TiN are sputtered: the standard one with high residual stress, and one with low stress. The major sputtering parameters are given in Table 1. The stress in the TiN is determined from the wafer curvature with a Tencor FLX 2908. The TiN layers are patterned by plasma etching with a chlorine-based chemistry similar to that used for etching aluminum. The contact windows are opened by a plasma etch which is fluorine-based. In both etches, the selectivity is low and an end point detection mechanism is essential. The Pt heaters consist of 185 nm Pt e-beam evaporated on top of a 15 nm Ta adhesion layer. They are patterned using a lift-off process.

The microstructure of the TiN is examined by using an FEI CM30T transmission electron microscope (TEM). The electrical characterization is done with an Agilent 4156C parameter analyzer. With this apparatus, the sheet resistance is measured on Van der Pauw structures. The spirals are heated up to failure by increasing the voltage, in 100 steps of 1 s each. The maximum applied voltages are 14, 100, and 9 V for the heaters of high-stress TiN, low-stress TiN, and Pt, respectively. The temperature of the heaters is estimated from the resistance of Pt and from the power dissipation. It is assumed that the resistance of Pt increases linearly with the temperature, with a TCR of $2.08 \times 10^{-3}\,{}^{\circ}C^{-1}$. This value is based on previously calibrated hotplates with the same Pt layer [31,32]. It also corresponds with the experience that Pt thin film heaters start to glow visibly at 600 °C [4]. More accurate measurements could be obtained from experiments in a furnace or on a hot chuck, from IR emission spectroscopy, or by using miniature thermocouples [1,21,33]. The time constant is evaluated by using a waveform generator and an oscilloscope. First, a block-shaped voltage wave of 10 Hz is applied, alternating between 0 V and halfway the maximum input voltage. Meanwhile, the resistance changes are monitored. Second, the small-signal bandwidth is determined by sweeping the

Table 1
Major parameters for sputtering of TiN with high and low residual stress

Parameter	High stress	Low stress
N2 partial pressure (Pa)	0.41	1.8
Total pressure (Pa)	0.53	2.3
Power (kW)	12	0.5
Substrate temperature (°C)	350	350
Bias voltage (V)	0	0