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Strain sensitive Pt–SiO_2 nano-cermet thin films for high temperature pressure and force sensors

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ABSTRACT

The strain sensitivity, i.e. the resistivity change due to mechanical strain of thin composite nano-cermet films of Pt–SiO_2 was investigated. We prepared films with a thickness of 400 nm by means of co-sputtering processes at substrate temperatures around 400 °C. The specimens respond to uniform strain ($\varepsilon = 0.2‰$) with gauge factors up to 18. These gauge factors remained high at least up to 250 °C in air and also after further annealing up to 600 °C in vacuum. Therefore we state these functional films might be suitable for high temperature pressure and force sensors. The films have a relatively high film resistivity of some MΩ/sq and exhibit temperature coefficients of resistance (TCR) in the range of −2000 ppm/K up to −600 ppm/K. X-ray diffraction revealed a single crystalline fcc platinum phase while transmission electron microscopy proved a typical granular structure of the films. Pt-clusters sized 5–10 nm are embedded in an amorphous insulating matrix of silica. The composition of such nano-cermet films displaying high gauge factors is approx. 40 at% Pt, 20 at% Si and 40 at% of O.

1. Introduction

For a wide range of applications there is a great demand for pressure and force sensors operating at elevated temperatures. Perhaps the most challenging one is the request for cost effective pressure sensors for combustion engines. The direct cylinder pressure monitoring of the combustion process will provide signals to further optimize fuel efficiency and reduce emission. Beside this a lot of processes in chemical production and engineering are requesting pressure and force sensors at higher working temperatures as well. The application of thin functional films as strain gauges on sensor bodies provides a commonly accepted concept. However the well-established thin films sensors of today, i.e. equipped with CrNi-functional layers have some major drawbacks. Their strain sensitivity (gauge factor, GF) is often very small and their operation does not allow temperatures in excess of 200 °C. Hence, there is still a need for a thin film sensor material having a high gauge factor and a high stability at elevated temperatures. The materials have to be thermodynamically stable and very corrosion resistant.

Researchers have identified some piezoresistive materials as possible candidates for high temperature applications. A review by Wrbanek et al. [1] covers AlN (GF ~ 15), ITO (GF ~ −11) and Al-ITO (GF ~ 8). Reports on TaN (GF ~ 4) [2] and Pt-ITO (GF ~ −26) [3] demonstrate thin sensory films capable of withstanding temperatures as high as 900 °C. The cermet system of Cr–SiO [4,7] provides GF ~ 9 while the system NiCr/Au–SiO_2 [5] shows GF ~ 10. Even gauge factors of 38 were published in a paper of 1972 by Meiksin et al. [6] for the Au–SiO cermet. Fricke et al. [8] published the possible use of sapphire as a substrate for pressure sensing bodies together with platinum functional films. However, none of the mentioned thin films has acquired industrial importance so far. In our study we start with materials that are well known for their high temperature potential. That is platinum with its extraordinary resistance to oxidation and corrosion and its well-approved usage for instance as Pt-100 temperature sensor. On the other hand we select SiO_2, being a dielectric also known as a good choice for elevated temperatures. We are aiming at a composite functional film for pressure and force sensors under harsh environmental conditions. This functional layer should have a high sensitivity for strain, a controllable temperature coefficient of resistance and most important a very good stability over time and environmental influences.

2. Experimental methods

Thin films of Pt–SiO_2 are co-sputtered from two targets positioned perpendicular to each other (see Fig. 1). The silica target

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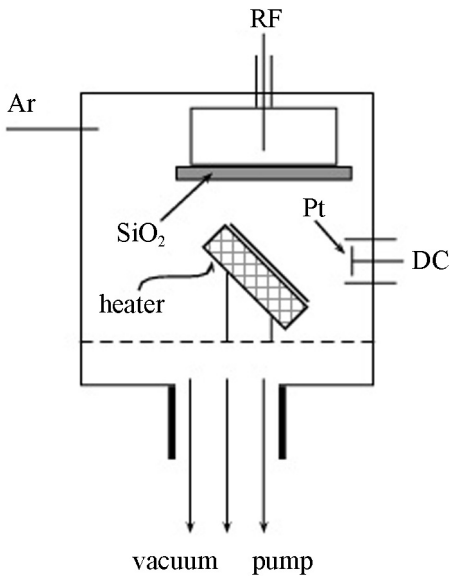

Fig. 1. Schematic diagram of the sputtering device with targets of Pt and SiO_2 at right angles. The samples are heated during the deposition process of the nano-cermets.

with a diameter of 5 in. was RF (13.56 MHz) magnetron sputtered while a small Pt-target (2 in. diameter) was simultaneously sputtered under DC magnetron conditions. The target to substrate distance was about 7 cm (SiO_2) and 14 cm (Pt) respectively. The majority of the films was deposited onto substrates of Si-wafers having a thermally grown SiO_2 insulation layer with a thickness of 300 nm. The substrates, tilted at an angle of 45° to each of the targets have dimensions of 12 mm × 30 mm and a thickness of 0.38 mm. The test pattern of a thin film resistor consists of a U-shape and four conductor paths, which were structured with the help of a shadow mask (Fig. 2). During deposition the substrates were heated to a maximum of approx. 400 °C by means of a heat radiating device placed behind the samples. In general two substrates were placed in one deposition run. Due to the small platinum target the uniformity of deposition is very limited. The other relevant sputtering parameters are listed in Table 1. After breaking the vacuum the samples were subsequently equipped with a contact layer of 50 nm thick CrNi solely on the conductor paths. For the determination of the elemental composition by EDX alumina substrates were used while XRD requires sapphire specimen. Various deposition runs were conducted with different powers on the two targets.

The gauge factor is measured with a fixture that clamps the sample on one side while it may be bent by applying a force on the other side. The force to bend the sample is realized with a mass

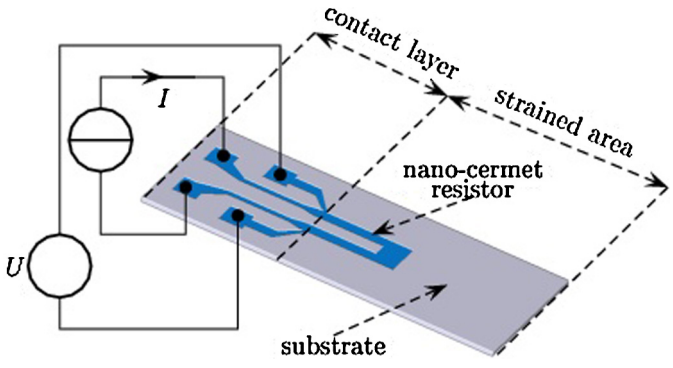

Fig. 2. Schematic layout of a sample. The U-shape resistor is made of the Pt–SiO_2 composite while the conduction paths and pads carry a layer of CrNi on top.

Table 1
Parameters of the co-sputtering process.

Background pressure	<2e−6 mbar
Working pressure	5e−3 mbar
Gas flow	20 sccm Ar
Substrate temperature	Up to 400 °C
Power SiO_2-target	325 W (5″) RF magnetron
Power Pt-target	60 W (2″) DC magnetron
Sputtering time	15 min

of 8 kg pressing the sample with a yoke onto the fixture. The sample is then repeatedly released and bent onto the fixture with a known radius of curvature (Fig. 3). During bending we have a homogeneous strain on the samples surface, that is determined by the radius (r = 842 mm) and the substrate thickness (d = 0.38 mm) to ε = 0.22‰ (per mill). Four spring-loaded pins, that withstand temperatures up to 250 °C, are connecting the resistor electrically to a Keithley 2612 sourcemeter. The gauge factor GF is then calculated taking the difference ΔR of the resistance R_0 in the released or non-loaded mode and the resistance R_{bend} in the bent or loaded mode [9].

$$\mathrm{GF} = \frac{\Delta R \cdot 2 \cdot r}{R \cdot d}$$

The gauge factor device is placed inside a temperature chamber and may be heated to a maximum of 250 °C. The precision and repeatability of the device is about ±0.5 gauge factor points. For measurements beyond that temperature a new measuring device still needs to be constructed. In this way, the gauge factor may be determined at different temperatures. If the samples resistance is taken in the non-loaded mode alone, the temperature coefficient of resistance (TCR) may be calculated referring to the base temperature of 30 °C [9].

$$\mathrm{TCR} = \frac{R(T) - R(30\,^\circ\mathrm{C})}{R(30\,^\circ\mathrm{C}) \cdot \Delta T}$$

After a first annealing step at 180 °C (3 h) in ambient air the samples were measured. Subsequent annealing steps were performed either at 300 °C in ambient air or at a maximum of 600 °C in a vacuum furnace. The samples were then measured again to evaluate their strain sensitivity. Structure and morphology of the thin films were analyzed by means of X-ray diffraction (XRD) and transmission electron microscopy (TEM) while their elemental composition was determined using a scanning electron microscope (SEM) with energy dispersive X-ray analysis (EDX). For the XRD investigation

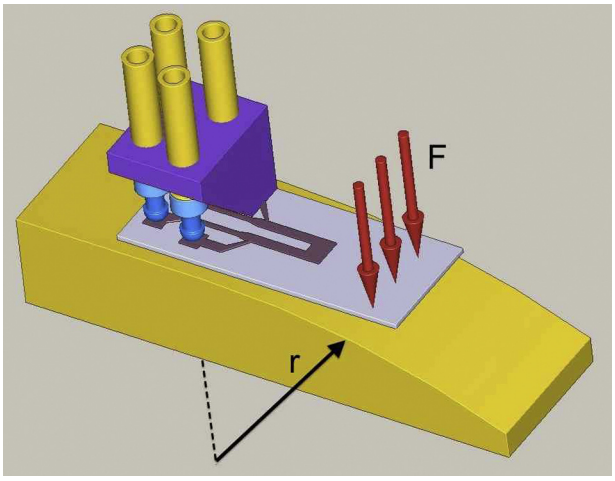

Fig. 3. Schematic of the gauge factor measurement fixture. The sample is clamped and electrically connected on the left while alternately pressed with a force F onto a device having a known radius r of curvature.