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Hydrogen microsensors based on NiO modified thin films

ABSTRACT

In this work, the surface modification of NiO thin films using Pt will be presented. The surface modified NiO thin films have been investigated with respect to structural, surface morphological and gas sensitive properties. The Pt very thin overlayers with a thickness of about 3 and 5 nm were formed on top of NiO surface by sputtering. Then the modified NiO films have been analysed by TEM, AES and AFM. The electrical responses of the NiO-based sensors towards H₂ concentration (500-5000 ppm) have been considered.

INTRODUCTION

Nickel oxide, which belongs to metal oxides, is usually taken as a model for *p*-type materials and is an attractive material well known for its chemical stability as well as for its excellent optical and electrical properties. In particular, the field of gas-sensing has benefited from the production of prospective materials characterized by a high surface-to-volume ratio [1]. Nanostructured materials exhibit more attractive properties compared with conventional materials, such as very small particle size, large exposed surface areas and high surface energy. The gas-sensing properties of metal oxides are more or less related to the material surface, its high porosity and a nanostructure with small particles. Also, these properties can be essentially improved by doping of their surfaces by catalytic metals. These efforts have been made to investigate the prospective thin film materials based on metal oxide, but there is no available information about nanostructured films with surface modification for gas detection.

EXPERIMENTAL

The NiO films were deposited by dc reactive magnetron sputtering from a Ni target in a mixture of oxygen and argon. A sputtering power of 600 W was used. Both the inert argon flow and reactive oxygen flow were controlled by mass flow controllers. The relative partial pressure of oxygen in the reactive mixture O₂-Ar was 20%. The total gas pressure was kept at 0.5 Pa. Details of these sputtering deposition conditions have been described elsewhere [2]. The NiO films were prepared onto unheated silicon and KCl for physical characterization. On top of these base films, thin Pt overlayers (3 and 5 nm thick) were deposited through a suitable photolithographic mask by magnetron sputtering. In order to stabilize the properties, all films have been annealed in a furnace at 600°C in dry air for 2 hours. The structural features of the films were investigated by means of TEM. Tecnai 20 S-TWIN transmission electron microscope has been operated at 200 kV. It is equipped with electron energy loss spectroscopy (EELS) and energy dispersive X-ray (EDX) facilities for high resolution chemical analysis. Auger electron spectroscopy (AES) measurements were carried out by the spectrometer ASC 2000 (Riber). The electron gun was operated at 3 kV, the electron beam had a spot diameter approximately 10 μm and the current was 1 μA. The surface morphology was observed by atomic force microscopy (AFM) using a Topometrix Discover TMX 2000 under normal air conditions. In our case a 70 μm x,y,z linear scanner with a minimal z-resolution of 0.2 nm was used. The NiO-based sensor devices prepared over alumina substrates

were electrically characterized as gas sensors by using a fully automated system

RESULTS

Identification of the deposited films was based on the observed electron diffraction patterns. The diffraction pattern is of a continuous ring type indicating a polycrystalline film. The grains are not oriented homogeneously, but into certain prominent directions. We found the reflections from the same oriented grains in the diffraction patterns. TEM observations (Fig. 1) both of unmodified and Pt-modified NiO films confirmed that the films were formed by nanocrystals and an amorphous phase and showed a fine-grained structure. The size of the nanocrystals ranges from a few nanometres to 10 nanometres depending on the position in the film. We revealed that the samples contain small grains that are partially bonded into clusters. According to AES measurements the presence of the Ni in the spectra recorded from Pt pad indicated that the Pt layer is either not completely closed or not homogenous in its thickness. The H₂ gas-sensing properties were considerably improved by the catalytic activity of the thin Pt overlayers.

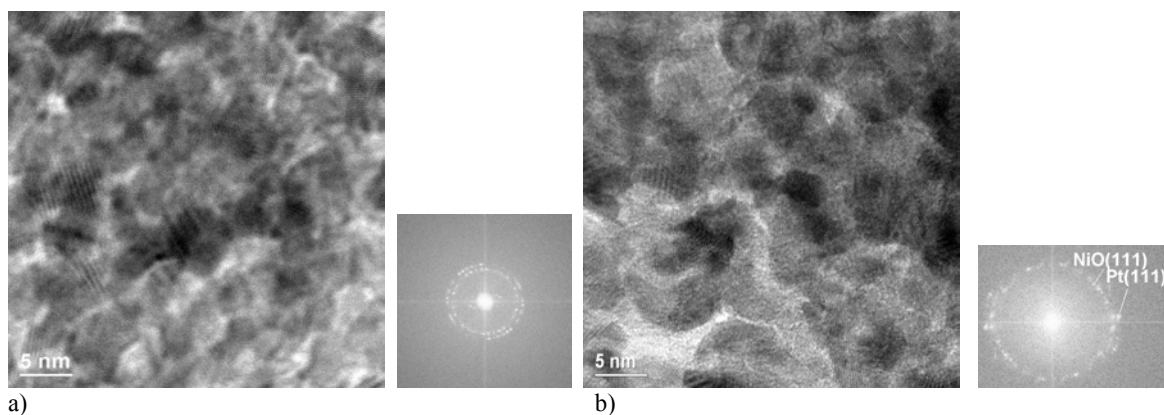


Fig. 1: TEM images of NiO film achieved after deposition (a), of Pt-3 nm formed on NiO film (b) with the related electron diffraction patterns.

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