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Post annealing effects on low temperature deposited Mn-Co-Ni-O films by radio frequency magnetron sputtering

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ABSTRACT

$Mn_{1.56}Co_{0.96}Ni_{0.48}O_4$ (MCN) thin films were deposited on amorphous Al_2O_3 substrates by radio frequency magnetron sputtering at a relatively low temperature (450 °C). The films were annealed at 450 °C, 600 °C and 750 °C for 20 min respectively. The structural property was characterized and the result indicated post annealing had important impact on crystallinity and surface morphology of the films. Temperature dependent resistivity test revealed that the MCN films possess moderate resistivity, low negative temperature coefficient and favorable characteristic temperature. The method of preparing MCN films with favorable performance at 450 °C is expected to be compatible with standard silicon industry process and has great significance for developing linear or focal plane devices with MCN thin film.

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1. Introduction

Mn-Co-Ni-O (MCN) material is a transition metal oxide with spinel structure, its formula can be written as AB_2O_4, which possesses excellent negative temperature coefficient (NTC), moderate resistivity, long term reliability, has been widely used in bolometers and uncooled infrared detectors [1–5].

For the past decades, MCN and copper doped MCN bulk materials have been studying extensively for their favorable performance, and the sintering method is a superior technique for preparing the bulk materials [6–9]. However, MCN films is more suitable for developing linear and focal plane infrared detectors. Therefore, various kinds of methods have been developed for preparing MCN films, such as chemical solution deposition (CSD) [10], pulsed laser deposition (PLD) [11], screen printing [12] and radio frequency (RF) magnetron sputtering [13]. It has great significance to develop a method to prepare MCN films which is compatible with standard silicon industry process to develop MCN films based linear or focal plane detectors. The methods of preparing MCN films mentioned above need to be annealed over 600 °C, which is much larger than the upper limit of silicon process temperature. Researchers developed different ways to solve this problem, Ji et al adopted laser molecular beam epitaxy (LMBE) technique to grow MCN thin films below 300 °C [14], Ko et al used spin spray-deposited method to prepared nano-crystalline Mn-Ni-O films at 90 °C and chemical solution deposition prepared Mn–Ni–O spinel thin films at 400 °C [15,16]. However, compared with above methods, RF magnetron sputtering method shows higher production efficiency and possesses high potential for industrial applications [17–19]. In this work, MCN films were deposited on amorphous Al_2O_3 substrates by RF magnetron sputtering at 450 °C, the structural and electrical properties of MCN films were investigated.

2. Experimental

The MCN films were deposited on amorphous Al_2O_3 substrates sized $20 \times 20\ mm^2$ by RF magnetron sputtering system operating at 450 °C. A wafer of MCN polycrystalline ceramic target sized Φ 60 mm × 5 mm was prepared via sintering method. The working pressure was 0.3 Pa. High purity (greater than99.99%) argon was applied to provide plasma, MCN films were deposited under power of 50 W with deposition time about 25 h. The as deposited MCN films were annealed at 450 °C, 600 °C and 750 °C for 20 min respectively, which were marked as S_2, S_3 and S_4, the as deposited was S_1. The resistance elements sized 500 μm ×500 μm which were patterned by photolithography process, thin layers of Cr/Au films with 30 nm and 150 nm thickness were deposited by double

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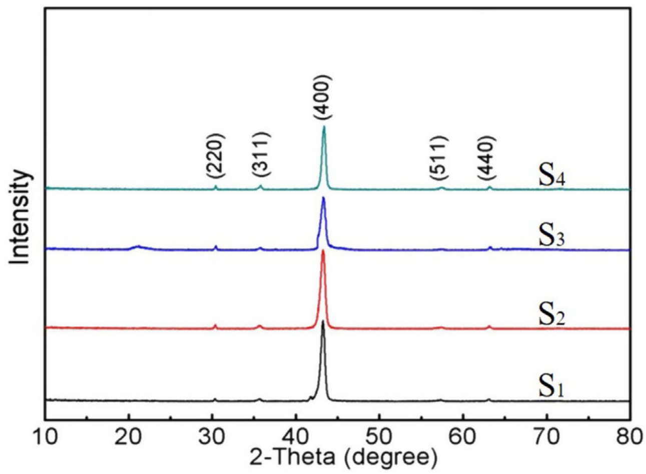

Fig. 1. XRD patterns of the MCN films with various annealing temperature.

ion-beam sputtering and patterned using lift-off technique. The phase identification of thin films was studied by XRD in the (θ, 2θ) configuration using a RigaKu D/MAX-2550 x-ray diffractometer with Cu-K_α radiation (λ = 1.5418 Å). The micrographic characteristics of the films were studied by HITACHI S-4800 field-emission SEM (FESEM). The electrical properties of films were analyzed with Keithley 2400 source meter and affiliated temperature controlling system.

3. Results and discussion

XRD patterns of MCN films annealed at different temperature are shown in Fig. 1. It can be seen that an obvious diffraction peak (4 0 0) revealed for MCN films, indicating MCN films deposited at 450 °C possess crystallization with preferred orientation. In MCN films, (4 0 0) crystal plane corresponding to the lowest surface free energy, thermal activation energy beyond surface free energy while growing films at 450 °C, causes films grow with (4 0 0) crystal plane easily. It also can be seen that the diffraction peak (4 0 0) intensity gradually decreases and then increases with the rising annealing temperature. The reason should be considered from two aspects, first, the surface free energy will be decreased through the post annealing process, the diffraction peak (4 0 0) intensity will decreases owing to the preferred orientation (4 0 0) become worse. However, when the annealing temperature further enhanced to 750 °C, the surface free energy will increase again because of the expanding surface area and preferred orientation (4 0 0) will improve [20–23].

The surface morphology of S_1, S_2, S_3 and S_4 four samples are investigated by FESEM as shown in Fig. 2, it can be seen that all samples have dense surface and well crystallization which is illustrated by XRD result. It can be seen that the grain size will be larger with the annealing temperature increase, for each film, an average of short and long diameters of a grain is calculated for more than 20 grains [24]. The average grain size of S_1 is 74.6 ± 65 nm, and the annealed films have bigger grain and better uniformity: 78.5 ± 43 nm, 109 ± 51 nm and 161 ± 60 nm for S_2, S_3, S_4 respectively. As we know, usually grains grow larger with higher annealing temperature. The surface morphology results indicate post annealed process can improve films' microstructural properties. Inset picture is the cross section of S_2 with 2 µm thickness.

In MCN material, the temperature dependent resistance can be described by small-polaron hopping model:

$$R(T) = CT^{\alpha}\exp\left(\frac{T_o}{T}\right)^{p} \quad (1)$$

where $R(T)$ is the resistance corresponding to working temperature, C is a constant, T is the working temperature, T_o is the characteristic temperature, for small-polaron nearest neighbor hopping(NNH) model, $\alpha = p = 1$, while for small-polaron variable range hopping (VRH) model, $0.25 < p = \alpha/2 < 1$ [25]. It can be seen that the resistivity of MCN films decreases with the increasing temperature as shown in Fig. 3(a), which is typical characteristic of MCN material.

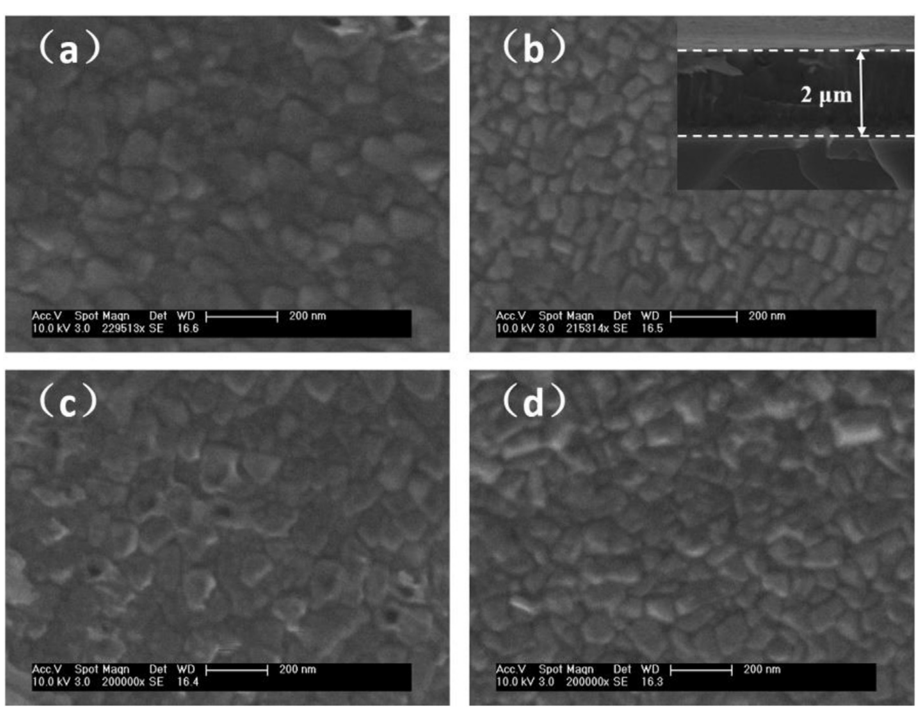

Fig. 2. SEM pictures of MCN thin films: S_1 (a), S_2 (b), S_3 (c), S_4 (d). *Inset: Cross section of SEM to measure the thickness of S_2.*

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