

## Nano Congress 2017-A Study on the Characteristics of Ni-Cr-Mn-Y-Dy Thin Film Resistors Using High Entropy Method - Ying-Chieh Lee - National Pingtung University of Science & Technology.

Ying-Chieh Lee

National Pingtung University of Science & Technology.

A Ni-Cr-Mn-Y-Nb resistive thin film was prepared in this study using DC and RF magnetron co-sputtering from Ni<sub>0.45</sub>-Cr<sub>0.27</sub>-Mn<sub>0.13</sub>-Y<sub>0.16</sub> cast alloy and niobium targets. The electrical properties and microstructures of Ni-Cr-Mn-Y films with Nb addition under various annealing temperatures were investigated. The phase evolution, microstructure and composition of Ni-Cr-Mn-Y and Ni-Cr-Mn-Y-Nb films were characterized using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), field-emission transmission electron microscopy (HRTEM). All Ni-Cr-Mn-Y-Nb films annealed at 300°C exhibited an amorphous structure. The Ni<sub>17</sub>Y<sub>12</sub> crystalline phase was observed in Ni-Cr-Mn-Y-Nb films with or without lower Nb content when annealed at 400°C. When the annealing temperature was set to 300°C, the Ni-Cr-Mn-Y films exhibited a resistivity ~480 mΩ-cm with the temperature coefficient of resistance (TCR) at ±30 ppm/°C. However, Ni-Cr-Mn-Y films with 14 at.% Nb exhibited the smallest temperature coefficient of resistance (±5 ppm/°C) with the resistivity ~585 mΩ-cm after annealing at 300°C in air.

### Introduction

The rapid development and improvement of information and telecommunication technologies as well as the expansion of digital industries are based to a substantial degree on high precision, reliable, integrated, low noise and low power consuming electrical components. The resistor is one of the fundamental components used primarily in electronic circuits. The demand for thin film resistors with low temperature coefficients of resistance (TCR) and high precision has dramatically increased in recent years. An important technical parameter of thin film resistors is the temperature coefficient of resistance (TCR). A high TCR will result in the resistance value drifting, influencing the resistor accuracy as the

temperature changes. The main factors influencing TCR include the film composition, sputtering process and annealing temperature. The film composition plays a decisive role among these three factors. Therefore, employing an appropriate method for depositing a suitable film composition is essential to obtaining high-resistance resistors with a low TCR. Extensive rapid development in high entropy alloy (HEA) films were obtained recent years by Yeh. High entropy alloys are multicomponent systems composed of elements displaying an nearly equiatomic configuration with contents ranging between 5 and 35 at.%. It was generally found that high entropy alloys form simple solid solution structures (rather than many complex phases) at elevated temperatures because of their large mixing entropies. However, it is possible to enhance the resistivity of alloy films using the high entropy alloy method. According to Matthiessen's rule:

$$\rho_{\text{total}} = \rho_{\text{defects}} + \rho_{\text{impurities}} + \rho_{\text{thermal}}$$

### 2. Experimental procedure

2.1. Ni-Cr-Mn-Y thin film Nickel (Ni), Chromium (Cr), Manganese (Mn), and Yttrium (Y) powders, as the main raw materials, were chosen to smelt the target for high-resistance thin film resistors. Alloy films were deposited onto polished alumina (Al<sub>2</sub>O<sub>3</sub>) substrates. These alumina substrates were scribed for the TCR measurement into 1.6 × 0.8 mm cell sizes using a laser. Glass and Si wafers were used as substrates for the sheet-resistance measurements and thin film thickness, respectively. These substrates were cleaned using a D.I. water-cleaning procedure and dried in nitrogen before loading into the sputtering chamber. Ni-Cr-Mn-Y thin films that were 80 nm in thickness were deposited onto the substrates using a DC magnetron sputtering system. A Ni<sub>0.45</sub>-Cr<sub>0.27</sub>-Mn<sub>0.13</sub>-Y<sub>0.16</sub> alloy with a diameter

76.2 mm was used as targets. The DC power was fixed at 50 W. The sputtering chamber was evacuated to a background pressure of  $5 \times 10^{-7}$  torr using a cryo-pump. Sputtering was performed using argon gas with a purity of 99.999% at flow of 60 sccm using mass flow controllers at a working pressure of  $3 \times 10^{-3}$  torr for gas introduction into the chamber.

**2.2. Ni-Cr-Mn-Y-Nb thin film** The niobium target was made from Nb powders, which is helpful to improved TCR and stable film structure. Ni-Cr-Mn-Y-Nb thin films 80 nm in thickness were deposited onto the substrate using a DC and RF magnetron co-sputtering system. A  $\text{Ni}_{0.45}\text{-Cr}_{0.27}\text{-Mn}_{0.13}\text{-Y}_{0.16}$  alloy and niobium with a diameter 76.2 mm were used as targets. The Ni-Cr-Mn-Y alloy target was set at the DC position. The niobium target with a diameter 76.2 mm was set at the RF position. To obtain different niobium contents in the Ni-Cr-Mn-Y film, the DC power was fixed at 50 W and the RF power was changed from 20 W to 120 W. The sputtering chamber was evacuated to a background pressure of  $5 \times 10^{-7}$  torr using a cryo-pump. Sputtering argon gas with a purity of 99.999% at flow of 60 sccm was performed using mass flow controllers with a working pressure of  $3 \times 10^{-3}$  torr.

### 2.3. Analysis

Thin films deposited onto glass plates at room temperature were subjected to transmission electron microscopy (TEM) and X-ray diffraction (XRD) studies. Thin films deposited onto  $\text{Al}_2\text{O}_3$  substrates (size: 400 mm<sup>2</sup>) were used to measure the electrical properties. The substrate temperature was 25°C. The as-deposited films were annealed at 250-400°C for 2 h, at a heating rate of 5°C/min in air. The sheet resistance  $R_s$  of the films was measured using the four-point probe technique. The thickness of the films was measured using FE-SEM (cross-section). The resistivity measured using the four-probe method was consistent with the resistivity obtained by the  $R_s$  and  $t$  samples. The TCR values of the thin films were measured using thin long strips cleaved from the substrate. Electrical contacts at the two ends

of the resistive strips were obtained by selectively coating the ends with sputtered silver. The DC resistance of the strips was measured using a digital multimeter (HP 34401A) at different temperatures (25°C and 125°C). The TCR of the thin films was measured using the following equation

$$\text{TCR} = \frac{1}{R} \left[ \frac{dR}{dT} \right] \times 10^6 \text{ ppm/K}$$

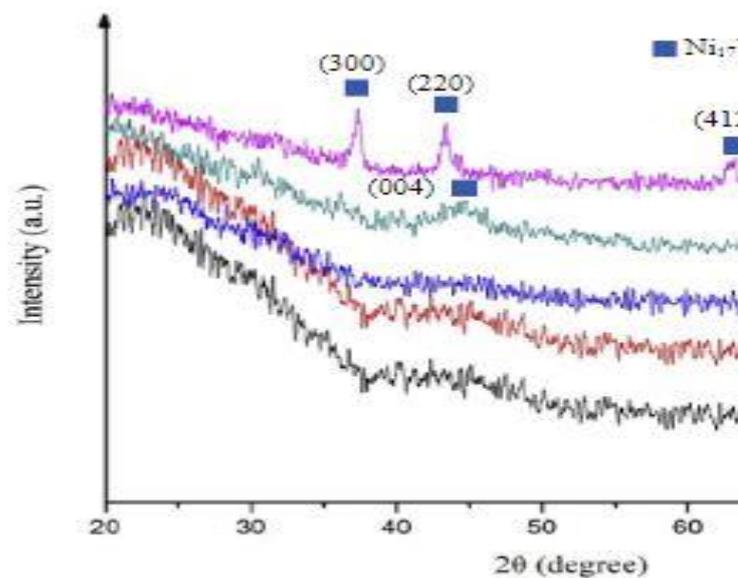


Fig. 1. X-ray diffraction patterns of Ni-Cr-Mn-Y thin films at different annealing temperatures.

### Conclusion:

This study investigated thin films fabricated for the purpose of preparing high resistivity with low-TCR thin film resistors. The dependencies of the Ni-Cr-Mn-Y-Nb thin film electrical properties on the annealing temperatures and the film compositions were also investigated. Our conclusions are summarized as follows: A Ni-Cr-Mn-Y resistive thin film was prepared using DC magnetron sputtering from  $\text{Ni}_{0.45}\text{-Cr}_{0.27}\text{-Mn}_{0.13}\text{-Y}_{0.16}$  cast alloy targets. When the annealing temperature was set to 400°C, Ni-Cr-Mn-Y films with some amounts of  $\text{Ni}_{17}\text{Y}_{12}$  nanocrystalline phases were observed. The Ni-Cr-Mn-Y film annealed at 300°C presented an amorphous structure using TEM analysis. The Ni-Cr-Mn-Y films annealed at 300°C exhibited the smallest temperature coefficient of resistance ( $\beta = 30 \text{ ppm}^\circ\text{C}$ ) with a resistivity of  $\sim 480 \text{ m}\Omega\text{-cm}$ . A Ni-Cr-

Mn-Y-Nb resistive thin film was prepared using DC and RF magnetron co-sputtering from  $\text{Ni}_{0.45}\text{-Cr}_{0.27}\text{-Mn}_{0.13}\text{-Y}_{0.16}$  cast alloy and niobium targets. All of the Ni-Cr-Mn-Y-Nb films annealed at  $400^\circ\text{C}$  exhibited an amorphous structure, except for specimens with 0% and 6.3% Nb addition at  $400^\circ\text{C}$ . Ni-Cr-Mn-Y films with 6.3 at.% Nb annealed at  $400^\circ\text{C}$  presented amount of  $\text{Ni}_{17}\text{Y}_{12}$  crystalline phase. Amorphous structures were obtained for Ni-Cr-Mn-Y films with higher Nb content ( $\square 14$  at.%), which is attributed to the high entropy alloy effect. The TCR values gradually shifted from positive to negative with increasing in niobium content. As the annealing temperature was increased, the TCR shifted from negative to close to zero. This shift is attributed to the crystalline and oxidation. The TCR of Ni-Cr-Mn-Y-Nb thin films could be adjusted to close to zero using the annealing process and adding Nb content. The oxidation layer thickness in the films was increased significantly from 8 nm at 14 at.% Nb to 11 nm at 31.6 at.% Nb. This result indicates that the film surface oxidation becomes thicker with more added Nb. The electrical properties indicated that Ni-Cr-Mn-Y films with 14% Nb addition annealed at  $300^\circ\text{C}$  exhibited the smallest temperature coefficient of resistance ( $\approx 5$  ppm/ $^\circ\text{C}$ ) with a resistivity of  $\sim 585$  m $\Omega\cdot\text{cm}$ . For practical purposes, it is important that films with a small TCR possess high resistivity water-based binders modified with nanoparticles materials and its application in the leather finishing.