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Research Article

A Study of Thin Film Resistors Prepared Using Ni-Cr-Si-Al-Ta High Entropy Alloy

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Ni-Cr-Si-Al-Ta resistive thin films were prepared on glass and Al_2O_3 substrates by DC magnetron cosputtering from targets of $Ni_{0.35}$-$Cr_{0.25}$-$Si_{0.2}$-$Al_{0.2}$ casting alloy and Ta metal. Electrical properties and microstructures of Ni-Cr-Si-Al-Ta films under different sputtering powers and annealing temperatures were investigated. The phase evolution, microstructure, and composition of Ni-Cr-Si-Al-Ta films were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), and Auger electron spectroscopy (AES). When the annealing temperature was set to 300°C, the Ni-Cr-Si-Al-Ta films with an amorphous structure were observed. When the annealing temperature was at 500°C, the Ni-Cr-Si-Al-Ta films crystallized into $Al_{0.9}Ni_{4.22}$, Cr_2Ta, and Ta_5Si_3 phases. The Ni-Cr-Si-Al-Ta films deposited at 100 W and annealed at 300°C which exhibited the higher resistivity 2215 $\mu\Omega$-cm with −10 ppm/°C of temperature coefficient of resistance (TCR).

1. Introduction

Rapid technological advancements in many electronics industries, such as in the telecommunication and information, the aerospace, and the precision measurement sectors of industry, require the continuous development of electronic components to achieve higher precision, reliability, and integration [1]. Among these components, the resistor is one of the fundamental components and is primarily used in electronic circuits. In this respect, the demands for thin film resistors with low temperature coefficients of resistance (TCR) and high precision have been increasing dramatically in recent years [2–5].

The temperature coefficient of resistance (TCR) is an important technical parameter of thin film resistors. A high TCR will result in resistance value drifting and will consequently influence the accuracy of resistors as the temperature changes [6]. The main factors influencing the TCR include the sputtering process, annealing temperature, and film composition, whereas film composition plays a decisive role among these three factors. Therefore, employing an appropriate method for depositing a suitable film composition is the key to obtaining high-resistance resistors with a low TCR.

Extensive and rapid development in high entropy alloy has been conducted since the recent years by Yeh et al. [7]. These alloys are defined to have five or more principal metallic elements, with the concentration of each element varying between 5% and 35%. It is generally found that high entropy alloys form simple solid solution structures (rather than many complex phases) at elevated temperatures because of large mixing entropies. The simple crystal structures possess many excellent properties [8], such as easy of nanoprecipitation, high hardness, and superior resistance to temper softening, wear, oxidation, and corrosion. The recent progress in HEAs, such as many new face-centered cubic (fcc) and body-centered cubic (bcc) HEAs and some high entropy bulk metallic glasses have been developed. They display an excellent mechanical response and good wear resistance at low and high temperatures, as well as good plastic behavior [9–12].

Ni-Cr thin films are employed in integrated circuits, where low noise, good power dissipation, and a near-zero temperature coefficient of resistance are important requirements. Several studies have reported on the deposition of Ni-Cr resistive films by thermal evaporation [13–16] and radio frequency (RF) sputtering, primarily for use as hybrid resistors [17, 18]. Extensive work has been reported on the control of the sheet resistance R_s and TCR of Ni-Cr resistors by doping the films with different impurities. If silicon is added to the alloys, Ni-Cr-Si thin film resistors with very low temperature coefficients of resistivity are obtained, but the alloy resistivities are not significantly increased [1]. In our previous study, the effects of aluminum addition and annealing on the microstructure and electrical properties of Ni-Cr-Si films have been reported [19]. The electrical resistivities of the Ni-Cr-Si-Al films were higher than those of the Ni-Cr-Si films annealed at temperatures below 400°C, and the annealed Ni-Cr-Si-Al films exhibited a TCR close to zero. However, aluminum element has lower melting point (660°C), which is not good for stability. The tantalum, with high melting point is at 3020°C, will be being beneficial for thermal stability of resistive thin films [20].

To obtain resistive thin films with high resistivity and a low TCR, the concept of high entropy alloy was introduced to investigate the Ni-Cr-Si-Al-Ta composition as thin film resistors based on previously study of Ni-Cr-Si-Al thin resistor [19]. The effects of sputtering power and annealing temperature on the phases and microstructural and electrical properties of Ni-Cr-Si-Al-Ta thin films were investigated.

2. Experimental Procedure

Ni-Cr-Si-Al-Ta thin films of 80 nm in thickness were deposited on the substrates using a DC magnetron cosputtering system having a sputtering rate of 5 nm/min. $Ni_{0.35}$-$Cr_{0.25}$-$Si_{0.2}$-$Al_{0.2}$ and tantalum of diameter 76.2 mm were used as targets. The sputtering chamber was evacuated to a background pressure of 7×10^{-7} Torr by a cryopump, and then the sputtering gas Ar with a purity of 99.999% at flow of 60 sccm was introduced into the chamber using mass flow controllers, by which the working pressure was 4.3×10^{-3} Torr. Thin films deposited on glass plates at room temperature were subjected to electron probe microanalysis (EPMA) and X-ray diffraction (XRD) studies, while thin films on Al_2O_3 substrates (size: 25 mm^2) were used for measuring the electrical properties. The DC power was adjusted at 100 W and 200 W, respectively. The as-deposited films were annealed at 250 to 500°C for 4 hours, with 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, and the thickness t of the films was measured by FE-SEM (cross-section). The resistivity measured by the four-probe method was consistent with the resistivity obtained by the product of R_s and t. The TCR values of the Ni-Cr-Si-Al-Ta films were measured on thin long strips cleaved from the substrate. Electrical contacts to the two ends of the resistive strips were obtained by selectively coating the ends with sputtered Ag. The DC resistance of the strips was measured on digit multimeter (HP 34401A) at different temperatures (25°C and 125°C). TCR of the Ni-Cr-Si-Al-Ta films was measured by the following relation:

$$\mathrm{TCR} = \left[\left(\frac{\Delta R}{\Delta T}\right) \times \frac{1}{R}\right] \times 10^6 \text{ ppm/K}. \quad (1)$$

The composition of the deposited films was determined by Auger electron spectroscopy (AES). AES depth profiles were obtained in a PHI 550ESCA/SAM Auger microprobe (Physical Electronics, USA). The crystallinity of the films was analyzed by X-ray diffraction (XRD, Bruker D8A Germany), using Cu K_α radiation for 2 θ values from 20° to 70°, with a scan speed of 3° min^{-1} and a grazing angle of 0.5° at 40 kV and 40 mA. Microstructural and thickness observations of the cross-sectional and plane-view morphology of thin films grown on glass substrates were analyzed by field-emission scanning electron microscopy (FE-SEM, Hitachi S-4700 Japan) with an accelerating voltage of 20 kV. The microstructure of the films was also investigated by a field-emission transmission electron microscope (FE-TEM, FEIE.O. Tecnai F20) equipped with an energy-dispersive spectrometer at an acceleration voltage of 200 kV. TEM specimens were prepared by disk cutting, mechanical polishing, dimpling, and ion milling. A Fischione 150 ultrasonic disk cutter (Fischione Instruments Inc., Export, PA) was used to cut a 3 mm sample, which was then mechanically polished to approximately 100 μm in thickness using a diamond lapping film. The sample thickness during lapping was accurately controlled by a tripod polisher (SPI Supplies, West Chester, PA). Next, the polished sample was dimpled to a thickness of approximately 4 μm using a Fischione 150 dimpling grinder, followed by ion milling on both sides with a Fischione 1010 ion miller to achieve an electron transparent thickness (<100 nm).

3. Results and Discussion

3.1. The Compositions, Phase Transformation, and Microstructure of the Sputtered Films. Electron probe microanalysis (EPMA) was used to determine the film compositions. The relative concentrations of nickel, chromium, silicon, aluminum, and tantalum were measured at three points in the sputtered films, respectively. The average values are listed in Figure 1. The results indicate that the composition of the sputtered Ni-Cr-Si-Al-Ta films with different sputtering powers were 23.5% Ni, 14.6% Cr, 23.6% Si, 16.8% Al, and 21.5% Ta at 100 W and 24.5% Ni, 13.8% Cr, 23.6% Si, 17.6% Al, and 20.5% Ta at 200 W, respectively. It was found that the Ta content was about 20%. However, these films are accorded to the rule of high entropy alloys which have five or more principal metallic elements with the concentration of each element varying between 5% and 35%.

Figure 2 shows X-ray diffraction patterns of Ni-Cr-Si-Al-Ta films at 200 W, for the as-deposited samples and those annealed at various temperatures for 240 min. All of the Ni-Cr-Si-Al-Ta films annealed at ≤400°C exhibited an amorphous structure, indicating that none of the elements crystallized or oxidized. The term amorphous is a general term that refers to a solid state with a nonperiodical atomic

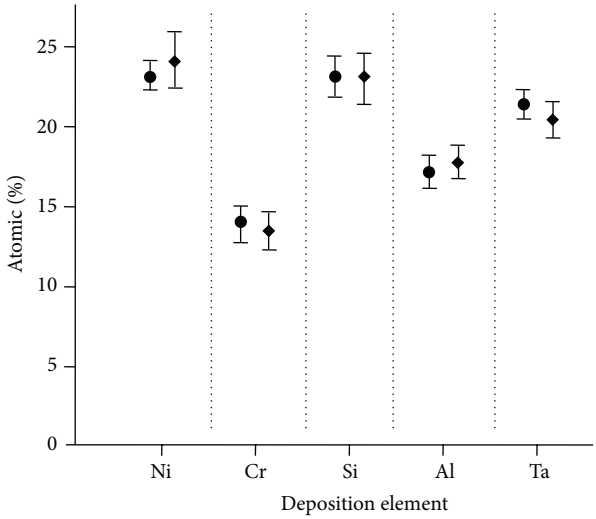

Figure 1: Compositions of Ni-Cr-Si-Al-Ta thin films with different DC sputtering powers sputtered on cooper sheet.

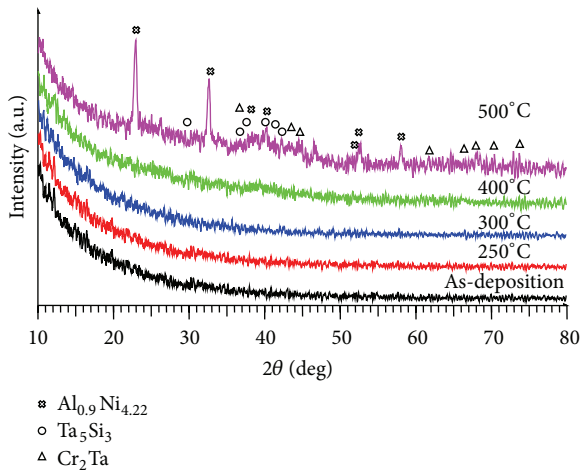

Figure 2: X-ray diffraction patterns of Ni-Cr-Si-Al-Ta thin films sputtered at 200 W with different annealing temperatures.

arrangement. A particular feature of an amorphous material at the atomic level in comparison with crystalline material is the absence of long-range order in the atoms. However, the atomic arrangement at the atomic scale (distance of a few diameters of atoms) is periodical [21]. The structure of crystalline materials can be easily determined by describing the unit cells of the crystals. The characterization of an amorphous structure is much more difficult due to the broadening of diffraction patterns and the lack of reflections during X-ray investigations. Further study is needed to use transmission electron microscopy for crystalline analysis. It is interesting to point out that the Ni-Cr-Si-Al-Ta films did not oxidize after 500°C annealing in air; there are only alloy phases formed in the Ni-Cr-Si-Al-Ta films. Yeh et al. [7, 22] reported that the HEAs can enhance the high temperature strength, corrosion, oxidation resistance, and so on.

Figure 3 shows X-ray diffraction patterns of Ni-Cr-Si-Al-Ta films with different sputtering powers on glass substrates, for as-deposited samples and those annealed at 500°C for 240 min. The figure shows that the as-deposited films had an amorphous structure. When the annealing temperature was set to 500°C, the crystallization of $Al_{0.9}Ni_{4.22}$, Ta_5Si_3, and Cr_2Ta was clearly discernible. In Figure 3(b), which also shows the results at different sputtering power, the intensity of XRD peaks clearly increases with increasing sputtering power up to 200 W. It indicates that the crystalline film is strongly influenced by the sputtering power when the films annealed at high temperature. This may be attributed to the fact that, at high sputtering power, the ejected metal atoms possess higher kinetic energy when they arrive on the substrate [23]. Consequently, these Ni, Cr, Si, Al, and Ta atoms have sufficient kinetic energy to rearrange themselves to form closer packing layer, resulting in a highly nanocrystalline film structure. The numbers or energy of electrons and ions in glow discharge plasma will increase with an increment in sputtering powers, in the case of using metal target [24, 25]. That is, the energy of sputtered atom which is obtained from the positive ions colliding to target will increase with increasing sputtering power. It is also possible that the highly energized electrons with increasing sputtering power will bombard the surface of growing film at the substrate, providing in the form thermal energy. This energy might act as an additional energy to promote the growth of crystalline. Therefore, it can be concluded that as the sputtering power increases, the number and the momentum of the sputtering particles will also increase. The atoms become more mobile on the surface of the film as the result of the higher bombardment rates on them [26].

Figure 4 shows SEM micrographs of the Ni-Cr-Si-Al-Ta thin films with different sputtering powers and annealing temperatures. Some heterogeneous phases appeared after annealing at 400°C in the Ni-Cr-Si-Al-Ta thin films deposited at 200 W, as shown in Figure 4(b), and it obviously appeared after annealing at 500°C as shown in Figure 4(c), which is consistent with the XRD analysis (Figure 2). These crystal phases should belong to $Al_{0.9}Ni_{4.22}$, Ta_5Si_3, and Cr_2Ta. For the Ni-Cr-Si-Al-Ta thin films deposited at 100 W and annealed at 500°C, the heterogeneous phases were also observed, as shown in Figure 4(d).

Figure 5 shows a typical TEM bright field image and selected-area electron diffraction (SAED) patterns of the Ni-Cr-Si-Al-Ta thin films with different sputtering powers and annealing temperatures. For Ni-Cr-Si-Al-Ta thin films deposited at 200 W and annealed at 300°C, it shows that the film with nanocrystalline structure appeared as shown in Figure 5(a). This result can be confirmed using the SAED patterns as shown in Figure 5(b). Therefore, the increase of sputtering power obviously causes the formation of a crystalline phase. With the annealing temperature increasing, more nanocrystallites were observed significantly to nucleate homogeneously throughout thin films and the SAED patterns changed to Debye-Scherrer-type rings (Figures 5(c) and 5(d)). However, the crystallites of the films annealed at 500°C were obviously larger than the films annealed at 300°C. The phase analysis performed from the electron

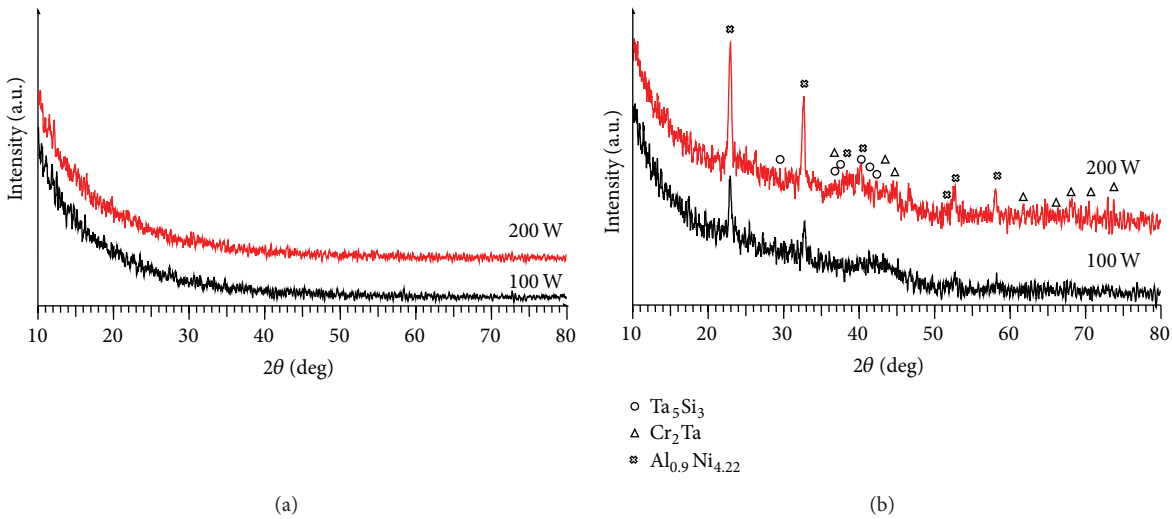

FIGURE 3: X-ray diffraction patterns of Ni-Cr-Si-Al-Ta thin films with different sputtering powers: (a) as-deposited and annealed at (b) 500°C.

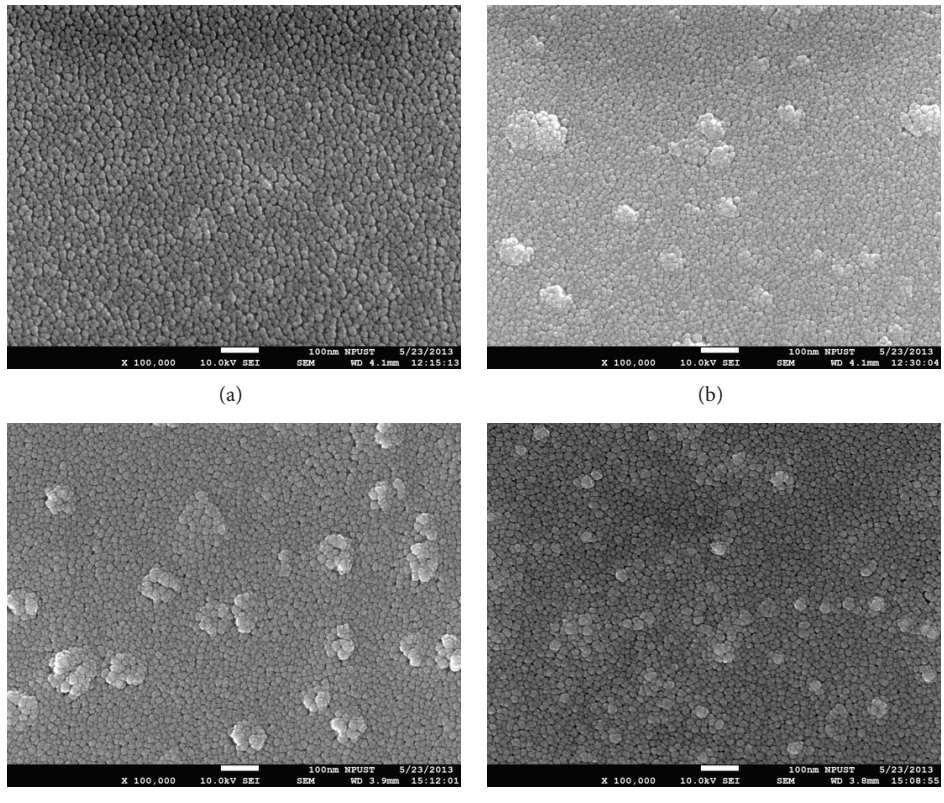

FIGURE 4: SEM micrographs of Ni-Cr-Si-Al-Ta films sputtered and annealed at (a) 200 W/300°C, (b) 200 W/400°C, (c) 200 W/500°C, and (d) 100 W/500°C.

diffraction patterns enables the identification of $Al_{0.9}Ni_{4.22}$ and Cr_2Ta phases. The increase in peak intensities indicates an enhancement in the crystallinity of films because it is generally noted that the crystallinity can be enhanced while increasing the annealing temperature [27, 28].

It is still of interest when considering phase competition between the solid solution phases and potential compounds (intermetallics). In other words, it is important to understand the thermodynamic nature of the phase stability of the solid solutions in Ni-Cr-Si-Al-Ta thin films at elevated

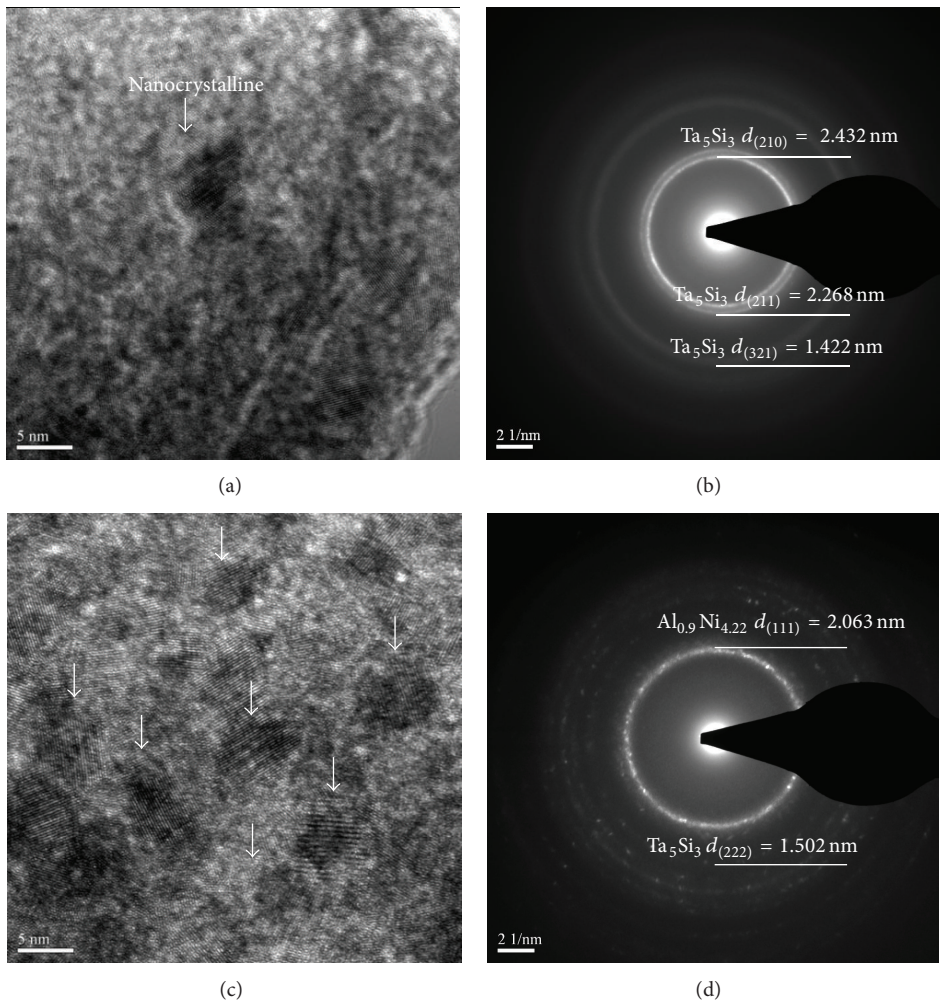

Figure 5: TEM micrographs and selected-area electron diffraction of Ni-Cr-Si-Al-Ta films sputtered and annealed at 200 W/300°C for (a) and (b) and 200 W/500°C for (c) and (d).

temperatures. Solid solutions are stable when the mixed crystal has lower free energy than the alternative-building up two crystals of different composition or building up a new structure in which the foreign atoms are put on ordered sites. The free energy is given by the following relation:

$$G = E + PV - TS, \quad (2)$$

where E is largely determined by the structural energy and the entropy is a measure of the randomness of the structure. If an atom added at random greatly increases the structure energy, the solid solution is unstable and two crystal structures form. On the other hand, if the addition of a foreign atom greatly lowers the structure energy, the system tends to form an ordered new phase. If the energy is not much changed, the entropy is increased by random additions so that the solid solution has the lowest energy and is the stable configuration [29].

3.2. The Electrical Properties of Ni-Cr-Si-Al-Ta Thin Films. To apply a material in thin film resistors, it is important to know (i) the range of resistivity in which a near-zero TCR can be obtained and (ii) the detailed resistance behavior in the range of working temperatures (usually −55°C to 125°C) [30]. Figure 6 shows the effect of annealing temperature and sputtering power on the electrical properties of the Ni-Cr-Si-Al-Ta films. The resistivity of Ni-Cr-Si-Al-Ta films decreases obviously with increasing of annealing temperatures. This result indicates that atomic configuration changes have occurred in Ni-Cr-Si-Al-Ta films. It is interesting to point that the resistivities of the Ni-Cr-Si-Al-Ta films are different between 100 and 200 W of sputtering power at 300°C annealing. The resistivity of Ni-Cr-Si-Al-Ta films is ~2200 and ~1600 $\mu\Omega$-cm for 100 W and 200 W, respectively. There is more than 35% difference in resistivity between them. The reason for this is due to different crystal structures of Ni-Cr-Si-Al-Ta films with different sputtering power at 300°C; one is an amorphous at 100 W and the other one is nanocrystalline at 200 W. It is also interesting to note that the resistivity of the Ni-Cr-Si-Al-Ta films decreases with increasing the annealing temperature. From the above XRD and TEM results, the crystallinity of Ni-Cr-Si-Al-Ta films was enhanced

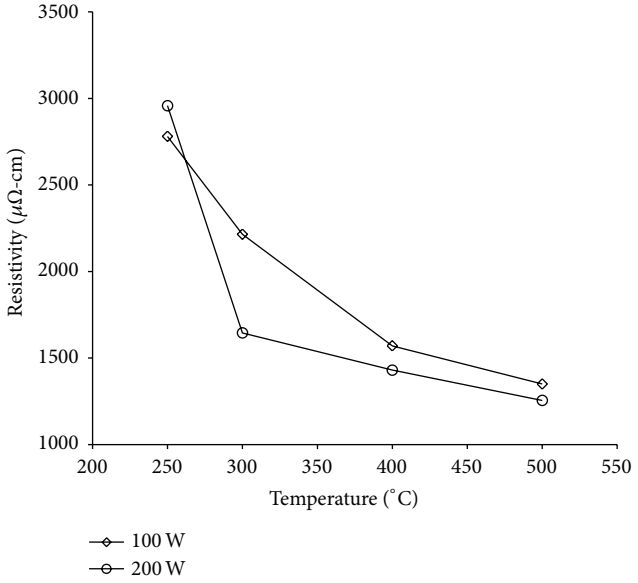

Figure 6: Room temperature resistivity of Ni-Cr-Si-Al-Ta films with different sputtering powers and annealing temperatures.

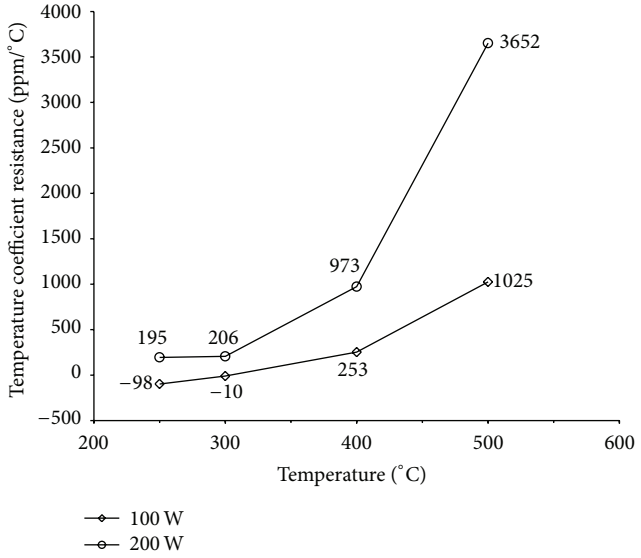

Figure 7: Temperature dependence of the TCR of Ni-Cr-Si-Al-Ta films with different sputtering powers and annealing temperatures.

with increasing the annealing temperature. In general, by increasing the annealing temperature, the resistivity of Ni-Cr-Si-Al-Ta films increases since the grain boundaries, crystal defects, and oxides generation of the film were increased [6]. In this investigation, the resistivity was decreased which can be attributed to an increase in alloy phases ($Al_{0.9}Ni_{4.22}$, Ta_5Si_3 and Cr_2Ta) with increasing the annealing temperature, as shown in Figures 2 and 5. According to Matthiessen's rule, the resistivity of a continuous film is a cumulative effect of various electron scattering processes in the film. The resistivity ρ_T of the film is given by the following relation:

$$\rho_T = \rho_B + \rho_S + \rho_I, \tag{3}$$

where ρ_B, ρ_S, and ρ_I are contributions made to the total resistivity of the film due to scattering at ideal lattice (same as bulk), scattering at film surface (dependent on film thickness), and scattering at imperfections (grain boundaries and impurities), respectively.

Figure 7 shows the effect of annealing temperature and sputtering power on the temperature coefficient of resistivity (TCR) of the Ni-Cr-Si-Al-Ta films. The TCR values were increased with increasing of annealing temperature. TCR values were about 200 ppm/°C annealed below 300°C at 200 W. Moreover, the Ni-Cr-Si-Al-Ta films deposited at 100 W annealed at 300°C exhibit a TCR close to zero because the films retain an amorphous structure after annealing. However, the TCR values of the films annealed at 500°C are about 1000 ppm/°C. The TCR value becomes worse when the annealing temperature is increased. It can be explained that the annealing response of the TCR is the result of competition between a negative contribution from weak localization effects in the amorphous region and a positive contribution from crystalline phase grains [31]. For practical purposes, it is important for films with a small TCR to possess high resistivity.

4. Conclusion

Ni-Cr-Si-Al (30/20/20/30 at.%) alloy and Ta targets were deposited on the glass and Al_2O_3 substrates as thin film resistor materials by cosputtering method. The compositions of film are accorded to the rule of high entropy alloys which have five elements with the concentration of each element varying between 5% and 35%. There is an amorphous structure observed in the films with sputtering power 100 W at 300°C. However, the annealing temperature was at 500°C; both films (100 W and 200 W) crystallized into $Al_{0.9}Ni_{4.22}$, Cr_2Ta, and Ta_5Si_3 phases. Electrical properties indicated that the Ni-Cr-Si-Al-Ta films with 100 W exhibited the smallest temperature coefficient of resistance (−10 ppm/°C) with higher resistivity ~2200 $\mu\Omega$-cm after annealing at 300°C in air. For practical purposes, it is important for films with a small TCR to possess high resistivity. In this study, the introduction of HEAs concept can effectively enhance the electrical properties of Ni-Cr based thin films, satisfying the requirements for thin film resistor application.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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