XRD INVESTIGATIONS OF CO FILMS DEPOSITED BY CVD

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Introduction

Thin films of cobalt, cobalt-based alloys, and Co/X multilayers, where X can be another metal or a dielectric, have been the subject of significant scientific research and have attracted great interest with respect to applications in data storage devices and sensors [1].

There are currently many deposition techniques for metal films. However, chemical vapor deposition (CVD) has a special place among them. CVD is a technique which offers potential for producing films with high uniformity of thickness and composition, high purity, minimal substrate damage, high deposition rates and the possibility for selected area growth [2].

The dependence of structure and phase composition on deposition conditions is necessary to know for film formation with specified operational characteristics. One of the modern methods that is used for the identification of crystal phase composition is X-ray diffraction analysis. The results of X-ray diffraction investigations of Co thin films deposited by CVD are presented in this work. The aim of this research is to reveal the effect of substrate and vaporization temperatures on the structural parameters of Co thin films.

Experimental details

Co films were deposited on Si(100) substrates by CVD. Co(*N'acN'ac*)$_2$ was used as a precursor. The deposition conditions were as follows: gas-carrier flow rate (Ar) was 1 l/h, gas reagent flow rate (H_2) - 4 l/h, the operating pressure was atmospheric (~760 *Torr*), the deposition process was carried out during a period of 4h. Three sets of samples were investigated. In the first set the vaporization temperature (T_{vap}) was fixed and was equal to 120 °C, while the substrate temperatures (T_s) were varied in the range of (310 – 420) °C. In the second set the vaporization temperature was also fixed (130 °C) and the substrate temperatures were varied in the range (300 – 340) °C. In the third set the substrate temperature was constant (330 °C) and the vaporization temperature was varied in the range from 120 to 155 °C.

The X-ray diffraction (XRD) analysis of the samples was performed on a DRON-SEIFERT-RM4 diffractometer (Cu Kα radiation, λ = 1.54051 Å). All measurements were carried out at an atmospheric pressure and at room temperature.

Results and discussion

According to XRD data, only one of the diffraction peaks centered at around 44,2 – 44,7° in 2Θ is observed in the samples deposited at T_{vap} = 120°C and at different substrate temperatures (Fig. 1). The asymmetric broadening of the diffraction peak can be considered as the superposition of several peaks for α and β –Co. Detailed analysis of XRD pattern reveals that this diffraction peak is positioned between fcc (111) of β-Co (face centered cubic) at 44,3° and the (002) reflection of α-Co (hexagonal close-packed) at 44,6°. Therefore, it is difficult to uniquely determine the phase condition of the studied Co films.

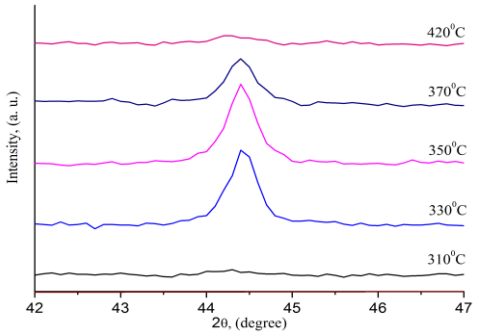

Fig. 1. XRD patterns of the Co samples deposited at T_{vap} =120 °C.

The height of the diffraction peak (*2*Θ = 44, 2 – 44, 7°) depends on the substrate temperature (Fig. 1). XRD analysis of the samples deposited at T_s = 310 °C revealed no peaks corresponding to the Co phase. Co films evaporated at T_s = 330 °C are characterized by the appearance of the peak. The observed diffraction peak becomes higher with increasing substrate temperature and reaches its maximum at T_s = 350 °C. However, further temperature growth results in a decrease in height of the X-ray diffraction peak until its loss at T_s = 420 °C.

The increase in vaporization temperature up to T_{vap} = 130 °C causes some changes in the crystalline texture of Co thin films (Fig. 2). The XRD pattern for Co samples deposited at T_s = 300 – 340 °C contain additional peaks, which could be indexed to the α-Co (100) (2Θ = 41.7°), α-Co (101) (2Θ = 47.6°) and β-Co (200) (2Θ = 51.7°). The height of the diffraction peaks is governed by the substrate temperature. The XRD peak becomes higher with increasing substrate temperature from T_s = 300 to T_s = 320 °C. However, further growth of substrate temperature up to T_s = 330 and 340 °C results in a gradual weakening of the peaks. The Co film deposited at substrate temperature T_s = 320 °C is noted to be characterized by well-pronounced texture (the maximum height of the diffraction peaks).

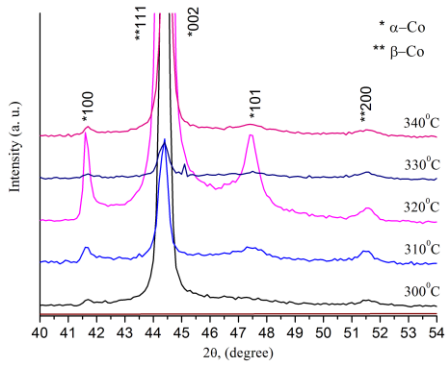

Fig. 2. XRD patterns of the Co samples deposited at T_{vap} =130 °C.

The comparison of fig. 1 and 2 shows that the increase in vaporization temperature of Co films from 120 up to 130 °C causes a considerable change in diffraction patterns. Fig. 3 presents the results of detailed investigations of vaporization temperature's effect on a metal film's structure.

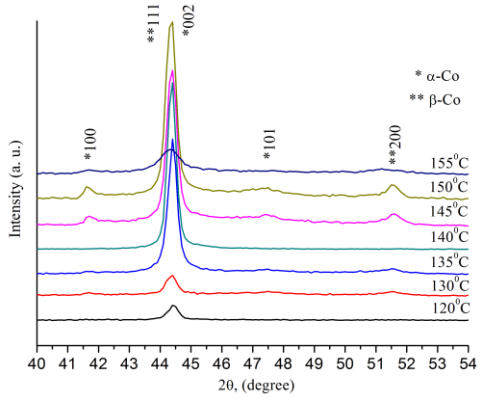

Fig. 3. XRD patterns of the Co samples deposited at T_s =330 °C.

The sizes of the coherent scattering region (CSR) and microstress values of Co films under study are presented in table 1. As seen from the table, the CSR size of films deposited at T_{vap} = 120 °C weakly depends on the substrate temperature in the range of temperatures from 300 to 350 °C. However the CSR size starts to decrease after increasing substrate temperature over 350 °C. A similar substrate temperature dependence of the CSR size is observed for films deposited at T_{vap} = 130 °C. Nevertheless, a comparison of samples from the first and second sets allows us to conclude that a temperature range in which films are characterized by a constant CSR size decreases with increasing vaporization temperature from 120 up to 130 °C.

The vaporization temperature dependence of the CSR size has a maximum (table 1). Actually, the growth of T_{vap} from 120 up to 135 °C results in a more than twofold increase in the CSR size. Further increase in vaporization temperature up to T_{vap} = 140°C causes a sharp decrease in CSR size. After this point, the CSR size remains unchanged until T_{vap} = 155 °C.

Microstresses of Co films deposited at T_{vap} = 120°C decrease with increasing substrate temperature, whereas values of microstresses are kept constant within the measurement error in films deposited at T_{vap} = 130 °C (table 1). It should be noted that the rise of T_{vap} from 120 up to 130 °C causes a threefold decrease in microstresses. However, further growth of vaporization temperature up to T_{vap} = 155 °C does not affect them.

Table 1. CSR size and microstresses σ of Co films deposited at different vaporization T_{vap} and substrate T_s temperatures.

T_{vap}, °C	T_s, °C	CSR,nm	σ, GPa
First set			
120	310	17	1,7
120	330	15	1,4
120	350	19	1,2
120	370	13	1,4
120	420	11	0,8
Second set			
130	300	35	0,4
130	310	26	0,5
130	320	33	0,2
130	330	26	0,4
130	340	20	0,3
Third set			
120	330	15	1,4
130	330	26	0,4
135	330	39	0,3
140	330	16	0,4
145	330	20	0,2
150	330	16	0,3
155	330	16	0,1

Conclusion

The results of the performed investigations show that Co films deposited by chemical vapor deposition consist of α-Co and β-Co crystals. Varying the substrate and vaporization temperatures allows us to widely modify the microstructure and texture of Co films. An increase in substrate temperature causes a decrease in the sizes of the coherent scattering region and values of microstresses. The degree of substrate temperature's effect on the structural parameters of Co thin films decreases with increasing vaporization temperature from 120 up to 130 °C. Moreover, the texture of Co films is modified – crystals with a new orientation appear.

References

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2. Chioncel M.F, Nagaraja H. S. Domain structures of MOCVD cobalt thin films // Journal of Magnetism and Magnetic Materials. - 2007. - № 313. – P. 135–141.