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Investigation of phase transformation for ferrite–austenite structure in stainless steel thin films

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In this work we report on phase transformation of 304 stainless steel thin films due to heat treatment. Ex-situ annealing was applied for evaporated 304 stainless steel thin films inside an ultra-high vacuum chamber with a pressure of 3×10^{-7} Pa at temperatures of 500 °C and 600 °C. The structure of thin films was studied by X-ray diffraction (XRD) and conversion electron Mössbauer spectroscopy (CEMS) techniques. The results revealed a transformation from α-phase that exhibits a body-centered cubic structure (BCC) to γ-phase that exhibits a face-centered cubic (FCC) due to annealing. In addition, the percentage of γ-phase structure increased with the increase of annealing temperature. Annealing thin films increased the crystal size of both phases (α and γ), however, the increase was nonlinear. The results also showed that phase transformation was produced by recrystallization of α and γ crystals with a temporal evolution at each annealing temperature. The texture degree of thin films was investigated by XRD rocking curve method, while residual stress was evaluated using curvature method.

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1. Introduction

Stainless steel is a highly demanded material in different fields due to its wide range of industrial applications in fields such as chemical processing, oil and gas, food production, and power generation [1–3] due to its outstanding mechanical properties as well as resistance to corrosion [4–6]. In the last decades, many investigations were carried out to study structural modification of 304 stainless steel films fabricated by sputtering technique, where the sputtering target is normally made of 304 stainless steel of γ-phase [7–9]. It was reported that the structure of these sputtered films contains both phases: γ and α [10]. Herein, if stainless steel films are generated by sputtering at a substrate temperature between 375 °C and 500 °C, they are likely to develop a structure that contains both phases [11–13]. If the sputtered 304 stainless steel films are formed on substrates at room temperature, they can develop a structure constitute by both phases. The α-phase in those films is an unstable phase, due to rapid quenching of the sputtered atoms and small clusters when they are deposited on a substrate at room temperature [14].

α-Phase in sputtered stainless steel thin films was reported to undergo a transformation to γ-phase at 477 °C [15] because α-phase is unstable at 477 °C or above. In addition, it was also observed that a full $\alpha \rightarrow \gamma$ phase transformation occurs at 677 °C for the above thin films. It should be noted here that sputtered 304 stainless steel thin films that are fabricated at room temperature are ferromagnetic due to the majority of α-phase [7] unlike bulk 304 stainless steel which is a non-magnetic material [16]. In addition, the transformation temperature of sputtered stainless steel thin films from α-phase to γ-phase is defined as Curie temperature, which is about 600 °C [7]. Beyond this temperature sputtered thin films would have a full phase transformation to γ-phase and they become non-magnetic similar to bulk 304 stainless steel [16].

Recently, we used the thermal evaporation method to prepare 304 stainless steel thin films [17,18]. It was demonstrated that the evaporated 304 stainless steel thin films exhibit nanocrystals ferrite–austenite mixture structure with a ratio of 6% for the γ-phase. In this work, the structural modification of ferrite–austenite mixture of 304 stainless steel thin films fabricated by thermal evaporation due to heat treatment is investigated by XRD and CEMS. Thin film stress is determined using the curvature method.

2. Experimental

2.1. Thin film preparation

Stainless steel thin films were deposited on quartz substrates with a thickness of 166 nm inside a high vacuum chamber by thermal evaporation, and used to investigate phase transformation. The thickness of the films was measured using a quartz crystal monitor placed close to the substrates. The details of the fabrication method were described elsewhere [17]. Table 1 presents the fabrication conditions. Thin films of

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Table 1
Conditions of thin film preparation.

Thin film fabrication conditions	
Distance between substrate and crucible	20 cm
Residual pressure in deposition chamber	10^{-4} Pa
Evaporation temperature of 304 SS bulk	1400 °C
Substrate temperature during deposition	25 °C
Deposition speed of 304 SS thin films	10.3 nm/s

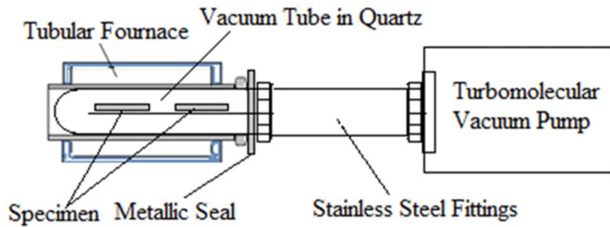

Fig. 1. Description of annealing system and its components.

different thicknesses were also prepared on silicon substrates and used to study residual stress of thin films.

2.2. Thin film annealing

Ex-situ annealing for thin films was carried out at 500 °C and 600 °C in an ultra-high vacuum chamber at 3×10^{-7} Pa placed inside a tube furnace for 1 h at each temperature. Figure 1 shows a schematic diagram of the annealing system that employed a turbo-molecular pump to achieve such a high vacuum. The annealing temperatures were chosen to cover the temperature range of phase transition. Quartz substrates were utilized since they are mechanically adequate for heat treatments, and they permit realization of annealing without atomic diffusion at the interface between thin films and substrate. The ultra-high vacuum ensured avoiding sample contamination or oxidation during the annealing. Furthermore, annealing and cooling rates of thin films were sufficiently slow to prevent their deterioration due to temperature gradient effect.

2.3. Characterization

2.3.1. XRD

X-ray diffraction (XRD) analyses of stainless steel thin films were performed using a SIEMENS D500/501 diffractometer using Cu-K_α radiation ($\lambda = 1.5406$ Å). XRD data were collected for 2θ in the range of 40°–140°. The diffractometer was equipped with a graphite monochromator placed before the detector to eliminate K_β radiation.

2.3.2. X-ray diffraction rocking curve method

The texture degree of thin films annealed at 500 °C for 1 h has been determined by X-ray diffraction rocking curve method [19–21]. Herein, the rocking curve method was applied to the (110) peak of α-phase taken from the X-ray diagram. A Lorentzian fit was applied to the (110) peak and the parameters are given in Table 2, where it is noted that the (110) peak corresponds to $2\theta = 44.80°$ (i.e. $\theta = 22.40°$).

In X-ray diffraction rocking curve method, X-ray source and detector were fixed in a position that corresponds to 2θ of Bragg's peak (110).

Table 3
Chemical composition of stainless steel thin film and the initial bulk material.

Chemical composition (wt.%)						
Elements	Fe	Cr	Ni	Mn	Si	C
304 SS thin films	71.75	17.06	7.60	3.48	–	–

The sample was rotated with an angle (Ω) about an axis perpendicular to the focal plane crossing an adjusted angular space at θ. In this experiment, Ω was varied from 2° to 42° with a step size of 0.5°, where this step size permitted to define the disorientation degree of diffraction planes relative to thin films surface. The maximum of the peak her is θ. The full-width at half maximum (FWHM) of the rocking curve peak was determined after the Lorentzian fit which permits to estimate the texture degree of the produced thin films.

2.3.3. Residual stress

The curvature method was utilized for determination of thin film's stress based on the change of curvature of silicon substrate (Si-(100)) of deposited thin film. Silicon substrate of high elasticity of 180.5 GPa [22], and dimensions of 200 μm (thickness) × 0.3 mm (width) were used for this study.

304 stainless steel thin films with thicknesses of 110, 120, 133, and 166 nm, were deposited on the substrates and used to study the influence of thickness and annealing on stress. The 166 nm thick film was annealed at 500 °C for 1 h and used to study the effect of annealing on stress. Fabrication and annealing conditions of thin films deposited on silicon substrate were similar to those deposited on quartz substrates above.

The application of curvature method is based on the measurement of the curvature size (R) that results from substrate bend (f) and the substrate length (L). The radius of curvature can be calculated using [23]:

$$R = \left(\frac{f}{2} + \frac{L^2}{8f}\right) \quad (1)$$

Eq. (1) expresses the geometrical relationship between R, L, and f of the curvature for a silicon substrate. It should be noted her that (f/2) is very small compared with $(\frac{L^2}{8f})$, thus, it can be ignored.

L and f were measured in this work using a mechanical profilometer: DEKTAK II. The measurements of both quantities were carried out before and after thin film deposition and treatment. L was 8 mm for all samples. Furthermore, measurements were carried out in both directions along the length of each substrate eight times per sample to reduce the error of manipulation. The stress can be calculated for a thin film by Stoney's relation [24]:

$$\sigma = \left(\frac{E}{1-\nu}\right)_S \left(\frac{H_s^2}{6h_f}\right)\left(\frac{1}{R} - \frac{1}{R_0}\right) \quad (2)$$

where E and ν are substrate's Young's modulus and Poisson's ratio, respectively, $(E/(1-\nu))_s$ is the term that expresses the elasticity of the substrate and it is equal to 180.5 GPa, and H_s and h_f are the thicknesses of substrate and thin film, respectively.

Table 2
Parameters of the fitting for the peaks: (110) α-phase and (111) γ-phase.

Diffraction peaks	Non-annealing		Annealing: 1 h—500 °C		Annealing: 1 h—600 °C	
	FWHM (°)	Intensities ratios I_γ/I_α	FWHM (°)	Intensities ratios I_γ/I_α	FWHM (°)	Intensities ratios I_γ/I_α
(111)-γ $2\theta = 43.85°$	1.13	0.0424	0.2447	0.5125	0.2502	0.7015
(110)-α $2\theta = 44.80°$	0.55		0.2787		0.3033	

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