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SHORT COMMUNICATIONS

In Situ Investigations of Magneto-optical Properties of Thin Fe Layers

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Abstract—A thin polycrystalline Fe film on a single-crystal Si substrate with natural SiO_2 oxide is obtained by thermal evaporation in ultrahigh vacuum. The magneto-optical properties of the resultant structure are investigated in situ by the methods of spectral ellipsometry. The values of the coercive force for the Fe film are obtained, and the magnetization reversal loop and the energy dependence of the equatorial Kerr effect are constructed. The effectiveness of magnetoellipsometry for in situ analysis of the geometrical and magneto-optical properties of thin ferromagnetic layers is demonstrated.

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INTRODUCTION

Analysis of the formation of heterostructures based on iron and silicon is stimulated by the need in magnetic semiconductors obtained using the developed silicon technology. For obtaining a high-quality heterostructure, information on all possible chemical interactions at the interface between the materials is required. A large number of investigations on the Fe/Si system have been carried out and various variants of phase diagrams have been constructed for subnanometer film thicknesses [1–3]. However, the magnetic and magneto-optical properties of Si and Fe compounds in the course of their formation have been investigated insufficiently in view of considerable technological difficulties in obtaining a magneto-optical system directly in an ultrahigh-vacuum growth chamber. It was shown in [4] that single-wave ellipsometry makes it possible to carry out magneto-optical experiments after an insignificant modification of the measuring circuit.

Standard methods of single-wave and spectral ellipsometry make it possible to determine the optical properties of various surfaces to a high degree of accuracy [5]. These control methods proved to be successful in the synthesis of nanostructures in which a continuous nondestructive monitoring of the processes of formation is required [5–9].

This study aims at analysis of the optical, magneto-optical, and magnetic properties of thin Fe films on the SiO_2/Si(100) surface before and after the sputtering directly in the measurement–growth complex designed by the group of authors [10].

1. EXPERIMENTAL

The sample was in the form of a single-crystal silicon substrate with a SiO_2 buffer layer. A detailed description of the chemical preparation of the substrate is given in [11]. Base vacuum in the processing cell was 10^{-7} Pa. Prior to the iron film deposition, we performed in situ spectral ellipsometry of the substrate for determining the thickness of the SiO_2 layer. The spectral range of ellipsometry was 360–900 nm. The measurements were taken in the four-zone regime [12] in zero magnetic field. The angle of incidence of light in all in situ measurements was fixed at 56°; for ex situ measurements, this angle was 70°.

Further, the iron film was obtained under the control of parameters of its growth. Iron was evaporated from a molecular source with a boron nitride crucible. The metal deposition processes was monitored by single-wave ellipsometry at a probe beam wavelength of 500 nm, and the effective thickness of the growing iron film was calculated using the Newton numerical method [13]. The resulting iron deposition rate was ~0.14 nm/min. The temperature of the substrate during iron deposition was about 250°C.

After the deposition and cooling of the sample to room temperature, we performed in situ ellipsometric experiments in zero magnetic field in a wavelength range of 360–900 nm for determining the structural properties of the resultant Fe layer.

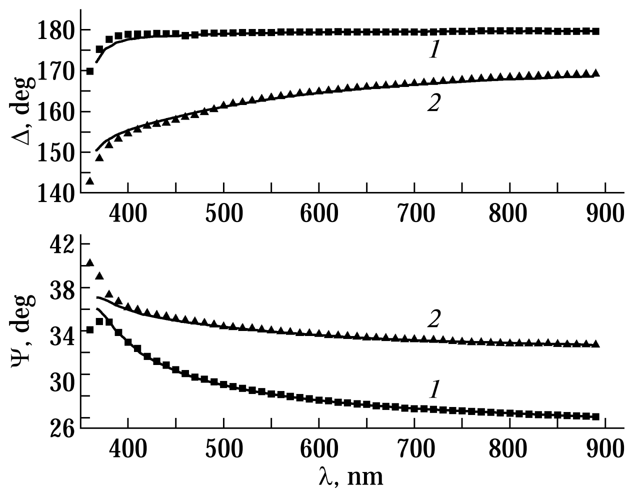

Fig. 1. Spectrograms of experimental and calculated ellipsometric angles (*1*) before and (*2*) after deposition of iron; solid curves—theory, squares and triangles are the results measured before and after sputtering, respectively.

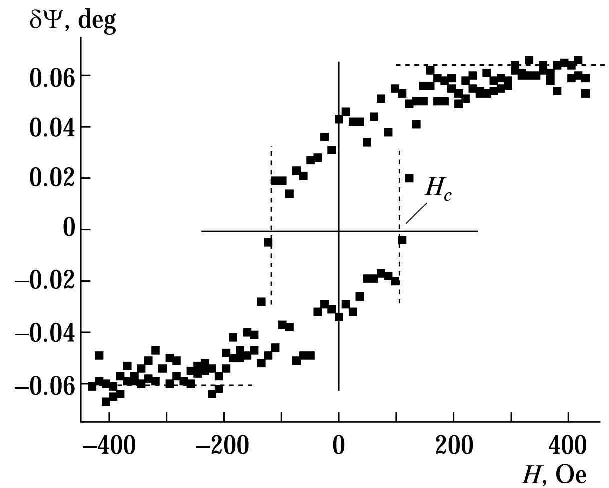

Fig. 2. Dependence of the variation of ellipsometric parameter Ψ on the magnetic field for the $Fe/SiO_2/Si(100)$ structure.

Single-wave (λ = 500 nm) ellipsometric experiments were carried out with a magnetic field varied in the range of sample saturation (±430 Oe). The magnetic field variation step was 12 Oe.

Magneto-optical measurements were carried out based on the equatorial Kerr effect (EKE) using an Ellips-1891 high-speed spectral ellipsometer and a modulated external magnetic field depending on the incident radiation energy in the range ~1.4–3.2 eV for in situ and ~1.4–3.8 eV for ex situ methods. The data were obtained by measuring the amplitude ellipsometric parameter Ψ for the magnetization reversal of the sample in saturation fields at each step in the wavelength of the chosen spectral range. The magnetic field applied for magnetization reversal of the sample to saturation was ±430 Oe.

2. RESULTS AND DISCUSSION

The spectral dependences of ellipsometric angles Ψ and Δ measured prior to iron sputtering are very close in absolute values to those for pure silicon with a slight deviation in phase parameter Δ. This indicates the presence of a thin layer of transparent natural silicon dioxide on the polished substrate surface. Since the effective thickness of the oxide layer is not sufficient for determining the porosity, we constructed an optical model of a continuous homogeneous SiO_2 layer and of a semi-infinite Si medium with sharp interfaces for determining its parameters. The sought parameter in fitting was the thickness of the oxide layer, which was 0.4 ± 0.1 nm. Figure 1 shows the measured spectral dependences of Ψ and Δ for the sample before the deposition of Fe and the spectra calculated on the basis of the optimized model. The discrepancy between the experimental and calculated dependences in the short-wavelength spectral region is due to the low intensity of the useful signal.

In monitoring the thickness and optical properties of the growing Fe film by single-wave ellipsometry, we determined the resultant value of its thickness (12.5 ± 0.1 nm). To verify the results of calculations, we studied the resultant structure ex situ by X-ray spectral fluorescent analysis (XSFA). The thickness of the iron film obtained using XSFA was 12.4 ± 0.6 nm. Thus, we can state that the values of the Fe film thickness obtained by XSFA and single-wave ellipsometry coincide.

As well as for the silicon substrate with a SiO_2 buffer layer, we carried out the fitting of parameters of the optical model with sharp interfaces between the homogeneous continuous Fe film, the homogeneous continuous SiO_2 film, and the semi-infinite Si medium from the results of measurements [14] and determined the structural parameters of the sample after sputtering; the thickness of the SiO_2 layer was 0.4 ± 0.1 nm as before and the effective thickness of the Fe film was 12.4 ± 0.1 nm. The fitting of thicknesses in all spectral ellipsometric experiments was carried out by the Nelder–Mead simplex method. The results of measurements of the spectral dependences of ellipsometric angles after sputtering of the iron film are also shown in Fig. 1 together with the data calculated using the optimized model.

The results of measurements of the magnetization reversal loop based on the magneto-optical Kerr effect are shown in Fig. 2. The wavelength of probe radiation in this case was 500 nm. Analysis of the results leads to the conclusions concerning certain magnetic properties of the resultant structure; in particular, the coercive force of the Fe film in our experimental geometry was 120 ± 6 Oe and the saturation field was ~300 Oe.

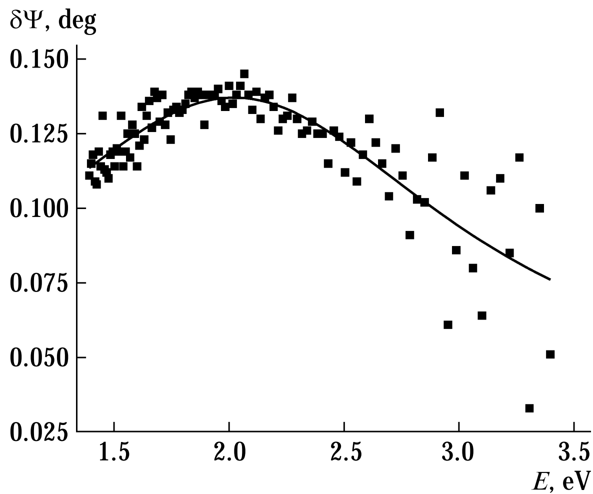

Fig. 3. Energy dependence of the magneto-optical Kerr effect, measured for the $Fe/SiO_2/Si(100)$ structure by the in situ method.

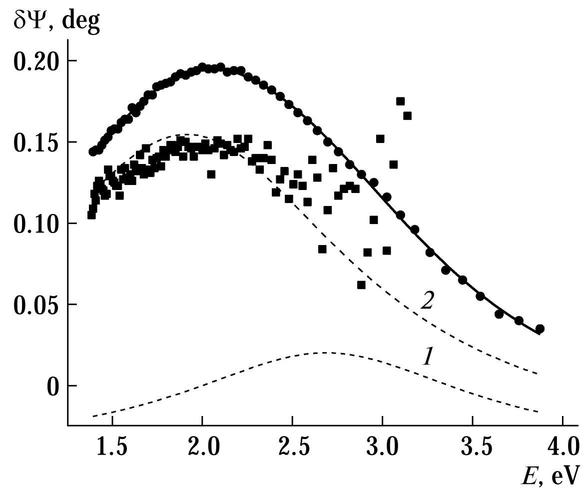

Fig. 4. Energy dependence of the magneto-optical Kerr effect, measured for the $Fe/SiO_2/Si(100)$ structure by the ex situ (circles) and in situ (squares) methods. Dotted curves are the results of decomposition of the spectrum into Gaussian peaks. The solid curve is the sum of these Gaussian peaks.

The resultant value of the coercive force corresponds to the data obtained in [15, 16] for the corresponding thicknesses in the case of a homogeneous film, but not of the island structure [17]. For example, according to the assumption made in [18], an increase in the porosity of the layer leads to a considerable increase in the coercive force only for a ferromagnetic layer with a thickness larger than 100 nm. For homogeneous Fe layers with a thickness slightly exceeding 8 nm, for which the coalescence of Fe islands in the surface into a continuous film is completed at a temperature of about 250°C, the value of the coercive force varies insignificantly upon a further increase in the thickness and remains in the interval 100–200 Oe [19], which is confirmed in this study.

Figure 3 shows the results of in situ measurement of the energy dependence of the magneto-optical Kerr effect in the range 1.4–3.4 eV. A peak in the region of 2.04 ± 0.03 eV can be clearly seen. However, in view of the weak signal intensity in the short-wavelength spectral region in the in situ magnetoellipsometer, the data exhibit a considerable energy spread, which complicates their analysis. For this reason, additional measurements of the EKE spectrum were taken on an ex situ magnetoellipsometer in the range 1.4–3.8 eV.

Figure 4 shows the results of measurements of the EKE spectrum. The differences in the peak intensities for the two spectra are due to different angles of incidence of light for in situ and ex situ measurements. The resultant energy dependence of the EKE coincides qualitatively with the results of investigations performed in [20] for bulk iron.

The energy dependence of the magneto-optical Kerr effect is determined by a number of factors, including the density of electron states of iron atoms. In [21], experiments were made on a polycrystalline 10-nm-thick Fe film using photoemissive spectroscopy; the results indicate the existence of an analogous peak in the density of electron states at 2 eV.

After the decomposition of the peak obtained by us and shown in Fig. 4 into two Gaussians with the maxima at 1.92 ± 0.10 and 2.69 ± 0.19 eV, we can compare them with the results of calculations of the density of states for iron [22, 23] and see that these peaks are formed by the t_{2g} states (curve *1* corresponds to electrons with spin up and curve *2* corresponds to electrons with spin down). Calculations [22] of the temperature-dependent band structure were performed by the LDA + DMFT method taking into account electron correlations. Calculations in [23] were performed using the spin-dependent methods in the theory of the density functional. It should be noted that the resultant exchange splitting 0.8 ± 0.3 eV of the bands with spin up and spin down is close to that obtained by inverse photoemission spectroscopy in [24, 25].

CONCLUSIONS

Summarizing the result of this research, we can note that the developed methods of simultaneous measurement of ellipsometric and magneto-optical parameters (we use the term magnetoellipsometry for such methods) directly in the growth chamber under ultrahigh vacuum conditions make it possible to analyze the structure of films with a thickness on the order of 1 nm as well as their magnetic properties in a single experimental cycle. Processing of the Kerr effect spectra has made it possible to obtain information on the spin-dependent density of electron states, which is in conformity with the results of theoretical calculations and with the results of the previous ex situ measurements.

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