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In Situ Conductance of Fe/Si and Fe/Ge Multilayers

P. Chomiuk[a,*], M. Wróblewski[b], M. Błaszyk[a], T. Luciński[a] and B. Susła[b]

[a]Institute of Molecular Physics, Polish Academy of Sciences
M. Smoluchowskiego 17, 60-179 Poznań, Poland
[b]Institute of Physics, Faculty of Technical Physics
Poznań University of Technology
Nieszawska 13a, 60-965 Poznań, Poland

In this paper we study Fe/Si and Fe/Ge multilayers prepared at room temperature by magnetron sputtering. *In situ* conductance measurements reveal the formation of interfacial Fe–Si and Fe–Ge mixtures. During the Fe deposition a modification of growth mode is noticed. Deposition of Si (or Ge) onto Fe leads to the reduction of the Fe layer thickness due to interdiffusion, and Fe–Si (or Fe–Ge) structures appear. Above about 1.3 nm of deposited Si (1.5 nm of Ge) nominally pure Si (Ge) starts growing. Surface topography of the Fe/Si multilayers is studied by atomic force microscopy.

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1. Introduction

The metal/semiconductor multilayered systems attract a lot of attention due to potential application in integrated metal–semiconductor devices. One of the most interesting of these structures seems to be Fe/Si multilayers (MLs) because of the strong antiferromagnetic (AF) coupling (see [1–4] and references therein). A lot of efforts have been made in order to clarify the origin of the interlayer interaction and structural profile of the Fe/Si MLs [1–4]. Although the Fe/Ge system shows no AF coupling, studies on it may show similarities and differences between these systems and bring a lot of useful information about the origin of the exchange coupling. In both systems intermixing at interfaces occurs and can lead to the appearance of various structures similar to Fe–Si and Fe–Ge phases [2, 4].

*corresponding author; e-mail: chomiuk@ifmpan.poznan.pl

In our previous papers we investigated Fe/Si and Fe/Ge MLs by the Hall effect and the Mössbauer spectroscopy [2–4]. In this contribution we compare and discuss their changes of conductance vs. deposition time.

2. Experimental

The *in situ* conductance measurements were performed with two-point method during deposition of the following samples: $[\mathrm{Fe}(3\ \mathrm{nm})/\mathrm{Si}(d_{\mathrm{Si}})]_{15}$ + Fe(3 nm) for Si thicknesses $d_{\mathrm{Si}} = 1.3$ and 2.5 nm, corresponding to the maximum and the absence of AF coupling [2, 4], respectively; and $[\mathrm{Fe}(3\ \mathrm{nm})/\mathrm{Ge}(2\ \mathrm{nm})]_{15}$ + Fe(3 nm). The specimens were deposited onto oxidised Si substrates at ambient temperature by magnetron sputtering. The vacuum during the experiment was better than 10^{-5} Pa, and argon partial pressure was in the range of 0.1 Pa. The deposition rates of iron, silicon, and germanium were 0.0293 nm/s, 0.0547 nm/s, and 0.0552 nm/s, respectively. Temperature T during the deposition was controlled by a constantan–copper thermocouple with the reference terminal kept at 0°C. Surface topography images were obtained by atomic force microscope operating in a tapping mode, on a series of samples $[\mathrm{Fe}(3\ \mathrm{nm})/\mathrm{Si}(1.1\ \mathrm{nm})]_N$ + Fe(3 nm) for $N = 5$, 10, and 15. The analysis of the atomic force microscopy (AFM) images was performed with the use of WSxM program [5].

3. Results and discussion

The exemplary plot of conductance vs. deposition time $G(t)$ for Fe(3 nm)/Si(1.3 nm) ML is shown in Fig. 1. All the $G(t)$ dependences discussed here exhibit a similar behaviour. The oscillations due to alternate deposition of metal and semiconductor result in the increase and decrease of the conductance. The conductance oscillates around a base line, which can be fitted by a straight line. This behaviour is opposite to metallic MLs (Py/Cu, e.g. [6]), where the base line is bent and tends to saturate. This indicates that the iron sublayers are embedded between almost insulating Si or Ge layers, thus the conductance of the whole ML is a sum of conductances of individual Fe layers. The presence of potential barrier between Fe layers in the Fe/Si system was previously confirmed by current–voltage characteristics, which exhibited a nonlinear dependence [3].

Let us analyse step-by-step the $G(t)$ dependences. The percolation threshold for iron deposited on oxidised Si substrate occurs at $d_{\mathrm{Fe}} \approx 0.9$ nm. Since the first Fe layer is deposited on the silicon oxide surface, and every other onto Si or Ge layer, the deposition of a few initial Fe/Si and Fe/Ge bilayers results in a different shape of the conductance dependence. The G vs. t plot stabilises and becomes repeatable after about 5 bilayer cycles. A further development shows minor changes in its shape.

Figure 2 shows an exemplary G vs. t plot of Fe/Si bilayer extracted from Fig. 1 and a schematic presentation of model describing the bilayer's growth. The section corresponding to Fe deposition can be divided into 3 parts. (i) Initially,

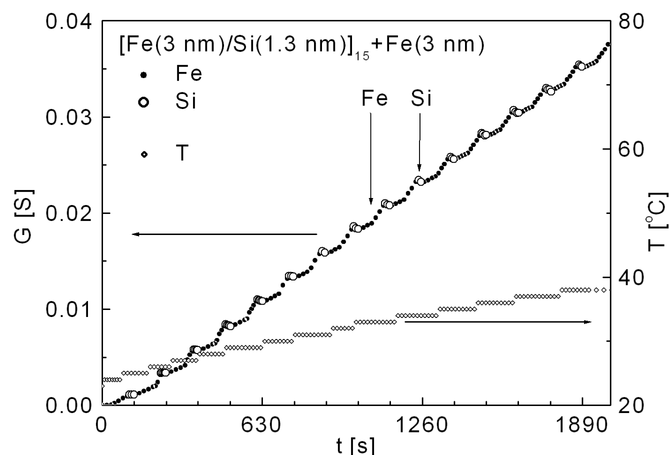

Fig. 1. The *in situ* conductance and the sample temperature as a function of deposition time of Fe(3 nm)/Si(1.3 nm) ML.

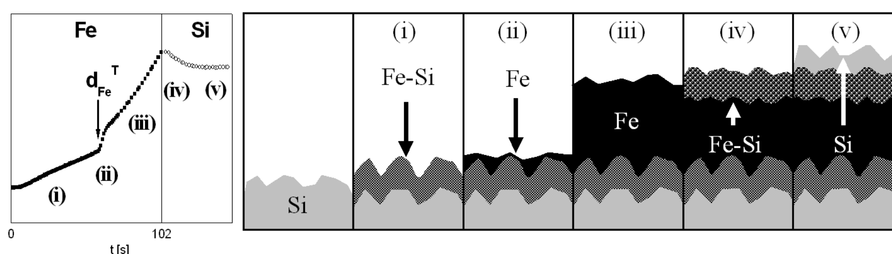

Fig. 2. The model describing deposition stages of Fe/Si MLs.

iron deposited onto Si layer interdiffuses into Si and a Fe–Si mixture is formed, whose conductivity is less than that of pure Fe layer. This is reflected by slow increase in conductance in the plot. (ii) For $d_{\mathrm{Fe}} \approx 1.7-2.6$ nm (see discussion related to Figs. 3 and 4) of sputtered Fe, the growth of pure iron layer starts. The steep increase in the $G(t)$ dependence is supposedly due to the surface flattening. (iii) Next the growth of the bcc-Fe phase is continued, which results in a further increase in conductivity in a stable way. The presence of the bcc-Fe phase and Fe–Si mixtures has been confirmed by the Mössbauer spectroscopy [2, 4]. The deposition of Si onto Fe can be divided into 2 steps: (iv) The initial decrease in conductance can be explained as an effect of Si and Fe intermixing. In such a case a part of the top iron layer is transformed into a Fe–Si mixture, thus a low conductive Fe–Si mixture layer appears. This process leads to the reduction of the effective Fe layer thickness. (v) A further deposition of Si (or Ge), i.e., for $d_{\mathrm{Si}} > 1.3$ nm ($d_{\mathrm{Ge}} > 1.5$ nm), gives rise to the growth of nominally pure, almost insulating Si (or Ge) layer. Such layers do not contribute to the conductivity of the whole stack, thus the saturation of the G vs. t dependence is observed.

Interestingly, the AF coupling is observed strictly for a very narrow range of silicon spacer thicknesses ($d_{\mathrm{Si}} \approx 1.1-1.3$ nm) [2]. For thicker spacers, consisting of nominally pure Si besides the Fe–Si mixtures, the AF coupling disappears. This is in agreement with our previous research that for the occurrence of the AF coupling nonmagnetic Fe–Si mixtures must be formed [3]. In the case of Ge deposited onto Fe intermixing occurs up to $d_{\mathrm{Ge}} \approx 1.5$ nm, but as shown in [2, 4] only Fe–Si mixtures present in the spacer can mediate the AF coupling.

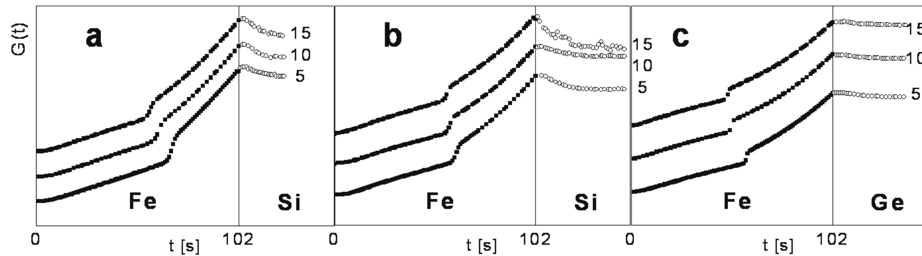

Fig. 3. The detailed view on G vs. t dependences for 5th, 10th, and 15th bilayer of Fe(3 nm)/Si(1.3 nm) (a); Fe(3 nm)/Si(2.5 nm) (b); and Fe(3 nm)/Ge(2 nm) MLs.

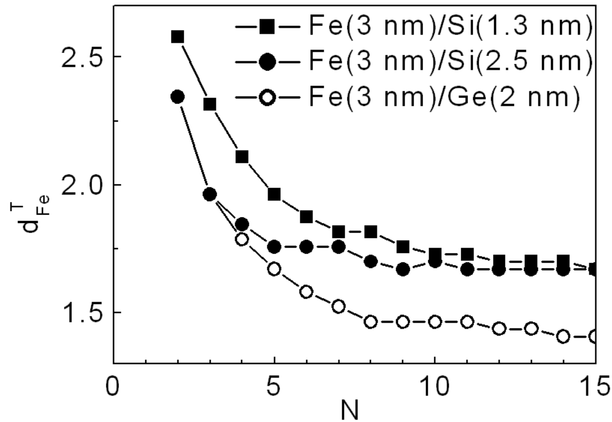

Fig. 4. Iron thickness of growth mode transition as a function of number of deposited bilayers.

A further part of this paper is devoted to the evolution of the $G(t)$ dependence of the investigated MLs. The G vs. t plots for 5th, 10th, and 15th bilayer of the examined MLs are exhibited in Fig. 3. The iron thickness corresponding to its growth mode transition is designated by $d_{\mathrm{Fe}}^{\mathrm{T}}$ (see Fig. 2). As it is clearly seen in Fig. 3, $d_{\mathrm{Fe}}^{\mathrm{T}}$ decreases with increasing number of deposited bilayers. The dependence of $d_{\mathrm{Fe}}^{\mathrm{T}}$ vs. bilayer number N is shown in Fig. 4. In the Fe/Si MLs $d_{\mathrm{Fe}}^{\mathrm{T}}$ varies between 2.6 nm (for $N = 2$) and 1.7 nm (for $N > 10$). For low N the changes are

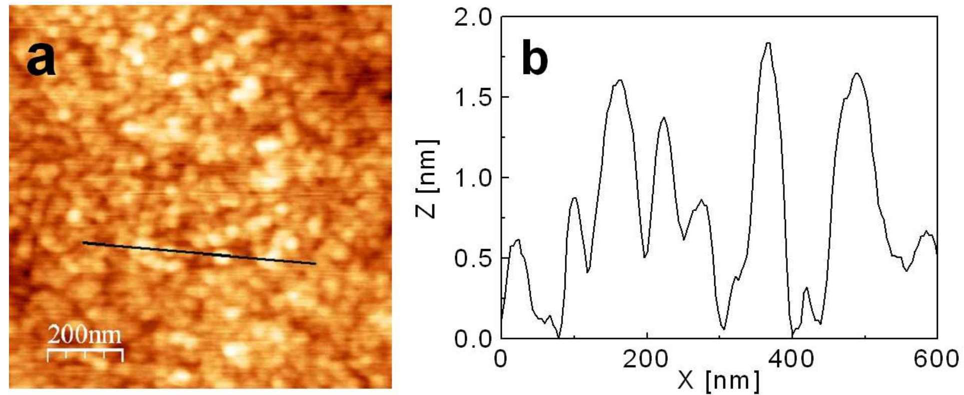

Fig. 5. An AFM image of $1 \times 1\mu\text{m}^2$ area (a); and an exemplary profile (b) of $[\text{Fe/Si}]_{10}$ ML.

more pronounced and the plot stabilises for higher N. Moreover, for the thicker Si spacer the saturation in the d_{Fe}^{T} vs. N dependence occurs more rapidly. For the Ge spacer d_{Fe}^{T} decreases to about 1.5 nm. This is not due to warming up the sample during deposition, because the temperature increase during the whole process is less than 20°C (see Fig. 1).

In order to examine the roughness at Fe/Si interfaces, the AFM study was performed on a series of $[\text{Fe(3 nm)/Si(1.1 nm)}]_N$ + Fe(3 nm) MLs with $N = 5$, 10, and 15. We presume that the roughness measured after deposition of several bilayers reflects the roughnesses at corresponding interfaces in the ML. Figure 5a shows an exemplary AFM surface image of 1×1 μm^2 area of a Fe/Si ML with $N =$ 10. The surface is rather flat with rms roughness much less than the considered sublayer thicknesses, and it slightly increases with growing number of repetitions N (from 0.31 up to 0.45 nm). Figure 5b exhibits an exemplary surface profile of the ML shown in Fig. 5a. The lateral grain size is within the range of 30–50 nm, and the average height is 1.43 nm. Since the lateral grain size is much larger than its height, we cannot correlate the roughness and the d_{Fe}^{T} dependences.

4. Conclusions

In situ conductance measurements provide real-time observations of intermixing processes during multilayer growth. In the Fe/Si and Fe/Ge MLs, during deposition of Fe onto Si (or Ge), a Fe–Si (Fe–Ge) mixture is formed up to about 1.7–2.6 nm (1.5–2.3 nm). Then a bcc-Fe phase starts growing. When Si or Ge is deposited onto Fe, a decrease in conductance is observed, due to the transformation of iron into Fe–Si (Fe–Ge) mixture, which reduces the Fe layer thickness. A further deposition of Si (or Ge), above $d_{\text{Si}} \approx 1.3$ nm (or $d_{\text{Ge}} \approx 1.5$ nm) leads to the growth of highly resistive Si (or Ge).

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