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Thermal stability of epitaxial Fe films grown on Si substrates by molecular beam epitaxy

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ABSTRACT

Epitaxial Fe films are grown on Si(0 0 1) and Si(1 1 1) substrates by molecular beam epitaxy at room temperature. Several samples of one Fe/Si structure are subjected to rapid thermal annealing from 100 to 500 °C. The annealing impact on the morphological, magnetic properties and interfacial heterostructures of these samples is examined by atomic force microscopy, vibrating sample magnetometer and transmission electron microscopy, respectively. The results demonstrate that the material system Fe/Si grown at room temperature exhibits an abrupt interface and is thermally stable up to a temperature of 150 °C.

1. Introduction

Fe and Si, the two common elements in Earth, are two very promising materials. For example, Fe exhibits a high spin polarization as well as a Curie temperature well above 300 K [1,2] and Si is convenient to achieve and use as a flat or stepped substrate for material epitaxy [3,4]. As one of their combination, Fe/Si heterostructure has captivated the imagination of many researchers over the past decades because of its importance in fundamental studies and potential applications [5–11]. For this purpose, it is desirable for an ideal heterostructure to inhibit chemical reactions between the Fe and Si. Hence, one has to make it clear the thermal stability of the Fe/Si system prior to its research or application design.

In this respect, the Fe/Si interface has been sensitively investigated by many groups [12–24]. Unfortunately, their results demonstrate an intense contradiction for the state of the interface with Fe grown at room temperature (RT). We classified most of the representative reports and found that the state of Fe/Si interface strongly depends on the deposition methods and conditions of Fe on Si. Specifically, on Si with a substrate temperature nearby RT, Fe deposited by resistively heated filament [13,14,17], pulsed laser deposition [18] and electron beam evaporation [16,20] usually results in a reactive silicide-like interface. On the contrary, Fe evaporated from an evaporation cell in a molecular beam epitaxy (MBE) system could grow chemically pure Fe films on Si [15,25]. The thermal stability of the Fe/Si structure with abrupt interface grown by MBE under consideration is thus of utmost importance for its use in applications.

In this letter, we report the growth of epitaxial Fe films on Si(0 0 1) and Si(1 1 1) substrates by MBE at RT and their subsequent annealing at temperatures between 100 and 500 °C. We find that the surface morphology, the saturation magnetization and the abrupt interface of the Fe/Si heterostructure are unaffected up to 150 °C. Evidence for obvious chemical reaction at interface are observed only at 200 °C and above.

2. Experimental

The epitaxial growth of Fe on Si(0 0 1) and Si(1 1 1) at RT was performed in an MBE system equipped with solid-source effusion cells for Fe. Prior to growth, Si substrate was chemically cleaned by trichloroethylene, acetone, methanol, and a final HF dip (5%, 2 min). After the substrate was loaded into the growth chamber, the substrate was further thermally cleaned at an annealing temperature of 800 °C. By cooling the substrate down to RT, Fe then grew at a rate of 0.13 nm/min to a final thickness of 30 nm. Nucleation and growth were monitored *in situ* by reflection high-energy electron diffraction (RHEED). The RHEED patterns (not shown here) evidence that on Si(0 0 1) three crystallographic orientation relationships coexist in contrast to on Si(1 1 1) a pure single orientation relationship exists. Namely, on Si(0 0 1) multi-domains with Fe(0 0 1)[1 0 0]||Si(0 0 1)[1 0 0], Fe(1 1 0)[$\bar{1}$ 1 0]||Si(0 0 1)[1 1 0] and Fe(1 1 0)[0 0 1]||Si(0 0 1)[1 1 0] coexist, which is in agreement

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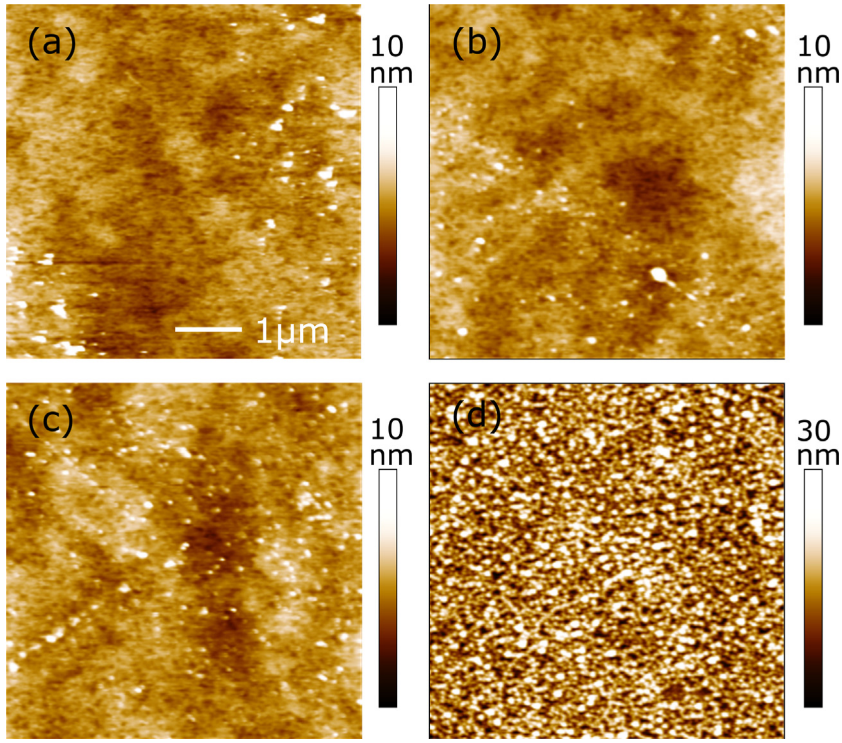

Fig. 1. AFM micrographs (5×5 μm^2) of: (a) the as-grown RT Fe/Si(0 0 1) sample, and samples annealed at (b) 100 °C, (c) 150 °C and (d) 300 °C, respectively.

with the result reported in Ref. [26]. While on Si(1 1 1) only a single domain with Fe(1 1 1)[$\bar{1}$ 1 0]||Si(1 1 1)[$\bar{1}$ 1 0] is observed, which agrees with the result reported in Ref. [25]. The surface morphology of the films was examined by atomic force microscopy (AFM). In-plane hysteresis loops were recorded at RT by vibrating sample magnetometer (VSM). All magnetization data presented here were corrected for the diamagnetic background of the substrate. The interface state of Fe/Si heterostructure was investigated by cross-sectional transmission electron microscopy (TEM). Rapid thermal annealing (RTA) of samples cut from one wafer was carried out in an Ar ambient for 1 min at temperatures between 100 and 500 °C.

3. Results and discussion

Fig. 1(a) shows the AFM micrograph of an as-grown RT Fe/Si(0 0 1) structure with a 30-nm thick Fe layer including a surface natural oxide. The surface exhibits a root-mean-square (rms) roughness of 0.9 nm over 5 × 5 μm^2, which is quite smooth in comparison with Fe films grown on GaN layers [27]. No obvious changes are observed for this structure annealed at 100 and 150 °C as depicted in Fig. 1(b) and (c), respectively. However, Fig. 1(d) shows that annealing at 300 °C induces a larger rms roughness of 4.7 nm over 5 × 5 μm^2. For annealing temperatures higher than 300 °C, the surface of this structure is induced even rougher up to an rms value of 6.3 nm at 500 °C (not shown here).

Fig. 2 shows in-plane hysteresis loops for the Fe/Si(0 0 1) heterostructure measured by VSM with the magnetic field (H) parallel to the Si[1 1 0] direction at RT (i.e. H parallel to Fe⟨0 0 1⟩ and Fe⟨1 1 0⟩ for different orientation domains as aforementioned). The as-grown Fe film exhibits a square hysteresis loop with a coercivity of 50 Oe. Upon annealing up to a temperature of 150 °C, coercivity and saturation magnetization (M_S) stay constant. Furthermore, we observe a progressive increase in coercivity with increasing annealing temperature. At temperatures above 200 °C, M_S drops rapidly, and the hysteresis loops develop an S-shape. This shape is related

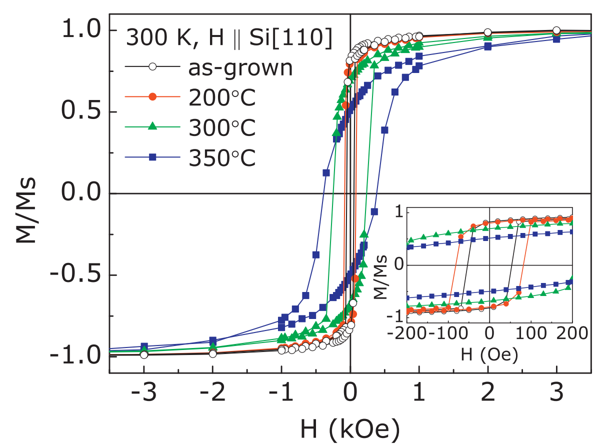

Fig. 2. Hysteresis loops obtained at RT for as-grown Fe/Si(0 0 1) sample and samples annealed at 200, 300 and 350 °C. The inset magnifies the low magnetic field range.

to a change of composition, which coincides with a starting and consequent reaction of Si with the Fe film as the state of interface detected by TEM. The properties and the state of the new phases at interface change the M_S, anisotropy, domain wall motion and thus the shape of the hysteresis.

To quantitatively show the impact of the annealing to M_S, we compare the saturation magnetization measured after annealing, $M_S(T)$, with that measured before annealing for the same piece of the sample, which could avoid errors from sample-area measurements. Fig. 3 quantitatively summarizes the results concerning the magnetic properties and morphology of the Fe films on Si(0 0 1), as well as on Si(1 1 1). Evidently, a thermal treatment of these films up to 150 °C demonstrate no obvious impact from annealing. Annealing temperatures higher than 200 °C should, however, be avoided, as they lead to the changes of the morphology and magnetic properties of the Fe films.