



# Magnetic properties of electrodeposited Fe/Au(1 1 1) layers: in situ AGFM measurements

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## Abstract

The magnetization state of electrodeposited Fe/Au(111) layers is investigated during the growth using in situ alternating gradient force magnetometry. It is shown that perpendicular magnetization anisotropy occurs as long as the thickness  $t_{\text{Fe}} < 2$  ML. The effect of depositing Cu on top is also studied. Results are compared to those obtained with MBE layers and discussed in the light of preliminary STM investigations of Fe growth. © 2001 Elsevier Science B.V. All rights reserved.

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It is well known that the magnetic moment of Fe atoms greatly depends on the crystal structure (FCC versus BCC) and its lattice parameter [1]. Therefore this makes the comparison of films prepared by different techniques highly interesting to eventually correlate different structure to the magnetic state. This work is a preliminary study of electrodeposited Fe/Au(111) layers. An in situ alternating gradient force magnetometer (AGFM) set-up was developed where the sample is immersed in an electrochemical cell and connected to a potentiostat for well-defined electrochemistry. The perpendicular ( $M_{\perp}$ ) and parallel ( $M_{\parallel}$ ) components of the magnetization may be probed from the very initial stages of the metal growth. Hysteresis loops can also be recorded. As shown below, results indicate that the occurrence of perpendicular magnetization anisotropy (PMA) depends on the surface chemistry of Fe.

The top of Fig. 1 gives the electrochemical response of the gold electrode in a 1 mM FeSO<sub>4</sub> solution of pH 4 (potentials are quoted versus the Hg/Hg<sub>2</sub>SO<sub>4</sub> reference electrode). The samples consisted of textured Au(111)

100 nm-thick-films evaporated on mica that were flame annealed before use to obtain the  $22 \times \sqrt{3}$  surface reconstruction [2]. Fig. 1 indicates that deposition occurs for  $U < -1.45$  V. Stripping of the layer is monitored by reversing the potential scan ( $U > -1.2$  V). Integration of the charge  $Q$  under the anodic peak was used to derive the iron thickness, assuming the reaction  $\text{Fe} \rightarrow \text{Fe}^{2+} + 2e^{-}$  [1 ML of  $\gamma$ -Fe(111)  $Q = 560 \mu\text{C}/\text{cm}^2$ ]. The negative wave at  $-1.3$  V is the reduction of protons. The bottom panel of Fig. 1 shows the transients of the magnetization  $M_{\perp}(t)$  and  $M_{\parallel}(t)$  recorded during Fe deposition at  $U = -1.45$  V ( $t > 20$  s). No external field  $H_0$  was applied (remanent field between poles is 30–50 Oe). The primary information is the ferromagnetic state of the iron layer at room temperature for a thickness well below  $t_{\text{Fe}} < 4$ –5 ML (this is the thickness measured after 50 s of deposition). The second remark is the occurrence of PMA for  $t_{\text{Fe}} < 1.8$  ML. In fact,  $M_{\perp}(t)$  peaks at  $t = 42$  s while  $M_{\parallel}(t)$  remains zero over the same time. Note that the delay between the step of potential ( $t = 20$  s) and the detection of an  $M_{\perp}(t)$  signal ( $t = 27$  s) is attributed to the progressive nucleation of the Fe layer. As the film thickness increases,  $M$  switches to become totally in-plane ( $M_{\perp}(t) = 0$ ). This transition is completed above a critical thickness  $t_{\text{Fe}}^* \sim 2.5$  ML.

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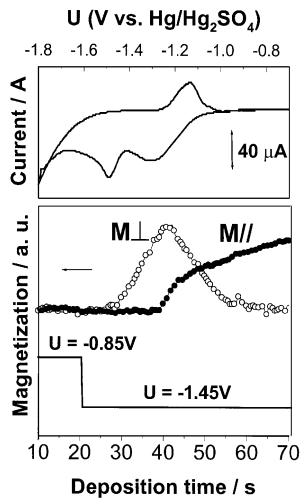


Fig. 1. Top panel: electrochemical response of the Au(111) electrode in the Fe solution; Bottom panel: transient curves  $M(t)$  in  $\perp$  (open symbols) and  $\parallel$  (bold symbols) configurations. The applied potential  $U$  is also shown as a function of time.

The hysteresis loops (HL) displayed in Fig. 2 were obtained after stopping deposition by setting  $U = -1.25$  V (the deposit neither grows further nor dissolves). The Fe thickness was  $\sim 1.5$  ML. Bold symbols correspond to the *naked* Fe layer. With  $H_{\perp}$  the square and open ( $H_C = 50$  Oe) loop is a confirmation of PMA. The corresponding HL is reversible with  $H_{\parallel}$ . For thicker layers ( $t_{\text{Fe}} > 2$  ML) reversible HL's are obtained with  $H_{\perp}$  and square ones with  $H_{\parallel}$  (not shown). The experiment in Fig. 2 was continued by depositing in situ a Cu layer atop the Fe film (trace amounts of  $\text{CuSO}_4$ , were added to solution, still keeping the potential  $U = -1.25$  V, so as to obtain a thin Cu deposit, without exposing the Fe to air). The resulting HL's (open symbols) indicate that  $M$  becomes in-plane upon Cu deposition.

The results above demonstrate that electrodeposited Fe layers exhibit remarkable magnetic properties at *room temperature*, showing ferromagnetism down to 1 ML equivalent thickness. This is in contrast with evaporated Fe(110) layers on Au(111) [3] and pulsed laser layer deposited Fe(111) films on Cu(111) [4] for which the Curie temperature remains well below RT. Given Ref. [1] and preliminary in situ STM observations which indicate that Fe grows nearly layer by layer up to 4–5 ML [2], we suspect that FCC  $\gamma$ -Fe(111) layers are growing electrochemically. From STM there is no clear indication of a phase transition  $\gamma$ -Fe(111)  $\rightarrow$   $\alpha$ -Fe(111) below 5 ML thickness. Such a transition occurs around 3 ML during

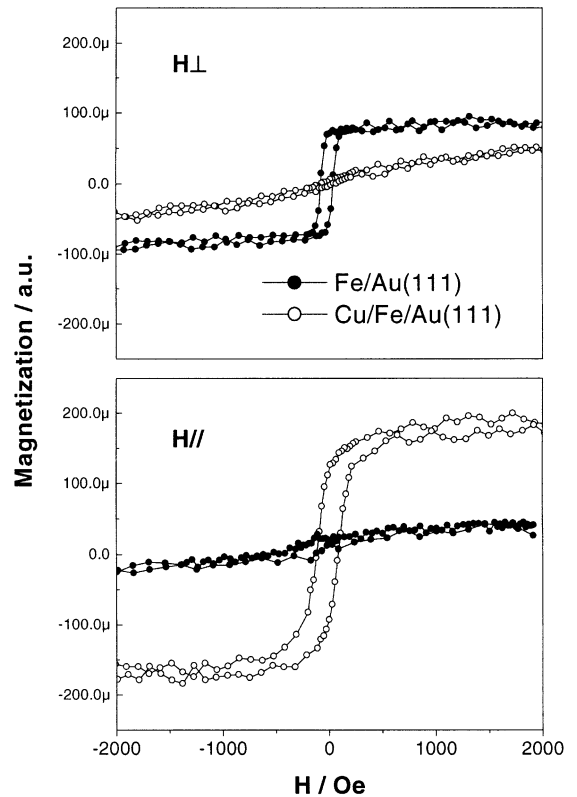


Fig. 2. In situ AGFM Hysteresis loops in  $\perp$  (top panel) and  $\parallel$  (bottom panel) configurations. Bold and open symbols correspond to the naked and Cu-covered Fe layers. The iron thickness is 1.5 ML.

MBE deposition [5,6]. This difference is attributed to the possible formation of an anion layer at the Fe/Solution interface. This layer likely stabilizes the metastable  $\gamma$  phase under tensile stress since anion layers often generate compressive stress on metallic layers. Under this hypothesis, the disappearance of the PMA after Cu deposition (Fig. 2, open symbols) might arise from a phase transition inside the Fe layer. Namely, the removal of the anionic layer would lead to collapse of the metastable Fe/Au structure.

## References

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