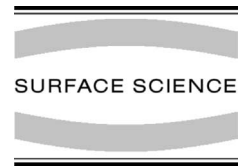




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# Conservation of uniaxial symmetry in Fe/Ag multilayers grown on stepped Ag(0 0 1)

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## Abstract

We obtained a stepped Ag(001) surface by growing a 100 nm thick Ag film on a polished MgO(001) crystal with a miscut of  $1.8^\circ$  in the  $[\bar{1}10]$  direction. With scanning tunneling microscopy we observe terraces of  $(7 \pm 3)$  nm wide separated by mono-atomic steps that are straight over at least 200 nm in the  $[110]$  direction. The interface structure of Fe/Ag multilayers grown at room temperature (RT) on vicinal Ag(001) is investigated for 15 monolayers (ML) of Fe and a Ag thickness ranging from 2 to 10 ML. During Fe growth the step structure disappears at the surface of the film. When Ag is deposited on top, steps already reappear at 2 ML. For increased Ag thickness, the steps straighten out and the roughness on terraces decreases. At 8 ML of Ag the step length and surface roughness is comparable to the uncovered Ag surface. However, on larger terraces, Ag islands are observed. In addition we compare the direction of magnetization with respect to the surface normal of an  $[\text{Fe}(15 \text{ ML})/\text{Ag}(4 \text{ ML})]_{16}$  multilayer grown on vicinal and flat Ag(001). For the vicinal substrate, at RT, we observe in-plane magnetization whereas for the flat substrate the magnetization is  $29^\circ$  out-of-plane. The results are discussed with respect to step anisotropy and step-related symmetry. © 2001 Elsevier Science B.V. All rights reserved.

**Keywords:** Iron; Silver; Magnetic films; Interface states; Vicinal single crystal surfaces; Mössbauer spectroscopy; Scanning tunneling microscopy

## 1. Introduction

Step-induced anisotropy in magnetic films and multilayers can be studied using vicinal substrates. Furthermore, a high density of steps in one crystallographic direction creates a uniaxial symmetry at the surface. For a film of Fe on Ag(001) it was

shown that the uniaxial anisotropy caused by the presence of steps depends quadratically on the step density [1]. Step-induced anisotropy was also observed in Fe/Ag multilayers. A considerable canting out of the film plane was reported for a 15 ML Fe film [2] whereas the spin reorientation transition (SRT) from out-of-plane to in-plane usually takes place at about 6 ML [3,4]. This canting disappears for increasing Ag film thickness corresponding to a decrease of steps (in the  $[110]$  and  $[\bar{1}10]$  directions) at the Fe on Ag interface in the multilayer. In this work we report on the interface structure in Fe/Ag multilayers grown on vicinal

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Ag(001) with steps in the [110] direction, using scanning tunneling microscopy (STM). Furthermore we investigated the magnetization with conversion electron Mössbauer spectroscopy (CEMS) and vibrating sample magnetometry (VSM).

## 2. Experimental

The experiments are carried out in an ultra high vacuum (UHV) system described elsewhere [5]. Vicinal Ag(001) is prepared on a polished MgO(001) crystal with a miscut of  $1.8^\circ$  in  $[\bar{1}10]$  direction. The MgO is cleaned in isopropanol and annealed in UHV to 875 K prior to growth. First, a 5 nm Cr buffer layer is grown at 450 K to improve epitaxy of a 100 nm thick Ag film, which is grown at room temperature (RT) [6]. The Ag is evaporated from a thermal Knudsen cell at 1265 K with a growth rate of  $1 \text{ \AA/s}$ . The Ag film is annealed at 475 K after deposition to obtain a smooth surface. We note that this is exactly the same procedure for flat Ag(001) as reported previously [5]. Natural Fe is evaporated from a mass spectrometer stabilized e-gun at a deposition rate of  $0.12 \text{ \AA/s}$ ,  $^{57}\text{Fe}$  (used as a probe for CEMS) from a thermal Knudsen cell at 1575 K with  $0.07 \text{ \AA/s}$ . The Fe/Ag multilayers are grown at RT on Ag(001) (both vicinal and flat).

## 3. Results and discussion

In Fig. 1a, an STM topograph is shown of the vicinal Ag(001) surface. From evaluation of a large area, a value of  $(7 \pm 3) \text{ nm}$  for the width of the terraces is obtained. We also observed occasional screw dislocations (3–4 every  $400 \times 400 \text{ nm}^2$ ) due to crystallographic imperfections in the MBE-grown film. The root mean square (rms) roughness is calculated for both flat and vicinal Ag(001). For the flat surface we obtain an rms of  $0.14 \text{ nm}$  and for the vicinal case  $0.4 \text{ nm}$ . This increased roughness is due to the spread in width of the terraces on the vicinal surface, which leads to a surface rippling on a scale of a few hundreds of nm whereas flat Ag(001) consists mainly of large terraces with no preferred step directions.

After deposition of 15 ML of Fe, the step structure has disappeared (Fig. 1b). Islands with a diameter of  $(7 \pm 1) \text{ nm}$  are observed separated by valleys of 2 ML deep. When this film is covered by 2 ML of Ag (Fig. 1c), the step structure starts to reappear, although the steps are still rough (Table 1). On the terraces, there are islands with an irregular shape, in the order of a few nanometer long and 1 ML high. We also observed depressions of the order of  $0.1 \text{ nm}$  deep. All these features lead to a high rms value of  $0.07 \text{ nm}$  measured on a single terrace (Table 1). For an additional 2 ML of Ag (Fig. 1d), the steps straighten out and the terraces become smoother (Table 1). No islands are observed. Depressions are still visible though. For 6, 8 and 10 ML Ag, the steps become longer and the rms values become comparable to the rms on a terrace of uncovered vicinal Ag(001). However, on the larger terraces, Ag islands start to grow leading to increased rms values as shown in Table 1. In Fig. 1f such a large terrace is shown. From 8 ML onwards we observe areas of a few nanometer in diameter, that are 1 ML deeper than the terrace level.

$[^{57}\text{Fe}(15 \text{ ML})/\text{Ag}(4 \text{ ML})]_{16}$  multilayers were grown on vicinal and flat Ag(001) in the same run. We did not observe drastic changes in the RHEED patterns during growth for both multilayers. The direction of magnetization at RT with respect to the surface normal was determined with CEMS. Since the measurements are performed ex situ, the multilayer was covered with a Ag layer of 10 nm. The obtained spectra are shown in Fig. 2. The two spectra have different relative line intensities, indicating that the direction of magnetization is different. The orientation of the spins can be calculated from the ratio of the line intensities given by  $3:x:1:1:x:3$  with  $x = 4\sin^2\theta/(1 + \cos^2\theta)$ ,  $\theta$  the angle between the incident  $\gamma$ -ray and the direction of the magnetization [7]. For in-plane magnetization one should observe 3:4:1:1:4:3, whereas out-of-plane results in 3:0:1:1:0:3. The spectra taken from the multilayers (15 ML of  $^{57}\text{Fe}$  in each bilayer) were fitted with two sextets. A first sextet with a relative population of  $\sim 11/15$  corresponds to a site in the middle of the film with a hyperfine field of 31.3 T. The other sextet was fitted with a Gaussian distribution ( $\sigma = 1.5 \text{ T}$ ) around 29.8 T

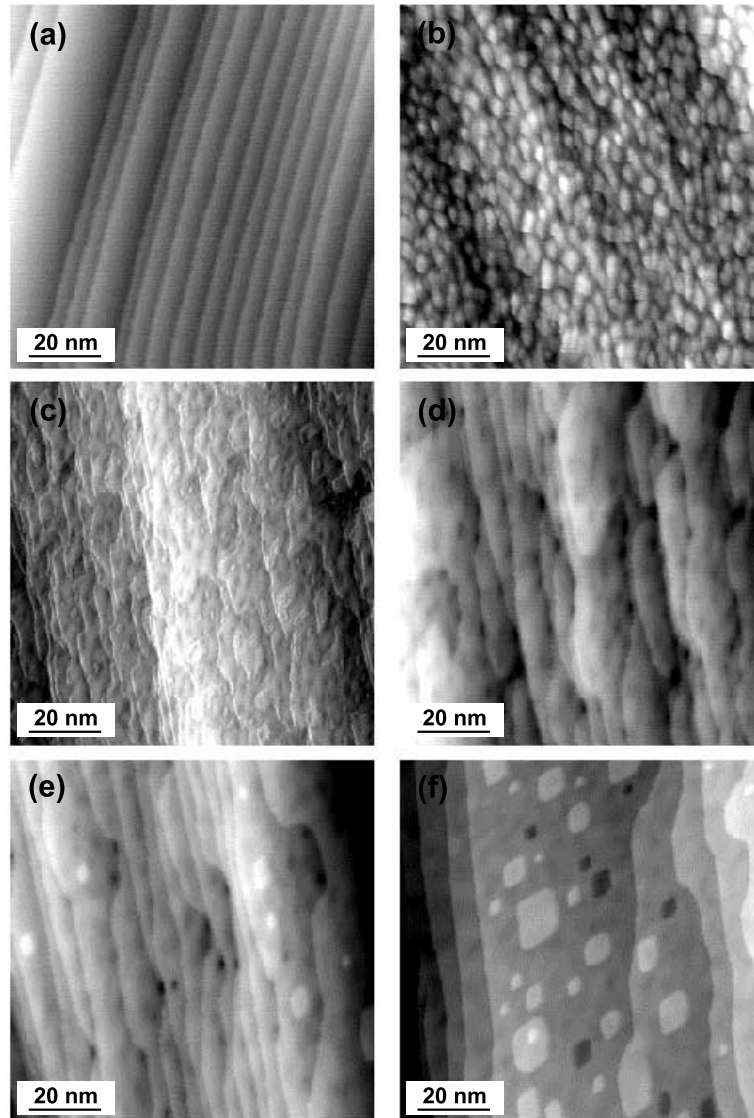


Fig. 1. STM topographs of: (a) vicinal Ag(001) with  $1.8^\circ$  miscut in the  $[\bar{1}10]$  direction, (b) deposition of 15 ML of Fe, covered with (c) 2, (d) 4, (e) 6, (f) 8 ML of Ag. All depositions were made at RT.

corresponding to Ag/Fe and Fe/Ag interface sites and have a relative population of  $\sim 4/15$ . The results of the fit are in good agreement with results obtained by Schurer et al. [8]. We used the relative line intensities of the site in the middle of the film to determine the angle  $\theta$ . For vicinal Ag(001) we find  $\theta = (85 \pm 3)^\circ$  and for flat Ag(001)  $(61 \pm 2)^\circ$ . Thus, for the vicinal substrate the magnetization is

almost in-plane whereas for the flat substrate the magnetization is  $29^\circ$  out-of-plane. To explain this difference, we consider the influence of the step density and the direction of the steps at the Fe/Ag interface. Kawakami et al. found that on vicinal Ag(001), the step-induced uniaxial anisotropy depends quadratically on the miscut angle  $\alpha$  [1]. This corresponds to  $H_s \sim \alpha^2$  with  $H_s$  the split field

Table 1

Rms values measured on a single terrace and length of steps for increasing Ag film thickness on vicinal Ag(001) covered with 15 ML of Fe

	Rms value on a terrace (nm)	Length of steps (nm)
Before deposition	0.02	>200
15 ML Fe + 2 ML Ag	0.07	10–25
15 ML Fe + 4 ML Ag	0.04	30–80
15 ML Fe + 6 ML Ag	0.03	>60
15 ML Fe + 8 ML Ag	0.02	>200
	0.10 <sup>a</sup>	
15 ML Fe + 10 ML Ag	0.02	>200
	0.12 <sup>a</sup>	

<sup>a</sup> Rms values on large terraces.

in the magnetization loop, taken along the direction of the steps, caused by the uniaxial anisotropy. We measured magnetization loops for the Fe/Ag multilayer grown on vicinal Ag(001) and found no difference between the parallel and perpendicular direction to the steps, meaning that there is no uniaxial anisotropy and  $H_s = 0$ . This is in agreement with Ref. [1], since only for a miscut angle higher than  $2^\circ$  an  $H_s$  differing from zero is observed experimentally.

For the Fe/Ag multilayer grown on flat Ag(001) a step density of 1 per 5 nm with steps in the [110] and  $[\bar{1}10]$  directions was reported for the Fe on Ag interface [2]. Note that for a vicinal Ag surface a terrace size of 5 nm corresponds to a miscut of  $2.3^\circ$  where uniaxial anisotropy due to steps was observed [1]. Although the step density on a flat Ag(001) substrate is much lower than for vicinal Ag(001), the surface of a 4 ML thick Ag spacer layer in an Fe/Ag multilayer has a higher step density when grown on flat Ag(001) compared to growth on vicinal Ag(001) with a miscut angle of  $1.8^\circ$ . It seems that for the growth of Ag on Fe, it is easier to recover steps in one direction than to reconstruct an atomically flat surface. In the latter case steps in two perpendicular directions are formed and the density of these steps decreases as the Ag layer thickness increases. Indeed, for a Ag spacer layer of 6 ML in an Fe/Ag multilayer grown on flat Ag(001), a step density of 1 per 9 nm was observed [2] (with steps in the [110] and  $[\bar{1}10]$  directions).

As mentioned above, the step structure is lost at the surface of the Fe film (Fig. 1b) after growth on

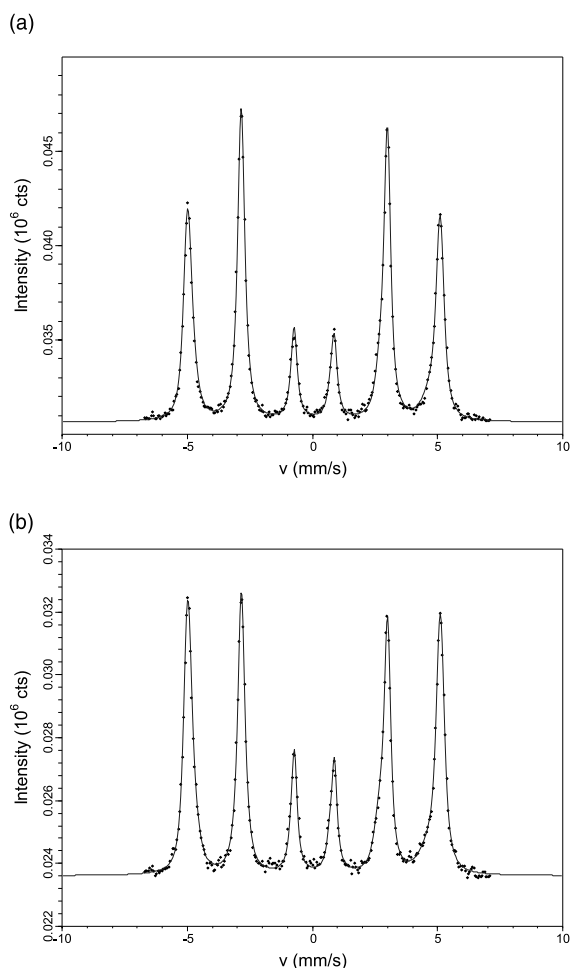


Fig. 2. Conversion electron Mössbauer spectra taken at RT from an  $[^{57}\text{Fe}(15 \text{ ML})/\text{Ag}(4 \text{ ML})]_{16}$  multilayer grown on (a) vicinal Ag(001) and (b) flat Ag(001).

Ag. However, for the multilayers that are studied here (with a Ag spacer layer of 4 ML) the step structure is preserved at the Fe/Ag interface as indicated by the CEMS results of Schurer et al. [8]. They have investigated the interface structure of Fe grown at RT on Ag(001) with terraces of about 100 nm. They observed only limited intermixing at the interface, but the terrace size was reduced to about 10 nm. Since the terrace size of the Ag surface, in both the vicinal and the flat multilayer, is already below 10 nm, we do not expect a further decrease during the Fe growth. This means that the step structure is preserved

at the interface during the growth of Fe. For a  $[\text{Fe}(15 \text{ ML})/\text{Ag}(4 \text{ ML})]_{16}$  multilayer grown on flat  $\text{Ag}(001)$ , the step-induced anisotropy becomes important due to the high density of steps. Since these steps are in two perpendicular directions, this can lead to frustration, resulting in an out-of-plane magnetization as observed by CEMS. For a similar multilayer grown on vicinal  $\text{Ag}(001)$ , the step density is too low to have an appreciable step-induced anisotropy. As a result there is no uniaxial behavior. Moreover, since there are only steps in one direction, there is no reason for out-of-plane magnetization.

#### 4. Conclusion

We presented a flexible method to obtain high quality vicinal  $\text{Ag}(001)$  surfaces using  $\text{MgO}(001)$  polished crystals with a miscut in the  $[\bar{1}10]$  direction. The interface structure of Fe/Ag multilayers grown on vicinal  $\text{Ag}(001)$  with a miscut of  $1.8^\circ$  was investigated. We found that the step structure is recovered during the growth of the Ag film. Using a 4 ML Ag spacer layer, the step density at the Fe/Ag interface is lower for growth on the vicinal  $\text{Ag}(001)$  surface compared to flat  $\text{Ag}(001)$ . In addition, no uniaxial anisotropy was observed in the vicinal multilayer since a miscut angle of  $1.8^\circ$  is too small. If the step structure during Ag

growth is also recovered for substrates with higher miscut angles, multilayers grown on such substrates are expected to have uniaxial magnetic behavior due to step-induced anisotropy.

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