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Crystal perfection by strain engineering: The case of Fe/V (001)

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ABSTRACT

We study the effect of bilayer thickness at fixed volume fraction on the structural quality of Fe/V (001) superlattices. We find that such artificial metallic superlattices can be manufactured with excellent crystal quality and layering up to at least 50 Å in repeat distance ($\Lambda = L_{Fe} + L_V$). For an intended fixed ratio of the constituents: $L_{Fe}/L_V = 1/7$, out-of-plane coherence lengths comparable to the thicknesses of the samples were obtained. We evaluate the strain in- and out-of-plane of both layers as a function of the bilayer thickness and comment on the growth using the framework of linear elasticity theory. We interpret the stability of the superlattice against crystal degradation due to the alternating compressive and tensile strain, yielding close to ideal lattice matching to the substrate.

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1. Introduction

Physical properties are often strongly altered when the spatial dimensions are reduced to the nanometer length scale. Thin films are used to explore the influence of strain and finite size on physical properties [1]. The crystal quality of the films can be essential for both obtaining well founded understanding as well as obtaining desired device performance, if minimum strain and defect density are of importance. Crystal perfection can be achieved through epitaxy, which refers to the formation of a single crystal thin film, on a single crystal substrate. The single crystal growth proceeds if both the in-plane lattice parameters as well as the orientation of the unit cell of the film and the substrate fulfil an epitaxial relationship [2]. In the case of heteroepitaxy, the inherent lattice mismatch of substrate and film will result in residual strain and ultimately the formation of defects in the film. For layer-by-layer growth, the strain can be accommodated up to a critical layer thickness.

Frank and van der Merwe introduced a one-dimensional model for describing mismatch between an epitaxial layer and a substrate and coined the term critical misfit in their seminal work [3] (9% in a one-dimensional model). This work was later extended to two dimensions by the same authors (see references in Ref. [4]). In the context of semiconductors, Mathews and Blakeslee [5] confirmed experimentally the critical thickness of a fully strained crystal, but reported discrepancies regarding the extent of accommodation by misfit dislocations. Their work included an expression for the critical thickness of superlattice growth whereby the critical thickness is a factor of four larger than single overlayers. Whereas epitaxial growth of semiconducting thin films based on Si and III/V semiconductor systems is by now the standard bearer of thin film crystal perfection (see for example reviews by Fitzgerald [4] and Segmüller [6] and references therein), defect free metallic systems are still more difficult to achieve. The question arises therefore whether metallic superlattices can be grown with a quality approaching to those of semiconductor heterostructures.

Here we have chosen Fe/V superlattices as a model system for exploring the crystalline quality in metallic superlattices. Fe/V superlattices have been used for addressing a range of basic research questions within statistical mechanics and condensed matter physics: Finite size effects on structural phase transitions [7,8], the use of hydrogen to switch the interlayer exchange coupling of Fe layers [9,10], site dependence of hydrogen diffusion [11] as well as superconducting spin valve effects [12]. In light of the extensive work done on Fe/V superlattices it is appropriate to report on a full investigation of the influence of thickness and strain on crystal quality.

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We build on the work of Isberg and Hjörvarsson and collaborators who investigated the influence of temperature on the crystal perfection of Fe/V grown by DC magnetron sputtering [13]. We will show that the thickness ratio of the layers (elements) can be used to obtain an ideal matching to the substrate. By using this approach, we will demonstrate the growth of high quality metallic superlattices, with crystal coherency comparable to the thickness of the samples. As a starting point for this study we were guided by the work of Liebig [14] for Cr/V superlattices and chose a ratio of the constituents to be 1/7, which was close to the ratio that yielded the best quality in that study.

2. Sample growth and design

The samples presented in this work were prepared using a quad d.c. magnetron sputtering system. The sample stage was rotated to obtain a homogeneous deposition across the surface. The temperature of the substrate was controlled within ≈1 K. The base pressure of the growth chamber was 2.7×10^{-7} Pa and was achieved using the combination of a turbo-molecular pump and a Ti sublimation pump. The targets were 99.99% pure and the Ar gas was of 99.9997% purity.

The dimensions of the substrates used were $10\times10\times1$ mm^3. Prior to deposition the substrates were exposed to 1273 K in vacuum, for 10 minutes, excluding heating up and cooling down time [15]. During the deposition of the superlattices, the temperature of the MgO (001), was kept constant at 618 K. The temperature reading was confirmed using a pyrometer pointed directly at the substrate holder, through a quartz window on the growth chamber. For the calibration of the pyrometer, materials of known emissivity were used and a series of temperatures were taken. The pressure of the Ar gas was 0.24 Pa (1.8 mTorr), as measured by a capacitance manometer and was held stable using an automatically adjustable gate valve combined with an Ar mass flow controller. A delay between the end and start of the growth of consecutive layers was set to 1 s to avoid co-deposition of the constituents, since the opening and closing of the shutters is approximately 0.3 s. The distance between magnetrons and substrate was 18.5 cm, the power of both magnetrons (Fe and V) was set to 50 W, and the rotation of the sample holder was set to 30 rounds per minute. The above pressure and power settings resulted in rates of 0.272 Å/s and 0.140 Å/s for Fe and V, respectively. The calibration of the deposition rates was performed by depositing thin films of the constituents separately on silicon substrates. The calibration samples were capped with Pd and were characterised by way of X-ray reflectivity. The reflectivity curves were fitted using Parratt's [16] formalism as implemented in the GenX [17] software.

Fe/V superlattices (SL) were grown epitaxially on a single crystal substrate of MgO (001), on which the [110] direction of MgO is parallel to the [100] direction of both the metals. The lattice parameter of MgO is 4.213 Å and along the diagonal [110] the interatomic distance is 2.980 Å. Iron has a lattice parameter [18] of 2.866 Å and the lattice parameter of vanadium [19] is 3.024 Å. Consequently, the two materials will exhibit tensile and compressive bi-axial strain in the plane, respectively.

Since the individual layer thicknesses in the largest superlattice considered in this work is below the theoretical critical thickness, if each layer were to be individually grown on MgO (39 Å for vanadium, and 22 Å for iron (see the Appendix)), we expect the optimum crystal quality to be found at the ratio of thicknesses that minimizes the elastic energy in the bi-layer. A schematic illustration of the sample design is shown in Fig. 1.

For convenience, we will refer to the intended number of monolayers as n_{Fe} and n_V, with bi-layer thickness $\Lambda = n_{Fe}(< c >_{Fe} /2) + n_V(< c >_V /2)$ where $< c >_{Fe(V)}$ is the average out of plane lattice parameter of Fe and V. The number of repeats for each superlattice was chosen so that the total V thickness would add up to ≈50 nm. In the case of 2/14 the number of repeats, $N = 23$. In the case of 3/21, $N = 15$ and in the case of 4/28, $N = 11$.

For transmission electron microscopy (TEM) observation, the samples were prepared in a cross-sectional geometry using mechanical polishing with subsequent grazing incidence bombardment with 4 kV Argon ion and final polishing at an ion energy of 1.8 kV. A FEI Tecnai F30-ST TEM was operated in scanning mode (STEM) and electrons scattered into angles larger than 50 mrad were acquired using a high angle annular dark field (HAADF) detector.

3. Results

3.1. TEM

Fig. 2 shows a HAADF image of the Fe/V 4/28 superlattice. The HAADF image predominantly shows Z-number contrast with heavier elements appearing brighter than the lighter ones. The image shows a smooth multilayer in the plane of the film. It can be seen in the image that there is an extra layer of vanadium between the superlattice and the palladium capping, as also indicated in the schematic of Fig. 1.

3.2. Reflectivity

X-ray reflectivity (XRR) and X-ray diffraction (XRD) were measured using a Panalytical X'pert MRD. For reflectivity, the operating

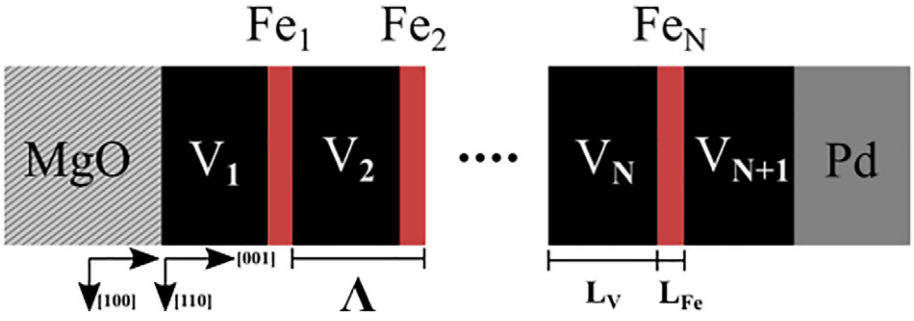

Fig. 1. Schematic representation of a superlattice. The subscript denotes the bilayer index and N is the total number of bilayers. $N + 1$ is the index for the final V layer.

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