Growth of ultrathin films of Fe on Au{001}

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The crystallographic aspects of the growth of ultrathin films of Fe on Au{001} are studied by means of quantitative low-energy electron diffraction (LEED) and Auger electron spectroscopy (AES). The first monolayer of Fe is found to be pseudomorphic to the Au{001} substrate with an interlayer spacing of 1.825 Å, and to be covered by a monolayer of Au at an interlayer distance of 1.85 Å, which acts as a surfactant. Thicker films of Fe [up to 45 layer equivalents (LE)] may be pseudomorphic and hence slightly strained (the lattice misfit is 0.6%) or equilibrium bcc with surface relaxation (the present analysis cannot distinguish between the two possibilities), but contain defects and disordered steps. Gold atoms are found to be segregated in the surface region of 45-LE Fe films, but mostly in a disordered arrangement. A comparison between the results obtained with two different computer programs for the calculation of LEED intensities finds that differences of 0.025 Å in the values of structural parameters may occur.

I. INTRODUCTION

Ultrathin films of $3d$ transition metals on nonmagnetic substrates are presently of great scientific and technological interest.[1] In this group, the films of Fe on Au{001} are unique, because they exhibit a number of remarkable magnetic and crystallographic properties.

At the outset, Fe/Au{001} is a good prospect for pseudomorphic epitaxy because there is a good lattice match (0.6%) of bcc Fe{001} to the primitive unit mesh of fcc Au{001}. Experiments have shown that single-crystal films of Fe can be grown on Au{001} up to thicknesses of about 20 layers.[2] These films have been found to be ferromagnetic; when grown at room temperature they have the magnetization in the plane of the films along a $\langle 100 \rangle$ axis, while when grown at 100 K they have the magnetization out of the plane for thicknesses of less than 2.8 atomic layers, but in the plane for larger thicknesses.[3] In addition, these films have been shown to contain Au atoms segregated in the surface region. This fact was demonstrated first by Bader and Moog[2] and Liu and Bader[3] only for rather thick (20-layer) Fe films, but was then assumed to be true for all thicknesses. The Au seems to act as a surfactant, which lowers the surface energy of the Fe and therefore favors growth in the layer-by-layer mode.

Bader and co-workers[2-4] grew their films at elevated substrate temperatures (150–250 °C) and claimed that the films grew layer by layer. These authors detected the occurrence of significant interdiffusion between Fe and Au only for growth at and above 300 °C, as judged from the time dependence of Auger signals.[2] It is interesting, however, that these authors also suggest that intermixing occurred when the temperature of films grown at 100 K was raised, based upon measurements of the direction of the easy magnetization axis.[1] Dürr, Germar, and co-workers[5,6] reported that deposition of Fe at room temperature or below was essential in order to obtain layer-by-layer growth and also in order to avoid interdiffusion. These and other[7] claims of layer-by-layer growth were based on the observation of breaks in the plot of Auger electron emission *versus* deposition time (so-called Auger breaks), and on the observation of sharp 1×1 low-energy-electron-diffraction (LEED) patterns for all thicknesses of the Fe films up to at least ten layers.[4-6] However, the layer-by-layer mode was *not* confirmed by Liew et al., who studied the growth of ultrathin films of Fe at room temperature by high-resolution LEED and found them to consist of "...a mixture of Fe-Au and Fe-Fe steps... indicating that the films grew in a three-dimensional (3D) mode."[8]

Himpsel exploited the unique properties of good lattice match and surfactant behavior of Fe/Au{001} in order to study variations of the electronic structure of Fe films on a layer-by-layer basis.[9] The Fe/Au system is particularly suited for the creation of narrow quantum wells, owing to the surfactant action; in addition, Fe and Au have very different band structures, thereby providing large band offsets and well depths. Extrapolating from the thick-film behavior studied by Bader and Moog,[2] Himpsel assumed that the surfactant Au layer would be present at the monolayer level as well. He thereby confirmed the theoretical predictions[10] of enhanced monolayer magnetism by measuring a ferromagnetic exchange splitting of 2.7 eV in a monolayer of Fe (the bulk splitting is 1.8–2.1 eV). This enhancement of the magnetic moment is larger than the one found previously by

Heinen et al. by spin-polarized photoemission,[7] but contradicts the no-enhancement results obtained by Kikuchi, Suzuki, and Katayama[11] with (SQUID) magnetometry on Fe-Au superlattices.

The work reported here was motivated by the desire to study the crystallographic aspects of a natural surfactant system, to get *quantitative* information about the atomic structure of ultrathin Fe films, and to test the growth mode by a technique other than Auger electron spectroscopy (AES). Wit these goals in mind, we present here the results of a study of the Fe/Au{001} system by AES and by quantitative LEED.

We recall that the primitive unit mesh of Au{001} has sides equal to 2.884 Å, only 0.6% larger than the unit-mesh edges of bcc Fe{001} (2.866 Å), a fact which favors pseudomorphic growth. A small lattice misfit, however, does not necessarily favor layer-by-layer growth—the misfit of bcc Fe to fcc Ag, which has unit-mesh sides of 2.889 Å, is also small (0.8%), but Fe does not grow layer-by-layer on Ag{001}.[12,13] Perhaps more important than the lattice misfit, in terms of the growth mode, are the surface free energies, which seem to indicate that Fe would *not* wet Au or Ag. [The calculated values of the surface free energies are 1.6 J/m^2 (Ref. 14) or 1.3 J/m^2 (Ref. 15) for Au; 1.3 J/m^2 (Ref. 14) or 1.1 J/m^2 (Ref. 15) for Ag; and 2.9 J/m^2 (Ref. 14) or 2.1 J/m^2 (Ref. 15) for Fe.] The main difference between the Fe/Au{001} and Fe/Ag{001} systems, at the outset, is the surfactant action that is reported to be present in the former, but does not occur in the latter, system.

II. EXPERIMENTS

The experiments were done in an ultrahigh-vacuum chamber with a base pressure of 6×10^{-11} Torr. The Au sample was a $12 \times 6 \times 1.5$ mm^3 platelet with the major surfaces perpendicular to the [001] direction within ±0.5°, polished with diamond paste and then electropolished. A {001} surface was cleaned *in situ* by cycles of sputtering with Ar ions (500 eV, 5×10^{-5} Torr) followed by anneals of 1 h at 300 °C. Higher annealing temperatures were avoided in order to prevent segregation of Sn and Ca on the surface, as detected by AES. The {001} surface was nevertheless satisfactorily ordered after the Ar-ion bombardments, as determined by the observation of bright and sharp 5×20 LEED patterns, and was completely free of Ca and Sn, as well as of the other common impurities such as S, O, and C to within the minimum detectable level of our cylindrical mirror analyzer, i.e., 1 at. %.

The iron source was a 99.999-at. %-pure wire wrapped around a tungsten spiral which was resistively heated typically to temperatures of 1300–1400 °C. The source was extensively outgassed after bakeout and subsequently outgassed prior to every deposition. While the source was heated, both the flange supporting the source and the surrounding chamber walls were air cooled to minimize outgassing and consequent contamination of the sample by oxygen and carbon. Deposition rates were kept roughly constant at about 0.1 Å/min, leading to pressures in the chamber of $5-9 \times 10^{-10}$ Torr. During depo-

sition the Au substrate was neither heated nor cooled—the substrate temperature was monitored by an infrared radiometer and was never at or above the minimum temperature measurable with this instrument (~ 150 °C). Use of identical deposition sources in previous experiments during which the sample temperature was monitored by a thermocouple indicated no significant increase in sample temperature above room temperature.

The film thicknesses were determined from AES by using the peak-to-peak height of the Fe (47 and 651 eV) and the Au (69 and 239 eV) signals. The standard procedure for this purpose, which is well documented in the literature,[16] had to be modified in the present case owing to the fact, now generally accepted[2,9] and directly proven below, that one layer of Au remains on the surface of the Fe film during deposition. The modification consists in the inclusion of new terms in the standard equation, which thus becomes

$$R = R_\infty \frac{(1 - e^{-d/\lambda_{Fe}})e^{-D/\lambda_{Fe}}}{e^{-(D+d)/\lambda_{Au}} + (1 - e^{-D/\lambda_{Au}})}, \tag{1}$$

where $R = I_{Fe}/I_{Au}$ is the ratio between the intensities (peak-to-peak heights) of the Fe (651 eV) and the Au (239 eV) AES signals; $R_\infty = I_{Fe}^\infty/I_{Au}^\infty = 5.4$ (Ref. 17); I^∞ denotes the intensity of the AES line from a very thick sample of Fe or Au; the λ's are the inelastic mean free paths of electrons with the appropriate energies, namely, 13.8 Å (Fe-651 eV) and 8.4 Å (Au-239 eV) (Ref. 18); d is the thickness of a film of Fe *assumed to be uniform* over the Au{001} surface; and D is the thickness of one layer of Au on top of the Fe film, also assumed to be uniform. Calculations based on Eq. (1) indicate that if D were assumed to vary by ±0.5 layer there would be errors in the calibration on the Fe thickness of approximately ±0.3 layer equivalent (LE) for low coverages and ±5 LE for high coverages. The accuracy in the thickness calibration is estimated to be about ±50% anyway because of uncertainties in the values of the inelastic mean free paths and the low intensity of the 239-eV AES line of Au.

The thicknesses of the Fe films as determined with Eq. (1) are quoted below in layer equivalents, representing the number of *uniformly distributed* layers of Fe that would produce the same value of the ratio R in Eq. (1) as that measured in the actual experiments. The conversion from Å to LE was done on the basis of the relation 1 LE=1.44 Å (for the thick Fe films) and 1 LE=1.80 Å (for less than two layers of Fe on Au{001}), but it should be clear that 1 LE is *not necessarily equal* to one layer, or, in the notation used by other authors, 1 ML.

LEED $I(V)$ curves were collected directly after each deposition had been made. They were then normalized to constant incident electron current and corrected for background prior to comparison with theoretical curves. The data-acquisition system consists of a rear-view LEED and a TV camera linked via a digitizer card to an IBM XT computer.[19] The software typically allows for the collection of all of the 24 degenerate low-index beams, i.e., the nondegenerate 10, 11, 20, 21, and 22 beams, within about one hour for good statistics. The LEED patterns and the corresponding $I(V)$ spectra were

all totally reproducible for similar film thicknesses on different occasions and for different deposition rates.

III. EXPERIMENTAL OBSERVATIONS

The 5×10 LEED pattern obtained from the clean Au{001} surface exhibited a high signal-to-background ratio only at low electron energies and, indeed, the fractional-order beams were only visible above the background for energies below about 200 eV. Above this energy, only the integral-order beams were discernible up to 450 eV. Deposition of a very small quantity of Fe (<0.2 LE) turned the clean Au 5×20 into a reasonably bright 1×1 pattern, the beam sizes of which varied slightly with energy. The $I(V)$ spectra at this very low concentration of Fe can reasonably be considered to represent the clean Au unreconstructed surface. Indeed, they are in good agreement with the spectra measured on a Au{001}-1×1 surface containing about 0.3 LE of Rh.[20]

Further deposition of Fe up to about 1.0 LE increased the background intensity of the LEED patterns slightly and progressively changed the $I(V)$ spectra as shown in Fig. 1 for the 10 and 11 beams. In order to perform a full-dynamical analysis, intensity data were collected for the 10, 11, 20, and 21 beams from the 1-LE pattern.

For Fe coverages from about 1.5 to 4 LE the LEED pattern visibly worsened—the background increased and the beams became more diffuse. The $I(V)$ spectra changed dramatically, as can be witnessed in Fig. 1. Intensity analysis was attempted for the data from the 2-LE film: various models, including pseudomorphic layers of pure Fe, pure Au, and random Fe-Au alloys of varying composition were tested, but none was found to produce an acceptable fit to experiment. This failure to find a satisfactory model, coupled with the increasing background and the worsening of the LEED pattern, suggests that probably layer-by-layer growth did not occur.

Above approximately 4 LE the 1×1 LEED pattern began gradually to improve. The $I(V)$ spectra were different from those taken at 2 LE, as can be seen in Fig. 1, and above about 20 LE they stabilized—no changes could be detected in the LEED patterns with increasing deposition of Fe except for a slight increase in background. The beam sizes of all LEED patterns above 3 LE were quite large and varied slightly with energy, indicating the presence of steps.

AES spectra indicated that even at high Fe coverages there remained an Au signal at 69 eV (the Au-239-eV signal was too weak to detect), which corresponds to about one layer. A thick (~45 layers) Fe film that was only briefly and lightly sputtered (500-eV Ar ions, 0.5 μA/cm² for 10 min) produced almost no Au AES signal and a much-enhanced Fe signal (a similar experiment was reported in Ref. 2). Thus, even on thick Fe films there was Au either on the surface or in the surface region. After the light sputtering the LEED pattern was still very bright, but the beams were slightly broader and showed slightly more noticeable changes in beam size with energy. Intensity data were collected from the 45-LE film both before and after the light sputtering and a LEED intensity analysis was carried out, as described below.

Deposition of Fe at elevated substrate temperatures was attempted in order to determine whether or not intermixing or alloying occurred as reported in previous studies.[2,5,6,9] Bader and Moog[2] reported intermixing about 300 °C, while Germar et al.[5] found agglomeration and/or intermixing at 150 °C, both cases judged by AES. Our attempts to grow Fe on Au at 300 °C produced slightly improved 1×1 LEED patterns but no change in the $I(V)$ spectra, compared to the room-temperature deposition, for films of thicknesses up to 1 LE. Further deposition up to about 2 LE did not change the $I(V)$ spectra either, but a very weak superstructure did develop in the LEED patterns which was rather 2×1-like (interpretation proved difficult). The 1×1 beams remained very sharp and intense. The origin of the superstructure is unknown, but it seems likely that intermixing or alloying was occurring in the surface region. This hypothesis
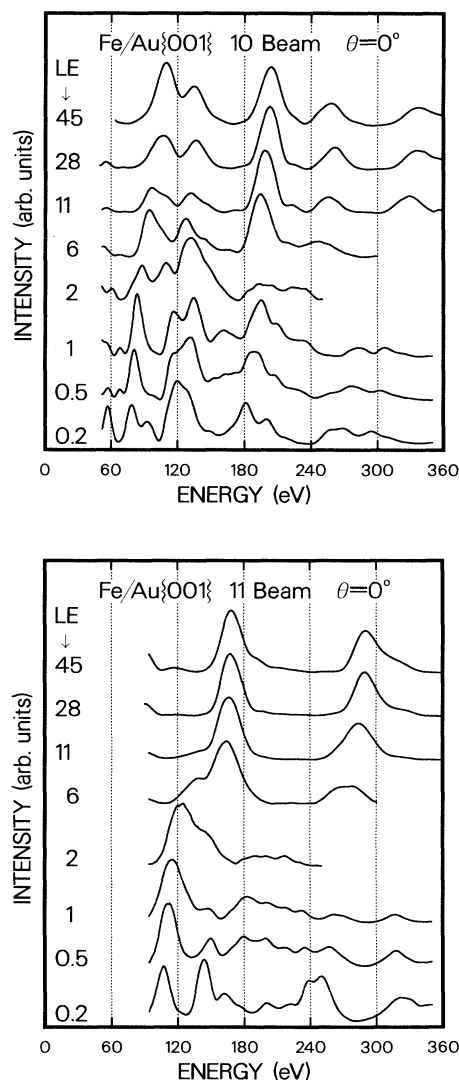

FIG. 1. Changes in the LEED 10 spectra (top) and 11 spectra (bottom) for Fe films on Au{001} with increasing film thickness. The thickness is expressed in layer equivalents (LE) as indicated on the left.

was partially confirmed by a subsequent 30 min of gentle argon-ion sputtering (400-eV ions, 5×10^{-5} Torr, ~ 0.5 $\mu A/cm^2$) which produced no change in the ratio of Auger peak intensities for Fe and Au.

In conclusion, the LEED observations suggest that the first Fe layer was very probably pseudomorphic, that growth of the Fe film beyond the first layer probably occurred by island formation, and that the thick Fe films had detectable amounts of Au in the surface region. LEED intensity analyses of both the monolayer and the thick Fe film are described below and provide more quantitative information.

IV. LEED INTENSITY ANALYSES

A. Monolayer film

The analysis of the 1-LE Fe film on Au{001} was carried out with two different computer programs, primarily in order to test the effect of different computer codes on the solution of surface-structural problem. One program was the CHANGE program of Jepsen,[21] the other was the LEEDFIT program of Moritz.[22] The input parameters were the same for both programs, in particular: relativistic phase shifts of both Fe and Au, inner potential $V_0 = -(8 + 5i)$ eV; Debye temperatures $\Theta_D = 165$ K for

Au and 440 K for Fe, corresponding to mean-square vibration amplitudes of 0.15 and 0.11 Å, respectively. The structural parameters were systematically varied in both programs for the two models tested, which were one pseudomorphic layer of Fe at various interlayer distances from the Au{001} substrate (model 1), and two pseudomorphic layers on top of the Au{001} substrate, namely, a monolayer of Fe covered by a monolayer of Au at various interlayer distances (model 2).

The analysis involved, for model 1, variations of the interlayer spacing d_{12} between the Fe monolayer and the Au substrate from 1.4 to 2.2 Å in steps of 0.1 Å; and, for model 2, variations of the interlayer spacing d_{12} between the Fe monolayer and the Au monolayer from 1.75 to 1.90 Å in steps of 0.025 Å, and of the interlayer spacing d_{23} between the Fe monolayer and the Au substrate from 1.75 to 1.95 Å, also in steps of 0.025 Å. The fit to experiment was evaluated visually and by three R factors: R_{VHT} (Ref. 23), r_{ZJ} (Ref. 24), and R_P (Ref. 25).

It appeared immediately obvious from visual inspection that model 1 does not fit the experimental data, whereas model 2 does. The refinement of model 2 was done by minimizing the three R factors independently. The contour plots are shown in Fig. 2 for the results obtained with CHANGE (left) and with LEEDFIT (right), and
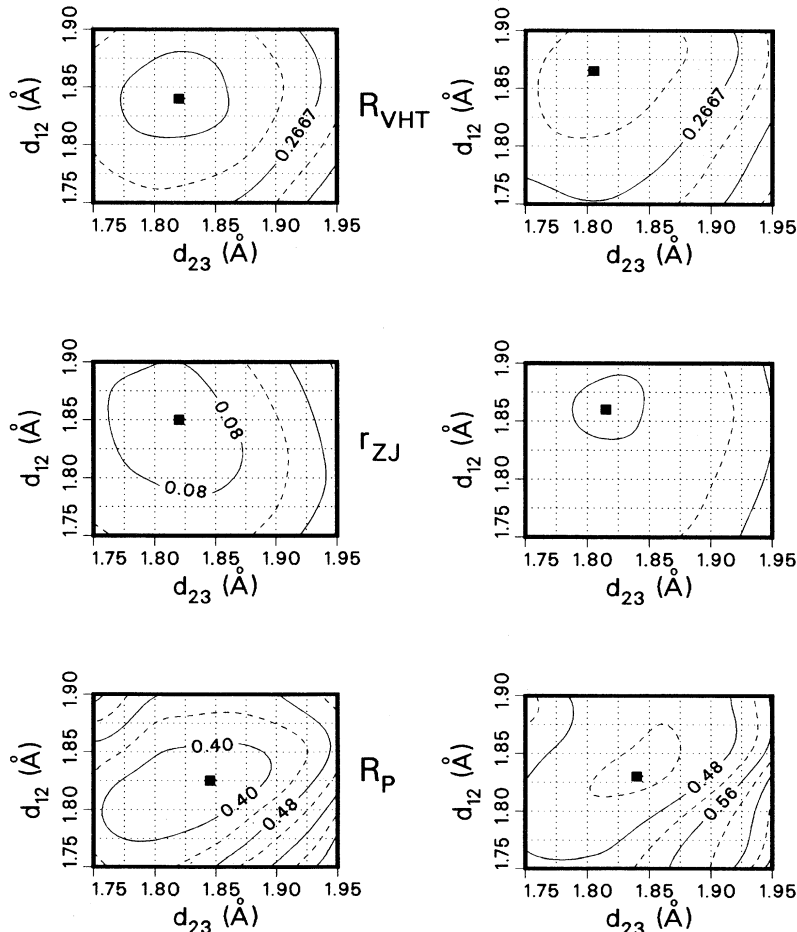

FIG. 2. Evaluation of the model involving a pseudomorphic monolayer of Au over a pseudomorphic monolayer of Fe on Au{001}. The figure depicts contour plots of the R factors R_{VHT} (Ref. 23), r_{ZJ} (Ref. 24), and R_P (Ref. 25) in the plane of $d_{12} = d_{Au-Fe}$ and $d_{23} = d_{Fe-Au}$ for calculations done with the CHANGE (left) and LEEDFIT (right) computer programs.

the corresponding best-fit parameters are listed in Table I.

It is interesting to note that fluctuations of the best-fit parameters are as high as 0.035 Å for different R factors (a fact already established in several other surface-structure determinations) and as high as 0.025 Å for different programs (a new finding, as far as we know). Since there is no reason for preferring one R factor over another or one program over the other, we take the average values of both structural parameters as the best solution of the problem, namely,

$$d_{12} = d_{\text{Au-Fe}} = 1.85 \pm 0.03 \text{ Å} ,$$

$$d_{23} = d_{\text{Fe-Au}} = 1.825 \pm 0.03 \text{ Å} ,$$

the error bars being consistent with previous LEED analyses. Figure 3 depicts the experimental $I(V)$ spectra compared to model 1 (a pure pseudomorphic monolayer of Fe on Au{001}) and to model 2 (a monolayer of Au over a monolayer of Fe on Au{001}) with the above parameters.

B. Thick film

The thickest Fe film grown in this work was about 45 LE. As mentioned above, the LEED pattern from such a thick film was still 1×1, but the diffracted beams were broad with somewhat energy-dependent width, and the background was high. These observations indicate that defects and high concentration of steps were present and that the ordered regions were relatively small in lateral dimensions.

We collected intensity data from a 45-LE film for the 10, 11, 20, and 21 beams, and carried out an intensity analysis for the purpose of determining the atomic arrangement in the ordered regions. In the corresponding calculations (which were done with the CHANGE program) the film was assumed to be semi-infinite, since LEED does not penetrate much more than about five or six atomic layers.

Two basic structure models were tested: a film of pure Fe and a film of Fe covered by a pseudomorphic monolayer of Au. The latter model was suggested by three experimental facts: (1) Since the monolayer film of Fe was found to be covered with a monolayer of Au (see Sec. IV A), it seemed reasonable to expect that the Au, acting in this case as a surfactant, would remain on top of the growing Fe film; (2) the Au/Fe AES intensity ratios were considerably larger than those calculated from the known
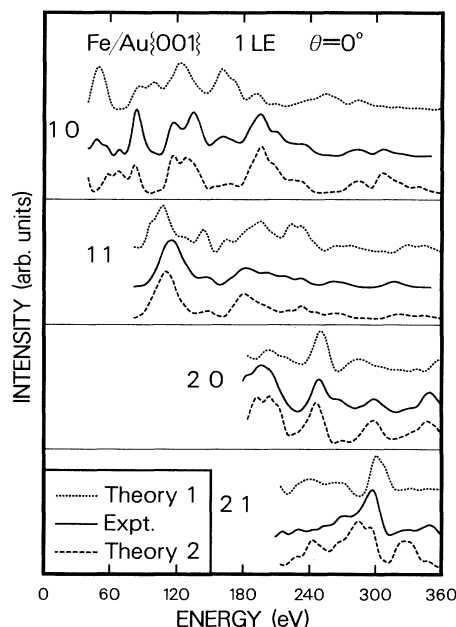

FIG. 3. Theory vs experiment for a 1-LE film of Fe on Au{001}. The dotted curves (Theory 1) are the results of calculations for a pure pseudomorphic monolayer of Fe on Au{001}. The dashed curves (Theory 2) are the results of calculations for a pseudomorphic Au layer over a Fe monolayer on Au{001}.

deposition rates and the standard calibration equation for growth of Fe *without* the Au surfactant; (3) as mentioned in Sec. II, even at high Fe coverages (judged from the deposition rates, which can be considered constant) there remained a Au AES signal at 69 eV which corresponds to about one layer of Au.

In testing the model of a pure Fe film we have to decide whether the bulk structure of the film was the equilibrium bcc structure of α-Fe or a strained body-centered-tetragonal structure due to pseudomorphism with the Au substrate (recall that the misfit between bcc Fe and fcc Au is only 0.6%, so that a 45-LE pseudomorphic film of Fe on Au is not impossible). As it turns out, $I(V)$ spectra calculated for either structure are practically indistinguishable from one another and hence the analysis cannot decide with any confidence between the two.

Figure 4 depicts the comparison between theoretical and experimental $I(V)$ spectra. In each panel, the top

TABLE I. Fe/Au{001}, monolayer film: best-fit parameters corresponding to minima of the R factors R_{VHT}, r_{ZJ}, and R_P as calculated with the CHANGE and the LEEDFIT programs. The quantities d_{12} and d_{23} (in Å) are the interlayer distances between the top Au monolayer and the Fe monolayer, and between the Fe monolayer and the Au{001} substrate, respectively.

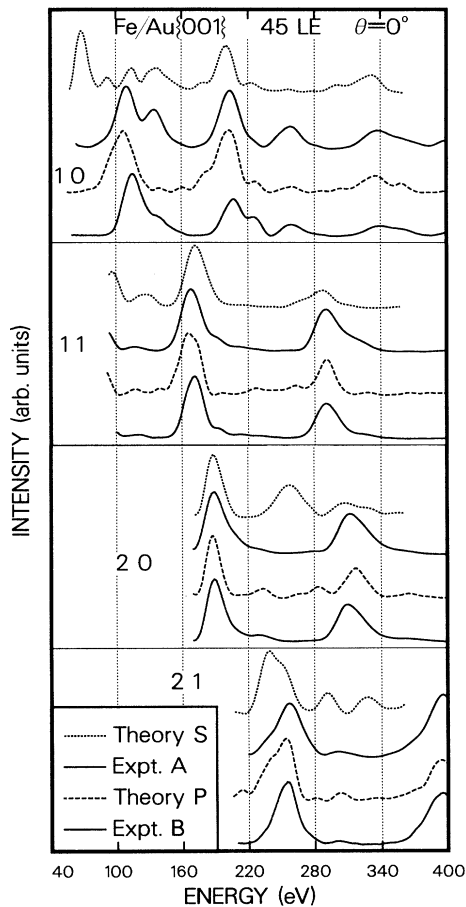|	CHANGE			LEEDFIT		
R factor	d_{12}	d_{23}		R factor	d_{12}	d_{23}
$R_{VHT} = 0.20$	1.840	1.820		$R_{VHT} = 0.23$	1.865	1.805
$r_{ZJ} = 0.07$	1.850	1.820		$r_{ZJ} = 0.08$	1.860	1.815
$R_P = 0.37$	1.825	1.845		$R_P = 0.43$	1.830	1.840

FIG. 4. Experimental $I(V)$ spectra from a 45-LE film of Fe on Au{001} (upper solid curves, Expt. A) compared to calculations for a semi-infinite pseudomorphic film of Fe covered by a surfactant monolayer on Au (dotted curves, Theory S), calculations for a pure semi-infinite pseudomorphic film of Fe (dashed curves, Theory P), and experimental curves (lower solid curves, Expt. B) obtained after light sputtering of the film that produced the Expt. A curves.

curve (Theory S, dotted line) is the best-fit calculated curve for a pseudomorphic Fe film covered with a surfactant monolayer of Au ($d_{\text{Au-Fe}} = 1.80$ Å); the upper solid curve (Expt. A) is the experimental curve from the 45-LE film as grown; the third curve (Theory P, dashed line) is a curve calculated for a pseudomorphic film of pure Fe ($d_{\text{bulk}} = 1.42$ Å); and the bottom solid curve (Expt. B) is the experimental curve from the thick Fe film after light sputtering (500-eV Ar ions, 5 min).

Neither calculation represents a very good fit to Expt. A (film as grown), but both the visual and the R-factor evaluations[26] favor Theory P (pure Fe film with no Au surfactant layer). The spectra from the lightly sputtered film (Expt. B) are more similar to those calculated for the pure-Fe model than to those calculated for the Au-surfactant model. Nevertheless, the fact that *no* good fit to Expt. A was found with the Au-surfactant model, no matter which interlayer distance was chosen, seems to indicate that whatever Au was in the surface region was

predominantly *not* ordered or at least not all *on* the surface of the film. Attempts were made to find a better fit to Expt. A with various mixtures of Au and Fe in the first atomic layer, but they all failed. We are therefore led to conclude that while in the present experiments the thick Fe films *did* contain Au atoms in the surface region (we estimated 1.7 ± 0.4 Å of Au), nevertheless only a minority of these atoms contributed to the LEED signal, i.e., most of them were probably disordered.

V. DISCUSSION

Some of the observations reported by other authors have been verified by the present LEED study, some have not. We have proved that one monolayer of Fe can be grown pseudomorphically on Au{001} with an interlayer distance of 1.825 Å, and that this layer is covered with a monolayer of Au at an interlayer spacing of 1.85 Å. The results obtained in the present work are proof of the existence of a surfactant layer of Au on the first monolayer of Fe. It is noteworthy that only Bader and Moog[2] and Himpsel[9] refer to the presence of the surfactant Au layer.

However, our observations of worsening LEED patterns upon further deposition of Fe are in direct contradiction with reports of LEED patterns remaining good.[2,4-6] (Himpsel[9] and Heinen et al.[7] do not report any LEED observations for films thicker than one layer.) The $I(V)$ spectra from our ~2-LE film cannot possibly correspond to two flat pseudomorphic layers, according to our calculations. These negative results speak against continued layer-by-layer growth, and suggest rather that after the first monolayer the growth may continue by way of islands with different heights and many disordered steps. It may be interesting to note, in this connection, that a recent scanning tunneling microscopy (STM) study of homoepitaxy of Fe on Fe{001} by Stroscio et al.[27] provides evidence for island growth, despite the observation of reflection high-energy electron diffraction (RHEED) oscillations. In the present work, we found that the LEED pattern improved in quality after ~4 LE, presumably when whatever islands had been nucleated coalesced into a continuous, though not necessarily flat, film. The LEED pattern persisted then up to ~45 LE, the largest thickness attained in the present work, albeit with increasing background and diffracted beam sizes.

Thick Fe films were shown to consist of either unstrained epitaxial bcc Fe or slightly strained pseudomorphic Fe—the present analysis could not tell the difference. Approximately one layer of Au was indeed present in the surface region of the thicker films, but we found that only a small part of it was ordered as a surfactant monolayer—most of it was either disordered or alloyed in the top few layers of the film.

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