Formation of the BiAg$_2$ surface alloy on lattice-mismatched interfaces

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We report on the growth of a monolayer-thick BiAg$_2$ surface alloy on thin Ag films grown on Pt(111) and Cu(111). Using low energy electron diffraction (LEED), angle resolved photoemission spectroscopy (ARPES), and scanning tunneling microscopy (STM) we show that the surface structure of the $\sqrt{3} \times \sqrt{3}$R30$^\circ$ ML Bi$_x$-ML Ag/Pt(111) system ($x \geq 2$) is strongly affected by the annealing temperature required to form the alloy. As judged from the characteristic $\sqrt{3} \times \sqrt{3}$R30$^\circ$ LEED pattern, the BiAg$_2$ alloy is partially formed at room temperature. A gentle, gradual increase in the annealing temperatures successively results in the formation of a pure BiAg$_2$ phase, a combination of that phase with a $(2 \times 2)$ superstructure, and finally the pure $(2 \times 2)$ phase, which persists at higher annealing temperatures. These results complement recent work reporting the $(2 \times 2)$ as a predominant phase to the strain at the interface between the Ag film and the Pt substrate, which is almost negligible at the Ag/Au(111) interface. Surprisingly, the BiAg$_2$ alloy did not form on Pt(111) for a 5 ML Ag film although the LEED and ARPES data showed the $(1 \times 1)$ spots and the Shockley surface state at the clean Ag(111) film surface, respectively.

In this work we investigate in details the possible formation of BiAg$_2$ alloy on such lattice-mismatched interface systems. Two different substrates are considered, namely Pt(111) and Cu(111), where the Ag-film/substrate lattice mismatch amounts to $\sim 4\%$ and $\sim 12\%$, respectively. In both cases we observe the formation of the surface alloy as demonstrated by three complementary experimental techniques: low energy electron diffraction (LEED), angle resolved photoemission spectroscopy (ARPES), and scanning tunneling microscopy (STM). The characteristic $(\sqrt{3} \times \sqrt{3})R30^\circ$ superstructure and the Rashba surface state are obtained with different Ag film thickness on both substrates. In fact, the alloy formation takes place at room temperature (300 K), and is constrained to annealing temperatures below (350 ± 20) K. For the Bi-Ag/Pt(111) systems, higher annealing temperatures result in the formation of the previously reported $(2 \times 2)$ superstructure for Ag films with a thickness as high as 100 ML, while the annealed Bi-Ag/Cu(111) shows the formation of BiCu$_2$ alloy at the expenses of the BiAg$_2$ phase. These findings provide a way of growing the BiAg$_2$ alloy and similar Rashba systems on substrates where the mismatch at the film-substrate interface might hinder their formation at reduced films thickness.

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I. INTRODUCTION

Heavy metal alloys, such as BiAg$_2$, feature spin-orbit split (SOS) surface states, an essential prerequisite for the manipulation of the spin-polarized current in spintronic devices [1]. Widely explored is the control of such SOS surface states through, for example, doping with alkali metals [2], exposure to noble gases [3], or through the formation of ternary alloys where a second heavy element is added [4].

The BiAg$_2$ alloy. In Bi-Ag/Cu(111) the minimum Ag film thickness that allows a well defined $(\sqrt{3} \times \sqrt{3})R30^\circ$-BiAg$_2$ structure is 2 ML [9]. The attempt to grow BiAg$_2$ on thin Ag films (2–5 ML) on Pt(111) has been reported to form a novel $(2 \times 2)$ superstructure for a significant range of Bi coverages (0.2 ML to 0.6 ML) [10]. The authors attributed the absence of the $(\sqrt{3} \times \sqrt{3})R30^\circ$ phase to the strain at the interface between the Ag film and the Pt substrate, which is almost negligible at the Ag/Au(111) interface. Surprisingly, the BiAg$_2$ alloy did not form on Pt(111) for a 5 ML Ag film although the LEED and ARPES data showed the $(1 \times 1)$ spots and the Shockley surface state at the clean Ag(111) film surface, respectively.

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II. EXPERIMENTAL METHODS

Two ARPES setups have been used: for the Bi/Ag/Pt(111) system, ARPES measurements were performed using a SCIEN TA R4000 electron analyzer (Würzburg) equipped with a monochromatized He discharge lamp (MB Scientific) operating at excitation energies of 21.2 eV (He I) and 40.8 eV (He II). The energy and angular resolution at 21.2 eV were set to ∼5 meV and 0.3°, respectively. All measurements were undertaken at 40 K, unless specified. ARPES data on the Bi/Ag/Cu(111) system have been carried out using a SPEC S Phoibos 150 electron analyzer (San Sebastián) operating with monochromatized He Iα photons, and with energy and angular resolution of ∼15 meV and 0.3°, respectively. These data were collected at 150 K. STM experiments on the Bi/Ag/Cu(111) system were taken at an Omicron VT Setup operating at 300 K. The samples are transferred between ARPES and STM setups without breaking the UHV conditions. Both Pt(111) and Cu(111) substrates were cleaned by successive sputtering-annealing cycles until sharp ARPES bands and (1 × 1) LEED spots are obtained. Ag and Bi are separately evaporated from Knudsen cells with rates of 1 and 0.06 ML/min, respectively. The Ag evaporator was calibrated by depositing 1 ML on a Cu(111) substrate, and annealed to 400 K, until the corresponding gapped surface state is observed [11,12].

Likewise, a 1/3 ML Bi was calibrated by Bi deposition onto Cu(111) that resulted in a sharp Rashba surface state in ARPES and a pure (√3 × √3)R30° superstructure in LEED [13,14]. The Ag thin films (x-ML) were first grown onto Pt(111) and Cu(111) held at 300 K and 150 K and subsequently annealed to 600 K and 400 K, respectively. The 1/3 ML of Bi was then grown on top of the x-ML Ag/Pt(111) and x-ML Ag/Cu(111) systems at 300 K and 150 K, respectively, and later annealed to different temperatures. At each annealing temperature both LEED or STM and ARPES data were acquired. Owing to the surfactant effect of Bi, the data were found to be independent on the deposition order, i.e., equivalent results are obtained when Bi is grown first on both Pt(111) and Cu(111) systems followed by Ag film deposition.

III. GROWTH OF 1/3 ML Bi ON x-ML Ag/Pt(111)

In Fig. 1 we present LEED patterns for a 1/3 ML Bi film on ∼3 ML Ag/Pt(111). The green circles mark the (1 × 1) spots of Ag/Pt(111) system. After Bi deposition, and without any heat treatment, a clear BiAg2-(√3 × √3)R30° is formed [the unit cell is marked by the yellow lines in (a)]. Extra spots, which have been previously assigned to the Bi-(p × √3) structure [15], are also observed. Its unit cell is marked by the white lines. The coexistence of the two phases is frequent for BiCu2- and BiAg2- alloys on Cu(111) and Ag(111), respectively, when the Bi coverage exceeds 1/3 ML [15,16]. In the present case, it is rather an indication of the partial alloy formation, as the deposition of the equivalent amount on Cu(111), taking the different lattice parameters into account, results in pure BiCu2- (√3 × √3)R30° spots. Moreover, the extra spots completely disappear after annealing to ∼320 K, leaving a sharp BiAg2- (√3 × √3)R30° superstructure, Fig. 1(b). After annealing to ∼340 K, a partial dealloying takes place resulting in an additional (2 × 2) superstructure (red lines mark its unit cell).

Fig. 1(c). At higher annealing temperatures (>350 K) a pure (2 × 2) superstructure is obtained, Fig. 1(d). It is important to note that the SOS alloy phase is stable within a very narrow temperature region (<350 K), explaining the absence of this phase in Ref. [10], where the annealing temperature was probably higher. Nonetheless, the strain is likely affecting
the stability of the alloy, considerably reducing the annealing temperature window. We also note that the successive transition from the \((\sqrt{3} \times \sqrt{3})R30^\circ\) Bi to the pure \((\sqrt{3} \times \sqrt{3})R30^\circ\), mixed \((\sqrt{3} \times \sqrt{3})R30^\circ\) and \((2 \times 2)\), and finally the pure \((2 \times 2)\) superstructure is independent on the Ag-film thickness (see Supplemental Material, Fig. S1 [17]) [18–20]. However, the thicker the Ag film becomes, i.e., the lower the overall surface strain, the higher the dealloying temperature becomes.

The \((\sqrt{3} \times \sqrt{3})R30^\circ\) \((2 \times 2)\) structural transition could be traced by monitoring the Bi 5d core level at different annealing temperatures. Figure 1(f) presents these data taken at 300 K with He \(L_\alpha\) \((h \nu = 40.8 \text{ eV})\) for \(1/2\) ML of Bi on 3 ML Ag/Pt(111). The experimental and the overall fitting spectra are indicated with purple circles and black solid lines, respectively. The highly asymmetric background is accounted for by a Gaussian edge, made of a pure Gaussian peak on top of a linear background (see Supplemental Material, Fig. S2 [17]). For the as-deposited sample (300 K), we observed a single spin-orbit split peak (red) at binding energies of \(\sim 23.74\) eV (Bi-5d\(_{5/2}\)) and \(\sim 26.79\) eV (Bi-5d\(_{3/2}\)), i.e., the magnitude of the spin-orbit split amounts to \(\sim 3.05\) eV. Irrespective of the \((p \times \sqrt{3})\)-Bi traces and the Ag film thickness, their energetic positions are always found at \((23.70 \pm 0.05)\) eV and \((26.75 \pm 0.05)\) eV, being consistent with the reported values for the well-defined BiAg\(_2\) alloy on Ag(111) [21].

At 340 K, the intensity of BiAg\(_2\) core levels decreases and a new spin-orbit split peak (blue) appears at \(\sim 23.95\) eV (Bi-5d\(_{5/2}\)), i.e., \(\sim 210\) meV shifted towards higher binding energy with respect to the energetic position obtained for the BiAg\(_2\) alloy, being now consistent with the reported value for the elemental bismuth [22,23]. This indicates a partial dealloying and the formation of the BiAg\(_2\) alloy (red) is completely suppressed, and the overall spectrum can be fitted with a single spin-orbit split peak (blue), with its energetic positions at \(\sim 23.95\) eV (Bi-5d\(_{5/2}\)) and \(\sim 26.97\) eV (Bi-5d\(_{3/2}\)), i.e., corresponding to the \((2 \times 2)\) superstructure. The energetic position of this peak is, within \(\pm 50\) meV, independent of the Ag film thickness. For all cases studied, beside the characteristic shifts, the magnitude of the spin-orbit splitting is \(\sim 30\) meV larger for the alloyed phase. The correspondence between LEED and the core-level photoemission data is obvious, and demonstrates the possible formation of the BiAg\(_2\) at such strained interface provided that the annealing temperature is reduced. We note that the single BiAg\(_2\) phase is hardly obtained given the limited temperature range available; nonetheless, traces of the coexisting phases do not contribute significantly to ARPES spectra [16], as we show next. We also observed that, if the deposition order is reversed, i.e., when Bi is deposited first then Ag on top, the intensity of the Bi 5d levels are only weakly affected even for the as-deposited sample (see Supplemental Material, Fig. S3 [17]). This confirms the surfactant nature of Bi, where most of the Bi atoms float to the surface at room temperature [10].

In the following we present ARPES data exclusively for BiAg\(_2\) alloy [i.e., after annealing to 320 K (a),(b), 340 K (c), and 400 K (d)] for different Ag film thickness. Figure 2 shows the Rashba SOS surface state obtained for an Ag film thickness as low as 2 ML. Thus a pair of Rashba split bands (red and blue dashed lines) termed \(E_{1,2}\) are observed. Their inner and outer branches are, respectively, denoted by \(E^-'\) and \(E^+'\), as indicated in Fig. 2(d) for the 100 ML Ag film [9]. The major difference is that the quality of the surface states is improved for higher Ag film thickness. Although the Pt(111) substrate is known to significantly alter the dispersion of the surface states of noble metal overlayers for film thickness as high as 4 ML [24,25] due to hybridization with its downward dispersing bulk \(sp\) band, the behavior of the Rashba state is practically unaffected indicating its strong localization at the topmost surface layer. The blurred intensity of the surface states for reduced Ag films is likely due to the emission from the underlying bulk Pt band, in addition to the possible roughness of Bi/Ag film [see Fig. 3 on the related case of BiAg\(_2\)/Ag/Cu(111)].

IV. GROWTH OF 1/2 ML Bi ON x-ML Ag/Cu(111)

The Ag/Pt system involves a significant interfacial strain so it is interesting to compare it to a system that \emph{a priori} does not have such inherent stress, as is the Ag/Cu(111) interface, which presents a layer-by-layer growth with moiré formation. In Fig. 3 we show STM images taken for \(1/2\) ML Bi deposited at 150 K on (a) 1 ML and (b) 2 ML Ag films grown on Cu(111) after gentle annealing to 350 K in both cases. Although the STM images for the 1 ML and 2 ML Ag/Cu...
overlayer systems prior to Bi deposition exhibit a perfect layer-by-layer growth (see Supplemental Material, Fig. S4 [17]), the Bi deposition roughens the surface, giving rise to BiAg$_2$/Ag islands (bright areas) and Ag-free areas (dark areas). The BiAg$_2$-covered areas amount to $\sim$50% and $\sim$85% for the 1 ML Ag and 2 ML Ag, respectively, whereas the lateral dimension of the islands is clearly larger for 2 ML Ag. In both cases the height of the majority of the islands is $\sim$4.6 Å, which correspond to bilayer BiAg$_2$/Ag islands. Thicker islands are also observed, which make up for the total 2 ML Ag coverage. On the other hand, excess Bi may begin to alloy with Cu, at apparently structureless Cu areas (see discussion below). A zoom-in image of Fig. 3(a) is shown in Fig. 3(c). The image contains two distinct areas, the Ag-free structureless area and the bilayer BiAg$_2$/Ag region exhibiting a moiré-like pattern. Such a moiré superstructure is characteristic for Bi-free, 2 ML Ag/Cu(111), although the same moiré pattern may also be expected for the BiAg$_2$/Ag/Cu(111) bilayer, as alloying with Bi does not change the Ag-Ag nearest neighbor distance (see Supplemental Material, Fig. S5 [17]). Both the moiré periodicity and the atomic lattice parameter can be deduced from the atomic scale STM image shown in (d). The moiré periodicity is found to be the same as for the 2 ML Ag/Cu (i.e., $\sim$24 Å), whereas the atomic scale resolution reveals the characteristic BiAg$_2$-$(\sqrt{3} \times \sqrt{3})R30^\circ$ with a lattice parameter of 5.1 Å [26]. Furthermore, the moiré pattern is $\sim$30°-rotated with respect to the atomic lattice, as shown in the Fourier transform at the image’s inset.

In Fig. 4 the ARPES photoemission intensity plots for the $\frac{1}{3}$ ML Bi on 1 ML and 2 ML Ag annealed to 350 K are presented. A distinct BiAg$_2$ Rashba surface state is obtained for both Ag coverages. Only a relative improvement in the quality of the surface states is visible, i.e., sharper bands are obtained for the Bi-2 ML Ag sample, as expected for the larger lateral extent of the BiAg$_2$ bilayers in Fig. 3(b). In fact, the quality of the surface states is comparable to the Bi/2-3 ML Ag/Pt(111) presented in Figs. 2(a) and 2(b), indicating that the reduced quality for both systems is related to surface roughness and overall island size. Such a growth behavior is likely triggered by both the well-known high mobility of Ag atoms (kinetics) and the interface mismatch (energetics). The latter is negligible at Ag/Au(111) interface and, therefore, $\frac{1}{3}$ ML Bi on 1 ML Ag/Au(111) exhibits no BiAg$_2$ phase, indicating that a minimum of one Ag bilayer is required for the alloy formation. Another important similarity between the two systems is that the exclusive BiAg$_2$ alloy phase is only obtained at low annealing temperatures (350 K); however, the behavior at higher annealing is very different between both substrates, as we show next.

In Fig. 5 we show ARPES data for a $\frac{1}{3}$ ML Bi grown on 1 ML Ag/Cu(111) at 150 K, and postannealed to (a) 430 K and to (b),(c) 600 K. The BiCu$_2$ surface state can be identified in both cases [3], while the BiAg$_2$ shifts toward higher binding energy. Its Dirac point is now found at $(-520 \pm 10)$ meV. It is only at 430 K, where the BiAg$_2$ surface state intensity is comparable to the BiCu$_2$, which can be mistaken with the $E_{\text{2-3}}$ state coming from the unoccupied region. This indicates that the dark areas in Fig. 3(a) are nonalloyed Bi/Cu which transform into BiCu$_2$ alloy upon annealing. The formation of BiCu$_2$ alloy is further justified by measuring its characteristic lattice parameter, $\sim$4.4 Å, in atomically resolved STM images (see Supplemental Material, Fig. S6 [17]). Above this temperature, ARPES data consists mainly of BiCu$_2$ and very weak BiAg$_2$ bands. This mixture of BiCu$_2$ and weak
FORMATION OF THE BiAg₂ SURFACE ALLOY ON . . . PHYSICAL REVIEW B 94, 155447 (2016)

FIG. 5. (a),(b) Photoemission intensity plot for 1/3 ML Bi on 1 ML Ag/Cu(111) after annealing to (a) 430 K and (b) 600 K. BiCu₂ and BiAg₂ surface states coexist with the weight of BiAg₂ decreasing upon annealing. In both cases the BiAg₂ surface state shifts towards higher binding energy compared to the 350 K annealed sample. (c) The same photoemission intensity plot shown in (b) overlaid with the characteristic surface state bands for pure BiCu₂ (red lines) and BiAg₂ (blue lines). The dispersion parameters are the ones reported for BiCu₂/Cu(111) and BiAg₂/Ag(111) except that the latter is shifted towards higher binding energy. (d) Constant energy surfaces (CES) taken at the indicated energies (white lines) in (c).

BiAg₂ surface states is stable up to annealing temperatures of 800 K. The strongly reduced intensity of the BiAg₂ surface states clearly indicates a dominant clustering and dealloying at such temperatures. In Fig. 5(c) we overlay the photoemission intensity in (b) with the parabolic dispersion reported for BiCu₂ (red lines) and BiAg₂ (blue lines) surface states. The red lines coincide with BiCu₂ bands, whereas the blue lines are shifted by 110 meV to higher binding energy. The coexistence of the two surface states can also be seen in the constant energy surfaces (CES) taken at the white dashed lines in (c) and presented in (d). The Fermi surface (FS) cut shows the three contours for BiCu₂, the −350 meV cut shows the main BiCu₂ contour with some inner intensity from the BiAg₂, the −510 meV cut shows the main BiCu₂ bands and the Dirac point of the BiAg₂, and the −920 meV cut shows the BiCu₂ (outer) and BiAg₂ (inner) rings. The reduced intensity and the shift of the BiAg₂ surface state to higher binding energy are likely associated with the presence of two-dimensional BiAg₂ crystallites of very small dimensions (see Supplemental Material, Fig. S6 [17]) [27].

V. CONCLUSION

In conclusion, we have shown that it is possible to form the BiAg₂ alloy for ultrathin Ag films, even on very strained interfaces. Indeed, the structure of 1/3 ML Bi on thin Ag films grown on Pt(111) and Cu(111) is strongly affected by the annealing temperature. For both substrates, the formation of the BiAg₂ alloy is only possible for annealing temperatures lower than (350 ± 20) K. The electronic structure of the BiAg₂ surface alloy, in both systems, was found to be qualitatively similar to BiAg₂/Ag/Au(111). At higher annealing temperatures, the previously reported (2 × 2) superstructure is obtained on Bi/Ag/Pt(111) independent on the Ag film thickness. However, higher annealing temperatures on Bi/Ag/Cu(111) systems result in the formation of BiCu₂ in combination with BiAg₂ traces.

The finding that the formation of BiAg₂ surface alloy on strained interfaces is limited to a narrow temperature range should have some implications on the search for BiAg₂ and similar Rashba alloys on technologically relevant substrates. For example, Ag grows in a layer-by-layer mode on Ni(111), although the lattice mismatch is larger than that for both Cu(111) and Pt(111). Ni(111) supports spin-polarized electronic states and therefore the interplay between Rashba and exchange interactions could be investigated [28,29]. Although the BiAg₂ alloy has been previously grown on Si(111) [6–8], the Ag film thickness could be lowered and a Rashba surface state interface with a semiconductor substrate could be obtained.

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155447-5
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[17] See Supplemental Material at http://link.aps.org/supplemental/10.1103/PhysRevB.94.155447 for additional information on the Bi/Ag/Pt system at reduced Ag thickness, the fitting of core-level data, the surfactant property of Bismuth, the film roughness induced by Bi deposition, the BiAg₂ moiré pattern, and the simultaneous formation of BiCu₂ surface alloy and BiAg₂ nanocrystallite.
[18] One exception is the ∼2 ML Ag case, where, in addition to the \((\sqrt{3} \times \sqrt{3})R30^\circ\) and the \((2 \times 2)\) spots in Fig. 1(c), traces of the \((4 \times 4)\) superstructure spots were detected. In fact these extra spots could be due Bi/Pt(111) areas present at such a reduced thickness.
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