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Photoemission study of the epitaxial Ce/Pd(100) interface

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Abstract. The electronic state of cerium has been investigated by core-level and valence band photoemission spectroscopy in ultra-thin films of a Ce-Pd monocrystalline compound obtained by epitaxial growth. Careful characterization of the surface leads us to the conclusion that the Ce configuration admixture is strongly reduced in the surface layer. By evaporating one extra monolayer of Pd, we were able to obtain core-level photoemission spectra without any surface contribution and to evidence an exceptional configuration mixing for the bulk ground state. The $4f$ spectral function obtained from photoemission measurements at the $N_{4,5}$ threshold also exhibits a very singular behavior and shed some new light on the description of highly-hybridized $4f$ states in Ce compounds.

PACS. 71.28.+d Narrow-band systems; intermediate-valence solids – 79.60.-i Photoemission and photoelectron spectra

1 Introduction

Cerium based compounds often exhibit singular physical properties associated with an anomalous $4f$ electronic configuration. This is due to an interplay between the large $4f$ intra-atomic Coulomb interaction and the hybridization between $4f$ and conduction states [1,2]. These competing mechanisms lead to interesting and unusual behaviors like Kondo effect, heavy-fermion or valence fluctuation behaviors. These properties are usually modeled by the single impurity Anderson model (SIAM) which predicts the formation, at low temperature, of a very narrow resonant state near the Fermi energy due to the admixture of the local $4f$ orbitals with the itinerant band states [3]. Spectroscopic properties have then been analysed in this framework [4,5] and it is often claimed that cerium in intermetallic compounds remains in the Kondo limit of the SIAM, *i.e.* with a $4f$ electron number close to 1 ($n_f \geq 0.8$) [3]. However recent thermodynamical and spectroscopic measurements on polycrystalline $CePd_7$ suggest that this limit can be overstepped. The specific heat coefficient and zero temperature magnetic susceptibility are found less for $CePd_7$ than for YPd_7, a similar compound without occupied f states [6]. $CePd_7$ is the only compound where such a reduction of these thermodynamical quantities with respect to the Y-isomorph occurs. The enhancement of the renormalized density of states, characteristic of Kondo materials, is not observed in this compound. This unusual behavior leads some authors to suggest that $CePd_7$ is a free electron system instead of a highly correlated intermediate valence system [6]. High energy spectroscopies partially corroborate these thermodynamical results. X-ray absorption measurements on the L_3 edge show that the $4f$ occupation number is 0.5 [7] (a value never observed in intermetallic Ce compounds [8]), and a very strong valence admixture is also deduced from X-ray photoemission on the $3d$ level. Inverse photoemission confirms the core level spectroscopy results since the spectral structure associated with $4f^2$ final states and usually situated around 5 eV above the Fermi energy in Ce materials is not visible in $CePd_7$ [9]. In summary, both thermodynamical and spectroscopic results suggest that $CePd_7$ belongs to the very strong hybridization limit of the SIAM with a very large Kondo temperature; inverse photoemission data give $k_B T_K = 0.95$ [10].

Unfortunately these spectroscopies are very surface sensitive and Ce is known to have a different electronic configuration at the surface [11]. Therefore experimental spectra always contain a surface contribution which is usually estimated by angle or photon energy dependence of the spectra. For exemple, the analysis of the incident angle dependence in resonant inverse photoemission spectra of $CePd_7$ yields a strong difference between ground state configurations in the bulk and at the surface [10]. In order to overcome these difficulties and obtain information on the bulk contribution, it is necessary to investigate systems with very well characterized surfaces. Recently, some photoemission studies have been carried out in this direction on epitaxial compounds grown on monocrystals. Some spectroscopic measurements have been carried out in this direction essentially in the Ce/Pt(111) system [12]

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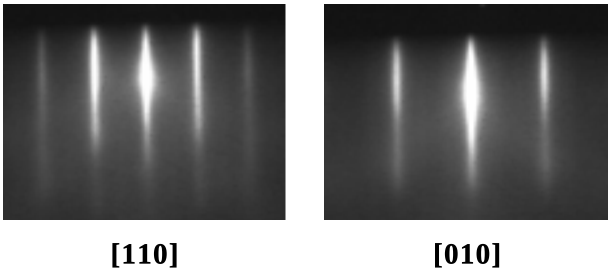

Fig. 1. RHEED patterns after Ce deposition and an annealing at 450 °C for two different azimutal angles. The ratio between streak separations is close to $\frac{\sqrt{2}}{2}$ as expected for FCC structure.

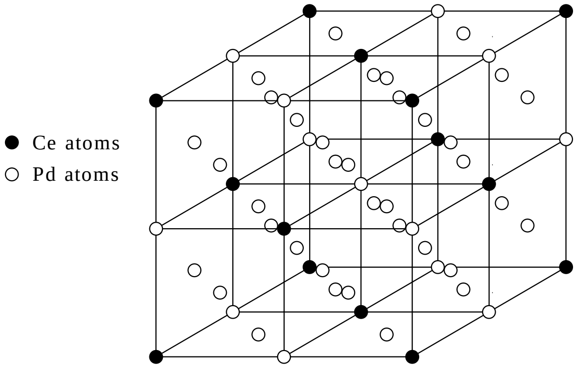

Fig. 2. Atomic structure of $CePd_7$. The solid-solution alloys correspond to a random Ce occupation of the different sites.

where an epitaxial compound $CePt_x$ ($x \simeq 2$) is formed at the surface. We present, in this paper, a core-level and valence band photoemission investigation of the Ce/Pd(100) system. *In situ* elaboration of epitaxial compounds and careful characterization with standard surface techniques lead to very well defined surfaces. In contrast, "bulk" polycrystalline or even monocrystalline materials have to be cleaned before spectroscopic measurements yielding poorly characterized surfaces. We develop an original method to eliminate the spectroscopic signature of the Ce atoms at the surface and then to determine the genuine bulk electronic configuration. By studying a thin crystalline film with a Pd overlayer, one completely elimitates the surface contribution and achieves a greater certainty about the bulk spectroscopic signature. We then demonstrate from core-level spectroscopy that the $4f$ states are strongly hybridized. We also carried out angle-resolved photoemission measurements at the $4d$ threshold and show a very singular spectral behavior which exhibits a significant deviation from the single impurity Anderson model.

2 Sample preparation and experimental details

Ce atoms are evaporated on monocrystalline Pd(100) substrates. The growth mode and surface crystallography are characterized by Reflection High Energy Electron Diffraction (RHEED) and Auger spectroscopy. Auger spectroscopy clearly demonstrates that interdiffusion occurs even at room temperature and an annealing at 450 °C yields a compound or an ordered alloy close to $CePd_7$ in composition. This behavior contrasts with deposition of Ce atoms onto Pd(111) surface which leads to the formation of $CePd_3$ after a subsequent annealing [13]. The thickness of the epitaxial films we prepare is about several tens of Å. In Figure 1, RHEED patterns for 2 different azimutal angles show the monocrystalline character of the prepared interfaces. The shape of the diffraction streaks clearly indicates a two-dimensional diffraction and a very flat surface with a small roughness. In this composition range, the Pd-Ce phase diagram exhibits a solid solution $Pd_{1-x}Ce_x$ with $0 \leq x \leq 0.125$ and an ordered compound: $CePd_7$. The crystallographic structure of $CePd_7$ consists of planes of Ce and Pd atoms separated by pure Pd planes as shown in Figure 2 [14]. The solid solution alloys present a face-centered-cubic structure like Pd metal. From the RHEED data, it is possible to deduce the surface structure. The diffraction patterns appearing for 2 azimutal angles separed by $\pi/4$ (Fig. 1) suggest a square symmetry at the surface. The lattice parameter we deduced ($a = 4.0 \pm 0.1$ Å) is in good agreement with the value observed in the solid solution 3.89 Å $\leq a \leq$ 4.13 Å [15]. A $CePd_7$ surface would give a superstructure associated with a lattice parameter doubled by the ordering of the Ce atoms. As this superstructure is not observed, the epitaxial film we obtain is likely a solid-solution alloy close to $CePd_7$ in composition. In this alloy, Ce atoms are only surrounded by Pd atoms so that the Ce-Ce distance is fairly large ($d = 5.67$ Å in $CePd_7$). After deposition and annealing, the surface layer contains both Pd and Ce as suggested in Auger spectra. In order to suppress the surface contribution in the Ce $3d$ core-level photoemission spectra, we have deposited one Pd layer on the epitaxial film. We demonstrated in the Ce/Fe(100) systems [16,17] that the modification of the Ce electronic structure due to surface effects is only limited to the first layer. Any sizeable interdiffusion between the Pd and the Ce/Pd alloy is not detectable by Auger spectroscopy suggesting that in the Pd-covered thin films, no Ce atoms remain at the surface. Ce atoms have the same local environment and give the same contribution in the photoemission spectra. This system is interesting because the hybridization strength is known to be large between Ce-f and Pd-d states [18] but as Ce-Ce interatomic distance is large, the direct f-f overlapping is small suggesting that this Ce/Pd alloy should be described in a first approximation as an assembly of independent Ce impurities in the SIAM.

Core-level photoemission spectra were obtained at room temperature on a VG ESCALAB MKII apparatus with Al Kα excitation energy. The resonant photoemission experiments were carried out at the Swiss-French SU3 undulator beamline of the superACO (Orsay) with a VSW analyzer. The overall energy resolution at the photon energy used and the angular acceptance of the analyzer were, respectively, 60 meV and ± 2°. These spectra were recorded at $T = 40$ K.

3 Results and discussion

In Figure 3, we present Ce $3d$ core-level photoemission spectra recorded on the epitaxial Ce/Pd system. These spectra exhibit two series of peaks whose separation corresponds to the core-hole $3d$ spin-orbit interaction. Each spectral structure is associated with a final state configuration of the Ce atoms. The prominent features in Figure 3a have mainly a f^1 character whereas the satellites correspond to final states with f^2 and f^0 character. As demonstrated by Gunnarsson and Schönhammer [3], the electronic configuration of Ce in the initial state can be deduced from the satellite intensities. The energy separation between these satellites and the main line mainly depends on the interaction between the localized f electrons and the core-hole left behind the photoexcitation. Qualitatively, the intensity of the f^0 structure, clearly seen in the $3d_{3/2}$ part at $E = 571.8$ eV, reflects the weight of the f^0 configuration in the ground state whereas the f^2 feature, at $E = 606.6$ eV in the $3d_{5/2}$ part, is a measure of the hybridization between $4f$ and conduction states [3]. Recently, following an idea of Gunnarsson and Jepsen [19], we have modified this approach to take into account in the calculation of photoemission spectra the dependence of hybridization with the Ce configuration. A better agreement with experiments is then obtained [20]. As emphasized in the introduction, the Ce electronic configuration is usually different at the surface. Thus the experimental photoemission spectrum (curve (a) in Fig. 3) cannot be directly related to the electronic configuration of Ce because it contains two contributions associated with surface and bulk Ce atoms. This spectrum is very similar to the spectrum of polycrystalline $CePd_7$ sample [9]. In order to suppress the surface contribution, we covered the sample with one monolayer of Pd at room temperature. Spectrum (b) in Figure 3 corresponds to this covered sample. A spectacular increase in the satellite intensities is exhibited suggesting a very strong valence admixture in the bulk. This method we used to eliminate the surface contribution allows an accurate determination of the bulk contribution without ambiguity. Usually, in polycrystalline materials, photoemission spectra contain a surface contribution which is quite difficult to quantify. The surface and bulk contributions are generally deduced by using the energy dependence of the inelastic mean free path [21]. Our procedure gives directly the bulk contribution without any assumption about the mean free path. We simulate the bulk spectrum in the framework of the configuration-dependent hybridization Gunnarsson-Schönhammer model [19,20] (Fig. 3c). A good agreement with the experimental spectrum is obtained with the following main parameters: the $4f$ energy $\varepsilon_f = -0.5$ eV and hybridization energy $V = 0.7$ eV. With this set of parameters, the ground state is characterized by a very strong valence admixture since the weight of the $4f^1$ configuration is particularly low ($w(f^1) = 0.57$) corroborating previous calculations in $CePd_7$ [9]. We would like to point out the small value of the charge fluctuation energy ($\varepsilon_f = -0.5$ eV). Usually in Ce compounds, the charge fluctuation energy is significantly larger so that only spin fluctuations are important for the low-energy properties. In the Kondo limit of the SIAM, the thermodynamical quantities only depend on one parameter (the Kondo energy) [2]. With the parameters we found, charge fluctuations play also an important role and should affect the near ground state properties. The spin fluctuation energy, which corresponds to the Kondo energy ($k_B T_K$), can be estimated to be $k_B T_K = 0.8$ eV from these simulations. Therefore charge (ε_f) and spin ($k_B T_K$) excitation energies have the same order of magnitude and the Ce electronic configuration cannot be interpreted in the Kondo limit of the Anderson Hamiltonian. This result shows that, in contrast to usually claimed, cerium can be encountered in the intermediate valence regime. We think that Ce/Pd is not the only system in this situation. Highly-hybridized compounds like $CeRh_3$, $CeRu_2$, etc. should belong to the same regime as corroborated by recent photoemission measurements carried out at very high photon energy on several Ce compounds [21–23]. These different results suggest that the surface contribution is sometimes underestimated yielding an incorrect determination of the Ce bulk electronic configuration. The difference in the Ce-electronic configuration in compounds like $CeRh_3$, $CePd_7$ and $CePd_3$ can be simply understood by the position of the Fermi energy with respect to the transition

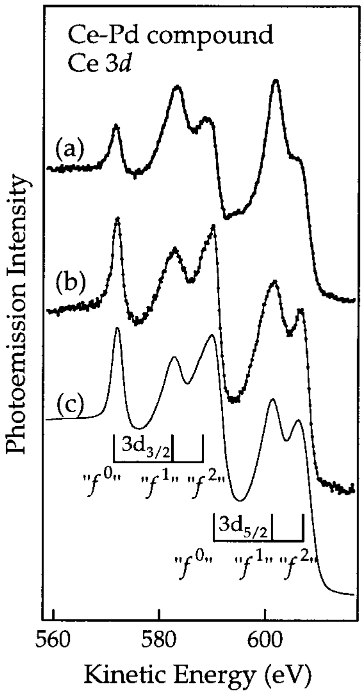

Fig. 3. Room temperature Ce $3d$ photoemission spectra: (a) uncovered epitaxial Ce/Pd thin film, (b) same film covered with one Pd monolayer, and (c) calculated in the Gunnarsson-Schönhammer model.

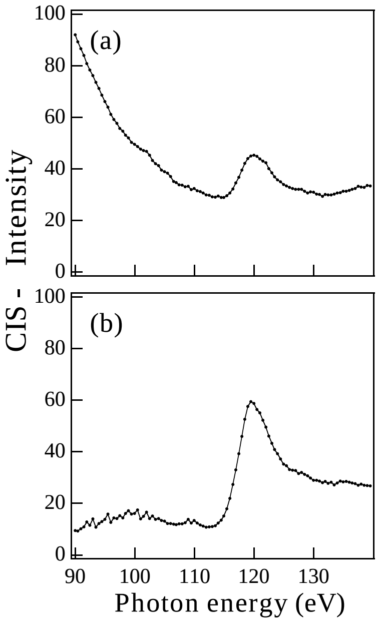

Fig. 4. Constant initial state spectra for two different binding energies, $E_B = -0.35$ eV (a) and $E_B = -1.5$ eV (b), near the $N_{4,5}$ threshold.

metal-*d* band. In $CePd_3$, the Fermi energy is situated in the gap between the Pd-4*d* and Ce-5*d* bands. As a consequence, the hybridization is weak. In $CeRh_3$ and $CePd_7$, the transition metal band is only partially occupied and a large density of states is found at E_F [13,18] leading to a large hybridization and then to a large configuration mixture. These core-level photoemission results are corroborated by valence band photoemission measurements near the $N_{4,5}$ threshold. In such experiments, an enhancement of the 4*f* signal is achieved by using the very strong energy-dependence of the 4*f*-cross section associated with the Fano effect. Figure 4 shows constant initial state spectra, which represent the evolution of the spectral weigth as a function of the photon energy, for two binding energies $E_B = -0.35$ eV (a) and $E_B = -1.5$ eV (b). For $E_B = -0.35$ eV, a pronounced peak is observed near $h\nu = 120$ eV (the $N_{4,5}$ threshold) suggesting that the spectral weight is dominated, at this binding energy, by Ce 4*f* states. By contrast the Fano resonance is strongly reduced for $E_B = -1.5$ eV indicating that the spectral intensity is mainly due to Pd states. As discussed in reference [24], the non-*f* states can resonate and the reminiscent Fano lineshape for $E_B = -1.5$ eV can be induced by the large hybridization between Ce-4*f* and Pd states. In Figure 5, we present low temperature ($T = 40$ K) valence band photoemission spectra of Ce/Pd thin films at normal emission near the $N_{4,5}$ threshold. As expected, a very strong photon energy-dependence is observed. Curves (a) and (b) in Figure 5 correspond respectively to the resonance ($h\nu = 120$ eV) and off-resonance ($h\nu = 113.5$ eV) spectra. Comparison of these two spectra clearly indicates that the peak just below the Fermi energy (E_F) can be attributed to the Ce 4*f* states. The spectral features near $E = -1.4$ eV and $E = -2.7$ eV probably result from Pd states. This statement is confirmed by measurements on pure Pd at the same energies (curves (c) and (d)) which exhibit the same structures. The off-resonance spectra in $CePd_7$ and Pd (curves (b) and (d)) are very similar. Moreover, the increase in the intensity of the $E = -1.4$ eV feature with increasing photon energy in Pd suggests that we have to take into account the modification of the Pd-derived spectral weight between resonance and off-resonance energies in the epitaxial $CePd_7$. These two observations lead us to the conclusion that the 4*f* spectral function is better estimated from the difference of the spectra of $CePd_7$ and Pd (curves (a) and (c)) at the resonance energy ($h\nu = 120$ eV). The 4*f* spectrum, obtained with this procedure, is only composed of one structure just below the Fermi energy (Fig. 6). What is the expected shape of the 4*f* spectral function in the single impurity Anderson model? Calculations carried out in the non-crossing approximation for the Kondo limit predict a broad feature around ε_f (usually experimentally observed between -1 and -2 eV [5,25]) and two narrow peaks at E_F and $E = -0.3$ eV interpreted as the tail of the Kondo resonance and the spin-orbit sideband [26]. However, these spectroscopic signatures are significantly modified in the intermediate valence regime [27]. The intensity of the broad f^0 structure vanishes whereas the E_F-peak broadens and increases in intensity. As a consequence the spin-orbit satellite at $E = -0.3$ eV is no longer resolved, and only one feature is observable.

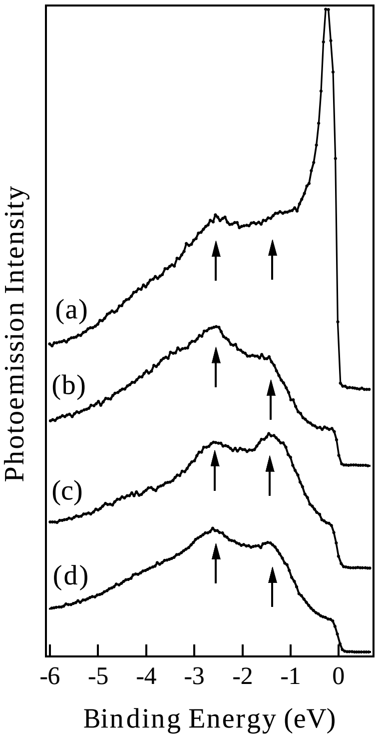

Fig. 5. Photoemission spectra at normal emission : (a) epitaxial Ce/Pd thin film at $h\nu = 120$ eV, (b) epitaxial Ce/Pd thin film at $h\nu = 113.5$ eV, (c) Pd buffer at $h\nu = 120$ eV, and (d) Pd buffer at $h\nu = 113.5$ eV.

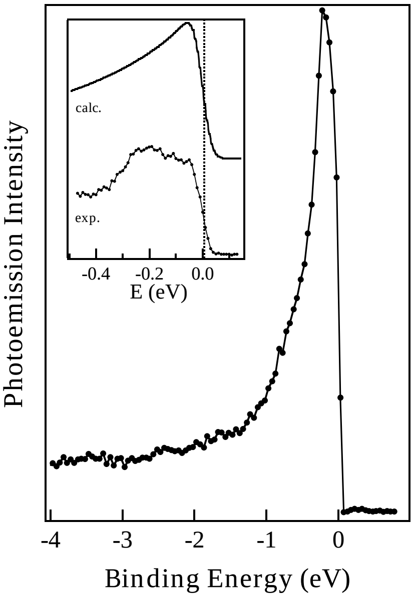

Fig. 6. $4f$ spectral function estimated from the difference of Ce/Pd thin film and Pd spectra at the resonance energy. In the inset, we show a close up of the experimental spectrum (exp.) and the spectral function calculated in the framework of the NCA of the infinite U single-impurity Anderson model (calc.).

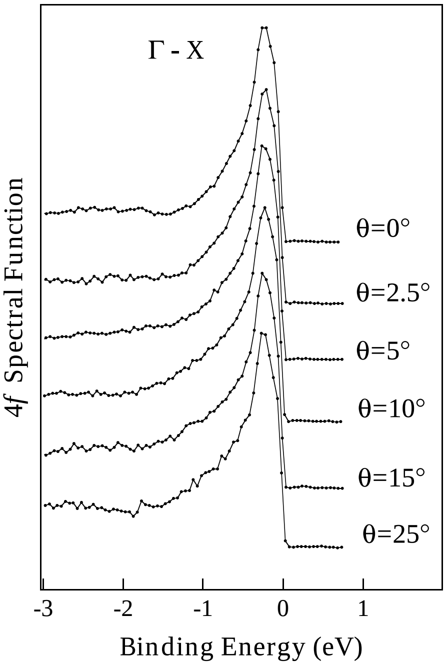

Fig. 7. Angle-resolved spectra in the ΓX direction at the resonance energy.

The experimental spectrum in Figure 6 is in qualitative agreement with this picture and then corroborates the results of our core-level photoemission measurements which suggest that Ce belongs to the intermediate valence regime in this compound. More surprisingly is the detailed shape of the spectral feature near the Fermi energy. As shown in the inset of Figure 6, the intensity maximum is observed near $E = -0.2$ eV. This shape of the spectral function has never been observed before in Ce compounds since high resolution photoemission data always exhibit a maximum at E_{F} which is interpreted to result from the tail of the Kondo resonance cut by the Fermi function [25]. In the inset of Figure 6, we also report the $4f$ spectral function calculated in the Non-Crossing-Approximation (NCA) of the SIAM in the infinite U limit [28]. The parameters used in the calculation were chosen to reproduce the weight of the f^1 configuration ($w(f^1) = 0.57$) we deduced from our core-level photoemission spectrum. In this calculation, a semi-elliptical conduction band was used. This schematic density of states (DOS) is far from realistic for $\mathrm{CePd_7}$ but it is well known that the precise shape of the Kondo resonance does not depend on the conduction DOS [2,25]. Therefore, comparison between experimental and calculated spectra clearly shows that the SIAM cannot describe the peculiar shape of the spectral function.

As previously suggested for $\mathrm{CeRh_3}$ [29], a band picture could be a more appropriate description of the $4f$ state in highly-hybridized materials. In order to evidence a possible band signature like dispersion or intensity variation, we carried out angle-resolved photoemission experiments. Recent measurements in several monocrystalline samples have shown an angular dependence of the intensity of the near-E_{F} structure revealing a momentum dependence of the $4f$ spectral function [30,31]. Our angle-resolved spectra of the epitaxial Ce/Pd shown in Figure 7, always exhibit the same shape without any sizeable evolution throughout the ΓX direction in the Brillouin zone. Dispersion can also be encoutered in the Anderson model as shown in recent numerical calculations of the spectral function of the periodic Anderson model [32]. These calculations, corresponding to a D-dimensional hypercube lattice in the infinite D limit, are far from realistic for Ce compounds and cannot be directly compared with experimental results. Nevertheless, they clearly indicate an enhancement of the spectral intensity near E_{F} with respect to the SIAM and a dispersion of the Kondo resonance. In our spectra, no Fermi energy crossing, which would at least manifest itself by an angle dependence of the spectral intensity, of an hypothetical $4f$ band is observed. But, this result does not allow us to completely exclude the existence of a $4f$ band or a dispersive Kondo resonance. As the momentum resolution is poor near the resonance energy, the dispersion would not be observed with our experimental angle and energy resolutions.

4 Conclusion

To summarize, by deposing Ce on a monocrystalline Pd(100) substrate, we succeed to prepare an epitaxial compound with a well characterized surface. An overlayer of Pd allows to eliminate the surface contribution in the core-level Ce-3d spectra. We then demonstrate that the bulk ground state of this epitaxial film is strongly hybridized. These data confirm the results obtained on polycrystalline $\mathrm{CePd_7}$ sample. This system does not belong to the Kondo limit of the Anderson model since the charge and spin excitation energies have the same order of magnitude. Although the core-level spectra reveal strong intra-atomic correlations and can be described in the SIAM, the $4f$ photoemission spectra only exhibit one feature whose energy position cannot be accounted for by this model. Since any dispersion and any angle-dependence of the $4f$ intensity are not observed, the f states do not exhibit a clear band signature. A satisfactory description of $4f$ electrons in such materials with strong intra-atomic correlations and a high degree of hybridization is still missing.

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