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Solid-state reaction in Ce/Ni multilayers studied by x-ray-absorption spectroscopy

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Taking advantage of the high sensitivity of the Ce valence to the local environment, we investigated the early stage of the solid-state reaction in Ce/Ni multilayers through x-ray absorption spectroscopy. Ce L_{III}-edge and Ni K-edge spectra have been compared to distributions of experimental spectra obtained from amorphous Ce-Ni alloys. Two possible diffusion profiles have been examined. It is found that the experimental data are compatible with the formation of a homogeneous amorphous-alloy interlayer instead of a Fickian profile.

I. INTRODUCTION

As noticed by Johnson[1] in his review of solid-state reactions, amorphization by diffusion in metallic multilaylayers can be considered to be a ubiquitous phenomenon. He observed from dimensional arguments that a special growth mechanism could operate for very thin interlayers (~ 10 Å) indicating that such thin interlayers also exist for nonreacting systems, as long as they are not epitaxially grown. To understand the properties of any multilayer, it is important to assess how sharp the interfaces are, and thus to investigate the conditions and mechanisms of formation of the interfacial region. We present a new method of studying the concentration profiles of very thin layers.

A set of experiments have been carried out to follow the early stages of diffusion in Ce/Ni multilayers by measuring the valence change in Ce with L_{III}-edge x-ray absorption spectroscopy. We found that at liquid-nitrogen temperature, almost all Ce is already mixed with Ni to form homogeneous Ce-Ni alloy layers separated by pure Ni layers. On annealing, the remaining pure Ni is absorbed by the amorphous Ce-Ni layer.

II. SAMPLE PREPARATION AND EXPERIMENT

Samples were made by alternate evaporation in an ultra-high vacuum chamber of Ni (99.998%) and Ce (99.99%) layers onto a Kapton$^{\mathrm{TM}}$ polyimide substrate cooled by a copper plate in contact with a liquid nitrogen tank. 130 bilayers of Ni (30 Å) and Ce (35 Å) were deposited. The pressure was 5×10^{-8} Torr, and the deposition rate was 2.0 Å/s for Ni and 2.4 Å/s for Ce. Finally, the multilayer was protected against corrosion by a 1000-Å Si coating.

The samples were kept in liquid nitrogen until they were transferred into a helium cryostat at the DISPERSIF station in the Laboratoire d'Utilisation du Rayonnement Electromagnétique. The maximum temperature reached by the sample during the short (<5 s) transfer was estimated to be less than 150 K by repeating the operation with a small thermocouple.

X-ray-absorption near-edge structure (XANES) spectra from 30 eV below to 100 eV above the Ce L_{III} edge and extended x-ray-absorption fine-structure (EXAFS) spectra from 50 eV below to 300 eV above the Ni K edge were recorded as the temperature was increased from 77 to 300 K. The temperature sweep was 2 K/min between 77 and 220 K (no change was observed in the 11 spectra taken in that range) and 1 K/min between 220 and 250 K (four spectra) where previous resistivity measurements had detected the onset of diffusion.[2]

To study the dynamics of solid-state reaction at constant temperature, three spectra were taken at 250 K (two spectra between 250 and 263 K), and four spectra at 263 K. Then six spectra were recorded between 263 K and room temperature (292 K). Two samples were measured at each edge, and reproducibility was found to be excellent.

We have used x-ray-absorption spectroscopy in a dispersive mode[3] to get full spectra within short times (1 s), so that the samples do not evolve during measurements. 30 spectra have been taken at each edge. Harmonics were suppressed with a gold mirror, and experimental resolution was 2 eV.

Subsequent transmission electron microscopy revealed that the diffused samples were amorphous.

III. EXPERIMENTAL RESULTS

A. EXAFS at the Ni K edge

Figure 1 shows the Ni K edge before and after diffusion. EXAFS undergoes few changes during diffusion.

For analysis of EXAFS in multilayers, two processes have been used in the past. Claeson *et al.*[4] have obtained EXAFS data from Nb/Zr multilayers of varying periods. They performed standard EXAFS analysis[5] to extract the number of neighbors of each species, the

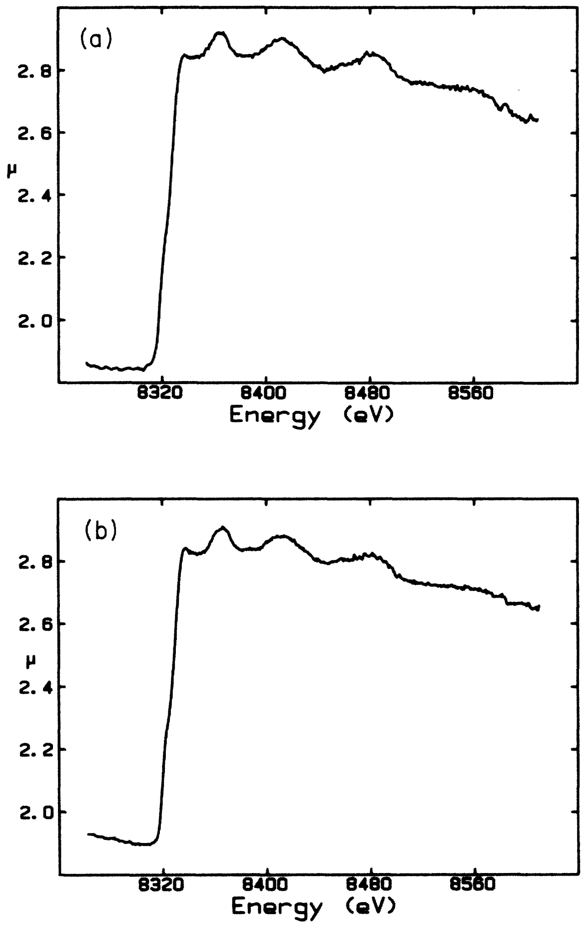

FIG. 1. (a) X-ray-absorption spectrum (absorption μ in arbitrary units) at the Ni K edge for the sample before annealing (77 K). (b) X-ray-absorption spectrum (absorption μ in arbitrary units) at the Ni K edge for the sample after annealing (up to room temperature).

average distances between species, and the Debye-Waller–like factors. This method was successful in discriminating between the bcc structure (eight first neighbors) and the hcp structure (12 first neighbors). Then they tried to take into account interdiffusive regions where concentration follows a linear or a step[8] profile. They obtained an interlayer thickness of 15 Å consistent with x-ray diffraction results, but they observed that the calculated number of neighbors could not be considered to be significant in the interdiffusive region.

Heald *et al.*[6] have performed EXAFS measurements on Cu/Hf multilayers with modulation periods of $\frac{1}{2}$–20 close-packed monolayers. They considered the multilayer interface as a layer of amorphous material which is surrounded by bulk metal, and they fitted their data by adjusting α in the equation

$$(\text{observed EXAFS}) = \alpha(\text{bulk-metal EXAFS}) + (1-\alpha)(\text{amorphous-alloy EXAFS}) .$$

They obtained consistent results from Cu and Hf edges with interfacial regions about 8–10 layers thick. This method has been used by Corni and Antonini[7] and by Brouder *et al.*[8] to study the solid-state reaction in Cu/Au and Co/Sn multilayers, respectively. However, they neglected in-layer structural reorganization during diffusion,[8] and the fact that EXAFS obtained from a (disordered) pure metal in a multilayer are different from EXAFS obtained from the same metal in a crystalline state.[8] Furthermore, the concentration of the amorphous alloy used as a standard has to be guessed, and can be nonuniform in the interlayer.

From Fig. 1 it appears that Ni EXAFS *by itself* is not sensitive enough to draw definite conclusions about the dynamics of diffusion. However, we shall see that the Ce L_{III} edge will enable us to describe these dynamics, even in the present difficult case.

B. XANES at the Ce L_{III} edge

Figure 2 shows the Ce L_{III} edge before, during, and after diffusion, where it appears that Ce XANES is much more sensitive to diffusion than EXAFS at the Ni K edge.

The L_{III} edge has become a standard measurement of valence in mixed-valent compounds. Cerium can have two configurations: $4f^0(5d6s)^4$ and $4f^1(5d6s)^3$. When Ce is mixed-valent, its L_{III}-edge spectrum shows two broad peaks typically 10 eV apart. According to the simple interpretation of these spectra,[9] the incident photon extracts an electron from a $2p_{3/2}$ shell of a Ce atom, and the core hole left behind can be screened either by a $4f$ or by a $5d$ electron. Screening with a $4f$ electron is more effective, because this orbital is very localized near the nucleus. Thus the photoelectron is not as strongly attracted by the core hole as for a $5d$ screening, and the threshold is at a lower energy. Consequently, the spectrum is analyzed as a sum of two contributions: the lower energy peak is due to a $4f$ screening ($4f^1$

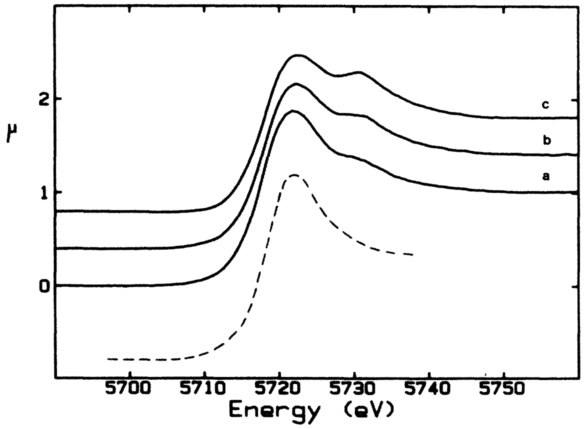

FIG. 2. XANES at the Ce L_{III} edge for sample a, before annealing (77 K); b, during annealing (250 K); c, after annealing (room temperature). Dashed line: XANES at the Ce L_{III} edge for the Ce/Cu sample (absorption μ in arbitrary units).

configuration), the higher one to the 5*d* screening ($4f^0$ configuration). In each case, the transitions to unoccupied states and the core-hole lifetime have been taken into account. The average valence is obtained from the relative intensity of these two contributions, fitted to the experimental spectrum.

We have used a standard computer program[10] to deduce valence from Ce XANES spectra (Fig. 2), and from amorphous Ce-Ni alloys of various concentrations used as references.[11] The valence obtained is 3.18 for the sample before diffusion; then it increases smoothly during annealing, up to 3.26. The valence of a homogeneous sample with the same amount of Ce and Ni is 3.27.[11]

At the DISPERSIF station, no mechanical parts move during the whole experiment, thus valence changes can be followed very precisely by fitting the difference between spectra.

Valence can also change because of oxidation[12] or pressure effects.[13] In order to determine whether oxidation occurs, the protective Si coating was scraped from a diffused sample with a low-energy ion gun in an ESCA (electron spectroscopy for chemical analysis) chamber, and photoemission measurements were performed. Only traces of oxygen were detected, so that oxidation effects can be discarded. We have not attempted to estimate the concentration gradient with ESCA because the facility cannot operate at liquid-nitrogen temperature.

To estimate pressure effects, a Ce/Cu multilayer was prepared under exactly the same conditions as the Ce/Ni sample. As Ce-Cu alloys exhibit no mixed valent behavior, if any valence change occurs, it can be wholly attributed to a pressure effect. As the structure and lattice parameters of Ni and Cu are similar, the stresses should be the same in Ce/Cu as in Ce/Ni. Ce L_{III}-edge x-ray-absorption spectra of the Ce/Cu sample measured at 77 K show that Ce is trivalent (Fig. 2). Thus the interfacial pressure is not strong enough to cause the transition from Ce_γ to Ce_α that occurs around 10 kbar, and the valence change observed in Ce/Ni can be attributed only to an alloying effect. Because of this low pressure, diffusion can be considered as a means for the system to release interfacial stresses.

IV. DISCUSSION

A. Method

The method used here to analyze L_{III}-edge spectra is related to the method described by Claeson *et al.*,[4] except that experimental spectra have been used instead of theoretical ones.

The layers are assumed to be periodic and planar, as experimentally observed on other systems.[14] Thus the diffused state is characterized by the concentration profile across a bilayer. To compute the valence corresponding to a given profile, thin imaginary slices are cut into the profile. Within each slice, the concentration is almost constant, and the valence is the same as in the co-evaporated amorphous compound of the same concentration (published by Malterre *et al.*[11]). Finally, the total valence of the sample is this local valence averaged over the full profile of a bilayer. Thus a valence is obtained as a function of a profile.

The present model is continuous and does not take into account the fact that layers are made of individual atoms. As noticed by Claeson *et al.*,[4] this atomic character is second order (proportional to the second derivative of the profile) and can be neglected for a smooth profile. For a steplike profile, there are very few atoms at the interface; moreover, as our samples are highly disordered, it is not much better to consider the multilayer as a set of regular atomic planes. On the whole, this assumption of continuity appears to introduce only small errors.

Our model relies on two main assumptions.

(1) In each slice, the local environment of the amorphous alloy formed during diffusion is the same as the local environment of the co-evaporated amorphous alloy with the same concentration. This is supported by the experimental evidence that generally, amorphous-alloy properties do not depend on their methods of preparation.[1,15] Furthermore, in our system, co-evaporated alloys and multilayers have been prepared under the same conditions in the same evaporator,[11] and Ce and Ni x-ray-absorption spectra of the diffused samples and of the corresponding co-evaporated alloy are very similar.

(2) Valence depends only on the local environment of Ce. This is supported by the successful use of the Jaccarino-Walker model to describe valence change in $\mathrm{Ce(Cu_{1-x}Ni_x)_5}$ alloys[16] (the Jaccarino-Walker hypothesis being much stronger than a local environment dependence).

A family of profiles must now be chosen to describe the diffusion process. A number of models of diffusion varying in sophistication are reviewed by Johnson,[1] and two simple one-parameter models have been tested in the present study, namely the "homogeneous model," and the "Fickian diffusion model."

Our samples are in three different situations: an initial state (as prepared at liquid-nitrogen temperature), in evolution (from 77 K to room temperature), and a final state (at room temperature). We assume that all these situations can be described by the variation of a single parameter, called λ for the homogeneous model and θ for the Fickian model.

B. Homogeneous model

In this model a homogeneous amorphous layer of uniform composition is considered to form between pure Ce and pure Ni. The parameter λ is the ratio of the amorphous layer thickness to the multilayer period. The uniform composition has been chosen to be the composition of the most stable amorphous Ce-Ni alloy according to de Boer *et al.*[17]: $\mathrm{Ce_{0.40}Ni_{0.60}}$. The coordinates z_A and z_B, where the amorphous layer begins and ends, are determined by mass conservation of both species. Let C_A^0 be the concentration of Ce in mass per unit volume corresponding to the constant atomic concentration α (here $\alpha=0.4$). Mass conservation yields

$$z_A = \frac{a}{2}\left[1 - \frac{\lambda C_A^0}{\rho_A}\right], \quad z_B = z_A + \frac{\lambda a}{2} .$$

Once one of the species has been completely transformed into the amorphous phase, the other species is absorbed by forming a homogeneous amorphous alloy of varying concentration. The mass concentration of Ce in the resulting alloy is $C_A = \rho_A/\lambda$. It should be stressed that this model is compatible with cross-section TEM micrographs observed in (thicker) Co/Zr multilayers.[14]

Figure 3(a) shows a number of profiles, and Fig. 3(b) plots valence as a function of the diffusion parameter λ. Between $\lambda=0$ and $\lambda=0.78$, pure Ce is absorbed by the $Ce_{0.40}Ni_{0.60}$ amorphous phase. Above $\lambda=0.78$, the Ce concentration in the amorphous phase decreases from $x=0.4$ to the final concentration $x=0.27$.

From Fig. 3(b), we obtain the values of λ corresponding to the valences of the experimental spectra. According to this model, the initial state of our sample is described by $\lambda=0.76$ and, on annealing, λ increases smoothly up to the final state $\lambda=0.96$.

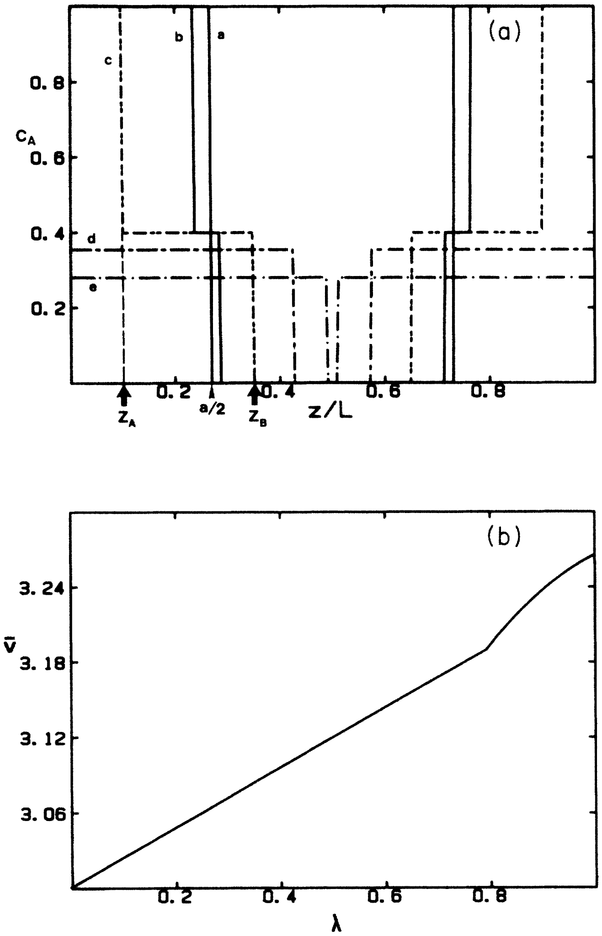

FIG. 3. (a) Profiles (atomic concentration of Ce vs depth) in the homogeneous model for *a*, $\lambda=0$; *b*, $\lambda=0.1$; *c*, $\lambda=0.5$: *d*, $\lambda=0.85$; *e*, $\lambda=0.98$. (b) Average valence as a function of the diffusion parameter λ in the homogeneous model.

C. Fickian diffusion model

In this model, Fick's diffusion equation

$$\frac{\partial C}{\partial t} = D\frac{\partial^2 C}{\partial z^2}$$

(where C is the concentration in mass per unit volume, D the diffusion constant, and z the depth in the layer) is solved for a constant-volume interdiffusion process[18] in a periodic material. Starting from a steplike profile (one layer of pure Ce and one layer of pure Ni), the following concentration is obtained as a function of time and depth:

$$C_A(z,t) = \frac{a}{L}\rho_A + \frac{2\rho_A}{\pi}\sum_{n=1}^{\infty}\frac{1}{n}\sin\left[\frac{n\pi a}{L}\right]\exp\left[-D\left[\frac{2\pi n}{L}\right]^2 t\right]\cos\left[\frac{2\pi n z}{L}\right] .$$

Defining the dimensionless parameter $\theta = 4\pi^2 Dt/L^2$, it is easily seen that each value of θ gives a unique profile.

Figure 4(a) shows a number of profiles, and Fig. 4(b) plots valence as a function of the diffusion parameter θ.

Thus a change in valence can be described by a change in θ. As for the homogeneous model, we find the values of θ corresponding to the observed valences from Fig. 4(b). We obtain the initial state with $\theta=0.84$ and the final state with $\theta=1.94$. The profile with $\theta=0.84$ is already an almost pure sine wave (the second harmonic is 1% of the first).

For this model more can be said about the system's evolution. At constant temperature, D should be the slope of the curve giving θ as a function of time. In Fig. 5 $\Delta\theta(t) = \theta(t) - \theta(t_0)$ (where t_0 is the time of the first spectrum taken at each temperature) is plotted for $T=250$ K and $T=263$ K. It is clear that $\theta(t)$ is not linear and that the interdiffusion coefficient D decreases with time. This is the typical behavior of diffusion in compositionally modulated films[19,20] where D is observed to decrease rapidly in the early stage of diffusion.

The maximum value of D is obtained as the slope of $\theta(t)$ for $t=0$, yielding $D(250\text{ K}) = 3\times10^{-18}$ cm^2/s and $D(263\text{ K}) = 0.8\times10^{-18}$ cm^2/s. Consequently, the diffusion coefficient appears to decrease with temperature, a result that casts doubt on the Fickian model.

D. Selection of the best profile

Even if EXAFS by itself is not sensitive enough to follow the diffusion process, it will be shown that it can be used to discriminate between different diffusion models.

To do this, an EXAFS spectrum is constructed from a profile, following the same method as for the valence: the profile is cut into thin slices, the contribution of each slice is the experimental EXAFS of the co-evaporated alloy with the same concentration, and the contributions are averaged over a bilayer, yielding an EXAFS spec-

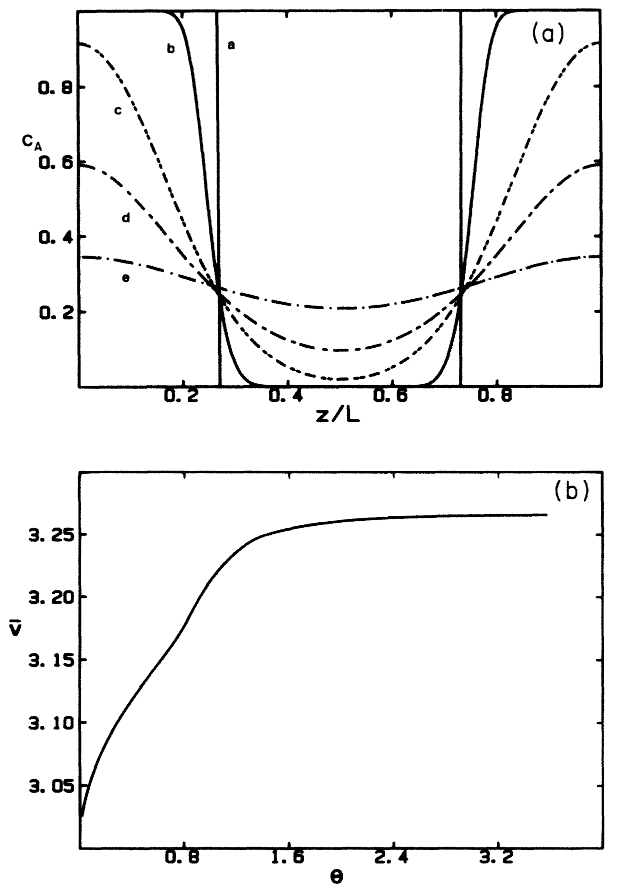

FIG. 4. (a) Profiles (atomic concentration of Ce vs depth) in the Fickian model for *a*, $\theta=0$; *b*, $\theta=0.02$; *c*, $\theta=0.3$; *d*, $\theta=0.8$; *e*, $\theta=2$. (b) Average valence as a function of the diffusion parameter θ in the Fickian model.

trum corresponding to the given profile. EXAFS at the Ni *K* edge were available to us only for a-$Ce_{0.30}Ni_{0.70}$ and a-$Ce_{0.57}Ni_{0.43}$. EXAFS for the other concentrations were linearly interpolated from these two spectra. This procedure is correct if the average local environment can be considered as a weighted sum of two specific local environments, which is consistent with the almost linear variation of valence as a function of concentration[11] in the concentration domain spanned by the Fickian model. For the homogeneous model, a disordered pure Ni EXAFS has been made by increasing the "Debye-Waller" factor of an experimental crystalline EXAFS spectrum at Ni *K* edge. This is a way to simulate the static disorder in Ni layers with the dynamic disorder due to temperature.

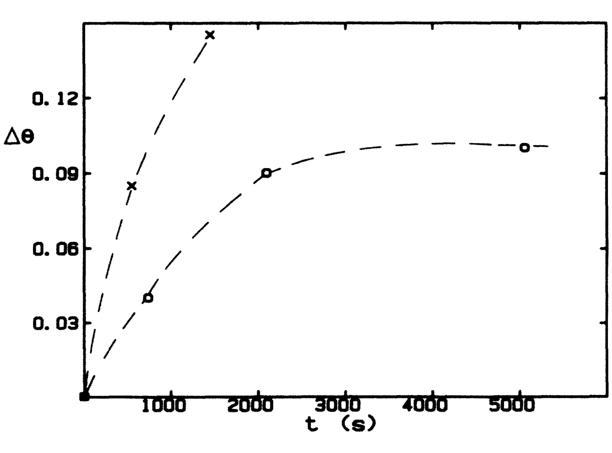

FIG. 5. $\Delta\theta$ as a function of time for $T=250$ K (crosses) and $T=263$ K (circles).

We would like to emphasize that no adjustable parameters are involved in this procedure. We take the concentration profile deduced from the models and the observed valences, then we simply add experimental Ni spectra (weighted by the profile) to obtain our EXAFS spectra.

EXAFS obtained from the Fickian and homogeneous models are shown in Fig. 6 for the samples before diffusion. Clearly the homogeneous model gives better agreement with the experimental results (the mean square error is 9.1×10^{-5} for the Fickian profile and

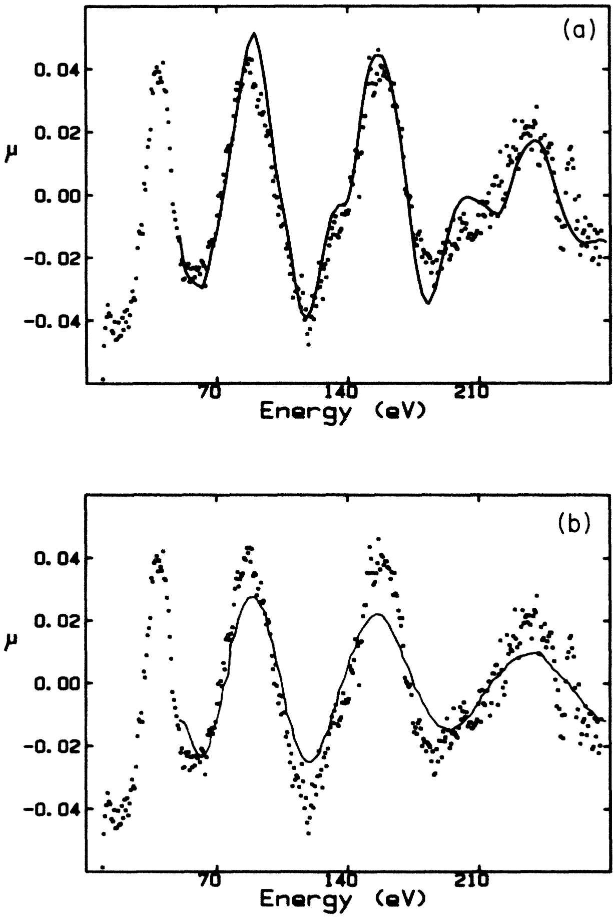

FIG. 6. (a) Experimental Ni *K*-edge spectrum of the sample before annealing (dots) compared with the EXAFS obtained from the homogeneous model (solid line). (b) Experimental Ni *K*-edge spectrum of the sample before annealing (dots) compared with the EXAFS obtained from the Fickian model (solid line). The solid line is wavy because it is generated from experimental spectra. (Absorption μ in arbitrary units.)

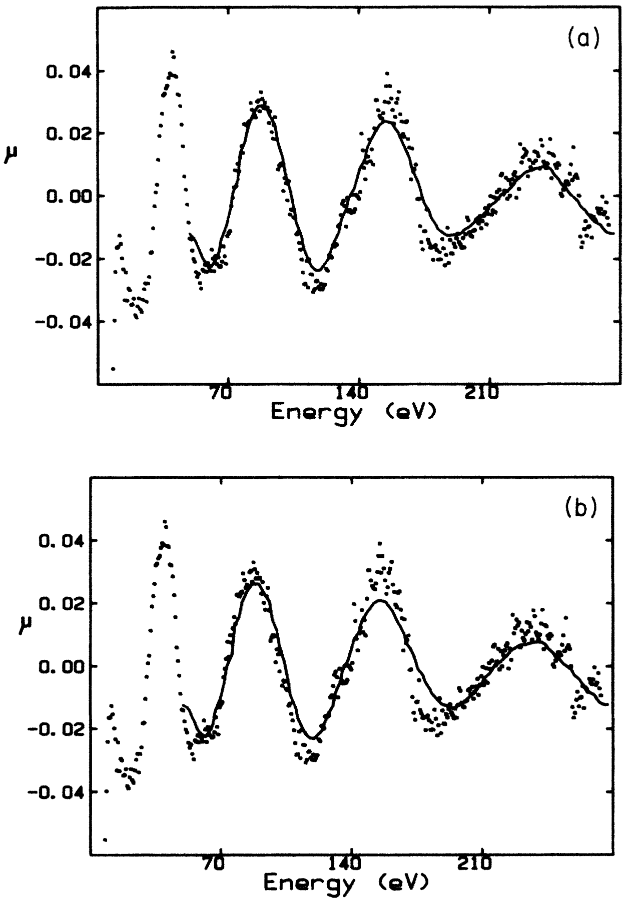

FIG. 7. (a) Experimental Ni *K*-edge spectrum of the sample after annealing (dots) compared with the EXAFS obtained from the homogeneous model (solid line). (b) Experimental Ni *K*-edge spectrum of the sample after annealing (dots) compared with the EXAFS obtained from the Fickian model (solid line). (Absorption μ in arbitrary units.)

6.7×10^{-5} for the homogeneous one). Enhancing the Debye-Waller factor is only a first approximation to disorder in Ni layers (the shape of the spectrum is not modified, it is only damped towards high energy), thus our homogeneous model simulation is more finely structured than the experimental spectrum.

Qualitatively, we see that the amplitudes of EXAFS oscillations are too low in the Fickian model, because we average over many different local environments, so that interference effects flatten the signal. In the homogeneous model, there is only one local environment for the amorphous phase, and Ni EXAFS of $Ce_{0.40}Ni_{0.60}$ and pure Ni are in phase, so that the presence of pure Ni enhances the signal up to its experimental value.

After diffusion, the simulations are still good (Fig. 7), but there is no longer any difference between the Fickian and homogeneous models, since the diffusion is almost complete (the mean-square error is 4.3×10^{-5} for the Fickian model and 3.6×10^{-5} in the homogeneous case).

V. CONCLUSION

The main point of this paper is to demonstrate that L_{III}-edge x-ray-absorption spectroscopy is a very sensitive method of studying diffusion in mixed-valent compounds. This is demonstrated by the fact that a glance at the Ce spectra is enough to know that layers are strongly mixed, independently of any model, because such a high valence can only be obtained from a large proportion of Ce alloyed with Ni.

Even for the very thin layers that have been investigated, it was possible to discriminate between two diffusion models, showing that the homogeneous amorphous interlayer model proposed by Johnson[21] in early papers on solid-state reactions is compatible with experiments. As the homogeneous profile appears before heating the sample, it seems that the process leading to this profile occurs at a very early stage, maybe during the preparation itself. This behavior has also been observed in Ni/Nb multilayers.[22]

Even if entering into the elaborate detail of crystal growth would take us too far afield, a tentative explanation of this phenomenon can be given. Rossi *et al.*[23] have deposited Pt atoms on a Si(111)7×7 surface. They observed that after saturation of the surface interstitial sites, the condensation of further Pt leads to diffusion within the substrate. This, added to the fact that interfacial pressure is less than 20 kbar, advocates the idea that the system might form an amorphous interlayer to release the enormous interfacial stresses that are found in epitaxial systems.[24]

The originality of our analysis is in the exploitation of the advantages of the two methods employed for studying x-ray-absorption spectra in multilayers: we use concentration profiles (as in Ref. 4), to avoid assuming uniform composition, and we use experimental spectra (as in Ref. 6), to eliminate the questionable use of theoretically generated quantities. Further experiments are now planned to test more sophisticated models of diffusion, where the dynamics of the solid-state reaction can be studied quantitatively.

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