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The ternary compound $Fe_3Ga_{2-x}As_x$: a promising candidate for epitaxial and thermodynamically stable contacts on GaAs

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Abstract

Solid state phase equilibria in the ternary Fe-Ga-As phase diagram have been established at 600°C using X-ray diffraction, scanning electron microscopy and EDX microprobe analysis as experimental techniques. The existence of a ternary phase, expressed by the general chemical formula $Fe_3Ga_{2-x}As_x$ ($0.20 \leq x \leq 1.125$), which crystallizes in hexagonal symmetry and derives structurally from the NiAs-type structure, has been evidenced. The original feature of the diagram is the occurrence of a tie line between this ternary phase and the semiconductor GaAs. It is the first time that such a ternary phase $M_x(Ga,As)_y$ (M = transition metal) has been found to be in thermodynamic equilibrium with GaAs. Based on hexagonal-pseudocubic crystallographic considerations, this phase $Fe_3Ga_{2-x}As_x$ presents real possibilities to be epitaxied on GaAs. Therefore, this compound appears as a unique candidate to obtain a quasi-ideal metal/GaAs contact, both thermodynamically stable and single-crystalline. For this purpose, we have studied the solid state interdiffusions in epitaxial (70 nm Fe)/GaAs(001) heterostructures grown at room temperature and annealed at 450°C in ultra-high vacuum conditions. The epitaxial growth of the $Fe_3Ga_{2-x}As_x$ compound on GaAs(001) has been proved but, for an annealing temperature of 450°C, the binary compound Fe_2As, which is not found in thermodynamic equilibrium with GaAs, coexists as patches at the interface. This indicates that annealing at higher temperature should lead to a quasi-ideal Fe_3GaAs/GaAs(001) contact. © 1997 Elsevier Science S.A.

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1. Introduction

The fabrication of stable and epitaxial contacts to III-V compound semiconductors is a formidable challenge to obtain 'ideal' metallizations for modern components technology (for recent general papers, see [1–3]). Up to now, the growth by molecular beam epitaxy (MBE) of three classes of metallic or semimetallic materials (mainly on GaAs) has been studied: the transition-metal gallides and aluminides like NiGa [4] and NiAl [5], the rare-earth monoarsenides such as YbAs [6] and ErAs [7] and the Fe-based intermetallic compounds $Fe_3(Al,Si)$ [8,9]. However, in silicon technology, to obtain metal/Si contacts (M/Si), another procedure has been extensively studied with success: the formation by solid phase interdiffusions of epitaxied and thermodynamically stable disilicides/silicon interfaces such as $CoSi_2$/Si, $NiSi_2$/Si and $CrSi_2$/Si [10,11]. This approach was tested in the case of GaAs, too. After depositing a metal thin film onto the semiconductor substrate by MBE and after annealings at various temperatures, the metal/GaAs interdiffusion couples

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(M/GaAs) have led to the formation of ternary $M_xGa_yAs_z$ phases, such as Ni_3GaAs, Co_2GaAs and $Pd_{12}Ga_5As_2$ [12]. Unfortunately, up to now, no ternary compound has presented all the required properties for ideal M/GaAs contacts, since these compounds are not at the same time epitaxied and thermodynamically stable with the substrate. In fact, at higher annealing temperatures, the final step of the interaction (M/GaAs) is always a mixture of binaries (e.g. NiGa and $NiAs_2$ in the Ni/GaAs case) and not a single-crystalline and single-phase film.

For the last 10 years, we have largely contributed to the metallurgy of the M/GaAs contacts by simultaneously studying the experimental ternary phase diagrams M-Ga-As and the solid state interdiffusions in M/GaAs structures, for example in the three systems Rh-Ga-As [13], Ni-Ga-As [14–16] and Er-Ga-As [17,18]. As previously proposed by Williams et al. [19], Beyers et al. [20] and Sands [12,21], these different studies confirm that the experimental determination of the M-Ga-As phase diagram is absolutely necessary to describe and understand accurately the solid-phase interdiffusions in the M/GaAs contacts.

In the same way, the Fe/GaAs interdiffusion couples have recently been studied under standard experimental conditions: after annealings for 1 h at 500°C, formation of the binary compounds Fe_3Ga and Fe_2As has been observed [22–24]. To our knowledge, no equivalent study has been published concerning the phase formation in Fe/GaAs contacts, fabricated under ultra-high vacuum (UHV) conditions for the GaAs surface preparation, iron deposition and annealing treatments.

The present paper deals with the solid state interdiffusions of an iron thin film deposited on GaAs. To rationalize the different steps of the interaction, the experimental determination of the bulk equilibrium phase diagram Fe-Ga-As has first been established.

2. The experimental Fe-Ga-As phase diagram and the $Fe_3Ga_{2-x}As_x$ ternary phase

Although the bulk Fe-Ga-As phase diagram has not yet been reported in detail, it is generally considered as closely related to the Ni-Ga-As one [1], owing to the fact that iron and nickel both have similar electronegativity factors and metallic radii. In both diagrams, neither iron nor nickel metal can be found in thermodynamic equilibrium with GaAs since, in the upper part of these diagrams, the occurrence of ternary phases with the general chemical formula $M_3Ga_{2-x}As_x$ (M = Fe, Ni) has been proved. Indeed the $Fe_3Ga_{2-x}As_x$ ternary phase, chemically and structurally similar to the $Ni_3Ga_{2-x}As_x$ one [14], has been observed in Fe-doped GaAs [25] and synthesized as a ternary bulk compound [26–28]. However, as previously mentioned for the Ni-Ga-As diagram, the ternary $Fe_3Ga_{2-x}As_x$ phase would not be found in thermodynamic equilibrium with GaAs since, in the case of nickel, a tie line connects the binary phases NiGa and $NiAs_2$, inhibiting the existence of a $Ni_3Ga_{2-x}As_x$-GaAs tie line. Moreover, it may be considered that, in the case of iron, the binary phases Fe_xGa_y and Fe_xAs_y are more likely to be in thermodynamic equilibrium with GaAs.

Firstly, in order to check this assumption, theoretical calculations have been made. In the absence of true experimental thermodynamic data, the standard formation enthalpies for the Fe-Ga and Fe-As binary systems have been calculated using the 'semi-empirical' Miedema model [29–31]. This has enabled us to elaborate a theoretical Fe-Ga-As ternary diagram using the simplifications proposed by Schmidt-Fetzer [32]. Astonishingly, this theoretical diagram (not presented here) differs significantly from the Ni-Ga-As one, because of the lack of tie lines between the Ga-containing binary compounds Fe_6Ga_5 or Fe_3Ga_4 and the As-containing ones $FeAs_2$ and/or FeAs. This suggests that, in contrast to the ternary $Ni_3Ga_{2-x}As_x$ phase, there is a possibility for the $Fe_3Ga_{2-x}As_x$ one (and in particular Fe_3GaAs which is stoichiometric in As and Ga atoms) to be in thermodynamic equilibrium with GaAs.

This ternary phase $Fe_3Ga_{2-x}As_x$ has been mentioned in the literature as existing over a broad homogeneity range corresponding to $0.21 \leq x \leq 1.125$ [26,27]. This phase has been indexed in hexagonal symmetry and exhibits a partly filled NiAs-type structure. However, since the structure has been found to be fully disordered for $0.85 < x \leq 1.125$, a structural change occurs at $x = 0.85$ down to 0.21, to a more ordered hexagonal structure which needs a doubling of the a unit cell parameter [26,27]. The magnetic structures of Fe_3GaAs and $Fe_3Ga_{1.7}As_{0.3}$ have recently been discussed [33,34].

To establish the experimental Fe-Ga-As ternary diagram, numerous samples with appropriate atomic compositions have been prepared by direct combination of the elements. The starting materials were either powders (iron and amorphous β-As) or ingots (gallium), all with a minimum purity of 99.99%. The samples were intimately mixed and placed in silica tubes which were evacuated to 10^{-3} torr, sealed under vacuum and placed in a resistance furnace for 24 h at 600°C. Numerous grindings, cold pressings and re-annealings at the same temperature for a long time were needed to reach thermodynamic equilibrium. Finally, samples were quenched in ice water. Then, they were analyzed using a powder diffractometer (CPS 120 INEL) equipped with a position-sensitive detector covering 120° in 2θ. Backscattered electron imaging was done with a Jeol JSM-35C scanning

electron microscope as well as standardless electron microprobe analysis.

Fig. 1 shows the experimental ternary phase diagram deduced from about 30 samples. Our results confirm the broad homogeneity range of the ternary $Fe_3Ga_{2-x}As_x$ phase ($0.20 \leq x \leq 1.125$), the hexagonal symmetry and the relationship with the NiAs-type structure [26,27]. Effectively, while for $0.85 < x \leq 1.125$, this phase has been indexed in a disordered hexagonal structure (e.g. Fe_3GaAs: $a = 0.4009(2)$ nm and $c = 0.5046(1)$ nm), for $x < 0.85$, extra reflections occur in the powder pattern which require a doubling of the a unit cell parameter (e.g. $Fe_3Ga_{1.6}As_{0.4}$: $a = 0.8133(2)$ nm and $c = 0.5010(1)$ nm). This ($2a$, c) hexagonal superlattice denotes a more ordered structure. Since the electron scattering factors of Ga and As are quite similar, one can deduce that this superstructure corresponds to an ordering of the extra Fe atom in the vacant metalloid sites of the NiAs-type structure. An X-ray diffraction powder refinement of the hexagonal superstructure, using the Rietveld method, is currently in progress in order to determine exactly the origin of this superlattice.

However, the original feature of the experimental Fe-Ga-As ternary phase diagram is that the ternary $Fe_3Ga_{2-x}As_x$ phase is in thermodynamic equilibrium with GaAs. Indeed, as presented in Fig. 2, the X-ray diffraction patterns for the initial atomic compositions '$Fe_2Ga_1As_1$' and '$Fe_2Ga_{1.4}As_{0.6}$' (labelled samples no. 2 and 5, respectively, in Fig. 1) show a mixture of the binary compound GaAs and of the ternary phases Fe_3GaAs and $Fe_3Ga_{1.6}As_{0.4}$, respectively. These results, which have been confirmed by SEM studies (not presented here), prove that the $Fe_3Ga_{2-x}As_x$ ternary phase is truly in thermodynamic equilibrium with GaAs at 600°C and probably also at higher temperatures. As shown previously for the solid state M/GaAs interdiffusions (M = Ni, Rh), the average atomic composition of the reacted layer remains on the vertical line connecting M to GaAs.

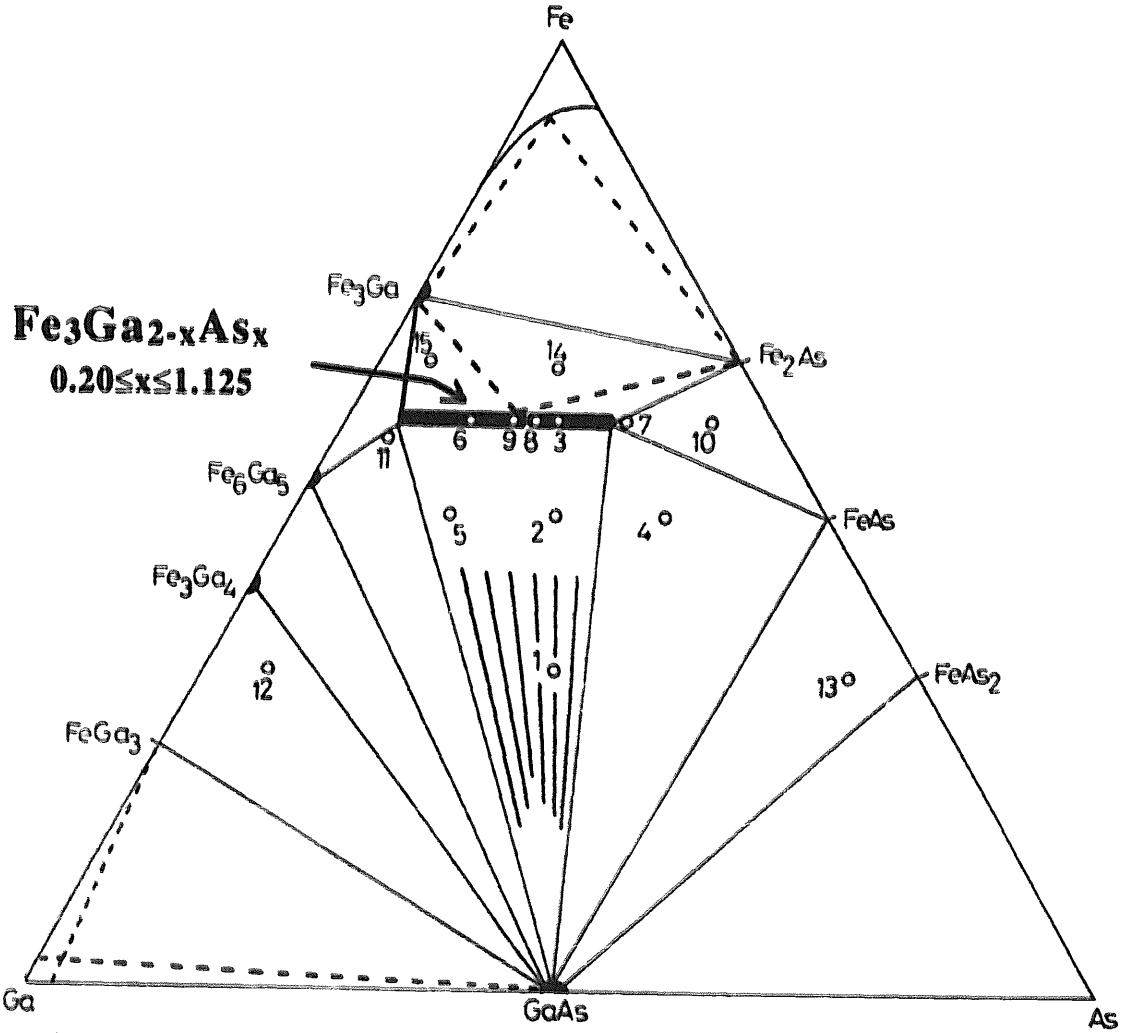

Fig. 1. Isothermal section of the experimental Fe-Ga-As ternary phase diagram at 600°C. Axes are in at.%. Only a part of the studied samples has been numbered. The two-phase region $Fe_3Ga_{2-x}As_x$-GaAs has been emphasized.

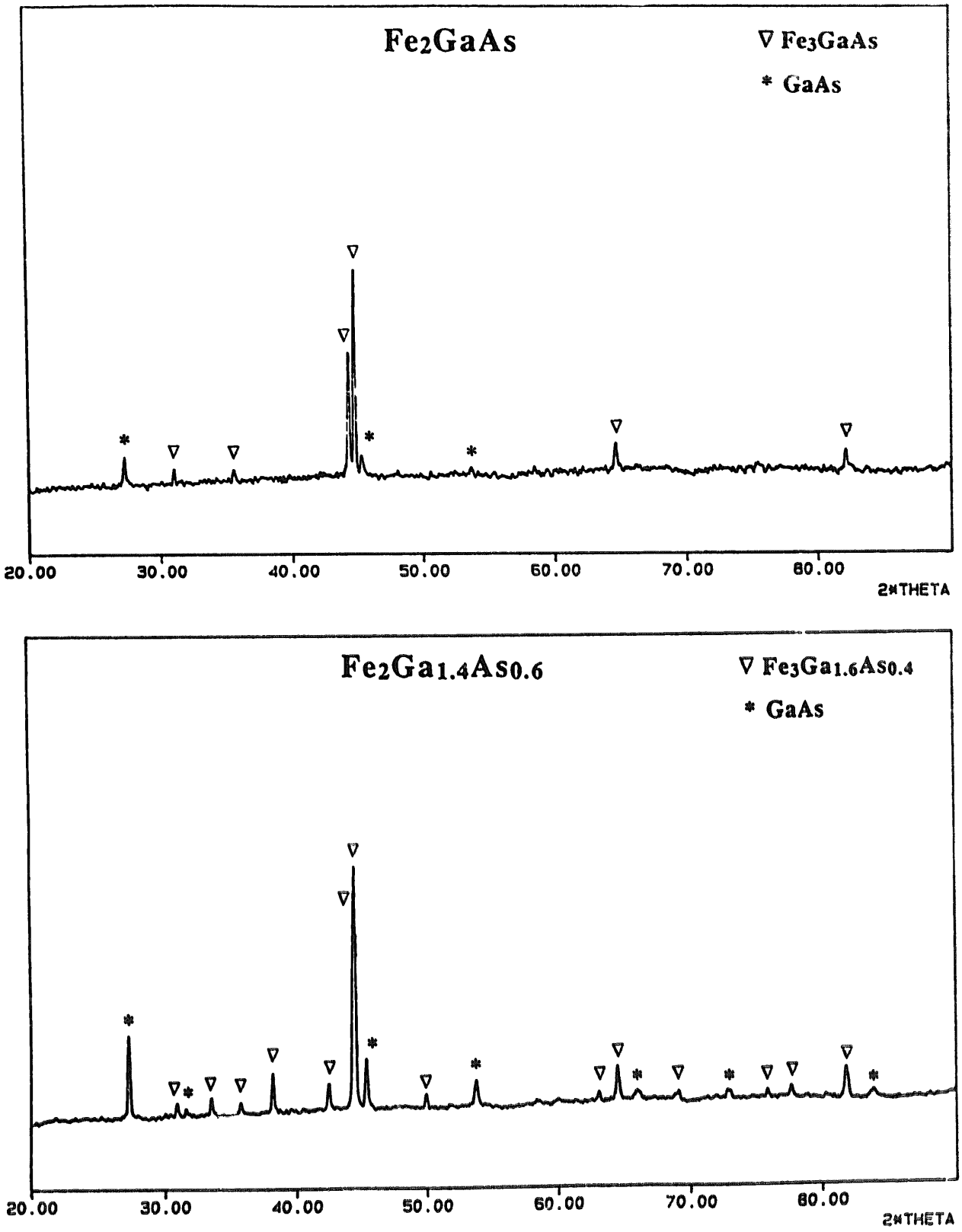

Fig. 2. X-ray diffraction patterns of sample 5 (atomic composition $Fe_2Ga_{1.4}As_{0.6}$) and sample 2 (atomic composition $Fe_2Ga_1As_1$) (λ_{Cu} = 0.154056 nm).

This result involves the occurrence of equiatomic ternary phases e.g. Ni_3GaAs, during the first steps of the interaction. In the same manner, in the case of the Fe/GaAs system, one might expect that, as long as the interfacial region may be considered as a closed system, the solid state interdiffusions in an

Fe/GaAs contact lead to a thermodynamically stable Fe_3GaAs/GaAs heterostructure which must now be the final step of the interaction.

After these thermodynamic considerations, it is interesting to have a look at the possibilities of epitaxial growth of the hexagonal $Fe_3Ga_{2-x}As_x$ phases on GaAs. As has previously been demonstrated, hexagonal phases can be considered to be pseudocubic because of their c/a ratio which is close to $\sqrt{3}/\sqrt{2} = 1.225$ (here for the $Fe_3Ga_{2-x}As_x$ solid solution, we found $1.23 \leq c/a \leq 1.274$). Such pseudocubic phases were previously encountered in the studies of Ni/GaAs [15,16], Ni/AlAs [35,36] and Ni/GaSb [37,38] interdiffusions. The accurate study of the epitaxy of hexagonal pseudocubic binary Ni_2Ga_3 ($c/a = 1.206$) on GaAs(001) and (111), carried out by transmission electron microscopy [39,40], gave a good illustration of this mechanism. For both substrate orientations, the relationships [100](001)hex.// [110](111)cubic {1} were observed between Ni_2Ga_3 and GaAs. In the case of (001) substrates, this implies a small α angle between the (101) plane of the hexagonal phases and the (001) plane of the GaAs substrate (Fig. 3). This α angle is a typical feature of each hexagonal pseudocubic phase because it is directly related to the c/a ratio. The angle α has been found to be equal to $\alpha \sim -0.5°$ and $+2°$ for Ni_2Ga_3 and Ni_3GaAs on GaAs(001), respectively. Because of the cubic symmetry of GaAs, four variants can exist, corresponding to the four $< 111 >$ axes of GaAs, leading to the formation of slightly twinned domains. In the case of the $Fe_3Ga_{2-x}As_x$ phase on GaAs(001), although the corresponding α angles are very small ($0.2° \leq \alpha \leq 1.1°$), a twinned structure of the epitaxial reacted layer is obviously probable, with the appearance of a mosaic structure.

3. Solid state interdiffusions in the Fe/GaAs(001) structures at 450°C under UHV conditions

The samples were prepared in situ in a RIBER 2300 molecular beam epitaxy (MBE) system connected by a UHV modutrack system to an analysis chamber. A 500-nm thick undoped GaAs buffer layer was first grown on n^+-type GaAs(001) substrates using standard MBE. Room temperature Fe depositions, about 70 nm thick, were made in the analysis chamber with a deposition rate equal to 9 Å/min using a high-temperature effusion cell with an alumina crucible. In the present work, the samples were annealed in situ for 15 min at 450°C, a temperature which allows a total reaction of the Fe thin film with the substrate without GaAs decomposition.

The MBE chamber is equipped with a 10 keV reflection high energy electron diffraction for in situ characterization. X-ray diffraction (XRD) patterns were drawn ex situ using a $(\theta-2\theta)$ powder diffraction set-up.

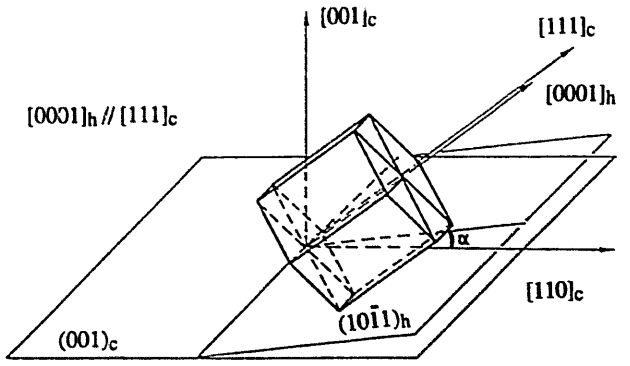

Fig. 3. Orientational relationships: $[10\text{-}10](0001)_{hexagonal}//[110](111)_{cubic}$ between a hexagonal pseudocubic phase and a cubic substrate oriented on a (001) surface.

Fig. 4a shows the RHEED diffraction patterns with the electron beam along the [011] direction of GaAs obtained from the starting (2×4) GaAs surface. After a 70-nm thick deposition of Fe at room temperature, the RHEED pattern (Fig. 4b) with a (1×1) reconstruction indicates that the Fe film grows as a single-crystal α-Fe with a (100) surface and with the in-plane $< 100 >$ axes of the Fe and GaAs aligned. This was expected, based on the fact that the GaAs lattice constant is almost exactly twice that of α-Fe ($\Delta a/a = +1.4\%$) and on the Fe growth on GaAs(110) and (001), previously reported [41,42]. Since the RHEED pattern does not form continuous streaks, we conclude that the Fe surface is not perfectly flat. During the temperature increase, an evolution of the diagram occurs till 300°C, with progressive occurrence of continuous streaks and of lines 1/2 leading to the diagram given in Fig. 4c at 450°C. This last result suggests the formation of an epitaxial single-crystalline reacted layer by solid state interdiffusion, with a surface flatter than the Fe one.

The XRD pattern of the unannealed samples, shown in Fig. 5a, confirms the epitaxial growth of Fe on GaAs. After a UHV annealing for 15 min at 450°C (Fig. 5b), four extra peaks occur, which are added to the GaAs ones. The peaks at $2\theta = 30.7°$ (not shown) and 63.7° correspond to the 101 and 202 reflections of the ternary hexagonal phase $Fe_3Ga_{2-x}As_x$. The XRD measurements indicate a mosaic structure with a full-width at half height of the 101 reflection equal up to 0.8°. The two other peaks at $2\theta = 35.20°$ and 74.40° are identified as the 110 and 220 reflections of the tetragonal compound Fe_2As presumably located at the interface [22–24]. The lack of other reflections of $Fe_3Ga_{2-x}As_x$ and Fe_2As shows that the two compounds are at least strongly textured with the fol-

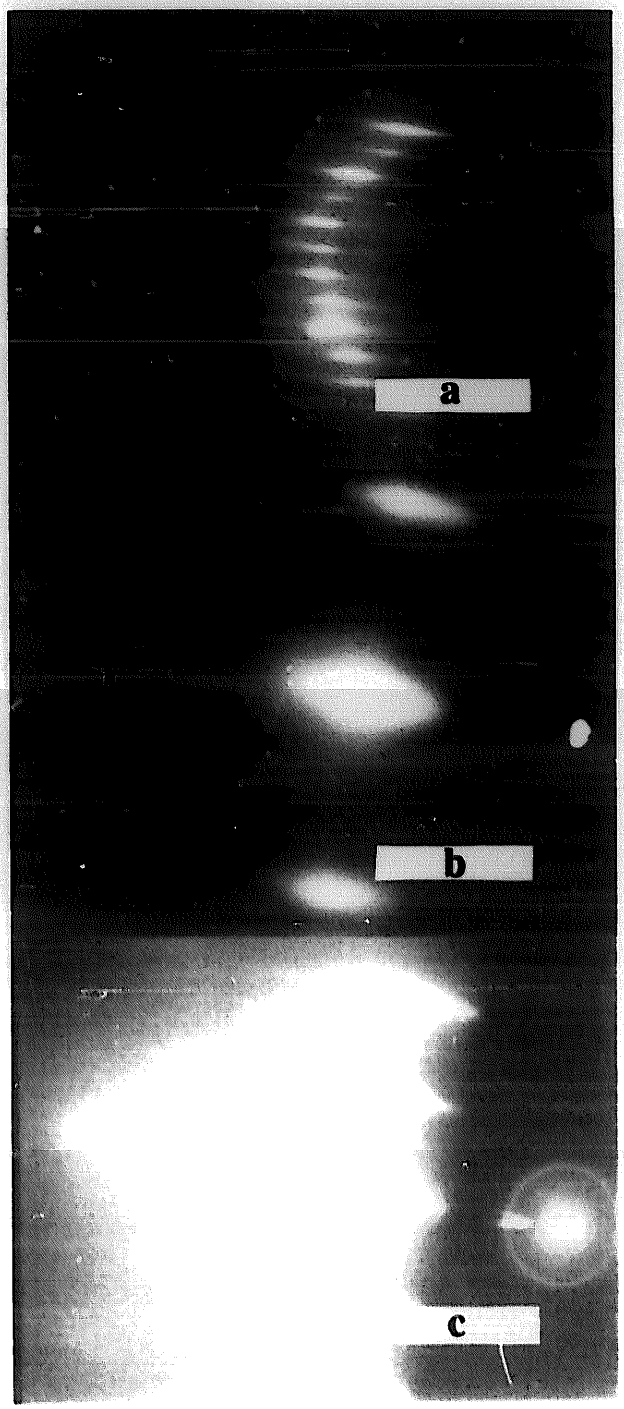

Fig. 4. RHEED patterns along [1-10] azimuth of the GaAs substrate taken (a) on the starting (2 × 4) GaAs(001) surface, (b) after a 70 nm Fe deposition and (c) after a 15-min UHV annealing at 450°C.

lowing relationships with the substrate: (101)$Fe_3Ga_{2-x}As_x$//(001)GaAs and (110)Fe_2As//(001)GaAs. In fact, the RHEED picture (which points out an ordering in the surface plane) and the XRD powder patterns reveal an epitaxial growth of $Fe_3Ga_{2-x}As_x$ on GaAs(001). Moreover, the structural relationship (1) expected for pseudocubic compounds on GaAs, was recently confirmed by transmission electronic microscopy.

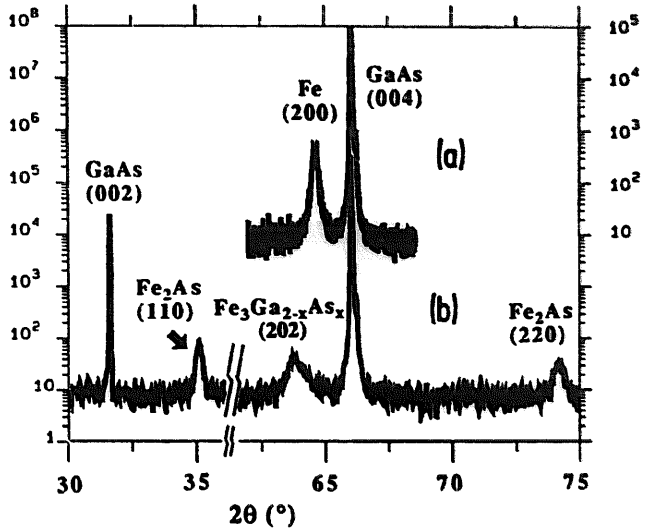

Fig. 5. X-ray diffraction patterns of a (70 nm Fe)/GaAs(001) structure (a) before and (b) after a UHV annealing treatment for 15 min at 450°C.

4. Concluding remarks

By working under UHV conditions, the solid state interdiffusions at 450°C in Fe/GaAs contacts lead to epitaxial ($Fe_3Ga_{1.8}As_{0.2}$ + Fe_2As)/GaAs(001) heterostructures. The mixture of binary compounds Fe_3Ga and Fe_2As previously observed after annealing at 600°C of a (polycrystalline Fe)/GaAs(001) contact [22–24] correspond to a previous step of the interdiffusion process which is probably due to the presence of impurities in the studied structures.

The fact that the stoichiometric composition Fe_3GaAs of the hexagonal ternary phase was not observed in the Fe/GaAs interdiffusions is not really surprising because of the presence of Fe_2As. This implies that the composition of the ternary phase obtained at 450°C is Ga-rich. Our work indicates that the UHV anneal at 450°C is not sufficient to reach the final step of the interaction Fe_3GaAs/GaAs because of the stability of Fe_2As on GaAs at this temperature. However, since Fe_2As is not in equilibrium with GaAs as shown in Fig. 1, a slightly higher annealing temperature should be necessary for the occurrence of Fe_3GaAs, with eventually an inert cap to avoid the As escape (for instance an Si_3N_4 film was previously used for Er/GaAs study [18]).

In conclusion, this set of experimental data shows the epitaxial growth of the $Fe_3Ga_{2-x}As_x$ compound by solid phase epitaxy on GaAs(001) at 450°C but at this temperature Fe_2As is stable on GaAs and the fabrication of quasi-ideal single-crystalline and single-phase Fe_3GaAs/GaAs(001) contacts will require UHV annealing treatments at higher temperatures. Work is now in progress to this end.

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