ISSN 1392–1320 MATERIALS SCIENCE (MEDŽIAGOTYRA). Vol. 9, No. 2. 2003

The Influence of Se Treatment and Annealing on Ferromagnetic-*n*GaAs Schottky Contact and Interface Properties

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Received 21 August 2002; accepted 18 April 2003

Effects of the selenium treatment on the interface structure and electrical characteristics of ferromagnetic-GaAs Schottky contacts were investigated. Selenium treatment significantly suppressed interfacial reactions between the Co and GaAs. However, in this case intensive Se segregation takes place at the Co(Se)-GaAs interface. The annealing results in the further CoAs formation and the increase of the amount of Ga in the Co film. Se treatment significantly suppresses thermally stimulated Co and GaAs interfacial reactions, too. Annealing in the N_2 gas ambient results in the decrease of the effective barrier height both of Co-*n*GaAs and Co(Se)-*n*GaAs Schottky contacts. It can be explained by Co and GaAs interfacial reaction for Co-GaAs contact and by Co and Se reactions for Co(Se)-GaAs Schottky contact.

Keywords: GaAs, ferromagnetic, Schottky contact, Se treatment, XPS, I-V characteristics.

1. INTRODUCTION

One of the most interesting and promising applications of the ferromagnetic thin films is as a source of spin-polarized carriers [1, 2]. In particular, ferromagnetic thin films can be used to inject an imbalance of spin-polarized electrons into the GaAs and other III-V semiconductors that opens an opportunity for making of a new class of devices such as spin transistors [2 – 4] and spin-based light emitting diodes [2, 5].

Therefore, it is very important to control electronic properties of the ferromagnetic-semiconductor contact. However, due to the Fermi level pinning effect, barrier of the Fe-nGaAs, Ni-nGaAs, and Co-nGaAs Schottky contacts do not depend on metal workfunction and it is relatively high [6, 7]. Different interface control techniques must be considered to ensure efficient injection of spin polarized electrons from ferromagnetic to semiconductor [2, 4].

It was found that chalcogenide (sulfur or selenium) treatment results in significant changes of the surface and interface properties of GaAs and other III-V semiconductors (eg. [7 – 10]). There are some studies on the influence of Se [11, 12] and S [13, 22] treatment on structure of ferromagnetic-GaAs interfaces and magnetic properties of Co [11 – 16] and Fe overlayers [17 – 22].

It was reported in [11], that the treatment by Se flux at 400°C temperature significantly suppressed As segregation. However, some Co-Ga intermixing at initial stages of growth occurred. Thus, epitaxial growth was achieved [11]. The Se-treated GaAs~001 surface was the best choice for obtaining homogenous crystalline growth with a better interface; and thus was made a better choice to use as a perfect spin filter in comparison with the Co/S/GaAs and Co/O/GaAs [12]. This way a perfect and well-ordered crystalline growth ensured a better growth for thicker film at the initial stage of deposition for Co/Se/GaAs [12].

S passivation by dipping in a ~$(NH_4)_2S_x$ solution for 1 h at room temperature eliminated the formation of a bcc interface layer when Co was deposited on S/GaAs, instead of GaAs [13]. It was shown that the absence of forced bcc structure in the Co/S/GaAs(100) system was a direct consequence of the effect of S passivation. In addition, an abrupt interface in the passivation-induced epitaxy was observed [13]. S-passivation on GaAs(100) by CH_3CSNH_2 treatment is an effective way of inhibiting the interdiffusion of As and Ga through the Co overlayer [14].

This way, weak interface reaction was achieved and a stable interface was formed at the coverage of 0.8 nm [15]. Ga atoms bonded with S at the surface exchanged with Co atoms and caused the formation of Co-S bonding; no segregated As appeared at the surface of Co overlayer, in contrast with the case of Co/GaAs(100) [15]. It was demonstrated in [16] that a Co epitaxial film grown on S-passivated GaAs(001) surface is smoother than the one grown on bare GaAs(001). It was established that the surfactant nature of sulfur plays very important and essential role in the formation of a smooth surface. The results of synchrotron radiation photoemission spectroscopy confirmed that S passivation greatly reduces the segregation of substrate atoms during film growth on a S-passivated surface [16].

Epitaxial Fe overlayers have been grown on the sulfur-passivated GaAs(100) surfaces by CH_3CSNH_2 treatment in [17]. The chemical surface properties were modified by changing the annealing temperature of the surfaces prior to the growth [17]. It was revealed, that the magnetization of Fe overlayers is characterized by the presence of Ga-S chemical bonds and As excess after the annealing [17]. It was confirmed that S passivation on the GaAs surface can effectively eliminate the magnetization deficiency previously attributed to interdiffusion of As into the Fe overlayer [17]. In the first stage of Fe deposition on S-passivated GaAs(100), Fe clusters were formed near S atoms. Compared to Fe/GaAs(100), the sulfur passivation weakened the reaction between As and Fe [18]. Ferromagnetic resonance (FMR) has been used to investigate the magnetism of Fe overlayer on S-passivated GaAs(100) pretreated by CH_3CSNH_2 [19]. It was shown, that sulfur passivation can prevent As diffusion into Fe overlayer and

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weaken the interaction of As and Fe [19]. As a result, the magnetism of Fe overlayer on GaAs(100) has been enhanced [19]. It was found, that the maximum effective magnetization can be obtained by S treated GaAs surface annealing at 400 °C. It was shown in [20], that magnetization of Fe overlayers is crucially determined by the Ga-S chemical bonds and by the presence of excess As after the anneals. CH_3CSNH_2 passivation of GaAs surfaces combined with the vacuum annealing can effectively eliminate the magnetization deficiency previously attributed to interdiffusion of As into the Fe overlayer [20, 21]. GaAs surface passivation by CH_3CSNH_2 can prevent deposited Mg and Fe from diffusing into or reacting with GaAs substrate. In such a way enhancement of the magnetism of Fe overlayer on GaAs surface was achieved [22].

However, there is a lack of information concerning the effects of selenium treatment on ferromagnetic-GaAs Schottky contacts [23]. Only "conventionally" prepared Co-GaAs, Ni-GaAs, Fe-GaAs Schottky contacts were investigated more thoroughly [24 – 35].

2. EXPERIMENT

In this investigation the Schottky barriers and samples for XPS (X-ray photoelectron spectroscopy) analysis were fabricated on VPE (vapour phase epitaxy) epitaxial *n*GaAs (100) wafers. Au/Ge/Ni ohmic contacts were evaporated on the n^+ substrate prior to Schottky contact formation after degreasing in dimethylformamide and acetone. Sintering of ohmic contact was completed by 15 min heating at 330 °C temperature in the N_2 gas ambient. After degreasing in dimethylformamide and acetone, the substrates were etched in $NH_4OH : H_2O_2 : H_2O = 3 : 1 : 144$ solution for ten seconds and rinsed in running deionized water for a few minutes. The etching procedure was used to obtain stoichiometric surface and to remove native oxides and possible defects, caused by ohmic contact annealing.

The substrate was then cleaved into a few pieces with one piece always used as a reference. The Schottky contact on it was formed without any other treatment. Other pieces were treated by H_2SeO_3 for 5 s in acid aqueous solution at 60 °C and again rinsed in running deionized water. The samples were immediately loaded into the LH A700QE vacuum deposition system. The Schottky contacts were e-beam deposited at 10^{-6} Torr base pressure at a substrate temperature of 100 °C. The diameter of the Schottky contact (diode) was 500 μm in all cases. After the initial measurements of the current-voltage (I-V) characteristics, samples were annealed in a furnace between 200 °C and 500 °C in ultrapure N_2 gas flow.

The measurements of the GaAs Schottky contacts I-V characteristics were carried out using an HP4062B semiconductor parametric test system. Effective barrier height (φ^*_b) and ideality factor (n) of the Schottky contact was calculated from the I-V characteristics using only the thermionic emission equation [35]. We use φ^*_b as a convenient I-V characteristic parameter, though the "real" Schottky barrier height, φ_b, of the Schottky barrier can differ very much from the calculated one while using the thermionic emission formula due to the presence of the other carrier transport mechanisms [8] and different barrier height areas [35]. This effective barrier height can serve as a measure of the minimum barrier height of the inhomogeneous Schottky contact as well [36, 37].

The thickness of the native oxide and selenide layers on the GaAs surface was estimated by a laser ellipsometer, Gaertner L115 (wavelength $\lambda = 632.8$ nm).

XPS measurements of the Se- treated GaAs surfaces were carried out using a KRATOS Analytical XSAM spectrometer operating at constant pass energy. The Al K_α line was used as a X-ray source. The resolution of the analyzer was about 1.0 eV for As 3d, Ga 3d, Se 3d peaks and 1.2 eV - for the rest of peaks.

3. EXPERIMENTAL RESULTS AND DISCUSSIONS

3.1. XPS SPECTRA

Fig. 1 and Fig. 2 show Ga3d and As3d XPS spectra of the samples. Co deposition onto the conventionally treated GaAs surface results in a shift of the As3d peak by ~ – 0.45 eV (Fig. 1). In the case of the selenious acid treated Co-GaAs interface position of the As3d peak is nearly the same. It can be explained by Co-As alloy formation as it was suggested in [14]. In both cases deposition of the 2 nm thick Co layer decreased intensity of the As3d peak. It can be seen, that Se treatment significantly suppressed formation of the Co-As alloy – ratio of the intensities of the As3d peak after and before Co deposition was respectively 0.15 in the case of conventionally treated Co-GaAs interface and by 0.06 in the case of selenium treated.

Position of the Ga3d peak of Co-GaAs structure is very close to the position of the elemental gallium 3d peak. It can be seen, that selenious acid treatment results in significant supression of the Co-Ga intermixing. – 0.9 eV shift of the Ga3d peak is very close to the value reported in [11, 38, 39]. The shift can be explained by alloying of Ga with the Co as it was suggested in [14].

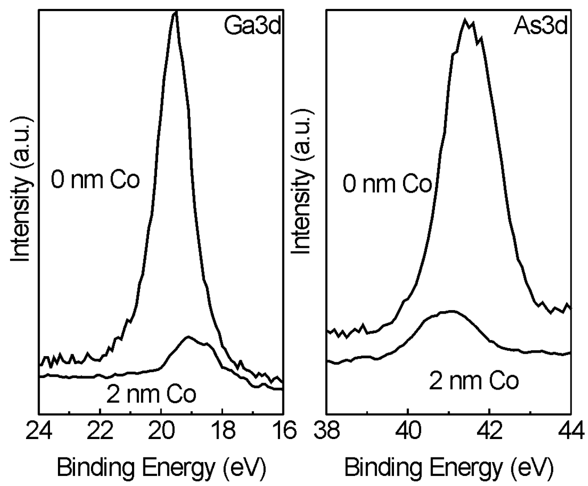

Fig. 1. Reference GaAs surface As3d and Ga3d XPS spectra (before and after deposition of the 2 nm thick Co film)

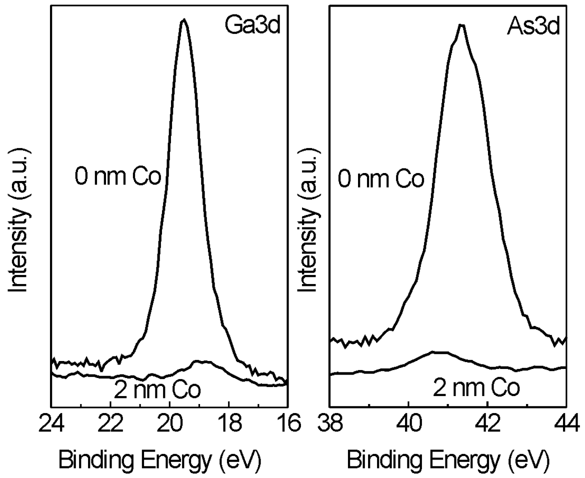

Fig. 2. Se treated GaAs surface As3d and Ga3d XPS spectra (before and after deposition of the 2 nm thick Co film)

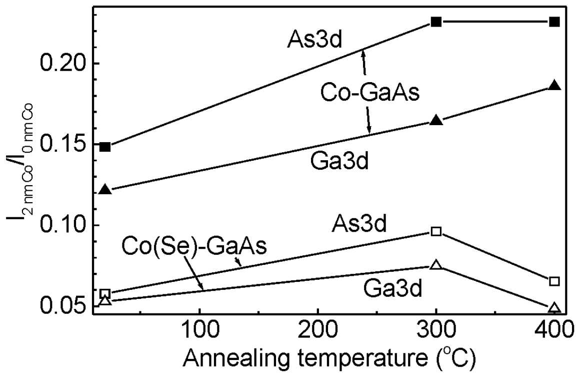

Fig. 3. Ratio of the As3d and Ga3d peak intensity after and before Co deposition as a function of the annealing temperature

It must be mentioned, that As3d and Ga3d peak intensity suppression, as a result of the Se treatment. achieved in our study was weaker in comparison with the [14]. It can be explained by additional 450 °C vacuum annealing of the S treated sample just before the Co deposition performed in [14].

Annealing at 300 and 400 °C results in further Co-As formation, elemental Ga segregation and intermixing for Co-GaAs (Fig. 3). In the case of Co(Se)-GaAs intensities of the As3d and Ga3d peaks increase as well as after annealing at 300 °C temperature. However, the ratio of peak intensity after and before Co deposition is much lower than the ratio for Co-GaAs. After annealing at 400 °C this ratio surprisingly decreases for the selenium treated sample. It should be assumed, that process of the As and Ga sublimation dominates over possible CoAs alloy formation and Ga segregation.

It can be seen in Fig. 4, that no shift of the Se3d peak occurred due to the deposition of 2 nm thick Co film. Surprisingly, the intensity of the peak remained nearly the same. Analysis of the intensities of Co2p (778 eV) and Co3p (60 eV) provides us additional information (Fig. 5). The intensity of Co3p peak of Co(Se)-GaAs is slightly higher than the intensity of Co-GaAs. However, the intensity of Co2p peak of Co(Se)-GaAs is more than

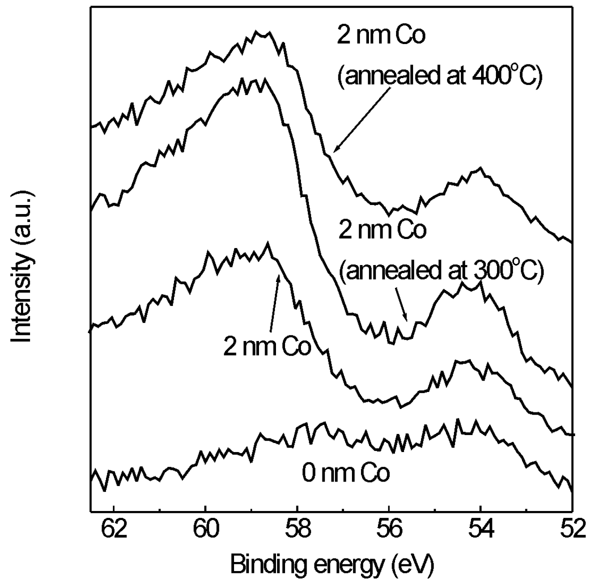

Fig. 4. Se treated GaAs surface Se3d and Co3p XPS spectra (before and after deposition of the 2 nm thick Co film)) as a function of the annealing temperature

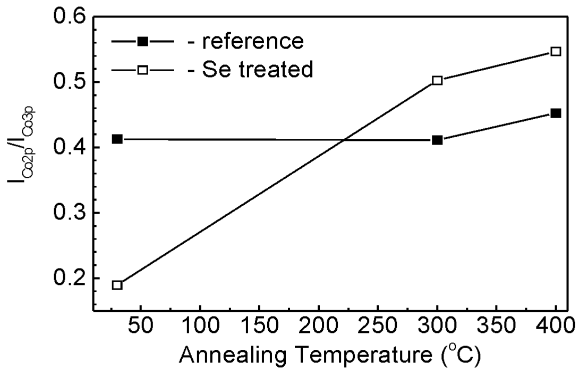

Fig. 5. Ratio of the intensities of Co2p and Co3p peaks as a function of the annealing temperature

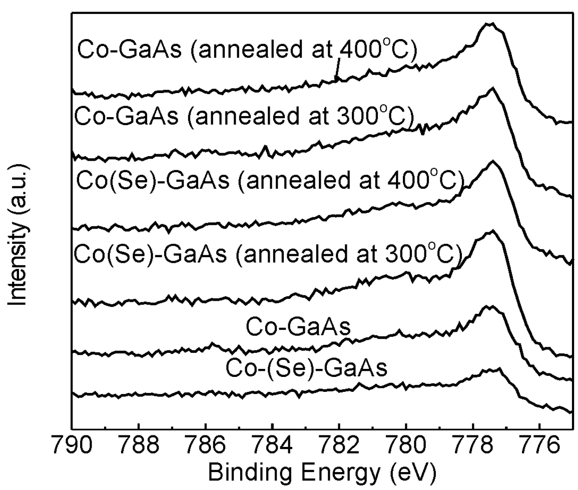

Fig. 6. Co2p XPS spectra of the Co-GaAs and Co(Se)-GaAs interfaces as a function of the annealing temperature

twice lower than the intensity of Co2p peak of Co-GaAs. It seems that in the case of Co(Se)-GaAs some overlayer is located on the Co film. Therefore, it would be assumed, that in the case of Co(Se)-GaAs interface intensive segregation of Se into the Co film takes place. It should be mentioned that Se segregation was reported for Co(Se)-nGaAs interface in [11] as well.

The shift of the Co2p peak both for reference and selenium treated sample after annealing was not observed (Fig. 6). However, annealing substantially increased the intensity of Co2p peak of the Se treated sample. On the other hand, the intensity of the Se3d peak did not decrease. Sharpening of the Se3d peak took place as well. Co2p peak shift caused by Co-Se bond formation reported by other authors is 0.3 eV [40] and even 0.1 eV [41] respectively. Therefore, it would be assumed, that Se reacted with Co, but possible shift of the Co2p peak was too small to observe it with our equipment.

3.2 ELECTRICAL CHARACTERISTICS

It can be seen in Fig. 7, that annealing at 200 °C resulted in a slight increase of the effective barrier height and decrease of the nonideality factor for the reference sample fabricated on the *n*GaAs epilayer doped to $3\cdot10^{15}$ cm^{-3}. While for Se treated Co-*n*GaAs Schottky contacts effective barrier height decreased and nonideality factor increased. The further increase of the annealing temperature resulted in nonmonotonical decrease of the effective barrier height both for reference and selenium treated contacts. However, for nonideality factor different approach was observed. The increase of the annealing temperature resulted in the increase of the nonideality factor of reference sample. It reached 1.5 after annealing at 500 °C temperature. However, the nonideality factor of the selenium treated Co-*n*GaAs Schottky contact decreased to the 1.06 as a result of the annealing at 500 °C temperature.

Annealing at 400 °C resulted in the decrease of the effective barrier height of all Co-GaAs Schottky contacts fabricated on the nGaAs epilayers doped to $2\cdot10^{17}$ cm^{-3} (Fig. 8.). However a considerable increase of the nonideality factor was observed for the reference and treated by 10 % selenious acid solution for 5 s Schottky contact. The nonideality factors of these samples were larger than 2. On the other hand, the increase of the nonideality factor of the Co-GaAs Schottky contact fabricated on the *n*GaAs surface treated by 0.1 % selenious acid solution for 5 s, was smaller. It increased from 1.2 to 1.4. The further increase of the annealing temperature resulted in the formation of the contacts with the ohmic I-V characteristics in the case of selenium treated contacts. However, it must be mentioned, that the effect of Se treatment by 0.1 % solution has poorer reproducibility in comparison with the 10 % solution. In some experiments I-V characteristics of Co(Se)-*n*GaAs Schottky contacts fabricated on the 0.1% H_2SeO_3 aqueous solution treated GaAs surface, had very large nonideality factor (1.5 and more). Similar approach was observed in the case of Co(Se)-GaAs Schottky contacts treated by 1 % solution. From the other hand, it can be mentioned, that for Au(Se)-*n*GaAs Schottky contacts, the lowest nonideality factor was observed for contacts treated by 0.1 % solution [42].

Surprisingly, in the case of Ni-GaAs Schottky contacts fabricated on the *n*GaAs epilayers doped to $2\cdot10^{17}$ cm^{-3},

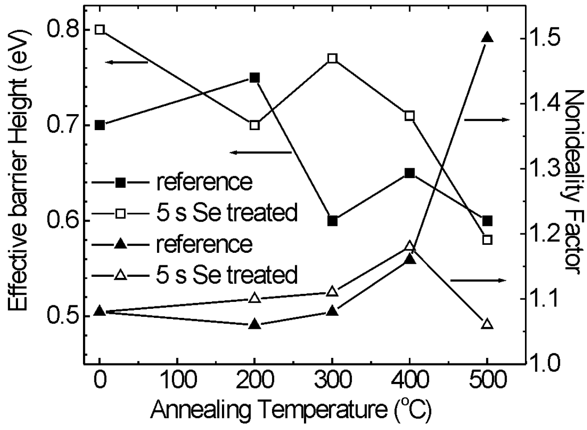

Fig. 7. Effective barrier height and nonideality factor of the Co-GaAs Schottky contacts as a function of the annealing temperature (GaAs epilayer was doped to $3\cdot10^{15}$ cm^{-3}). 0 °C data refer to the characteristics of the samples before the annealing

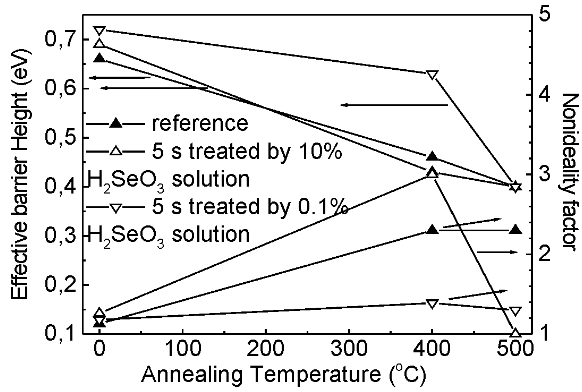

Fig. 8. Effective barrier height and nonideality factor of the Co-GaAs Schottky contacts as a function of the annealing temperature (GaAs epilayer was doped to $2\cdot10^{17}$ cm^{-3}). 0 °C data refer to the characteristics of the samples before the annealing

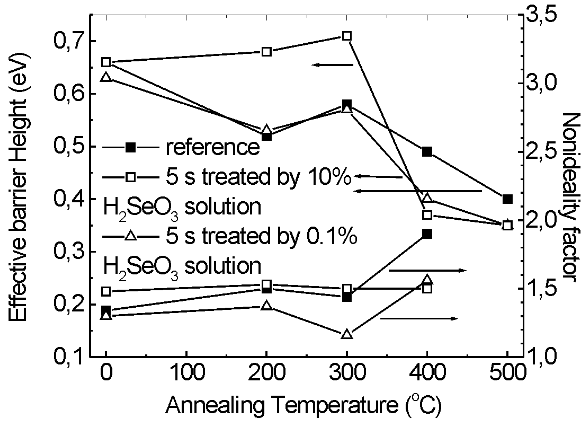

Fig. 9. Effective barrier height and nonideality factor of the Ni-GaAs Schottky contacts as a function of the annealing temperature (GaAs epilayer was doped to $2\cdot10^{17}$ cm^{-3}). 0 °C points refer to the characteristics of the samples before the annealing

the decrease of effective barrier height was already observed after the annealing at 200 °C temperature for the reference samples and the ones treated by 10 % selenious acid solution 5 s (see Fig. 9). On the other hand the increase of the effective barrier height of the Ni-GaAs Schottky contact fabricated on the *n*GaAs surface treated by 0.1 % selenious acid solution for 5 s was observed. Increase of the annealing temperature up to the 400 °C resulted in the decrease of the effective barrier height of the Se treated Ni-GaAs Schottky contacts to the 0.4 eV. However, the nonideality factor of these samples was relatively big – 1.5. The increase of the annealing temperature resulted in further decrease of effective barrier height and even the formation of the contacts with the ohmic I-V characteristics.

3.3 DISCUSSION

The source study on the influence of the annealing on the "conventionally" treated Co-*n*GaAs and Ni-nGaAs Schottky contacts showed rather contraversional results.

Some authors reported higher thermostability of the Co-*n*GaAs Schottky contacts in comparison with the present study. The effective barrier height value has increased by 0.03 eV with the increasing annealing temperature up to 550 °C for Co/*n*-GaAs(Te) Schottky barrier diodes in [24]. Even annealing at 700 °C caused very small decrease of the effective barrier height and some increase of the nonideality factor [24]. While in our study effective barrier height of reference sample already was decreased as a result of the annealing at 300 °C and substantial increase of the nonideality factor began at 400 °C. On the other hand, annealing at 420 °C temperature resulted in the Schottky barrier height increase from 0.69 eV (as-deposited state) up to 0.81 eV in [25]. However, annealing at higher (>460 °C) temperature caused the formation of low barrier contacts [25] as well as in present study. It was explained by Co reaction with GaAs at 380 °C temperature by formation of Co_2GaAs and subsequently the ternary phase decomposition as a result of the higher temperature annealing [25].

For comparison purposes some studies on the annealed Ni-*n*GaAs Schottky contacts can be mentioned. Slightly higher or even lower thermal stability of the Ni-*n*GaAs Schottky contact in comparison with the present study were reported in [26, 27]. Annealing at 350 °C temperature resulted in the Ni-*n*GaAs Schottky barrier heights increase from 0.76 eV (as-deposited state) up to 0.84 eV [26]. The further increase of annealing temperature resulted in the decrease of effective barrier height to the 0.48 eV (after 1 h 550 °C annealing). The increase of effective barrier height was explained by the formation of a metastable hexagonal phase Ni_2GaAs and the decrease – by formation of the NiAs and NiGa [26]. Chemically deposited Ni-*n*GaAs Schottky contacts became ohmic at 400 °C temperature while annealing temperature was increased continuously (annealing ambient was He) [27]. However, in the case of step increase of the annealing temperature, I-V characteristics of the Ni-*n*GaAs Schottky contacts became ohmic already after annealing at 290 °C temperature [27].

However, in [28 – 32], annealing at 300 °C temperature resulted in increase of the effective barrier height of the Ni-*n*GaAs Schottky contact. While subsequent NiAs formation [28 – 30] or higher temperature annealing resulted in only slight decrease of the φ^*_b [30 – 32].

It has been seen that the intimate Ni-GaAs contact is thermally more stable than the MIS [33]. After 450 °C heat treatment, the I-V characteristics of the MIS device contacts become near-ohmic [33]. However, φ^*_b of the intimate Ni-*n*GaAs Schottky contact was substantially larger in comparison with the reference sample even after the annealing at 600 °C temperature [33]. It was explained by NiGa formation [33]. When data was re-evaluated using flat band effective barrier height instead of the effective barrier height calculated using thermoelectronic emission formula, much more consistent results were obtained, indicating that flat band effective barrier height always increased (or at most stays constant) with increasing annealing temperature [34].

In our case CoAs is already present in the interface of the unannealed sample. It is more difficult to identify whether Ga is present in the Co film as a elemental Ga or as a CoGa compound. In the case of the selenium treated sample, intensive Se segregation into the Co takes place. It seems that the annealing results in the further CoAs formation and the increase of the amount of Ga in the Co film. Se treatment significantly suppressed thermally stimulated processes mentioned above, as well. On the other hand, it seems that annealing results in formation of the Co selenide.

Therefore, the decrease of the effective barrier height of the Co-*n*GaAs Schottky contacts, caused by annealing, can be explained by Co interfacial reactions with the GaAs. It seems that it is caused rather by Co alloying with the Ga, because *n*GaAs surface enrichment by Ga using vacuum annealing just before evaporation of Co results in the decrease of the effective barrier height of the Co-nGaAs Schottky contact [43]. Relatively poor thermostability of Co-*n*GaAs Schottky contact, reported in this study can be explained by the presence of the binary Co and GaAs compounds already after the deposition of the Schottky contact metallization. As well as by peculiarities of the initial surface cleaning. There were reported in [44], that effective barrier height of the Ni-*n*GaAs Schottky contacts formed on the GaAs surface treated by HCl and NH_4OH can differ by 0.2 eV. Low thermal stability of the chemically deposited Ni-nGaAs Schottky contact [27] as well as influence of the native oxide layer [33] can be mentioned, too.

On the other hand, it seems that in the case of Co(Se)-GaAs Schottky contact the decrease of the effective barrier height caused by annealing is the result of the interfacial reactions between the Co and Se. Possible effects of the suppression of interfacial reactions between the Co and GaAs can be mentioned as well considering decreased thermostability of the Schottky contact with the native oxide interlayer [33]. In the case of the Ni(Se)-*n*GaAs Schottky contact similar approach can be supposed.

CONCLUSIONS

In conclusion, selenium treatment significantly suppressed interfacial reactions between the Co and GaAs. However, in this case intensive Se segregation takes place at the Co(Se)-GaAs interface. The annealing results in the further CoAs formation and the increase of the amount of Ga in the Co film. Se treatment significantly suppresses thermally stimulated Co and GaAs interfacial reactions as well.

Annealing in the N_2 gas ambient results in the decrease of the effective barrier height both of Co-*n*GaAs and Co(Se)-*n*GaAs Schottky contacts. It can be explained by Co and GaAs interfacial reaction for Co-GaAs contact and by Co and Se reactions for Co(Se)-GaAs Schottky contact.

Acknowledgments

This work is supported by the Lithuanian Science and Studies Foundation.

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DOI: 10.5755/j02.ms.26696