Microstructure and Properties of Aluminum Contacts Formed on GaAs(100) by Low Pressure Chemical Vapor Deposition with Dimethylethylamine Alane Source

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We report on a low pressure chemical vapor deposition of metallic thin aluminum films on GaAs (001) with a dimethylethylamine alane (DMEAA) source and H_2 as a carrier gas. The deposition temperatures varied in the range 130÷360°C. Integrated volumes for Al (111), (100), (110)R and (110) grains were estimated by the x-ray diffraction technique and the growth temperature values preferred for every type of grains were observed. The experimentally observed dominance of Al(110)R over Al(110), irrespective of the substrate miscut direction, supports the GaAs(100) inner anisotropy effect on the Al grain orientation.

Electrical resistivity was 5 μΩ·cm for best Al films. The Schottky barrier heights were near an 0.7 eV level and the ideality factor n= 1.1. Nonalloyed ohmic contacts were fabricated on an n-type GaAs epitaxial layer with an additional set of Si δ-layers near the Al/GaAs interface. Specific contact resistance ρ_c = 7 μΩ см^2 was measured. Best contacts were obtained at a deposition temperature lower than 250°C.

Key words: Aluminum films on GaAs, Schottky barrier, nonalloyed ohmic contact

INTRODUCTION

Chemical vapor deposition of metallic aluminum layers using a dimethylethylamine alane (DMEAA) precursor as a metalorganic source has been actively investigated in the last few years.[1-5] High purity Al films with electrical resistivity close to the bulk value,[3] and selective growth of Al into μm-size holes of SiO_2 mask[4] have been successfully demonstrated as a method of multilevel metallization in modern integrated circuit technology.

Other application of such Al films can be connected with fabrication of barrier or ohmic contacts to semiconductor structures. By deposition a metal is performed immediately after the epitaxial growth of semiconductor layer without interrupting the growth process and growth breaking it is possible to avoid external contamination and oxidation of the semiconductor-metal interface. This technique appears to be very attractive for making good nonalloyed ohmic aluminum contacts to n-type and p-type GaAs and InGaAs. It was successful with molecular beam epitaxy (MBE) growth apparatus.[6-8] The same approach based on another growth technique - metalorganic chemical vapor deposition (MOCVD) with trimethylamine alane as Al precursor - has been used for preparation of Al/n-GaAs Schottky barriers;[9] and their successful application as low barrier microwave detectors was reported.[10]

Furthermore, the origin and microstructure of Al epitaxial film on GaAs(100) surface remains the subject of interest.[1,2,3,9-13]

Here we report a low pressure MOCVD of aluminum films on GaAs (001) with a dimethylethylamine alane (DMEAA) source and H_2 as a carrier gas. An atomic force microscopy (AFM) analysis of morphology together with the microstructure evaluation based on X-ray diffraction have been performed. The aluminum film resistivity and the intrinsic properties of: Al/n-type GaAs contacts as Schottky barrier or a nonalloyed ohmic contact have been investigated. Note that the growth conditions of a carrier gas MOCVD of Al layers as well as the structural and electronic properties of thus-obtained metal-semiconductor barrier and ohmic contacts Al/n-GaAs have not been studied sufficiently so far.

EXPERIMENTAL DETAILS

Al films were deposited with a DMEAA precursor in a MOCVD reactor at a 50 mbar reactor pressure. High purity DMEAA was kindly supplied for experiments by the *Epichem company* (UK). The bubbler with DMEAA was cooled down to 15°C. Hydrogen was used as a carrier gas. An H_2 flow rate was 4000 ml·min^{-1} for the reactor and 200 ml·min^{-1} for bubbler. Flow rate for DMEAA was 12 μmol·min^{-1} The overall procedure included growth of a 150 nm thick GaAs layer at T = 650°C, immediately followed by Al deposition at temperatures T_g in the 130÷360 °C range in the same reactor. Epitaxial GaAs layers were grown on GaAs substrates 3° tilted from (100) toward [011]. Trimethylgallium and arsine precursors were used at a 50 mbar reactor pressure and hydrogen served as carrier gas.

Surface roughness, film thickness and an average linear grain size were evaluated with a Solver-P4 atomic-force microscope (NT-MDT, Russia). We use the common definition of roughness: $R = \Sigma |h_i - h_{mid}|/N$, where N is the total number of pixels in the AFM image, h_i the AFM reading for the *i*th pixel, and h_{mid} the averaged h_i. The average (nominal) film thickness was measured by means of AFM using a diluted KOH etch to remove the film from the selected areas of the substrate, similarly to that described in Ref. 1. The average linear grain size was estimated from AFM cross-sectional scans as l = L/k, where L is the length of a scan line and k is the number of distinct maxima in the scan. For comparison we obtained a few images with a scanning electron microscope JEM 2000 EX-II. The texture and microstructure of Al films were characterized by X-ray diffraction. The film composition was analyzed by Auger spectroscopy. Al resistivity measurements were taken on photolithographically formed Al microbridges formed on semi-

insulated substrates. The electrical properties of Al/GaAs contacts were measured with circular mesas of diameters varying from 7 to 500 μm, formed on heavily doped substrates.

RESULTS AND DISCUSSION

Film composition, surface and microstructure

Auger depth profiling showed no volume contaminations in Al layers grown at any temperature in the range of 130÷360°C. Usual traces of carbon and oxygen were found at the beginning of the profiling process.

Three AFM images in Fig. 1 demonstrate the evolution of Al grains at growth temperature T_g=240°C with an increasing deposition time: (a) - 15s, (b) - 120s, (c) - 240s. Average film thickness was found to be 20 nm for (b) and 30 nm for (c). This method proved no good for measurements of the thinnest film (a) because of the low contrast of etched areas boundary. It is clearly seen how a grain size increases with time. The grains are closely packed even in the thin film, so a coalescence of the grains takes place in the process. Coalescence is known as a common feature of Al growth.[1-6,11,12]

Fig. 2 shows the AFM measured surface roughness (triangles) and the average linear grain size (oval) *versus* deposition time for T_g=240°C - open signs and for T_g=160°C - closed ones. The nominal thickness shown on the top axis is estimated on the assumption of a constant growth rate value for all films. Both dependencies are close to the linear one on a double *log* scale and the tilt of the line gives the power *m* of the $y = x^m$ function. The obtained values of *m* for the grain size and surface roughness were 0.55 and 0.82 respectively. The dependence of roughness on time differs from $m = 0.5$ anticipated for a deposition process with a random particle flow without surface diffusion, and from m=0 for an ideal layer-by-layer growth mode, because a set of grains was formed on a real surface. On the other hand, it differs from the linear dependence (m=1) reported in Ref. 1 for Tg=400 °C. Zero incubation times measured for both dependencies in Fig. 2 are different from those obtained in Ref. 3 for Si and SiO_2 surfaces but in agreement with the one for GaAs surface, probably due to natural Al-As bonding in the first interlayer.

The roughness and the growth rate as functions of the growth temperature are plotted in Fig. 3 for a nominal film thickness of 200 nm. The growth rate remains near 2.7 nm/min with some deviations over the entire range in contrast to 160 °C maximum obtained with the growth mode without a flow of precursor[3] or with a low carrier gas flow[5], but in agreement with the flux limited growth mode for T_g>160 °C in Ref. 1. A nonlinear dependence for roughness is clearly seen in Fig. 3 but the function is nearly constant in the temperature range from 160°C to 240°C. This is reason why the 160°C points in Fig. 2 are located near to 240°C line.

An Al film microstructure is known as complicated due to a set of preferred grain orientations, and AFM allows us to analyze an individual large grain. The largest grains were observed at T_g= 360°C. Fig. 4 shows AFM images of Al triangle-, rectangle- and near square base islands with visible facets for a nominal Al coverage of 200 nm. An Al(111) threefold symmetry pyramid is uncommon for this growth temperature. In Fig. 4a we show such a pyramid specially selected for investigations. To explore the orientation of the facets, we measured the angle between each facet and the GaAs surface by AFM cross-section scans as was done in Ref. 1.

The angles obtained for the threefold symmetry pyramid shown in Fig. 4a were 33°±3°. For a crystalline Al island with a fcc crystal structure, the angle expected between the (111) and the (110) planes is 35.3°. The base of a threefold symmetry pyramid should be an (111) plane, so the facets in Fig. 4a are the (110)-type planes in agreement with those observed in Ref. 1.

For the twofold symmetry pyramid shown in Fig. 4b the obtained angles were 32°±2° for the wide facets and 45°±1° for the narrow but smoother pair of the opposite facets. This grain has the Al (110) base, the wide facets are (111) and the narrow ones are (100) in conformity with those for MBE grown Al.[1,12] A somewhat lower experimental value for the (111)-facets slope may be attributed to a visible vicinal surface of the facets, formed by an echelon of upward-going steps. The pyramid in Fig. 4c is nearly square based but it has two types of facets. One slopes at about 35°

and the other slopes at 45°. Therefore, the pyramid shown in Fig. 4c is an Al (110) grain, just as in Fig. 4b.

Estimation of integrated volumes for all Al grains with one orientation from AFM data is problematic due to the absence of flat facets on most grains and because more than one grain may lie in a film depth from bottom to surface. The x-ray diffraction technique is known to be useful for this purpose.[2,3]

X-ray diffraction (ω-2Θ), ω and ϕ-scans were used in an Al film analysis. The (ω-2Θ)-scans contained Al (111), Al (200) and low resolution Al (220) + GaAs (400) reflections. The Al (111) and Al (200) peak widths obtained from the ω-scans determine the angular spread for the texture axes Al (111) and Al (100). These peak positions correspond to the averaged angle positions for the texture axes. The ϕ-scans through the Al (311) reflections for Al (110) grains were performed with Al [110] as the ϕ-axis. Representative scan is shown in Fig. 5. The scan consists of two sets of four peaks: Al(311), (3,-1,1), (3,1,-1) and (3,-1,-1). The peaks in each set are nearly equal, inequality of the peaks in one set is connected with misalignment of the Al[110] direction and the ϕ-axes. These two sets correspond to two kinds of Al (110) grain turned by ϕ+90° about one another. Two GaAs in-plane directions are shown in the same scale of ϕ angle. The peaks Al(311) nearest to GaAs[011] are shifted by ±55° in the major set and by ±35° in the minor one. The calculated values for a cubic crystal are ±54.85° and ±35.15°.The major set is for the (110)[001] Al // (100)[011] GaAs epitaxial relation and the minor one is for the (110)[001] Al // (100)[0,1,-1] GaAs relation. The first one is known as the Al (110) R orientation.[11-13]

In this way we obtained the peak intensity for 4 Al-grain types: (111) for Al (111), (200) for Al (100), (311) R for Al (110) R and (311) for Al (110), instead of the conventionally examined two types.[2,3] The size of an x-ray beam spot on sample was about 2 mm^2, therefore, the relative intensity of the peak may be used as an unscaled estimate for a relative volume of the set of grains. The obtained ratios I(hkl)/[I(111) + I(200) + I(311)R + I(311)] *versus* deposition temperature are plotted in Fig. 6. There is a intervals of temperature preferable for every type of grains, as is clearly seen in the figure. For T_g< 140°C the Al (111) texture dominates. The ω-angle position for the Al (111) peak was not equal to Θ (111) but was shifted by 3°. Hence, the Al (111)-plane was parallel to the GaAs (100) step of the vicinal surface rather than to the average substrate surface. We, therefore, come to a conclusion that we had a clean GaAs surface even at low growth temperatures. With an increase in the deposition temperature the relative volume of Al (100) grains reaches a maximum near 140°C. Then the Al (110) has a maximum at T_g= 200°C, and then the epitaxial Al (110) R dominates up to the highest temperatures. Fig. 7 provides unambiguous evidence for the epitaxial Al structure grown at 240°C. It is a typical cross sectional SEM image along the film surface near the cleaved edge of the sample. Well-shaped pyramids with parallel facets are clearly seen in the figure in contrast to Al films without epitaxy[5]. These pyramids make the film surface rough.

The quantitative parameters of Al growth were found to be sensitive to the growth conditions such as gas flow, position of a substrate in the reactor, but the qualitative features remain unaffected. Our earlier results[9] on Al growth with the same reactor and a different precursor (TMAA - trimethylamine alane) showed the same sequence of Al grain orientations from Al(111) to Al(110)R when the growth temperature was increased. The Al(111) texture corresponds to the maximum surface density of Al atoms on the close-packed (111) plane. It is generally observed when the epitaxy is difficult at low deposition temperatures and a reduced surface mobility[2,3] or with an amorphous substrate. The Al(100) // GaAs(100) epitaxy relation leads to a good match of the crystal lattice of GaAs (100) surface and the 45°-twisted Al (100) one. This relation was realized in the MBE growth mode with a specially prepared surface.[11-13] At the same time, the (110) oriented Al was frequently observed as additional or major population.[1,2,11-13] Different reasons were suggested to explain why the (100) epitaxy is not ideal in this case. First, it is unsaturated bonds of Al atoms,[11] second a stepped surface of tilted GaAs(100),[12] and the third mechanism is compressive strain of the film.[13]

The next complicated question is why Al(110)R prevails at high deposition temperatures. There are two different Al(110) orientations on the GaAs(100) surface and the only difference is 90° rotation. The key factors may by the inner anisotropy of the GaAs(100) or the morphology anisotropy of GaAs stepped surface.[12] If the elongated hat-form Al(110) island (see Fig. 4b) along the step edge is energetically favorable, the result would depend on the direction of GaAs(100) tilt. When the direction is [011] the step edge is [0,-1,1] and the long [1,-1,0] edge of Al grain is parallel to [0,-1,1]GaAs. The [001]Al is parallel to [011]GaAs and this is Al(110)R epitaxy. If the hypothesis is true the [0,-1,1] tilt direction would lead to the other epitaxy - Al(110). We made this experiment with the two types of substrate.

Table 1 shows results of three experiments: #244, #245 and #246 with the two substrates in each and a TMAA precursor. Nominal film thickness was about 100 nm. The [011] and [0,-1,1] directions on the GaAs plate were specially tested by the of etched pits form following the methodology described in Ref. 14. The direction and the value of GaAs(100) tilt were measured by x-ray diffraction. One type of substrate was 3°tilted to [011] and the other 3° to [0,-1,1]. The ratio of I(311)R to I(311) was measured with the previously described ϕ-scan, see Fig. 5. In addition, the full width on half maximum in the ω-scan of (220)Al reflection gives the angular spread of the common [110]Al axis. It is clearly seen that the Al(110)R prevails in all cases. This gives support to the inner anisotropy of the GaAs atomic structure as a reason for Al(110)R dominance.

The Al (110) R phase is known as a result of strong interface bonding of Al atoms with the reconstructed GaAs (100) or AlAs (100) surfaces.[11] We relate the domination of Al (110) R in our films at $T_g > 200°$ to a clean substrate surface and absence of an intermediate layer, excluding may be AlAs. A sharp increase in roughness near Tg= 250°C (see Fig. 3) can be explained by this single mode domination with irregular spaced Al(110)R grains on the surface but such a conclusion needs further examination. On the other hand, (100)Al growth was found to lead to a smooth surface of thick film in the MBE growth mode.[12,13] This smoothing effect has not been achieved in our reactor so far. Mirror-like films were produced only on early stages of growth with a nominal thickness of below 50 nm for $T_g \leq 240°C$. When thickness exceeded 100 nm, a grain size compared with the visible light wavelength and the film became a white haze, which was observed earlier.[1] It should be noted that the facets of Al grains and the set of Al epitaxial orientations obtained in our growth kit are similar to those reported for MBE.

Electrical resistivity and contact properties

Fig. 8 shows the electrical resistivity of 200 nm thick Al films as a function of growth temperature. The resistivity slightly increases with temperature T_g. The lowest value of the measured electrical resistivity was ~ 5 $\mu\Omega$·cm. It exceeds 3 $\mu\Omega$·cm for bulk Al mainly due to the effect of a surface roughness on the measured film thickness value and, probably, due to defect incorporations.[3] The temperature coefficient of resistance $\alpha = 4.5 \cdot 10^{-3}$ K^{-1} was measured in a temperature range from 80K to 300K for an Al film deposited at 200°C. It exceeds the value $\alpha_{Al} = 3 \cdot 10^{-3}$ K^{-1} for bulk Al 1.5 times.

Characterization of Al/n-GaAs contacts was based on current-voltage (I-V) measurements of mesa-diodes at room temperature as described in Ref. 9. Usually, the contacts demonstrated a near ideal Schottky diode behavior. The dependence of the barrier height ϕ_b^{I-V} and ideality factor *n* of Schottky diodes on deposition temperature of Al film are shown in Fig.8. Up to Tg= 250°C the barrier height is near a 0.7 eV level. It is slightly lower than the value of 0.77 eV for evaporated Al.[15] The ideality factor has a good value of *n*= 1.1 in the same temperature range. When Tg exceeds 250°C, the barrier height increases and the ideality factor changes for the worse, too. Such a behavior can be explained by formation of an intermediate AlAs layer at high temperatures. Wide-band gap AlAs material is equivalent to a thin isolating layer between a metal and a semiconductor, which increases the effective barrier height[16] and the ideality factor.[17]

Nonalloyed ohmic contacts were fabricated on an n-type GaAs epitaxial layer with Si-doping at a $4 \cdot 10^{18}$ cm^{-3} level and thickness of 0.15 μm. Because this level is not sufficient for ohmic

contact formation, an additional set of three Si δ-layers equally spaced at 2 nm from the Al/GaAs interface and from each other, was introduced, as was done in Ref. 9. The procedure was finished by deposition of an 0.2 μm thick Al film at a 160°C temperature. Circular contacts of various diameters were photolithographically formed and an 0.5 μm deep mesa-structure was subsequently etched. The back-side contact was alloyed Au-Ge. The results of contact evaluation are shown in Fig. 9. The inset in Fig. 9 confirms the linear current-voltage dependencies for the ohmic contacts with diameters of 50 μm and 20 μm. Specific contact resistance ρ_c was determined from the dependence of resistance on the inverse contact area, as described in Ref. 18:

$$R - R_0 = (\rho d + \rho_c)/\pi r^2 \qquad (d<<r),$$

where R is the measured resistance; R_0 the back-side contact resistance; ρ, d the resistivity and thickness of epitaxial underlayer; r the contact radius. The R_0 value was measured at the largest contact, $\rho \cdot d \approx 0.2$ μΩ см^2, taking into account the underlayer doping level and thickness. Fig. 9 shows an experimental dependence, yielding $\rho_c = 7$ μΩ см^2, which is comparable with the data on the nonalloyed ohmic contact resistance obtained elsewhere.[8]

CONCLUSIONS

The process of a low pressure MOCVD of Al from DMEAA on GaAs (100) with hydrogen as a carrier gas and deposition temperatures ranging from 130 to 360°C was studied. The set of Al epitaxial orientations and the facets of Al grains are found to be similar to those reported earlier. The growth temperature intervals preferable for Al (111), (100), (110) and (110)R types of grain was observed with x-ray diffraction measurements. The experimentally observed dominance of Al(110)R over Al(110), irrespective of the substrate miscut direction, supports the GaAs(100) inner anisotropy effect on the Al grain orientation.

Fabrication of nonalloyed ohmic and Schottky barrier contacts Al/n-GaAs has been demonstrated in a non-stop MOCVD process with a carrier gas. Best contacts were obtained at a deposition temperature of Al films lower than 250°C. Schottky contacts have an 0.7 eV barrier height and the ideality factor n= 1.1. Nonalloyed ohmic contacts were fabricated on an n-type GaAs epitaxial layer with an additional set of Si δ-layers near the Al/GaAs interface. The contacts have specific contact resistance in the *mid*-μΩ см^2 range.

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Figure Captions

Fig. 1. AFM images of Al films deposited at T_g= 240°C over deposition times varying as (a) 15 s; (b) 120 s; (c) 240 s.

Fig. 2. Average grain size (ovals) and surface roughness (triangles) dependence on the deposition time for Al films deposited at T_g= 240°C (open signs) and T_g= 160°C (closed signs).

Fig. 3. Surface roughness and growth rate dependence on the growth temperature for Al films of about 200 nm thickness.

Fig. 4. Individual Al grains with a different habit on the surface of a T_g=360°C film. (a) Threefold symmetry pyramid; (b) Twofold symmetry grain; (c) Near square based pyramid.

Fig. 5. Representative x-ray ϕ-scan through the Al (311)), (3,-1,1), (3,1,-1) and (3,-1,-1) reflections with Al [110] as the ϕ-axis. The scan consists of two sets of four nearly equal peaks. These two sets correspond to two kinds of Al (110) grain turned by ϕ+90° about one another.

Fig. 6. X-Ray diffraction relative intensity dependencies on deposition temperature for different grain orientations: (a) I (111) is for Al (111) grains; (b) I (200) for Al (100) grain; (c) I (311) for Al (110) grains; (d) I (311) R for Al (110) R grains.

Fig. 7. SEM image of an Al film along the surface near the cleaved edge of a sample deposited at T_g= 240°C with a nominal film thickness of about 200 nm.

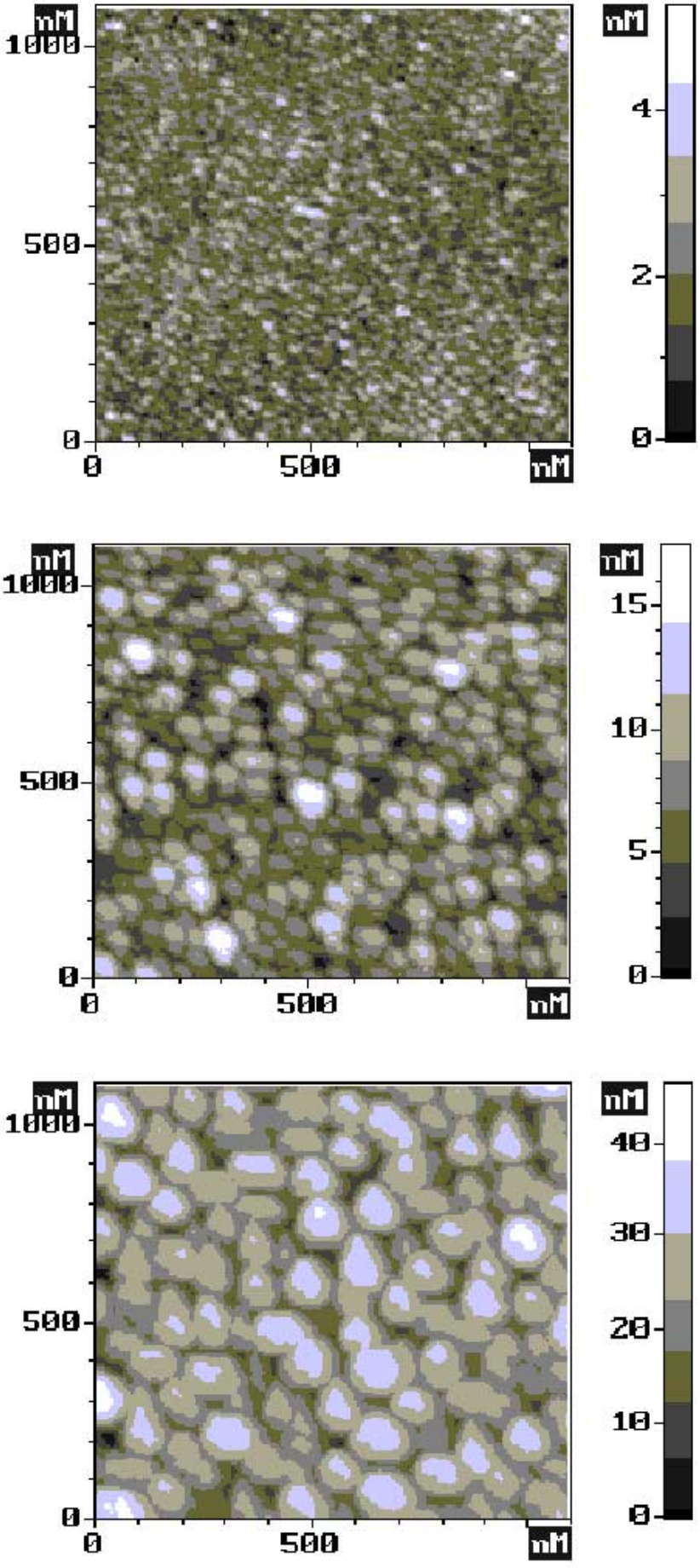
Fig.8. Resistivity of 200 nm thick Al films vs growth temperature - (a) and parameters of the Schottky diodes Al/n-GaAs : ideality factor - (b) and barrier height - (c).

Fig.9. Contact resistance of ohmic contacts as a function of the inverse contact area. The specific contact resistance ρ_c= $7x10^{-6}$ Ω cm^2 is obtained. Insert: current-voltage characteristics of ohmic contacts of 50 and 20 μm diameters.

Table 1

X-ray diffraction measurements of Al film on GaAs(100) substrates 3° tilted toward two different directions: the ratio of (311) intensities of two types of grain Al(110)R and Al(110), and the angular spread of the (110) axis.

Experiment number	Direction of GaAs(100) tilt	I(311)R/I(311)	$FWHM_{\omega}$ (220)Al, deg
#244	[0,-1,1] [011]	45 13	0.5 0.7
#245	[0,-1,1] [011]	6 5	1.0 0.6
#246	[0,-1,1] [011]	30 17	0.7 0.7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500
0
0
500
nM
nM
4
2
0
nM
1000
500
0
0
500
nM
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nM
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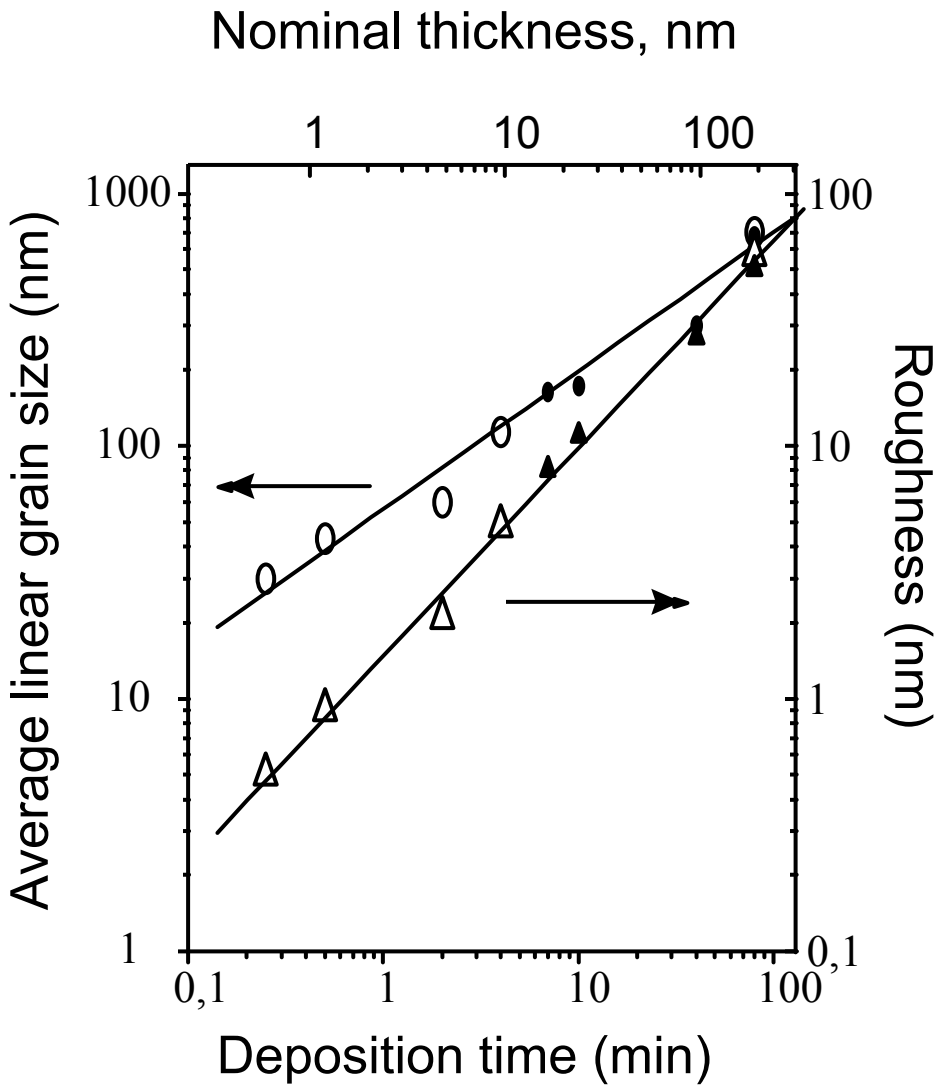
500
nM
nM
40
30
20
10
0

Fig.1.


Nominal thickness, nm
1
10
100
1000
100
10
1
Average linear grain size (nm)
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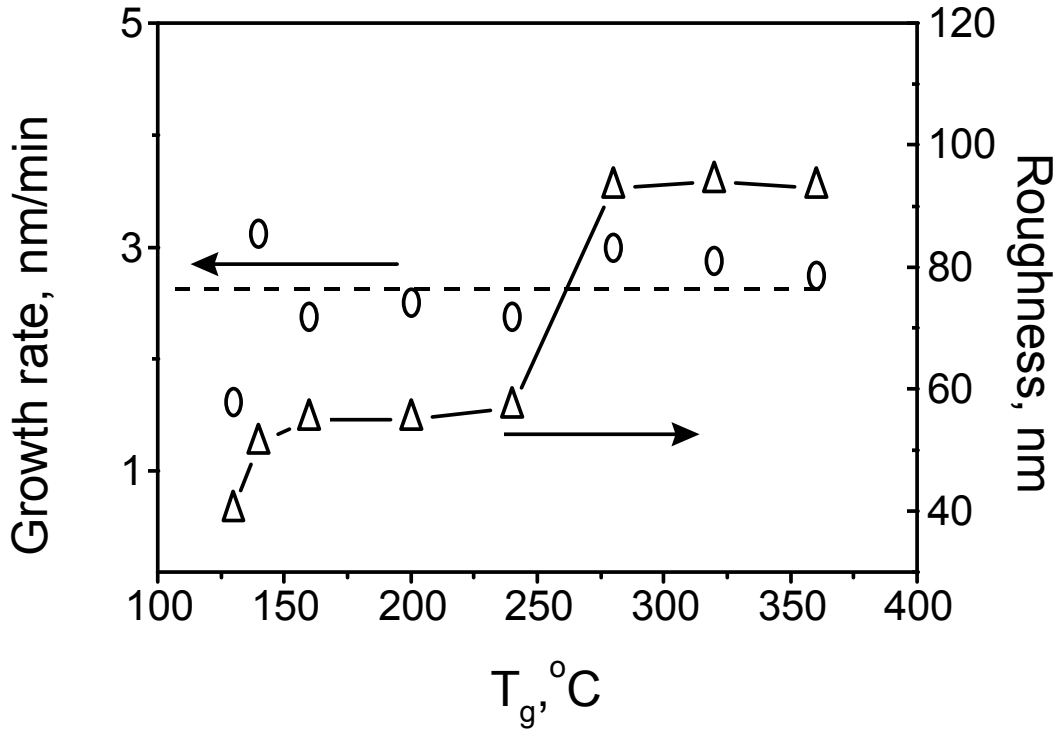
10
1
0,1
Roughness (nm)
0,1
1
10
100
Deposition time (min)

Fig.2.


Growth rate, nm/min
Roughness, nm
T_g, °C
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3
1
120
100
80
60
40
100
150
200
250
300
350
400

Fig.3.

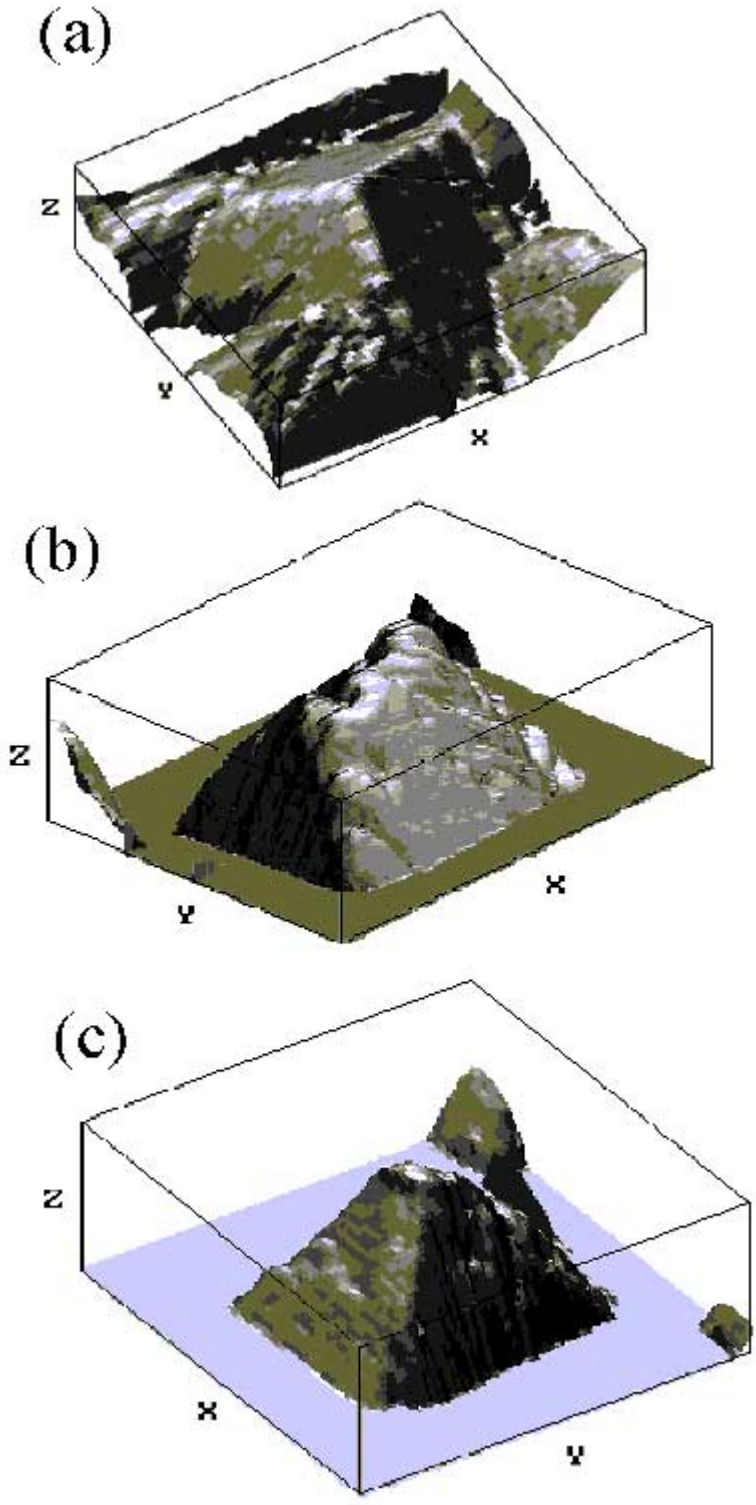

Fig.4.

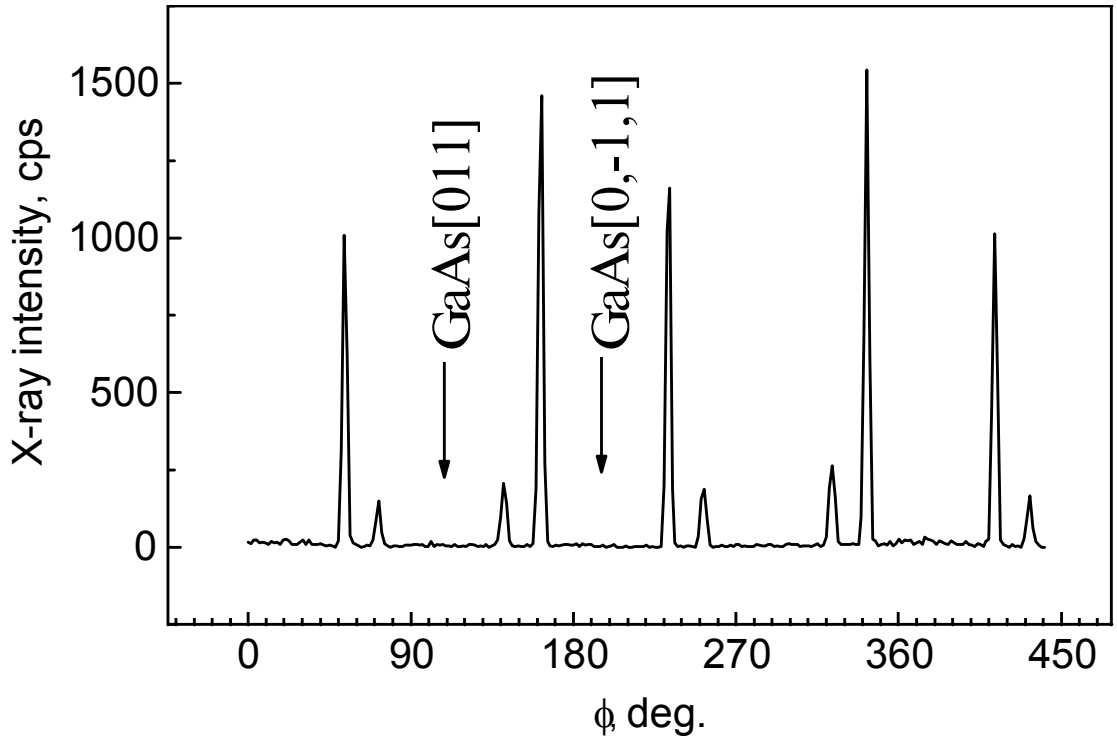
X-ray intensity, cps
1500
1000
500
0
GaAs[011]
GaAs[0,-1,1]
0
90
180
270
360
450
ϕ, deg.

Fig.5.

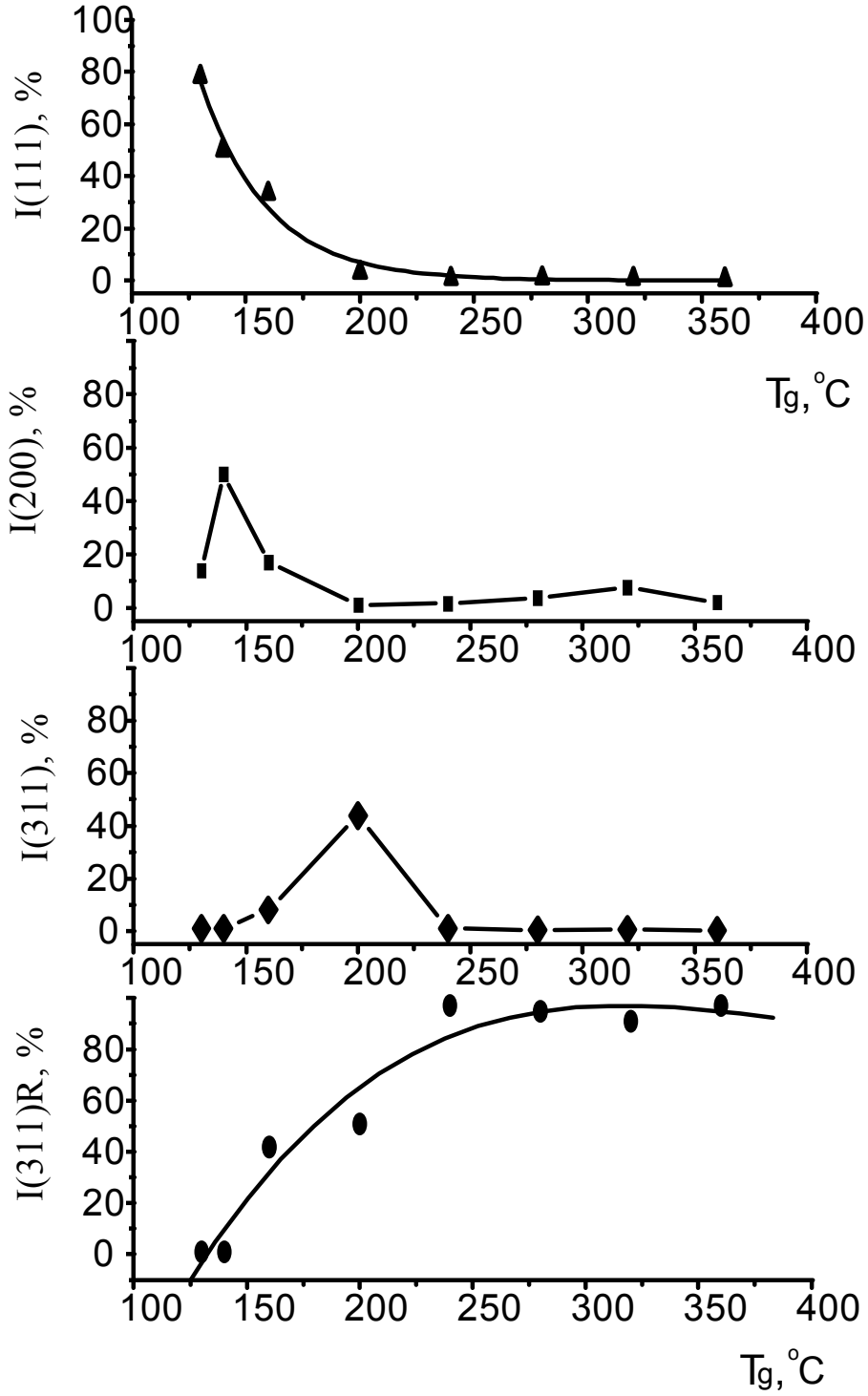

Fig.6.

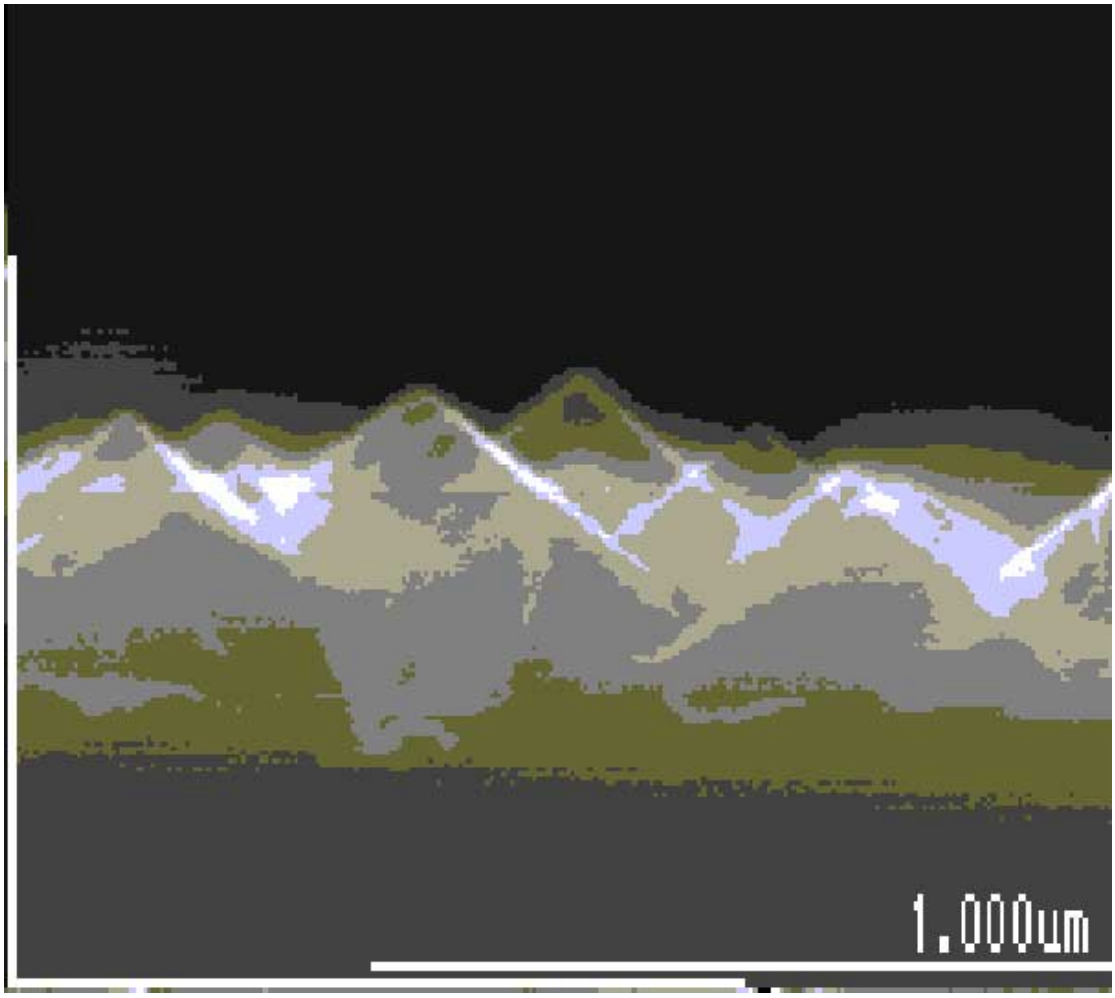

Fig.7.

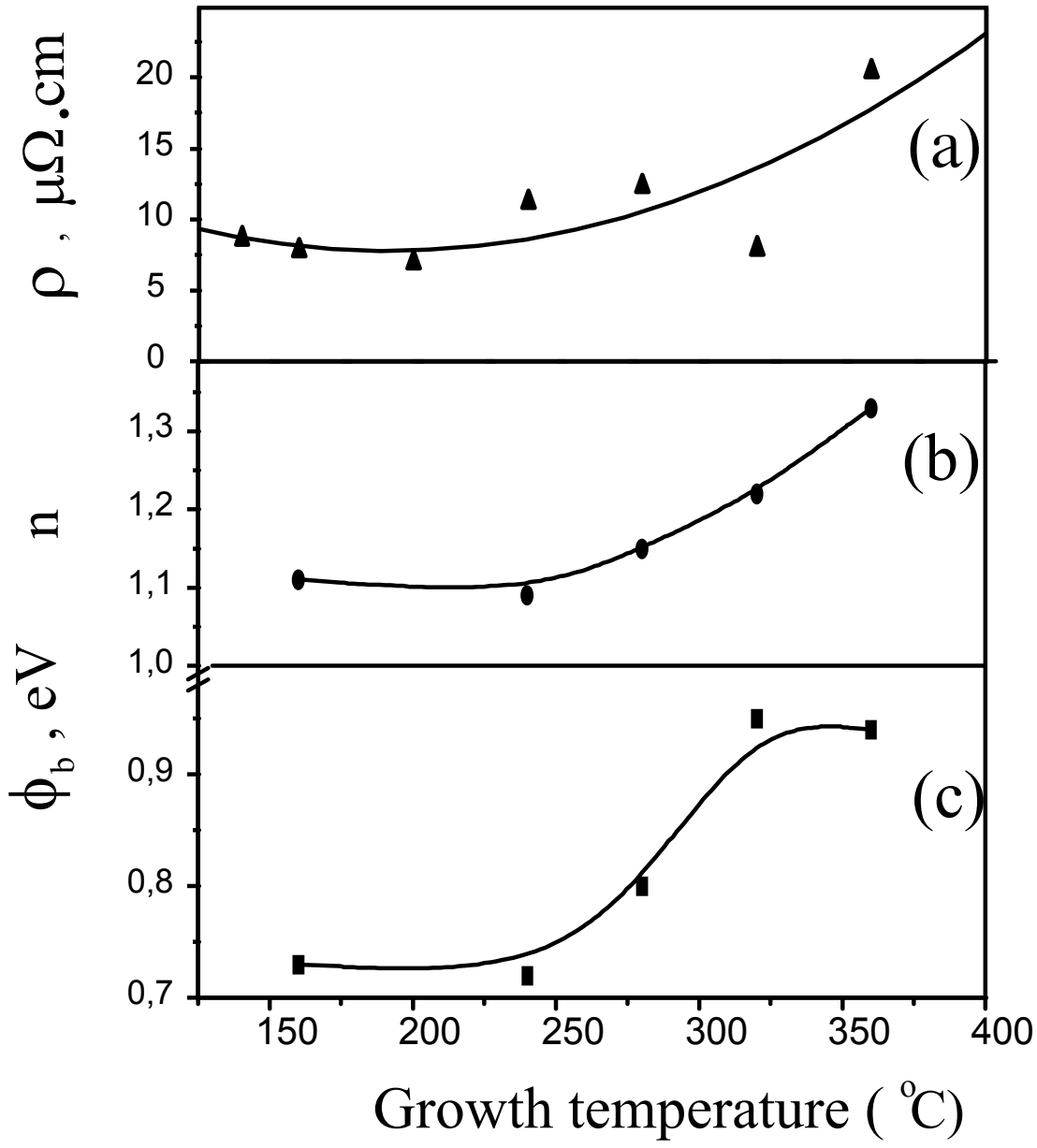
ρ , μΩ.cm
20
15
10
5
0
(a)
n
1,3
1,2
1,1
1,0
(b)
ϕ_b , eV
0,9
0,8
0,7
(c)
150
200
250
300
350
400
Growth temperature (°C)

Fig.8.

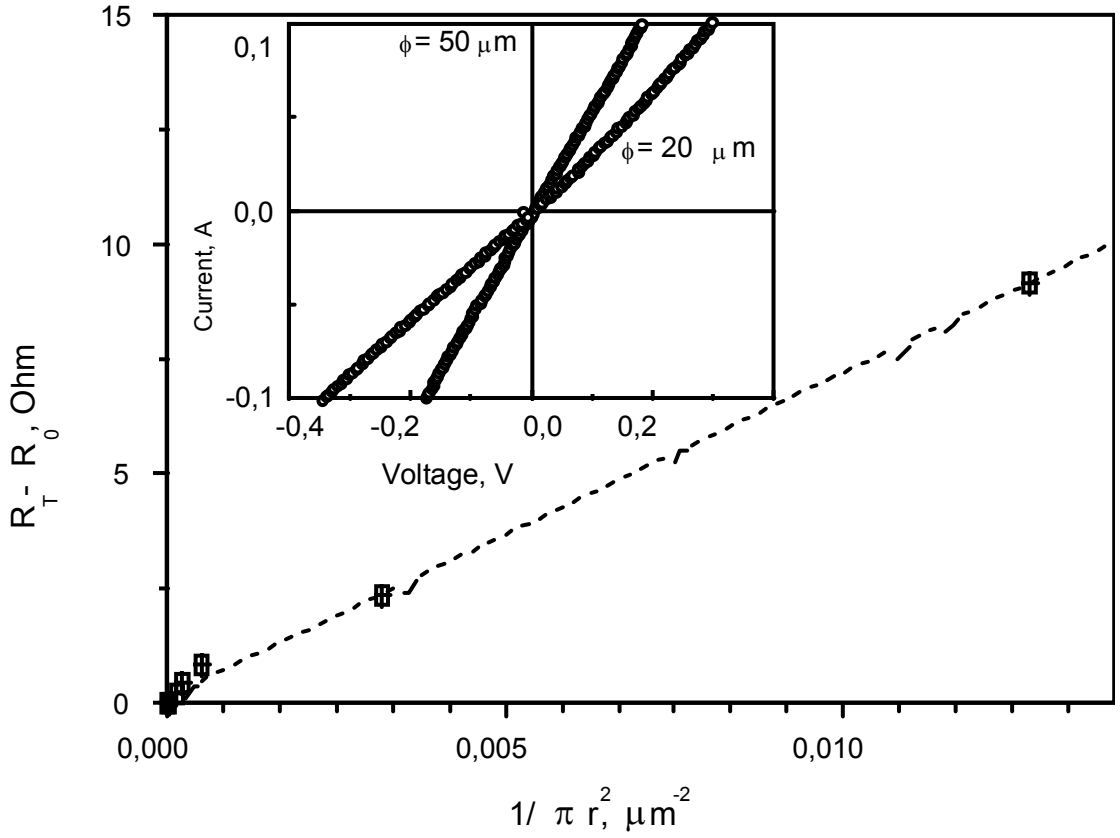
15
10
5
0
0,000
0,005
0,010
R - R , Ohm
T 0
1/ π r, μm
2 -2
0,1
0,0
-0,1
-0,4
-0,2
0,0
0,2
Current, A
Voltage, V
ϕ= 50 μm
ϕ= 20 μm

Fig.9.