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Radio frequency sputtering of tungsten/tungsten nitride multilayers on GaAs

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Thick tungsten nitride films have been deposited on GaAs substrates using a reactive rf-sputtering system. The optical indexes of the WN_x films (determined by *in situ* kinetic ellipsometry) and their nitrogen contents (determined by Rutherford backscattering), have been determined versus the pressure ratio P_{N2}/P_{tot}. Using reactive sputtering conditions which provide highly nitrogenated films, W/WN_x twenty period multilayers with nanometric layer thicknesses have been deposited. The experiment has also been monitored *in situ* by kinetic ellipsometry at 1.96 eV and the multilayer has been analyzed *ex situ* by grazing x-ray reflection measurements at 1.54 Å. The composition of the different layers has been determined precisely by Rutherford backscattering analysis. Both W–WN_x and WN_x–W interfaces in the multilayer appear very sharp by kinetic ellipsometry. This is confirmed by the occurrence of well defined Bragg peaks on the grazing x-ray reflectivity curves in spite of the low density contrast between W and WN_x layers.

I. INTRODUCTION

In GaAs self-aligned gate technologies the efficiency of the gate material as a diffusion barrier and the stability of its electrical properties are essential for reproducible device performance. The problem is to find a metallic material which has good electrical properties even after a post implantation annealing (typically around 800 °C). Up to now, numerous studies[1–6] have been devoted to the optimization of tungsten nitride based structures which have a good efficiency as diffusion barriers.[2] Nevertheless, a compromise of the nitrogen amount in the film must always be found to obtain at the same time, an efficient diffusion barrier and good electrical properties. In particular the efficiency of a diffusion barrier obtained by sputtering is optimum when the pressure ratio $r_p = P_{N2}/P_{tot}$ is greater than 20%.[4] On the other hand, the Schottky contact quality, the stress in the film and the electrical conductivity are compatible with a MESFET technology even if r_p is under 10%.[4] Nevertheless some self-aligned field effect transistors have shown interesting characteristics but with very low pressure ratio during the WN_x deposition (6% for Ref. 5, and 4% for Ref. 6 for example).

In this short paper, we want to demonstrate the feasibility of a new layered structure which can integrate the advantages of both W layers and highly nitrogenated WN_x layers. The idea is simply to deposit a multilayer which alternates thin W and WN_x layers. The multilayer electrical conductivity in the plan of the film (used in the gate of a transistor) will be provided by the W layers, and the diffusion barrier properties by the WN_x layers. In this paper, only the physical properties of this type of structure will be presented. Electrical measurements before and after annealing will be presented elsewhere.

II. EXPERIMENTAL SETUP

The deposition system used in this study has been recently applied to the deposition of a stack alternating copper and copper oxide layers.[7] The introduction and the regulation of a reactive gas near the sample have then been controlled accurately. In spite of the high reactivity of the oxygen, stacks with well-defined Bragg peaks have been realized. Then the same system has been used in this study to alternate W and WN_x layers at the same scale. Reactive rf-sputtering deposition of tungsten nitride films have been intensively studied in the literature.[4–6] It is well known that the introduction of a N_2 partial pressure in an rf-sputtering system induces more or less nitridation of the tungsten layers, depending on the pressure ratio of r_p. We have then calibrated our own system in terms of nitrogen content of the layers, depositing thick WN_x layers on GaAs substrates varying r_p and keeping constant all the other deposition parameters. Then stacks alternating nanometric layers of W and WN_x were deposited. All the experiments have been monitored *in situ* by kinetic ellipsometry (1.96 eV) and analyzed *ex situ* by grazing x-ray reflection (1.54 Å) and Rutherford backscattering.

The diode rf-sputtering system used in this study has been described elsewhere.[7,8] Between the sample (3 in. wide) and the W target (5 in. wide), a fast screen allows us to control very precisely the deposition times of the different layers. The fast screen is kept closed when changing the N_2 partial pressure in the system. The W layers are then deposited after a short prepulverisation of the target, and the WN_x layers when the nitrogen partial pressure is well stabilized. The target autobias and the total pressure are regulated at 300 V and 8 mTorr, respectively, during all the experiments to obtain constant deposition rates. The nitrogen partial pressure is introduced near the sample to minimize the target nitridation.

Se-doped CZ (100) 2-in. GaAs wafers were used. Each wafer has been submitted to a chemical native oxide removal [NaOH (10%) for 1 min] prior to the introduction in the plasma system.

The *in situ* kinetic ellipsometer (KE) has a rotating analyzer and fixed polarizer configuration, with a helium–neon laser source (1.96 eV). All the experimental details about ellipsometry have been described elsewhere.[9]

Grazing x-ray reflectivities (GXR) have been measured

TABLE I. Main characteristics of the tungsten and tungsten nitride thick films and multilayers deposited on GaAs. The total pressure and the autobias of the target have been fixed at 8 mTorr and 300 V, respectively. x is the atomic concentration ratio $[N]/[W]$ (x in the chemical formula WN_x).

Sample number	P_{N_2}/P_{tot} (%)	Thickness (Å)	Optical indexes (1.96 eV)		Rutherford backscattering ($\times 10^{15}$ at/cm^2)		
			n	k	W	N	x
1	0	···	4.00	3.95	483 ± 25	90 ± 20	0.19 ± 0.06
2	10	800	3.80	2.90	324 ± 16	356 ± 50	1.10 ± 0.20
3	20	715	3.75	2.65	277 ± 15	333 ± 50	1.20 ± 0.25
4	40	600	3.75	1.85	212 ± 12	318 ± 50	1.50 ± 0.30
5	60	190	3.20	1.20	50 ± 3	70 ± 15	1.40 ± 0.40
			W/WN_x multilayer (40 periods)				
	0	30	3.95	3.30			0.63
6					444 ± 25	466 ± 60	
	40	28	3.65	1.70			1.50

using a horizontal θ–2θ diffractometer PW1050. Experimental details can be found in Ref. 10.

The different thicknesses are determined by GXR and the optical indexes and the interface behaviors by KE as discussed in Ref. 10. Rutherford backscattering measurements were performed with a 2000-keV He^+ particles, produced by a 2500-kV Van de Graaff accelerator. The nitrogen content was determined by fitting the height of the W signal using the procedure given by Ziegler.[11] The reported errors were estimated from a direct evaluation of the N peak which is on top of the large GaAs background. The usual reduction of this background by channeling fails in this case because of the beam divergence due to the W layer just before entering the GaAs channel.

III. RESULTS AND DISCUSSION

A. Deposition of thick films

The main characteristics of the thick WN_x layers deposited on GaAs substrates have been reported in Table I. Five layers have been deposited varying only the pressure ratio r_p from 0% to 60%. KE trajectories measured during the deposition of these five films are represented in Fig. 1. These trajectories have been fitted using a single index layer along all the deposition, except at the beginning of the KE trajectory which includes certainly a modification of the substrate by the argon bombardment. The optical indexes deduced from these simulations show a little decrease of the real index n and a great decrease of the absorption k, increasing r_p. As

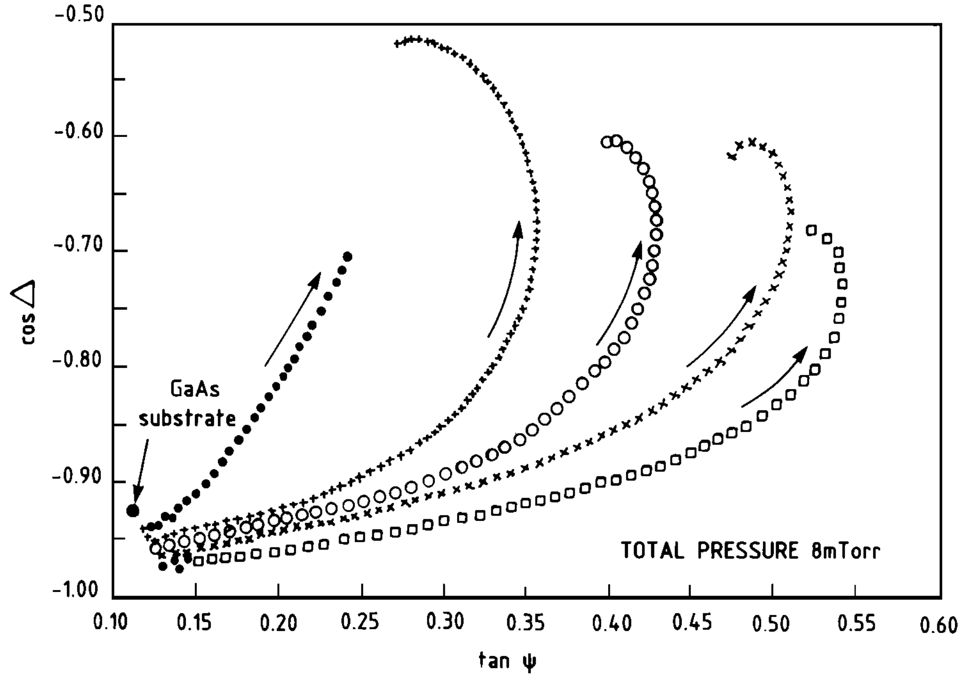

FIG. 1. Experimental kinetic ellipsometry trajectories measured during the deposition of five thick WN_x films on GaAs substrates. The pressure ratio P_{N_2}/P_{tot} was varied from 0% to 60% keeping constant all the other deposition parameters. 60% (●), 40% (+), 20% (○), 10% (×), and 0% (□).

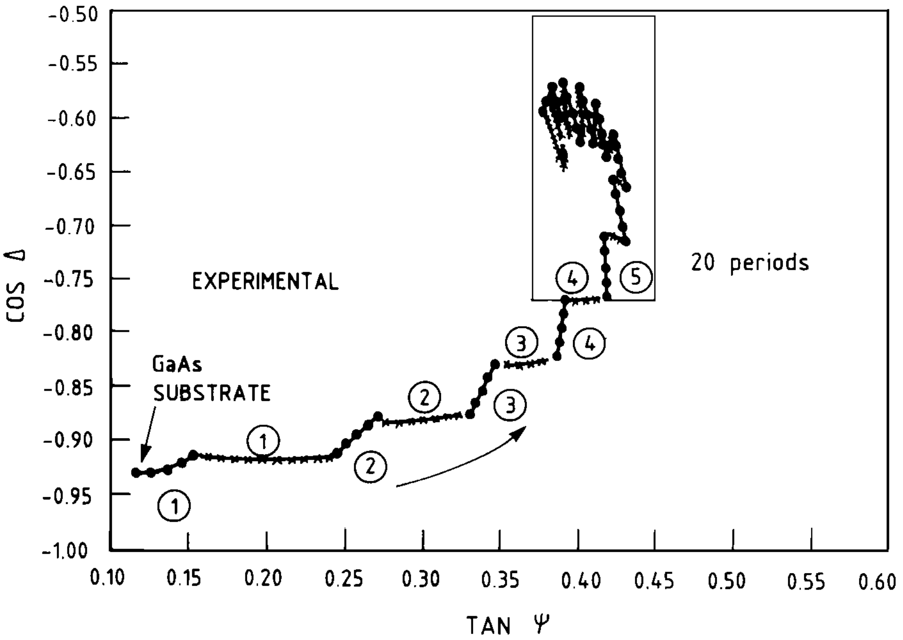

FIG. 2. Experimental kinetic ellipsometry trajectory measured during the deposition of 20 periods of a W/WN$_x$ multilayer on GaAs substrate. The inset in the figure is reported on Fig. 3. Tungsten nitride (●) and tungsten (×).

expected, the layers becomes more and more insulating. The deposition time has been fixed to $\simeq 3000$ s. The deposition rate of the pure W layers is ~ 0.4 Å/s. When we increase the pressure ratio r_p, we observe a great decrease of the deposition rate (0.2 Å/s for $r_p = 40\%$). For higher pressure ratios the deposition rate becomes negligible due to the target nitridation (0.06 Å/s for $r_p = 60\%$). The Rutherford backscattering analysis shows that the nitrogen composition in this case ($x \simeq 1.4$ for $r_p = 60\%$) is quasi the same than for $r_p = 40\%$. Moreover, the optical indexes of this sample are lower than that of sample 4. The nitridation of the target is certainly sufficient to decrease the sputtering efficiency and then the deposition rate without increasing the nitrogen content and the density of the layer is reduced due to the low sputtering efficiency. To make our W/WN$_x$ multilayers we have then selected the higher pressure ratio which provides a maximum nitrogen incorporation with a non-negligible deposition rate ($r_p = 40\%$).

B. Deposition of multilayers

The experimental KE trajectory measured during the deposit of twenty periods of a W/WN$_x$ multilayer is reported in Fig. 2. The deposition times have been fixed to 2 min for both the W and WN$_x$ layers. This low value produces thicknesses ~ 30 Å. We are then under the crystallization thickness of the tungsten layers which has been determined to be ~ 40 Å in our system by transmission electron microscopy.[12] The stack begins with a WN$_x$ layer to increase the diffusion barrier efficiency of the multilayer. The different parts of the trajectory due to the W and WN$_x$ layers appear perfectly. To make a simulation we have been interested in the middle of the stack to avoid possible substrate effects (inset in Fig. 2). This part of the trajectory is reported in Fig. 3 with the corresponding simulation. Both the W–WN$_x$ and WN$_x$–W interfaces have been assumed perfectly sharp. The thicknesses (28 Å for the WN$_x$ layer and 30 Å for the W layer) have been extracted from the GXR measurements which are reported in Fig. 4 (see below). Assuming these thickness values, optical indexes for W and WN$_x$ in the stack can be found very precisely. We found ($n = 3.9$, $k = 3.3$) for tungsten layers and ($n = 3.6$, $k = 1.7$) for tungsten nitride layers. This latter index is comparable with that obtained on

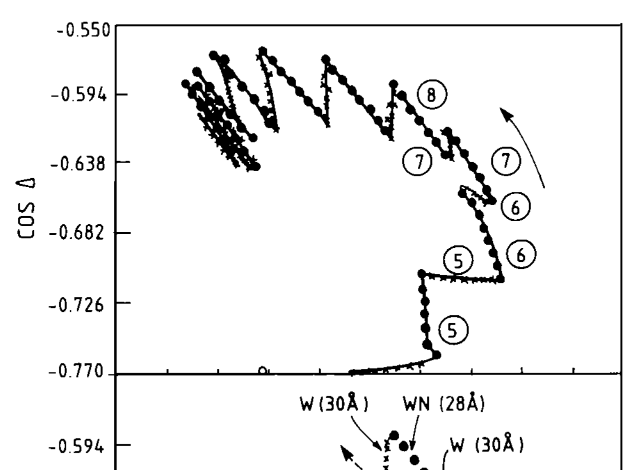

FIG. 3. Experimental and simulated kinetic ellipsometry trajectories measured during the deposition of the multilayer of Fig. 2. The simulation has been made assuming perfect interfaces and constant optical indexes for the W and WN$_x$ layers. Experimental (top): tungsten (×) and tungsten nitride (●); theoretical W ($n = 3.9$ and $k = 3.3$) (×) and WN ($n = 3.6$ and $k = 1.7$) (●).

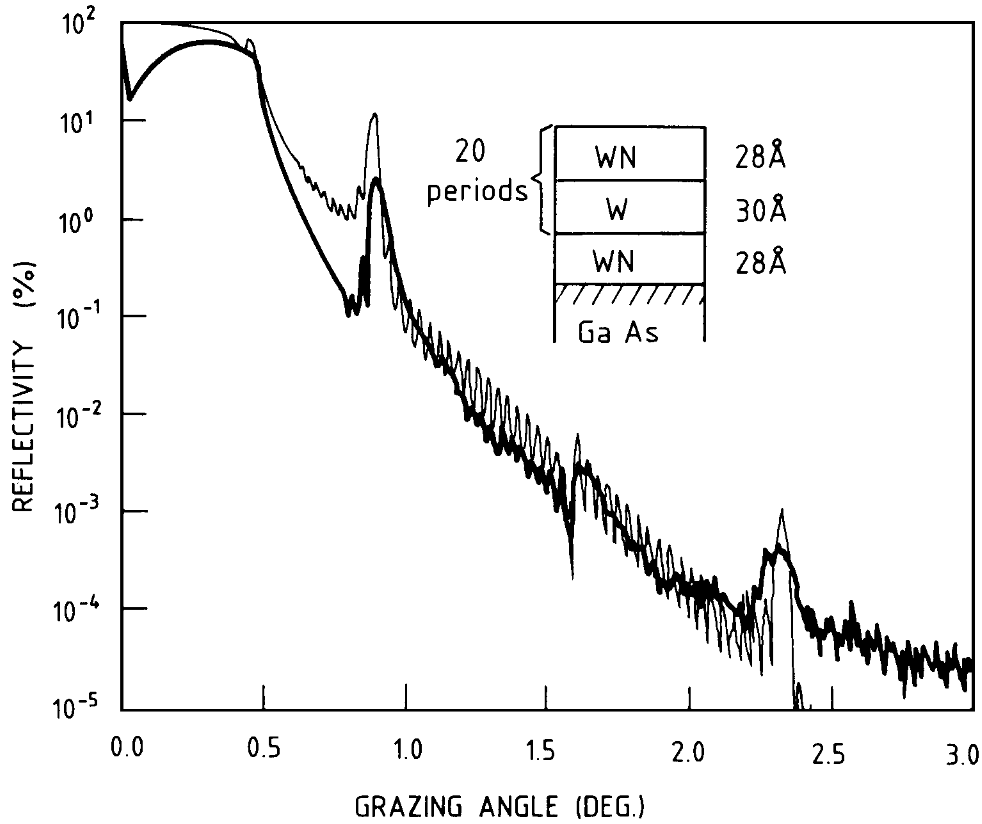

FIG. 4. Experimental (heavy line) and simulated (light line) grazing x-ray reflection of the W/WN$_x$ multilayer of Fig. 2. The model used for the simulation is also reported in the figure.

thick layers with the same pressure ratio (sample 4 in Table I), but it is not the case for the W layers. Indeed, if we compare these values with the optical indexes of quasipure tungsten (sample 1 in Table I) we see that a residual nitridation remains in the W layers, which is intermediate between that of pure W layers (sample 1 in Table I) and WN$_x$ layers deposited with $r_p = 10\%$ (sample 2 in Table I). This is confirmed by the Rutherford backscattering analysis of the W/WN$_x$ multilayer (sample 6 in Table I). If we assume the same nitrogen content in the WN$_x$ layers in the stack as for thick films deposited in the same conditions ($x \simeq 1.5$), we find a non-negligible nitrogen content in the W layers ($x \simeq 0.63$), perfectly in agreement with the value expected from the optical indexes. This excess of nitrogen in the W layers can have two origins. The pressure ratio used to make the multilayers can be sufficiently high to induce a slight nitridation of the tungsten target. When a W layer is deposited after a WN$_x$ layer the tungsten nitride of the target can be found inside the layer. The second origin can be a low residual nitrogen partial pressure inside the chamber even during the W layer depositions.

Experimental and simulated reflectivity curves of this multilayer are reported in Fig. 4. Well-defined Bragg peaks appear in the angle range 0°–3°. The second-order Bragg peak is weak because of the γ value ($\gamma = d_W/d_{\text{tot}}$) close to $\frac{1}{2}$. The simulation has been made assuming no interdiffusion at the different interfaces, but with an important physical roughness which is taken into account by a Debye–Waller factor σ_R of 8 Å. This very important value can explain the difference between the simulation and the experiment especially close to the first-order Bragg peak. Indeed, the Debye–Waller factor model is not very accurate in this case. The relatively low Bragg peak intensity is related to the low contrast between the indexes of tungsten ($\delta \simeq 4.07 10^{-5}$ and $\beta \simeq 3.5 10^{-6}$), and those of tungsten nitride ($\delta \simeq 3.07 10^{-5}$ and $\beta \simeq 0.6 10^{-6}$ at $\lambda = 1.54$ Å).

IV. CONCLUSIONS

In conclusion, in first time we have determine the reactive plasma conditions necessary to deposit highly nitridated WN$_x$ films. The nitrogen content and the optical indexes at 1.96 eV of the tungsten nitride films have been determined versus the pressure ratio $r_p = P_{N2}/P_{\text{tot}}$ during the film deposition. We have also realized for the first time a W/WN$_x$ multilayer with nanometric thicknesses. A twenty period stack presents well-defined Bragg peaks characteristic of a well-ordered structure. Moreover, the W–WN$_x$ and WN$_x$–W interfaces are very sharp as shown by *in situ* kinetic ellipsometry. The tunsgten nitride layers inside the multilayer have a great amount of nitrogen ($x \simeq 1.5$ in WN$_x$). The W layers are also not free of nitrogen ($x \simeq 0.6$), as shown by Rutherford backscattering analysis. Stability and electrical properties of this type of structure will be presented elsewhere.

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