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WSi_x formation in W–Si multilayers¹

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The formation of WSi_x phases from multiple thin layers of W and Si was investigated. Tungsten and silicon thin films of ~ 5 nm thickness were deposited sequentially onto GaAs substrates by dc magnetron sputtering from elemental targets. The total film thickness was ~ 300 nm. The W and Si layer thickness, and thus the overall composition of the films, was controlled by adjusting the power applied to the two sputtering targets. The films examined in this work had nominal compositions in the range WSi₂-WSi_{0.48}.

The multilayered structure was subjected to rapid thermal annealing at 900°C. The extent of the reaction, the grain size, and the crystal structure of the silicides were determined using transmission electron microscopy and X-ray diffraction. For WSi_x films with $x \le 0.52$, the silicides had the β W structure and were relatively large grained, ~450 nm, while for x > 0.52, the film contained the W₅Si₃ phase and consisted of grains ~45 nm in diameter.

Schottky contacts were of good quality for $x \le 0.52$, with a barrier height (ϕ) of 0.7 V and an ideality factor (*n*) of 1.15. Schottky contacts with higher silicon concentrations were poor.

The WSi, (x = 0.52) Schottky contact has been successfully incorporated into a self-aligned gate field-effect transistor.

On a étudié la formation de phases WSi_x dans des couches minces multiples de W et Si. Des films minces de tungstène et de silicium ayant environ 0,5 nm d'épaisseur ont été déposées en succession sur des substrats de GaAs par pulvérisation au magnétron CC, à partir de cibles élémentaires. L'épaisseur totale de l'ensemble des couches était d'environ 300 nm. L'épaisseur de chacune des couches W et Si, et par conséquent la composition de l'ensemble des films était contrôlée en ajustant la puissance appliquée aux deux cibles. La composition nominale des films examinés dans ce travail se situait dans l'intervalle WSi₂–WSi_{0.48}.

Chaque structure multicouche a été soumise à un recuit thermique rapide à 900°C. L'étendue de la réaction, la grosseur de grain et la structure cristalline des siliciures ont été déterminés par microscopie électronique en transmission et par diffraction des rayons X. Pour les films WSi_x avec $x \le 0.52$, les siliciures avaient la structure β W et étaient relativement à gros grains, environ 450 nm, alors que pour x > 0.52, les films contenaient la phase W_5Si_3 et consistaient en grains d'environ 45 nm de diamètre. Les contacts Schottky étaient de bonne qualité pour $x \le 0.52$, avec une hauteur de barrière (ϕ) de 0.7 V et un facteur d'idéalité

(n) de 1,15. Avec des concentrations de silicium plus élévées, les contacts Schottky étaient mauvais.

Le contact Schottky WSi_x (x = 0,52) a été incorporé avec succès dans un transistor a effet de champ à porte auto-alignée. [Traduit par la revue]

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1. Introduction

Tungsten silicide was first described by Yokoyama et al. (1) as the gate metallization in a self-aligned gate field-effect transistor (SAGFET) process for GaAs. In this process, the gate electrode also serves as the implant mask for the n^+ regions. It is therefore crucial that the WSi_x -GaAs contact be thermally stable under the n^+ annealing conditions required to make the implant electrically active. Until recently, the n^+ anneals were usually carried out in furnaces at temperatures of 750-850°C for several minutes; Onishi et al. (2) studied the silicide formation and thermal stability of WSix films after furnace annealing. Rapid thermal annealing (RTA) is used increasingly in the fabrication of GaAs metal semiconductor field-effect transistors (MESFET). The WSi_r-GaAs interaction during the shorter annealing times must be determined. Improved FET performance of WSi_x SAGFETs as a result of RTA was demonstrated by Onishi et al. (3) but no information on the physical nature of the film-GaAs interface was provided. In this work the formation of tungsten silicide and the thermal stability of the WSi_x-GaAs interface after RTA are investigated.

2. Experimental

The WSi_x films were deposited in a custom-built sputtering system equipped with three planar magnetron targets (Fig. 1). High-purity elemental tungsten and silicon targets (14 cm \times 22 cm) were dc sputtered in an Ar atmosphere at a pressure of 0.7 Pa. The wafers were mounted on the substrate carousel,



FIG. 1. Schematic of the deposition system.

which was then continuously rotated, resulting in the deposition of alternating layers of W and Si. The thickness of an individual layer depended on both the power applied to the target as well as the rotation speed of the carousel. Films described in this work were prepared at a rotation speed of 34 rpm to obtain W and Si layers less than 0.5 nm thick. The sputtering power for the Si target was fixed at 500 W, and the power applied to the W target was adjusted to give the desired composition. Table 1 summarizes the W sputtering power and the nominal composition.

For electrical characterization, WSi_x was deposited on Sidoped GaAs substrates ($n = 1-5 \times 10^{17} \text{ cm}^{-3}$), and test structures were defined by plasma etching in CF₄-O₂ mixtures. Ohmic contacts were formed by alloying small tin spheres. *I*-*V* measurements were performed on a HP4140B

TABLE 1. Electrical characteristics and phases observed in WSi_x films after RTA at 900°C, 6 s

Tungsten power* (W)	Nominal composition	Barrier height (V)	Ideality factor	Phases observed by X-rays
600	WSi _{0.48}	0.72	1.15	$W_3Si, \alpha W$
530	WSi _{0.52}	0.74	1.16	W ₃ Si, aW
440	WSi _{0.63}	0.47	2.95	αW, WsSi3
130	WSi ₂	0.53	2.65	WSi ₂ , W ₅ Si ₃

*Sputtering power of the Si target was fixed at 500 W

picoammeter with a built-in dc voltage source. Transistors with a 1 µm gate length were fabricated on semi-insulating GaAs substrates. Apart from the WSi_x gate, the process steps were similar to those used in a standard GaAs MESFET process. Samples for transmission electron microscopy (TEM) and X-ray analysis were deposited on semi-insulating GaAs substrates. The silicide contacts were examined in the TEM in both the horizontal and vertical sections to determine the grain size and crystal structure of the silicide and the extent of interaction between the GaAs and WSi, at the silicide-GaAs interface. The horizontal sections were obtained by dissolving the GaAs using a 0.5% Br₂-CH₃OH solution at room temperature. The vertical sections were prepared by mechanical and Ar⁺ ion thinning as outlined elsewhere (4, 5). X-ray powder-diffraction scans were taken on an automated Philips diffractometer.

3. Results and discussion

Figure 2 shows the I-V curves of both as-deposited and annealed films with three different nominal film compositions WSi_x, where x = 0.52, 0.63, and 2.0. The as-deposited films displayed good diode behaviour with ideality factors close to unity and barrier heights of ~0.65 V. After annealing at 900°C for 6 s (10 s for x = 2), the WSi_x diodes with x > 0.52 were seriously degraded, as indicated by the dramatic increase in the ideality factor and the accompanying drop in the barrier height. In contrast, the films with higher W content ($x \le 0.52$) appeared to improve after RTA, giving barrier heights of ≥ 0.7 V while the ideality factors remained close to unity. These observations are summarized in Table 1, together with the phases found from X-ray powder-diffraction scans.

The major phase observed for WSi_x, $x \le 0.52$, was W₃Si crystallized in the β W structure (6); α W was present as a minor component. For films with higher Si content, the phases were α W, W₅Si₃ (x = 0.63), and W₅Si₃, WSi₂ (x = 2.0). In all films there appeared to be a lower Si content than expected on the basis of the nominal composition. It is not clear at present whether this reflects a real discrepancy in composition or whether the Si is present in a form that cannot be detected by X-rays (e.g., in grain boundaries).

To gain some insight into the origin of the difference in electrical behaviour of WSi_x as x changes from 0.52 to 0.63, we have examined samples of these films by transmission electron microscopy. A marked difference in grain size is found in the annealed films (Fig. 3). The W-rich film (Fig. 3a) has an average grain diameter of 450 nm, which is about 10 times the average grain size of the Si-rich film (Fig. 3b). Selected-area diffraction patterns (SADP) for these samples indicate the presence of α W and W₅Si₃ phases in the Si-rich film, in agreement with the phases observed by X-ray diffrac-



FIG. 2. The I-V characteristics of as-deposited and annealed (900°C, 6 s) films: \blacksquare , 530 W, as-deposited; \Box , 530 W, 900°C, 6 s; \blacklozenge , 440 W, as-deposited; \bigcirc , 440 W, 900°C, 6 s; \blacklozenge , 130 W, as-deposited; \triangle , 130 W, 900°C, 10 s.

tion. The SADP of the W-rich film, however, shows only lines for αW . This observation is at odds with the X-ray diffraction results, which also indicates the presence of W₃Si (βW).

The silicide–GaAs interface was investigated by crosssectional TEM(XTEM). Examination of the cross sections in Figs. 4a and 4b reveals subtle but important differences in the appearance of the interfaces. The XTEM picture of the W-rich film (Fig. 4a) shows that the WSi_x–GaAs interface is no longer sharp after RTA. From the roughness at the interface, it is estimated that the silicide grains penetrate into the GaAs substrate to a depth of approximately 10 nm. The interface of the Si-rich film with the GaAs (Fig. 4b) remains smooth and abrupt after annealing. Little or no penetration of the silicide into the GaAs is observed, which is possibly related to the thin, continuous, interfacial layer visible in the micrograph.

The TEM investigation of the WSi_x contacts after RTA shows considerable differences in the physical structure of the films as well as the nature of the WSi_x-GaAs interface. The failure mechanism of the WSi_x diodes with x > 0.52 may be related to the fine grain structure of these films. Grain boundaries provide fast diffusion paths, and it is likely that the electrical degradation of these diodes is caused by the indiffusion of one of the film constituents. The C-V measurements of both as-deposited and annealed films show flat carrier profiles, indicating that gross in-diffusion had not occurred. However, the C-V technique cannot provide information on the carrier concentration in the vicinity of the film-GaAs interface.



FIG. 3. The TEM plan views of (a) WSi_x, x = 0.52, and (b) WSi_x, x = 0.63 after RTA at 900°C, 6 s.



FIG. 4. Cross-sectional TEM of (a) WSi_x, x = 0.52, and (b) WSi_x, x = 0.63 after RTA at 900°C, 6 s.



FIG. 5. (a) An SEM micrograph of a WSi_x SAGFET. (b) A trace of FET characteristics (10 mA/division, 0.5 V/division, 0.5 V/step).

The thermal stability of WSi_{0.52} films allows their incorporation in a SAGFET structure. A scanning electron micrograph (SEM) of a completed device is shown in Fig. 5*a*, while Fig. 5*b* gives a trace of the FET characteristics. From measurements of 80 devices with $L_g = 1 \mu m$ and $W_g = 2 \times 100 \mu m$ across a 2 in. GaAs wafer, the following results have been observed (1 in. = 2.54 cm).

Average values and standard deviations of the saturation current (I_{dss}) , pinch-off voltage (V_p) , and transconductance (g_m) are 41.9 \pm 2.2 mA, 2.04 \pm 0.11 V, and 133 \pm 5 mS·mm⁻¹, respectively. These are acceptable values for this structure and indicate that integrated circuits could be fabricated from SAGFETs with WSi_{0.52} Schottky gates.

4. Conclusion

The thermal stability of tungsten silicide on GaAs after rapid thermal annealing has been investigated. WSi_x films with $x \le 0.52$ (nominal composition) are found to be stable, whereas Si-rich films degrade significantly after RTA. X-ray diffraction analysis and electron microscopy reveal important differences in the structure and nature of the film–GaAs interface of W-rich or Si-rich films. The failure mechanism of WSi_x for x > 0.52 is not understood at present. The feasibility of incorporating $WSi_{0.52}$ into a SAGFET has been demonstrated.

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- N. YOKOYAMA, T. ONISHI, H. ONODERA, T. SHINOKI, A. SHIBATOMI, and H. ISHIKAWA. IEEE International Solid State Circuits Conference. 1983. p. 44.
- 2. T. ONISHI, N. YOKOYAMA, H. ONODERA, S. SUZUKI, and A. SHIBATOMI. Appl. Phys. Lett. 43, 600 (1983).
- T. ONISHI, Y. YAMAGUCHI, T. INADA, N. YOKOYAMA, and H. NISHI. Conference on Solid State Devices and Materials, Kobe, Japan. 1984. p. C-7-1.
- 4. B. J. KESTEL. Ultramicroscopy, 9, 379 (1982).
- 5. J. C. BRAUMAN and R. SINCLAIR. J. Electron Microsc. Tech. 1, 53 (1984).
- W. B. PEARSON. A handbook of lattice spacings and structures of metals and alloys. Vol. 2. Pergamon Press Ltd., Oxford, England. 1967.