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Investigations on the Structure of MOCVD AlN Layers on Silicon

By

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The AlN layers are deposited from NH_3 and TMA on (111) and (100) Si according to the MOCVD method. The influence of the substrate temperature and of the composition of the gaseous phase on the real structure is investigated in the temperature range from 1073 to 1573 K. At a temperature of 1573 K and a molar ratio of NH_3/TMA $> 400/1$ producing of monocrystalline AlN layers on (111) Si is successful.

Die AlN-Schichten werden nach dem MOCVD-Verfahren aus NH_3 und TMA auf (111) und (100) Si abgeschieden. Es wird der Einfluß der Substrattemperatur und der Gasphasenzusammensetzung im Temperaturbereich von 1073 bis 1573 K auf die Realstruktur untersucht. Bei 1573 K und einem Molverhältnis NH_3/TMA $> 400/1$ gelingt es, auf (111) Si einkristalline AlN-Schichten herzustellen.

1. Introduction

On the basis of the investigations conducted by Manasevit et al. [1] on the AlN preparation using MOCVD, we have developed an experimental variant permitting to deposit AlN layers with a satisfactory homogeneity on different substrates in an induction-heated vertical reactor [2].

For investigating the application possibilities of these AlN layers in microelectronics it is particularly important to find out the influence of experimental conditions on the physical properties and to make the preparation process reproducible.

In the following we will discuss the effects of the deposition temperature, the substrate orientation, and the composition of the gaseous phase on structural perfection. The methods used for analysis were based on reflection electron microscopy, X-ray diffraction, and scanning electron microscopy.

In general, reactions as, for example, that of trimethylaluminium (TMA) with NH_3 are determined essentially by the effects resulting from chemical kinetics. However, the course of reaction will not be examined in this paper.

In order to certain that the experimental results can be compared with each other, such factors as, for example, the distance between gas inlet and substrate, the total flow rate, the absolute concentration of NH_3, and the cooling rate have been kept constant. As substrate we used (111) and (100) silicon wafers with a diameter of 36 mm which have been treated according to [3].

2. Results

2.1 Temperature of the substrate

An exact investigation of the influence of the substrate temperature T_S on the structural perfection of the AlN layers performed by means of reflection electron microscopy revealed the following characteristic features:

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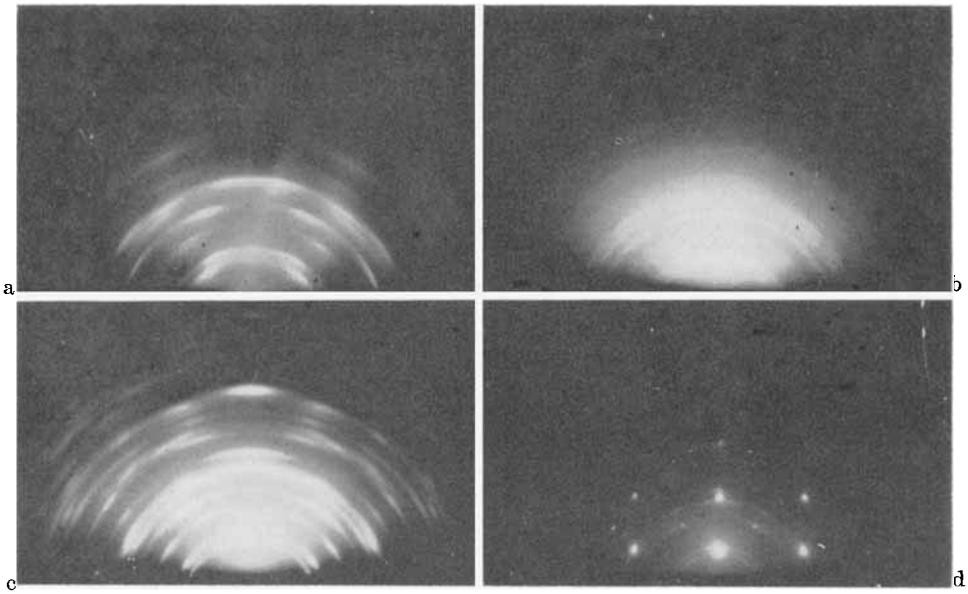

Fig. 1. RHEED patterns from AlN layers on (111)-Si a) $T_S = 1073$ K, $\langle 11\bar{2}1 \rangle$ fibre axis; b) $T_S = 1273$ K, textureless; c) $T_S > 1473$ K, $\langle 0001 \rangle$ fibre axis; d) $T_S = 1573$ K, electron beam azimuth in $[\bar{1}2\bar{1}0]$ direction

At a temperature of $T_S = 1073$ K, AlN layers with a good texture and a $\langle 11\bar{2}1 \rangle$ fibre axis are formed (Fig. 1a), with the axis being perpendicular to the surface of the substrate. Between 1073 and 1473 K, the degree of texturing is generally lower than below and above this temperature range, which is in many cases equivalent to being textureless (Fig. 1b). From 1473 K on, the structural quality of the layers improves again considerably. The samples show a good to very good texture at 1473 K. Above this temperature, a change in the preferred orientation occurs. The layers show no longer a distinct $\langle 11\bar{2}1 \rangle$ fibre axis, but predominantly a $\langle 0001 \rangle$ orientation (Fig. 1c). At 1573 K, we succeeded in depositing layers with the highest structural perfection namely single-crystalline AlN on (111) silicon (Fig. 1d). The determination of intergrowth resulted in the correlation (0001) AlN || (111) Si, $[\bar{1}2\bar{1}0]$ AlN || $[\bar{1}10]$ Si, which is in conformity with literature [1, 4, 5]. The electron microscopic examinations and X-ray analysis of the AlN layers made at different points of the substrate revealed a single-crystalline character with a minimum polycrystalline portion.

2.2 Orientation of the substrate

The investigation of the influence exerted by the orientation of the substrate showed that the deposition on (111) and (100) silicon substrates, taking place under the same conditions, brought no serious differences in texture up to 1473 K (e.g. different sharpness of texture, change in the preferred orientation), which was contrary to expectations. The situation was different at 1573 K in so far as we have succeeded in depositing single-crystalline AlN only on (111) silicon so far.

2.3 Composition of the gaseous phase

The analysis of the influence of the composition of the gaseous phase, i.e. of the molar ratio of ammonia to trimethylaluminium (NH_3/TMA), in the temperature range from 1073 to 1573 K showed that the degree of texturing changes only slightly as a function

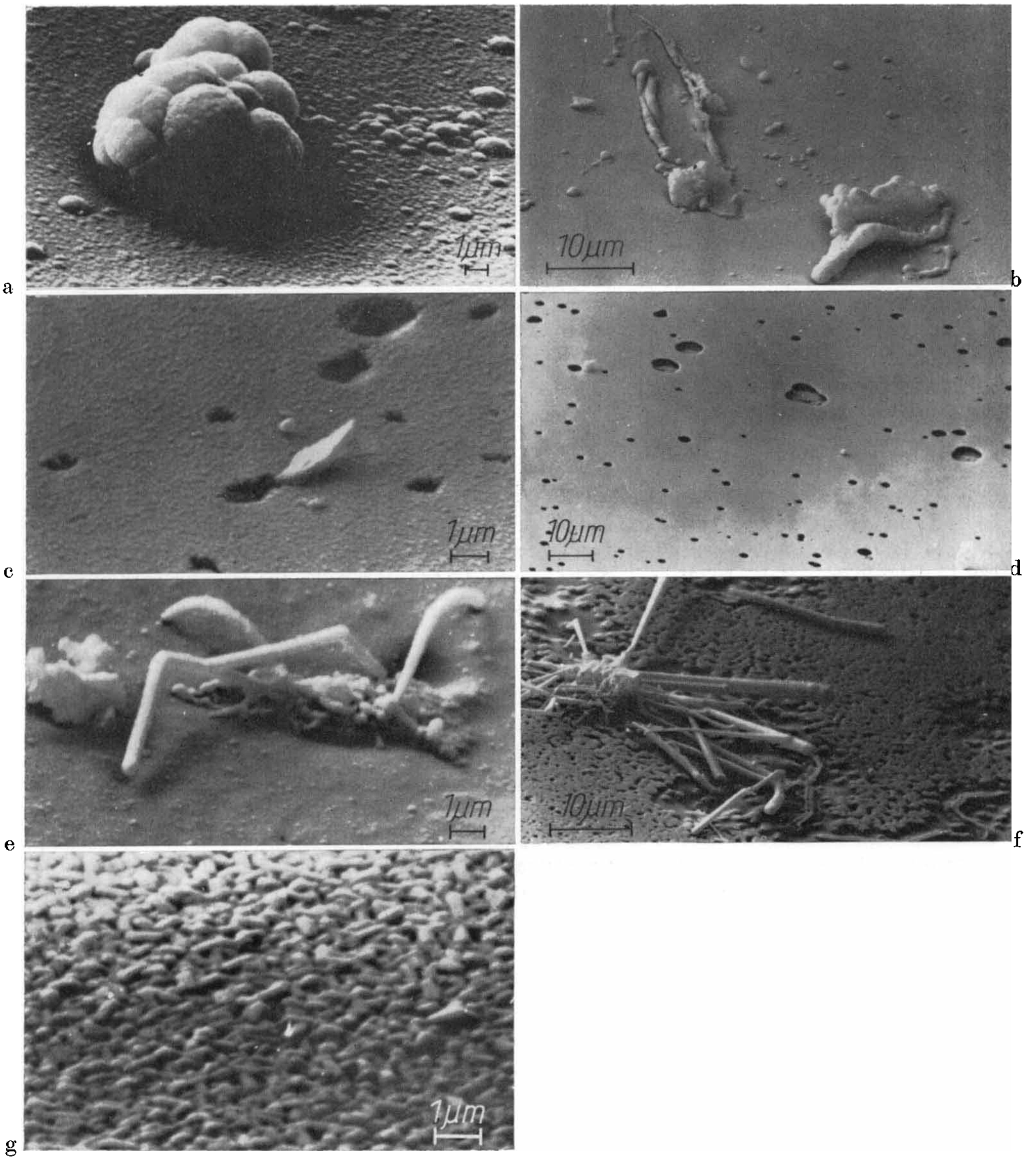

Fig. 2. Scanning electron micrographs of the surface of AlN layers a), b) particles with different shapes; c), d) pinholes; e), f) whiskers; g) surface roughness, film thickness ≈ 1 µm

of the molar ratio. In order to obtain good textures or single-crystalline AlN, the molar ratio should be $NH_3/TMA > 400/1$ [2], which also applies to other physical and chemical properties.

2.4 Surface morphology

For most applications the quality of the surfaces of the AlN layers plays an important part. Therefore, we examined the surfaces of the layers obtained under various conditions of growth by means of a scanning electron microscope. The layers show various defects which are found in a large number and great variety, especially at high deposition temperatures (1473 to 1573 K). Thus, particles having different shapes and sizes are found on the surface of AlN (Fig. 2 a, b). Furthermore, other disturbances of growth arise in the form of pinholes (Fig. 2 c, d) exerting an unfavourable influence especially on the electrical properties. Their density greatly depends on the perfection of the substrate and is 0 to 350 cm^{-2}. At the present state of the investigations, however, no systematic connection with the production parameters can be discovered.

The surface morphology of relatively thin layers (100 to 200 nm) deposited at 1573 K shows remarkable features. Here we have observed whiskers of different shapes (Figs. 2 e, f) the density of which increases with decreasing TMA content.

The shape of the whiskers suggests the VLS mechanism [6, 7]. The surface roughness increases considerably with growing thickness of the layer (Fig. 2g).

Layers of a thickness of about 1 μm have been polished in a mechano-chemical way for further physical investigations.

3. Discussion

For interpreting the experimental results, it is necessary to have a closer look at the rate of deposition R_D as a function of the substrate temperature T_S as it is represented in Fig. 3. In our experiments we stated the maximum of the rate of deposition between 1173 and 1373 K, with the shape of the curve flattening with increasing molar ratio. At temperatures below 1173 K, R_D decreases quickly. At a substrate temperature of 1073 K, the mobility of the nuclei is still so high that a good texture can develop.

With increasing substrate temperature also R_D goes on increasing, but despite of a higher temperature the quality of the developing textures is not satisfactory any longer, the layers are partially polycrystalline.

At temperatures above 1373 K the rate of deposition decreases because of the preponderance of the homogeneous reaction. This leads to favourable conditions of growth for the nuclei developing on the substrate.

In addition, an improvement is obtained by a more and more decreasing density of the structural defects at high temperatures so that sharply textured or even single-crystalline layers can deposit. In this temperature range, however, one has to expect junction layers that may develop near the phase boundary between AlN and Si, and which create more favourable conditions with respect to the geometry of the lattice. This assumption is confirmed by Auger investigations made by us.

In case of a molar ratio of 400, the rate of deposition is always distinctly greater (20 to 50%) than in case of a molar ratio of 3200 in the temperature range examined. This is reflected in the structural perfection which is lower in case of a molar ratio of 400.

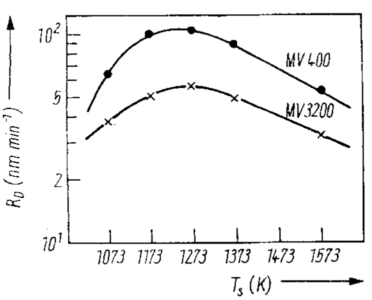

Fig. 3. Rate of deposition R_D as a function of the substrate temperature T_S

Only at temperatures above 1273 K the influence of the orientation of the substrate on the structure of the AlN layers becomes apparent in a distinct manner. Here again there exists a connection with the rate of deposition which is higher by 25% in case of (100) Si than in case of (111) Si at a temperature of 1573 K.

The higher perfection reached on (111) Si, observed also in [8], is certainly favoured by low R_D values. Moreover, the (111) Si surfaces are slip planes. A high mobility of dislocation offers favourable conditions for intergrowth.

4. Summary

The present work investigates the influence of the most important experimental parameters on the real structure of AlN layers. The preparation of the AlN layers is effected by the MOCVD method on the basis of NH_3 and TMA (111) Si and (100) Si served as substrate materials.

The substrate temperature and its influence on the reaction are of decisive importance for the structural perfection of the layers. These show a good texture at a temperature of 1073 K. A further increase of the substrate temperature brings on a deterioration of the structure, but above 1473 K the structure again becomes distinctly better. At a temperature of 1573 K, we succeeded in producing single-crystalline AlN layers.

The deterioration of the quality of the surface occurring in case of increasing substrate temperatures is to be ascribed essentially to the homogeneous reaction.

The course of the reaction and thus the rate of deposition are considered in connection with the perfection and the surface quality of the layers.

The influences exerted by the orientation of the substrate become apparent only above 1273 K. Single-crystalline layers were obtained only on (111) Si and in case of a molar ratio of $NH_3/TMA > 400/1$.

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