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Indium nitride crystals with flower-like structure

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Preparation of indium nitride at atmospheric pressure has been examined by means of halide chemical vapour deposition; from the SEM observations of the crystals deposited onto an Si(100) substrate it was found that they showed flower-like structure.

Science and technology of three-dimensionally ordered materials have led to great excitement during the last decade. Amongst them, subjects dealing with very tiny objects are of immense academic interest. There has already been much progress in the synthesis, assembly and fabrication of three-dimensionally ordered materials,[1] and also potential applications of such materials to a wide variety of technologies, such as glancing angle deposition (GLAD)[2] and template selectivity growth.[3]

Recently, we examined the preparation of InN utilising a reaction of gaseous $InCl_3$ and NH_3 under atmospheric pressure (AP-HCVD).[4] It was found that when the substrate temperature was in the range 723–823 K that indium nitride crystals with unusual flower-like structures were formed. Here, we report the results of the investigation on indium nitride with flower-like structure grown on an Si(100) substrate by means of atmospheric pressure halide chemical vapour deposition.

A horizontal hot-wall quartz reactor was used for the preparation of InN. The nitride was deposited onto an Si(100) substrate under atmospheric pressure. The growth temperature was measured just under the substrate stage with an error limit of 0.2 K. The substrate used was an n-type Si(100) wafer with a resistance of $5 \times 10^{-4}\ \Omega$ m. $InCl_3$ in a source boat was evaporated at a temperature of 523 K, and supplied to the growth zone of the reactor by purified N_2 carrier gas. NH_3 was also supplied to the growth zone of the reactor by purified N_2 carrier gas. The partial pressures of $InCl_3$ and NH_3 were adjusted independently by varying the flow rates of N_2 carrier gas. The partial pressure of NH_3 was varied in the range 9.3×10^3–2.8×10^4 Pa at a constant partial pressure of $InCl_3$ of 4.7×10^2 Pa. The purity of the N_2 and NH_3 gases (Air Liquide Co. Ltd) are 99.9999% and 99.9995%, respectively. The purity of $InCl_3$ (Kojundo Chemical Lab. Co., Ltd) is 99.99%. The

Table 1 Typical growth conditions

Substrate	Si(100)
$InCl_3$ partial pressure/Pa	4.7×10^2
NH_3 partial pressure/Pa	2.8×10^4
Carrier gas	N_2
Total flow rate/m^3 s^{-1}	1.25×10^{-5}
Growth temperature/K	823

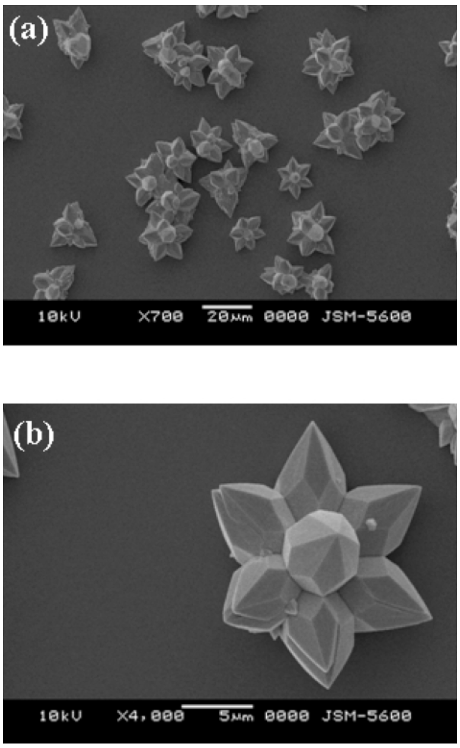

Fig. 1 SEM image of the InN grown under condition of Table 1. (a) magnification ×700, (b) magnification ×4000.

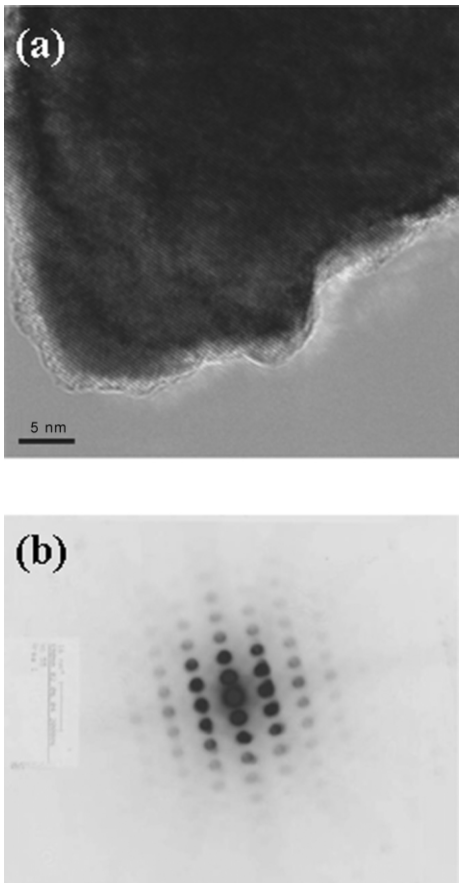

Fig. 2 (a) TEM and (b) electron diffraction images of an InN crystal.

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crystallographic structure of the deposited InN was examined by a Rigaku Rint 2000 X-ray diffractometer. Their crystallinity was evaluated by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and electron diffraction.

The crystal structure and orientation of the as-grown films were examined by means of the X-ray diffractometry (XRD). Only the diffraction peaks of InN and the Si substrate were observed. The observed diffraction lines reveal that the hexagonal structure is the only crystalline phase, exhibiting sharp and symmetric reflections for basal (002) and (004) planes, and broad and asymmetric reflections for the non-basal (101), (103) and (110) planes.[5] The lattice constant was estimated to be 0.570 nm utilising the observed (002) diffraction, which implies that the obtained lattice constant is similar to the reported value of 0.57033 nm for the polycrystalline InN powder.[5]

Fig. 1(a) and (b) show SEM micrographs of the InN grown on the Si(100) substrate under the conditions listed in Table 1. At a glance of the SEM micrographs in Fig. 1(a), it is immediately noticed that the deposited InN has an interesting geometry with flower-like structure. The enlarged SEM micrograph of a flower-like InN arrangement shows that it consists of six petal crystals and a style crystal (Fig. 1(b)). One can see that there is a sixfold axis along the style, and that the respective crystals constituting the petals and a style are staggered hexagonal bipyramids. The flower-like InN was formed only when the substrate temperature was kept at 823 K. As for the partial pressure of NH_3, it was found that the geometry with flower-like structure was hardly affected in the range of $p(NH_3) = 9.3 \times 10^3–2.8 \times 10^4$ Pa at a constant partial pressure of $InCl_3$ of 4.7×10^2 Pa examined in this study.

A representative TEM micrograph and an electron diffraction pattern of the indium nitride crystals are shown in Fig. 2(a) and (b), respectively, in which the top of the one of the staggered hexagonal bipyramidal crystals was measured. In Fig. 2(a), it is seen that there are a number of lines with a constant spacing of 0.2853 nm, which is assigned to the 002 spacing based on both the JCPDS data[5] and the XRD results mentioned above. Also, an intense spot pattern appearing in the electron diffraction image is a characteristic pattern of hexagonal structures (Fig. 2(b)). This means that the staggered hexagonal bipyramidal crystals are single crystals of InN.

In summary, we have found that InN with flower-like structure was deposited onto an Si(100) substrate by AP-HCVD upon reaction of $InCl_3$ and NH_3. Each crystal constituting the flower is a single crystal with staggered hexagonal bipyramidal structure. The formation of such highly unusual crystals is sensitive to the substrate temperature.

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