PAPER • OPEN ACCESS

Separation of stress-free AlN/SiC thin films from Si substrate

To cite this article: A V Redkov et al 2016 J. Phys.: Conf. Ser. **741** 012034

View the article online for updates and enhancements.

Separation of stress-free AlN/SiC thin films from Si substrate

A V Redkov[1,2], A V Osipov[1-3], I S Mukhin[1,3] and S A Kukushkin[1-4]

[1]St. Petersburg Academic University, St. Petersburg 194021, Russia
[2]Institute of Problems of Mechanical Engineering RAS, St. Petersburg 199178, Russia
[3]ITMO University, St. Petersburg 197101, Russia
[4]Peter the Great St. Petersburg Polytechnic University, St. Petersburg 195251, Russia

Abstract. We separated AlN/SiC film from Si substrate by chemical etching of the AlN/SiC/Si heterostructure. The film fully repeats the size and geometry of the original sample and separated without destroying. It is demonstrated that a buffer layer of silicon carbide grown by a method of substitution of atoms may have an extensive hollow subsurface structure, which makes it easier to overcome the differences in the coefficients of thermal expansion during the growth of thin films. It is shown that after the separation of the film from the silicon substrate, mechanical stresses therein are almost absent.

1. Introduction

Nowadays scientists are actively searching for new ways to obtain high quality semiconductor thin films and heterostructures, in particular, gallium nitride that is used in LEDs and other electronics [1,2] and aluminum nitride. For the growth of such films sapphire and SiC substrates are used mainly [3-5], less often silicon or other substrates, but all of these substrates has some disadvantages. Sapphire has very low thermal conductivity and is dielectric, so the additional conducting contacts should be added. SiC is more suitable, because its thermal conductivity is high and close to the conductivity of the copper. In addition, the lattice parameters of SiC and AlN/GaN are much closer to each other [6]. Since SiC substrates are already widely used in practice, it allows one to suggest, that the use of SiC as a substrate for the growth of AlN/GaN heterostructures looks very promising. Nevertheless, a significant drawback of using single-crystal SiC in the manufacturing of devices is the high price of SiC substrates of good quality and desired polytype. One of the promising types of substrates which solves these problems are substrates of nano-SiC/Si, grown by substitution of silicon atoms to carbon atoms [7] directly in the silicon substrate. In fact, the authors of that work for the first time in world practice implemented method of successive substitution of carbon atoms instead of native silicon atoms right inside the substrate without destroying its crystalline structure. The method resembles the "genetic synthesis" of protein structures in biology. The quality of SiC thin film obtained by this method are far superior than quality of films grown on silicon substrates by conventional methods. The method is simple and cheap, and the presence of pores beneath the surface of SiC allows to overcome the mechanical stresses resulting from the lattice mismatch. Thus the study of the method [7] is prospective for the development of thin film technology.

2. Experiments

For the experiment we have used silicon substrate with orientation <111>, which was subjected to growth process of SiC film [7] at 1250C, CO pressure 2 Torr. The time of the growth was 20 min. According to ellipsometric data, the total thickness of the film was ~100nm. Then in HVPE process on this substrate we have grown AlN film of 18 mkm thickness at 1080C and NH_3/H_2 ratio 1:5. Then, the sample was subjected to chemical etching in a solution containing a mixture of nitric and hydrofluoric acids and water, 4:1:5, respectively. Such a mixture effectively removes silicon [8]. Etching occurred for 15 minutes at 20°C, after which the film has been completely separated from the substrate. After that the resulting film was studied using a Raman microscope Witec Alpha 300 R and the scanning electron microscope.

3. Results and discussion

After 1-5 min of immersion into the etching solution one can note the beginning of the etching with the naked eye. It becomes apparent that the edges of the thin film start to peel off from the substrate. Then over time, the etching front moves towards the center of the substrate, until the film becomes completely separated from the silicon substrate. Probably such behavior is caused by a large number of pores and channels in the subsurface area of silicon, which are quickly filled with acid. This allows acid to penetrate deeper fast, and to etch only small amounts of silicon, which is located between the pores. There is no need to dissolve all of the silicon substrate as a whole, so the separation process takes only a few minutes. Usually this time at room temperature takes 10-20 minutes. The schematic view of this process is presented in Figure 1.
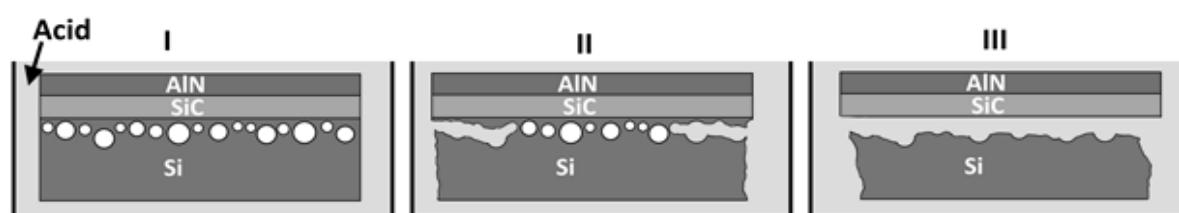

Figure 1. AlN/SiC film delamination mechanism in acid. I - the initial stage. II- etching silicon substrate under the AlN/SiC from the edges towards the center. III - the separation of the film from the substrate

Scanning electron microscope images of AlN/SiC thin film cross-section and "backside" of SiC film are presented in Figure 2. One can clearly see the developed structure of the channels with the thickness of 2 microns remained after etching below the surface of the silicon carbide film. The channels have thin walls about tens of nanometers in thickness and are hollow inside. It can be expected that the ratio of surface area to volume of the material in such a system is quite large. Since the channels have not been dissolved by acid, one can assume that during the growth of SiC film, the surface of the pores formed in the silicon substrate is also being covered by silicon carbide, that was suggested in [7], and channels remaining after etching are the carcass of these pores.

Comparing pores which were visible on cross-section of the SiC films cleaved in [7] and Figure 2, one can conclude that pores in SiC are not enclosed objects, but rather part of the channels which originate at the surface, and have a dendritic structure, which develops deeper into the silicon. The presence of such system of hollow channels can lead to the fact that the mechanical stress arising from the difference in thermal expansion coefficients and lattice parameters mismatch will be much less than in continuous films, because the contact area of the film and the substrate becomes much less. In addition, the presence of pores in silicon subsurface region creates an effective "mechanical decoupling" between the film and the Si substrate.
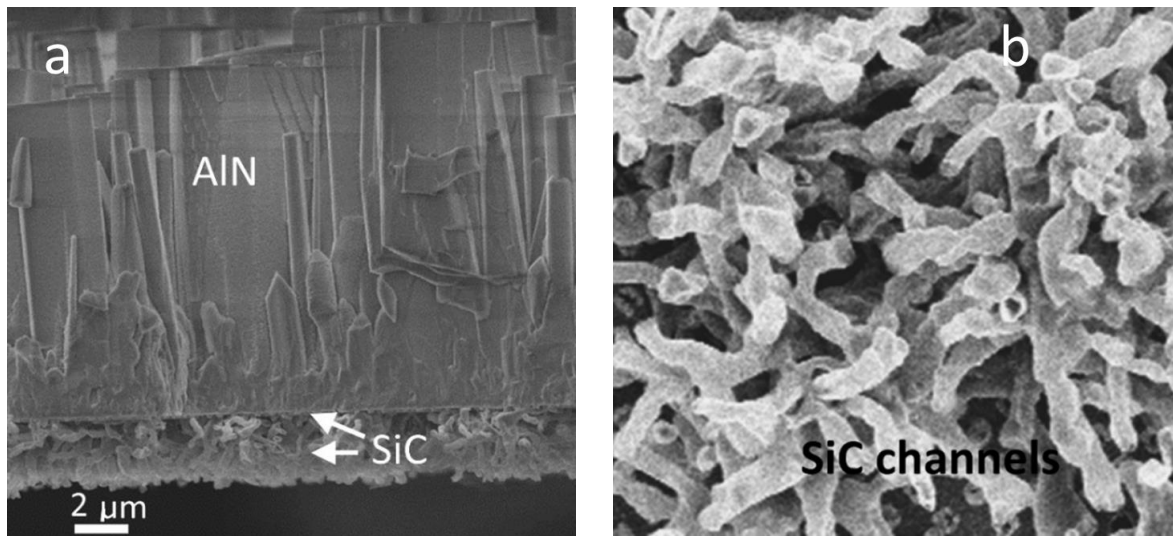
Figure 2. SEM image of the cross-section of AlN/SiC film (a) and the "backside" view of SiC substrate.

The Raman spectra of the initial AlN/SiC/Si structure and AlN/SiC thin film are presented in Figure 3. One can note that the silicon band 521 cm^{-1} after etching totally disappeared, and spectra contains only bands of AlN and SiC. Thus the silicon has been fully dissolved by acid. Inset in Figure 2 demonstrates the position of AlN band 650-665cm^{-1}. The center of this band after etching has been shifted to 657 cm^{-1}, which means that mechanical stress is almost absent in the film [9].
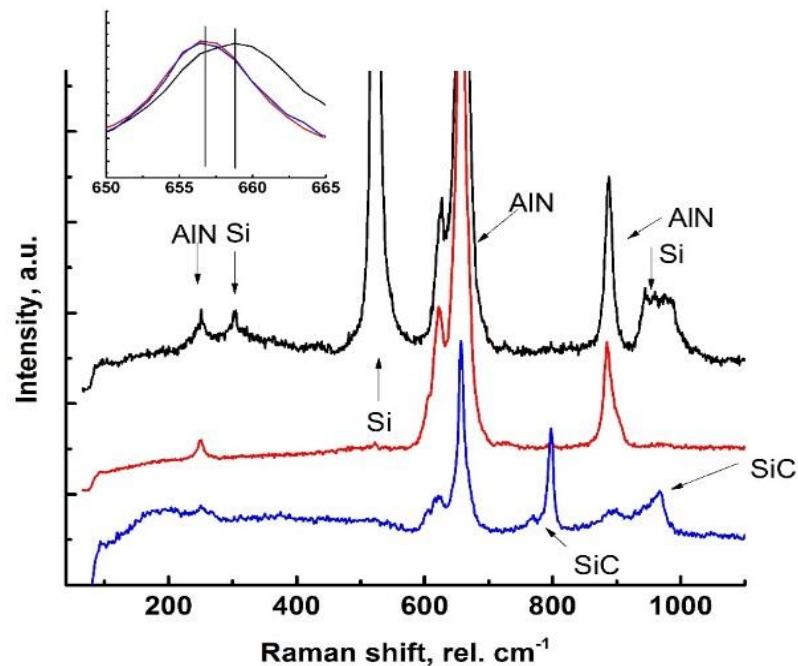

Figure 3. Raman spectra of initial AlN/SiC/Si structure (top graph) and AlN/SiC film from the AlN side (middle) and from SiC side (lowest one).

5. Conclusions

In this work we consider the mechanism of separation of AlN/SiC thin film from Si substrate by chemical etching. It is shown that the presence of SiC buffer which has extensive subsurface porous structure allows to separate thin films or heterostructures from silicon substrates effectively and in a short time without using of sophisticated technical means. This study allowed for the first time to explicitly see the SiC surface grown by substitution of atoms, from the "reverse" side, without the substrate. We identified the structure of pores and channels that appeared in the subsurface area of silicon during the growth of silicon carbide. Such a porous system allows one to "decouple" the mechanical stresses in thin film from the substrate and substantially reduces them, thus leveling the difference in lattice parameters and thermal expansion coefficients. It was shown that after separation from the substrate the mechanical stresses in AlN/SiC thin film are almost completely relaxed. The developed method of the heterostructure separation from Si substrate allows one to transfer it to another substrates, and creates unprecedented conditions for the development of micro and nano-electronics. The proposed method of separation of high-quality epitaxial layers and heterostructures of wide bandgap semiconductors grown on silicon nanocarbide may find applications in the study of various properties of free-standing structures. They can also be used for growth of low-defect crystals. For example, one can use such AlN/SiC/Si substrate as a seed for growth of thick layers of AlN or GaN and then separate them from the silicon substrate.

References

[1] Fujii T, Gao Y, Sharma R, Hu E, DenBaars S and Nakamura S 2004 *Appl. Phys. Lett.* **84** 855-57
[2] Khan M, Kuznia J, Bhattarai A and Olson D 1993 *Appl. Phys. Lett.* **62** 1786
[3] Yoshida S, Misawa S and Gonda S 1983 *Appl. Phys. Lett.* **42** 427-9
[4] Boćkowski M et al 2005 *J. Cryst. Growth* **274**, 55-64
[5] Rizzi A et al 1999 *J. Vac. Sci. Techol. B* **17** 1674-81.
[6] Kukushkin S, Osipov A, Bessolov V, Medvedev B, Nevolin V and Tcarik K 2008 *Rev. Adv. Mater. Sci* **17** 1-32
[7] Kukushkin S and Osipov A 2014 *J. Phys. D: Appl. Phys.* **47** 313001.
[8] Nikolaev A, Nikitina I, Zubrilov A, Mynbaeva M, Melnik Y and Dmitriev V 1999 *MRS Proc.* **595** F99W6-5
[9] Freitas J, Culbertson J, Mastro M, Kumagai Y and Koukitu A 2012 *J. Cryst. Growth 350* 33-7