TEM study of the AlN grain orientation grown on NCD diamond substrate

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Abstract

Piezoelectric AlN layer grain orientation, grown by room temperature reactive sputtering, is analyzed by transmission electron microscopy (TEM). Two types of samples are studied: (i) AlN grown on well-polished NCD (nanocrystalline diamond) diamond, (ii) AlN grown on an upside down NCD layer previously grown on a Si substrate, i.e. diamond surface as smooth as that of Si substrates.

The second set of sample show a faster alignment of their AlN grain c-axis attributed to it smoother diamond free surface. No grain orientation relationship between diamond substrate grain and the AlN ones is evidenced, which seems to indicate the preponderance role of the surface substrate state.

1. INTRODUCTION

Diamond properties make it a promising alternative to silicon for the manufacturing of future MEMS devices [1]. Previous studies showed the potential of diamond as active acoustic substrate for surface acoustic wave filters (SAW) [2]. The growth of AlN films on diamond can be performed by several methods. Pulsed DC reactive sputtering can be performed at room temperature which is a first step in reaching a technological compatibility with Si. This implies a polycrystalline micro- or nano-structure [3] in which AlN/diamond lattice relationship is still not clear for sputtered layer. In the other hand, in the case of MOVPE grown, a direct relationship seems to help the AlN grain orientation in a columnar grain configuration [4]. Indeed, K. Hirama et al [5] showed a direct relationship between the diamond (111) plane and the AlN (0001) at the interface. More detailed, other authors showed also by high resolution transmission electron microscopy (HREM) a relatively fast alignment of the c-axis, less than 0.5μm [6] for MOVPE growth while nearly 1μm is necessary for sputtering related process [7].
Thus, temperature seems to play an important role and for the room temperature case, interface state is more important than diamond/AlN relationship. To show this peculiar feature, two samples with different interlayer roughness are compared.

2. EXPERIMENTAL

In the two samples have been changed the underneath diamond surface state on which AlN is grown by sputtering [8]: (i) for sample A, the diamond is grown by microwave plasma chemical vapor deposition (MPCVD) on a commercial Si substrate and then polished before pulsed DC reactive sputtering AlN deposition at room temperature; (ii) sample B follows the same routine, the only difference is that the diamond layer is "inverted" and glued on Al_2O_3 before growing the AlN. Then the AlN is grown on a much smoother surface. To ensure the up-side down operation and the further glue on Al_2O_3 substrate, the NCD layer is thicker in sample B than that of sample A as it can be observed on the TEM observations.

Concerning the latter, HREM mode, diffraction contrast mode (CTEM) and selected area electron diffraction (SAED) patterns, are recorded using a JEOL 2010F (FEG gun) and a JEOL 1200EX electron microscopes working at 200 and 120kV acceleration voltage respectively. The sample preparation is achieved in a FIB-dual beam following a standard lift-off routine. Lamella thickness is estimated to stand around 80 nm.

3. RESULSTS AND DISCUSSION

The purpose of the present study is to compare the grain crystalline orientation between both approaches. For SAW filters, the induction of a surface wave within the diamond is fundamental. The key parameters to achieve it are AlN layer thickness and it crystalline orientation. The latter is an important factor to optimize its piezoelectric effect [9]. It c-axis of the hexagonal lattice should be perpendicular to the AlN/diamond interface. To evaluate it two techniques are here used allowing a very local evaluation: SAED patterns and HREM observations where fast Fourier transformation (FFT) allows to obtain similar information than that of SAED. Fig. 1a shows sample A grain configuration. SAEDs in CTEM correspond to the interface, mid-layer and top-layer locations. In the region 1, the SAED reveal the three materials diffraction signature, the Si substrate at the (110) pole (i.e. the electron beam oriented in the [110] direction respect to the Si lattice), the NCD (a=0.356nm) and the AlN (a=0.311nm) both with a circular shape diffraction spots that corresponds to polycrystalline behavior. If grains are randomly oriented, then a circularly shaped diffraction pattern is expected. In region 1, this is clearly the case but the more the aperture is located close to the surface, the less misoriented are the AlN grains. Indeed, at the surface, the pattern is close to correspond to the $(01\bar{1}0)$ pole as previously reported [10].

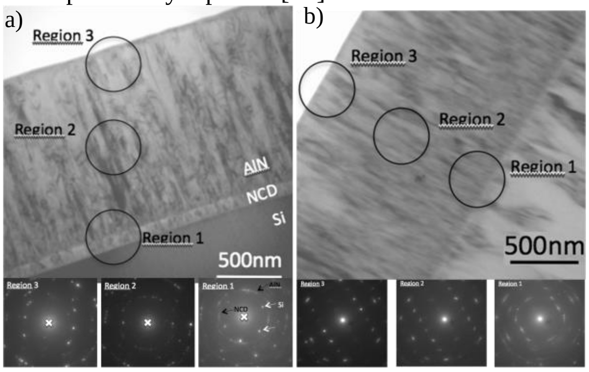

Fig. 1: CTEM observations of sample A (a) and B (b). The SAED are recorded on the region indicated in the bright field (BF) observations where, for sample A, the two AlN and diamond (NCD) layers are well revealed on the Si substrate (see white labels). In sample B, as the AlN is grown on a thicker diamond layer, and thus only these two materials are observed by SAED. The aperture location is indicated in black and it diameter is around 400nm.

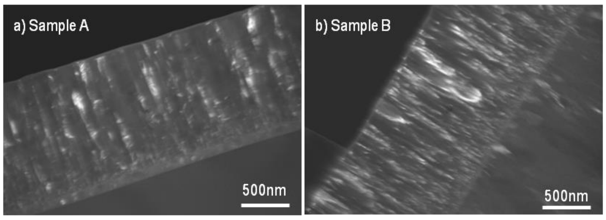

Fig.2: a) and b) show dark field (DF) observations of sample A and B respectively. The micrographs are recorded near the {0110} pole spot positions. Thus each micrograph evidences grains with similar orientation. Sample B columnar grains are showed to be longer, some of them well oriented from the interface.

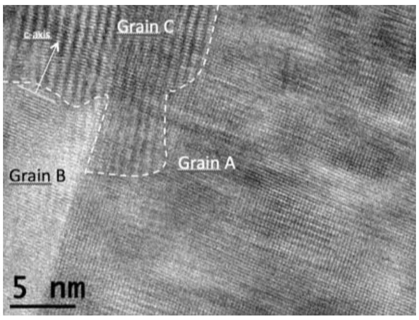

Fig.3: HREM observation of sample A showing three grains. Grain A and B are slightly rotated around the c-axis as the atomic column are visible only in grain A at the grain boundary. Grain C is located underneath grain A and B. His c-axis is slightly rotated respect to the perpendicular of the interface (that correspond to c-axis of grain A and B) as indicated by the Moiré effect.

To compare the sample A behavior to that of AlN grown on an up-side down smooth diamond layer, same observation routine is shown in Fig.1b. The first difference is that the SAED at the interface show grain better oriented as a full circle is not observed but only less than 10º arcs circles corresponding to each diffraction plane family. Indeed in Fig.2, sample B grains are shown to be thinner and longer than those of sample A. In sample B, some grains, generated just at the interface, show their c-axis nearly (within the angle defined by the DF aperture) perpendicular to the interface and more grains seem to be well oriented from those dark field (DF) observations while sample A seem to have larger grain but worse oriented.

Finally in Fig.3, a HREM observation shows a grain boundary between two grains with their lattice slightly rotated around the c-axis. Indeed, the high resolution is observed only in grain A while only the c-planes are observed in grain B. The third grain is located underneath the two others.

4. CONCLUSION

The present work, based on TEM grain analysis of the AlN grown, show the importance of the AlN/diamond interface state for the “self-orientation” of the lattice c-axis of AlN grains. The smoother is the interface, the more grains are well oriented in the first steps of the AlN grow. Indeed, the up-side down approach permit a diamond free surface as smooth as that of commercial Si substrates, i.e. much smoother than polished diamond, and reveal thinner but well oriented grain configuration. In contrast to that previously observed on MCD (microcrystalline diamond) [10] where two grain families were revealed, here, growth on NCD, only one principal orientation is observed by the SAED patterns with some slight disorientation around the c-axis as observed by the arc-shaped SAED spots and by the HREM grain boundary micrograph.

5. REFERENCES

[1] O. Auciello S. Pacheco, A.V. Sumant, C. Gudeman, S. Sampath, A. Datta, R.W. Carpick, V.P. Adiga, P. Zurcher, Ma. Zhenquiang, Y, Hao-Chih, J.A. Carlisle, B. Kabius, J. Hiller and S. Srinivasan, IEEE Microwave Mag. 8, 61 (2007).

[2] Satoshi Fujii, Phys. Status Solidi A. 208, 1072 (2011).

[3] V. Mortet, O. Elmarzria, M. Nesladek, J.D’Haen, G.Vanhoyland, M.Elhahiki, A. Tajani, E. Bustarret, E. Gheeraert, M. D’Olieslaeger, P.Alnot, Diam. Rel. Mat. 12, 723 (2003)

[4] Y. Taniyasu and M.Kasu, J. Cryst Growth **311**, 2825 (2009)

[5] K. Hirama, Y. Taniyasu, M. Kasu, J. Appl. Phys. 108, 013528 (2010)

[6] M. Imura, K. Nakajima, M. Liao, Y. Koide, H. Amano, Diam and Rel. Mat. 19, 131 (2010)

[7] J.G. Rodríguez-Madrid, G.F. Iriarte, D. Araujo, M.P. Villar, O.A. Williams, W. Müller-Sebert, F. Calle, Mat. Letters 66 339 (2012)

[8] G.F. Iriarte, J.G. Rodriguez, F. Calle, Mater Res Bull, 45, 1039 (2010)

[9] M. Benetti, D. Cannata, F. Di Pictrantonio, E. Verona, IEEE Trans. Ultrason. Ferroelectr. Freq. Control, 52, 1806 (2005)

[10] F. Lloret, D. Aráujo, M.P. Villar, J.G. Rodriguez-Madrid, G.F. Iriarte, O.A. Williams, F. Calle, Microelectron. Eng. 112, 193 (2013).