Comparative XPS Analysis of N Atoms in Graphite and Single Crystal Diamond in Studies of Chemical/Physical/Electronic Properties in SCD Implanted with Low Energy N^+ Ions

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1. Introduction

Published XPS analysis of materials with C-N bonded atoms (e.g., g-CN, N in poly/single crystalline diamond) revealed N1s peaks at 398-399 eV (N1), 399-400.5 eV (N2) and 401-403 eV (N3), with N1/N2 peaks assigned to C-N bonds and N3 peak assigned to N atoms bonded to O, or NH. New XPS analysis of N atoms implanted in C-materials, to be discussed in the NDNC's presentation, involved *in situ* cleaning of C-based surfaces, eliminating contaminants, before XPS analysis of *in situ* N atoms implantation in C-materials. The new XPS analysis revealed that the N3 peak relates to N-C bonded atoms (Fig. 1 (d)), The research to be discussed, involved N^+ ions implantation in Highly Ordered Pyrolytic Graphite (HOPG) and Electronic Grade Single Crystal Diamond (EGSCD). The *in-situ* N^+ ion implantation in EGSCD revealed new chemical/physical/electrical phenomena, revealing a structured a-C subsurface region (Fig. 1 (e), characterized by N1s peaks, and insulator to electrical conductor transformation of 5-8 nm diamond layer, both effects opening potential pathways for research relevant to diamond-based micro-/nano-electronics.

2. Investigation of Associated Chemical/Physical/Electronic Effects in Low Energy Nitrogen Atoms Implanted in SCD

Most prior research on N^+ ions implantation in diamond involved ion energies in the 10-100s KeV to MeV, resulting in relatively deep N atoms insertion under the surface, inhibiting production of subsurface Nitrogen-Vacancy (NV) centers investigated for application to quantum and biosensors-based devices. Thus, the R&D described here, focused on investigating low energy N^+ implantation in EGSCD and HOPG's subsurface regions, followed by *in situ* XPS analysis, to make critical comparison with prior research on N atoms implantation in C-materials. N atoms were implanted in EGSCD's and HOPG's surfaces via *in situ* 5,000 eV N_2^+ ions (2,500 eV into material, upon surface cracking), directed at 45˚ incidence to sample's surface; e-beam surface bombardment was used to avoid surface electrical charging by N^+ ions-induced electron emission. Surface layer peeling of HPGO and immediate insertion in XPS evacuated chamber provided atmospheric contaminants-free material (Fig. 1 (a)). Ar-Cluster-Ion-Beam bombardment of EGSCD eliminated O, C, N from atmospheric adsorption on sample's surface, and without incorporating Ar atoms in the subsurface layers (Fig. 1(b)). The shift of the C1s sp^3- diamond peak to 282.7 eV, from the real 285.5 eV peak in diamond, is due to insertion of neutralization electrons. N^+ ion implantation in HPSGO and EGSCD resulted in a low B.E. C1s peak at 284.3 eV, associated with C atoms with sp^2 bonds configuration on the top surface and a-C regions. The high B.E. C1s peak at ~285.9 eV is associated with C atoms with sp^3 diamond bonds, present in the a-C and deep EGSCD regions. The top surface and a-C regions exhibit a nanostructured arrangement (Fig. 1 (c)). The N1s signal relates to photoelectrons ejected from the surface and a-C regions and is formed by three peaks N1 (398.2 eV), N2 (400 eV) and N3 (402.4 eV) (Fig. 1 (d), all related to C-N or N-N chemical bonds. The N1s peak N3 is not due to N-O, N-H or $N\text{-}CH_x$ bonds. The N1 peak and N3 peak are related to weak C-N or N-N bonds linked to C atoms with sp^2 bonds, while the N2 peak appears as a stronger C-N bond involving C atoms with sp^3 bonds, characteristic of diamond.

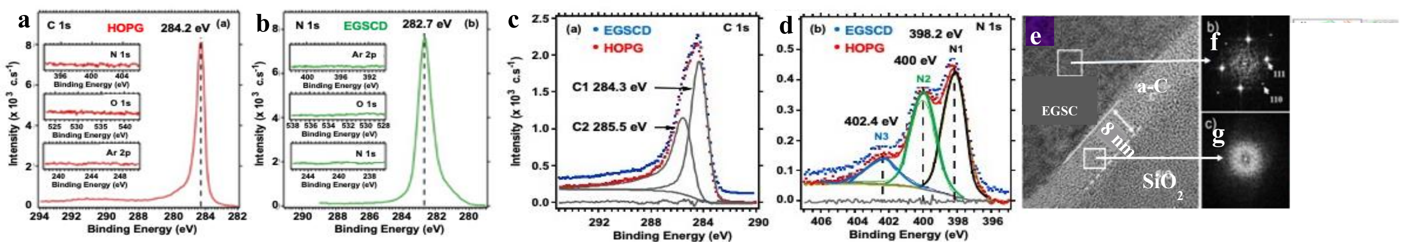

Figure 1.XPS: a) HPGO with delaminated surface layer (no impurities); b) EGSCD cleaned with ArCIB (no impurities); c) C1s peaks and d) N1s peaks in N^+ ion implanted HPGO and EGSCD, the latter showing all N1, N2, N3 peaks correlated to C-N or N-N bonds and NO impurities; e) HRTEM of N implanted EGSCD, showing the a-C layer with sp^3 C bonds; f) and g) show the FFT patterns from the EGSCD (diamond) and the a-C layer (nanostructured C, respectively (The SiO_2 layer was grown on the EGSCD surface to protect it during the FIB process to prepare the HRTEM sample.