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Polishing of Black and White CVD Grown Polycrystalline Diamond Coatings

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Abstract: Microwave plasma CVD growth can produce black and white varieties of polycrystalline diamond (PCD), depending on their as-grown purity. These two types of PCDs have been polished by mechanical and chemo-mechanical polishing respectively. It has been observed that initial roughness of 2.21µm for white PCD can be brought down to 175 nm after 70 hours of combined polishing, whereas, 85 hours of combined polishing could bring down the high initial roughness of 11.2µm for black PCD down to 546 nm at the end. Although, the material that was removed during polishing was higher for the black variety of PCD but it had lower polishing rate of 4nm/hr than white PCD (13nm/hr) during chemo-mechanical polishing. Such differential polishing rate was due to harder top polished surface of the black diamond than the white diamond. The nanoindentation study on the polished PCD surfaces revealed that the black PCD has a final nanohardness of 32.58±1 GPa whereas the white variety PCD had a polished surface nanohardness of 28.5±2 GPa. More conversion of diamond surface into harder amorphous sp3 than softer graphite during polishing action may have resulted such slow rate of anisotropic polishing for black diamond than white diamond.

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1. INTRODUCTION

1.1. Diamond Polishing – Relevance of the Research Topic

Polishing of diamond is very important technology [1-8]. Being the hardest known material, it is also the most difficult process of polishing. Diamond has been polished by laser, ion beam, hot metal plate, chemo-mechanical etc. techniques [9, 10]. It has been observed that there are faster techniques for microscopic polishing but generally authors' earlier work on polishing [11] found to be very slow process of diamond material removal. The typical removal rate varies from few microns per hour for chemo-mechanical polishing (CMP)/hot metal plate/electrical discharge machining and upto tens of microns per hour for mechanical polishing or reactive ion beam etching [18]. But it has been observed that laser polishing can remove hundreds of microns of diamond material per hour [12]. Unfortunately laser polishing is localised and scanning is required to polish large areas, whereas mechanical or CMP polishing can effectively cover wide areas. Ion beam and laser polishing are non-contact methods but the machines are also costly in comparison to mechanical or chemo-mechanical polishing machines.

1.2. Diamond Polishing Mechanisms – Literature Survey

CVD growth of diamond is columnar in perpendicular to the substrate surface which leads to very rough as-grown topography with high asperities and deep valleys [13]. In order to planarise or even out such rough surfaces, it is necessary to remove the protruding edges of the diamond crystals [14]. Diamond is the hardest known material which poses great difficulty in smoothening by mechanical abrasion only. There are techniques by which diamond crystals can be removed by chemicals, high temperature, high pressure, electrical arcs, ion beams in order to create surfaces devoid of uneven topography [18-21]. Mechanical removal of diamond material from uneven top surface can be performed by equally hard diamond grit abrasives only, or abrasive free dynamic friction polishing [15, 16] which actually uses thermo-chemical principle. Two hard materials in contact under pressure will result micro-cracks in the tip regions which will dislodge asperities. Whereas, in the presence of oxidising agents [17], the carbon atoms can form CO or CO_2

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and thereby remove diamond from the rough top surface by chemical means [18-20]. When thermal assistance is provided alongwith the chemicals, such oxidation reactions take place more easily [21]. Generally such abrasives and chemicals are added with application of vertical load and rotational shearing forces, which are high enough to cause the carbon atoms to dislodge covalent bonds from slurry adatoms. It has been observed that plasma etching/sputtering [22-25] (removal of carbon atoms by breaking C-C bonds with reactive plasma ion species or momentum transfer by heavy incident ions) of rough diamond surface with oxygen or CF_4/SF_6 plasma [26] makes the sharp crystal edges into obtuse ones which are basically graphitised and then subsequently removed by mechanical abrasion. Surface graphitisation is the primary phenomenon in creating smoother diamond surfaces. It is known that graphite is sp^2 bonded with weak van-der waals forces in between layers, whereas, diamond with sp^3 bonds are very strong to break and dislodge from rough surface. So to planarise diamond surface it is easy to first transform the diamond into graphite which afterwards can easily be removed by shear mechanical action. Graphite is the stable form of carbon under ambient conditions. So, if the energy barrier is crossed (by temperature, pressure rise), metastable diamond can transform into stable graphite phase. One of the easy methods of such conversion is incidence of laser onto diamond. Laser diamond interaction can be described in two ways [27-29]. One is the laser ablation where high temperature of 4000^{o}C causes to form liquid carbon which then sublimes and redeposited as graphite at around 1700^{o}C. High laser power causes the temperature of the diamond sub-surface layer also to get heated up and thus converted into graphite. The other simultaneous process is the atmospheric oxidation of the carbon liquid into CO_2 or CO gases. Similarly, ion beam polishing or electrical discharge machining are also principally the same procedures of melting/evaporation of the diamond surface at high temperature and simultaneous subsurface graphitisation with atmospheric/plasma oxidation. There is a well known problem of carbon atom diffusion into steel substrates during high temperature CVD growth of diamond films [30]. Such carbon diffusion problem has been overcome by inserting barrier layer [31], but such property of hot cast iron plate has widely been used [32] for high temperature diffusion based carbon material removal into the sliding plates [33] alongwith favourable catalytic graphitisation [34, 35] and oxidation. However, it is associated with surface contamination problem due to formation of metal-carbon complexes.

1.3. Work Objective

There exists polishing anisotropy in diamond [36-38]. Polishing takes place over preferential planes and along easy crystallographic directions. It has been observed that single crystal diamond (SCD) can easily be polished on (110), (100) and (111) planes in the decreasing order of ratio 1:0.6:0.1, along <100> direction, whereas, <011> direction is difficult to polish [39]. Since polycrystalline diamond (PCD) grains are randomly oriented, it is difficult to planarise over large area. It is simulated [40-42] that "soft directions" easily convert diamond into non-diamond phases whereas the sp^3 carbon atoms along "hard directions" are difficult to transform under mechanical pressure, which causes distortion and scratching of perfectly cubic carbon to form amorphous sp^0, sp^1, sp^2, sp^3 hybridisation and well arranged sp^2 carbon phases. The polishing directions, along which more sp^2 is created than amorphous sp^3 under stress, favours more material to remove.

The quality of diamond is also critical in determining the polishing rate. Diamonds are being grown into various grades, depending on the presence of defects inside their lattices. Poorer quality diamonds are black opaque in nature due to inherent defects present, whereas, better quality diamonds can be grown into translucent or transparent grade due to less number of CVD growth defects present. But such black and white as-grown diamonds need to be polished for any engineering applications. It is expected that optical grade diamond will be difficult to planarise in comparison to thermal grade diamond coating. Although there are reports of polishing anisotropy in SCD and PCD as discussed above but observation of differential polishing rates in between different grades of CVD grown diamond has so far not been reported. In the present work, authors investigated the relative easiness of polishing between two grades of diamond (black and white), as it is not available in the literature so far [43].

2. MATERIAL AND METHODS

Microwave plasma enhanced chemical vapor deposited (Table **1**) diamond of white variety (good quality with few defects) and black variety (poorer quality with more defects) were polished using mechanical polishing (MP) followed by chemo-mechanical polishing (CMP). Mechanical polishing was done using metal bonded diamond discs (Buehler, USA) with different diamond particle size (6 - 70 µm) at 200 RPM and 111 N normal load. Chemo-mechanical polishing was performed at 70ºC using a mixture of $K_2S_2O_8$ and H_2SO_4. The

Table 1: CVD Processing Parameters for Growing Black and White Diamond

CVD diamond sample	CVD Reactor	Precursor gases (sccm)	Pressure (Torr)	Microwave Power (kW)	Microwave Frequency	Substrate Temperature (^{o}C)
Black	ARDIS 100	H_2:CH_4:O_2=492.5:59.7:7.5	87	4.3	2.45 GHz	900
White	DT1800	H_2:CH_4=500:5	118	9	915 MHz	970-1070

Table 2: Parameter for Mechanical Polishing (MP) of CVD Diamond Wafer

SAMPLE	MECHANICAL POLISHING				
	REAGENT	LOAD (N)	DISC SPEED (RPM)	POLISHING DISC (µm)	TIME (hrs.)
Black	H_2O	111	200	6 to 70	55
White	H_2O	111	200	6 to 70	35

Table 3: Parameter for Chemo-Mechanical Polishing (CMP) of CVD Diamond Wafer

SAMPLE	CHEMO-MECHANICAL POLISHING						
	REAGENT	SAMPLE SPEED (RPM)	TIME (hrs.)	TEMP (^oC)	LOAD (N)	POLISHING DISC	SLIDING DISTANCE
Black	$K_2S_2O_8$ solution + H_2SO_4	200	30	70	89-133	Pure alumina	4-5 mm
White	$K_2S_2O_8$ solution + H_2SO_4	200	35	70	89-133	Pure alumina	4-5 mm

diamond samples were rubbed against pure alumina disc instead of metal bonded diamond discs during CMP. The parameter of mechanical and chemo mechanical polishing are shown in the Tables **2** and **3**.

Fischerscope nanoindenter (model H100-XYp) was used with tetrahedral Berkovich diamond indenter of tip radius 150 nm with semi-apex angle of 65.3^o for measuring hardness of the polished samples. The depth and load sensing resolutions were 1 nm and 0.2µN respectively, while it works with DIN 50359-1 standard. Data accuracy and reproducibility was ensured by calibrating the machine before each and every experiment with reference BK7 glass block. 25 individual measurements of nanohardness values are typically used for reported average data. At the beginning of each experiment the area function of the indenter tip is evaluated. Available machine software corrects experimentally obtained load (P) versus depth of penetration (h) data for tip blunting effect.

Roughness coherence scanning interferometer (CSI - Contour GTK, Bruker Nano GmbH, Germany) scan area was as large as 0.88×0.66 mm^2. Raman spectroscopy (STR500, Cornes Technologies, USA, originally from Seki Technotron, Japan) was used to characterise sp3 and non-diamond phases. Bench top scanning electron microscope (Phenom Pro X at 5 - 15 kV beam energy) with energy dispersive x-ray scan (EDAX) was used for surface morphology and elemental analysis of the polished and unpolished PCD surfaces.

3. RESULTS

Figure **1** is the typical scanning electron microscope images for CVD diamond (given pictures were captured before and after polishing of the white variety). They did not show any significant increase in grain size on polishing but there was concomitant decrease in rough surface topography. The SEM images indicate the presence of voids in between the flattened regions. Planarization is a phenomenon of bringing the height of adjacent diamond columns into same height, thereby increasing the apparent grain sizes on diamond surfaces. The SEM images after polishing (1b and 1c) are primarily bimodal distribution of grain sizes, i.e. there are predominant bigger grains with interstitial smaller grains. The polished surface has contrasting large flat regions which are separated from each other by grain boundaries. Such large flat region when viewed under higher magnification, it was found to be atomically flat with some pits present on the surface with grain pull out phenomenon during polishing. EDAX signals from such polished surface gives 100% carbon peak confirming no contamination from the contacting surfaces of the polishing consumables, like pad, disc or slurry. Both the white and black varieties of CVD diamond surfaces looked similar under SEM and EDAX scans.

Raman spectroscopy further confirms the purity of the polished white diamond surface (Figure **2**). It was found to be under tensile stress with a Raman peak downshift (3.77 cm^{-1}), but such shift was smaller than polished black variety diamond (4.74 cm^{-1}). Interestingly, the inherent stress present in the black diamond marginally increases (0.16 cm^{-1}) after polishing (Figure **3**). Raman spectroscopy also confirms that the polished surfaces are devoid of any non-diamond carbon which was clearly present for black variety of diamond before polishing (Figure **3a**). Amorphous carbon inclusions may not be completely absent for white quality diamond after polishing, as some undulations were still visible in the 1450-1600 cm^{-1} range (Figure **2**). The roughness and nanoindentation hardness results are listed in Table **4** with Figures **4** and **5**.

4. DISCUSSION

4.1. MP and CMP Polishing Rates for Black and White PCDs – CSI and SEM Analysis

Both the white and black diamond samples were first mechanically polished and then were chemo-mechanically

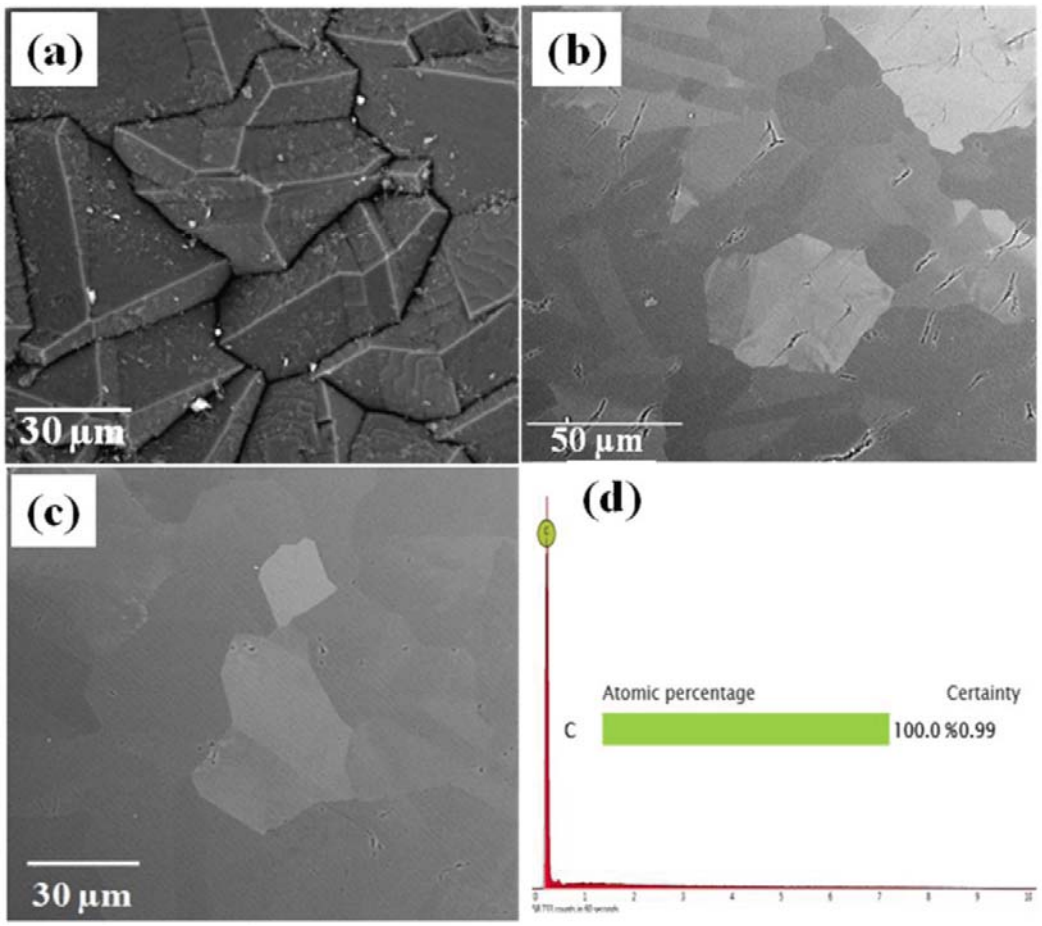

Figure 1: SEM images of white diamond (**a**) before polishing, (**b**) after chemo-mechanical polishing (low magnification), (**c**) after chemo-mechanical polishing (medium magnification), (**d**) area EDX.

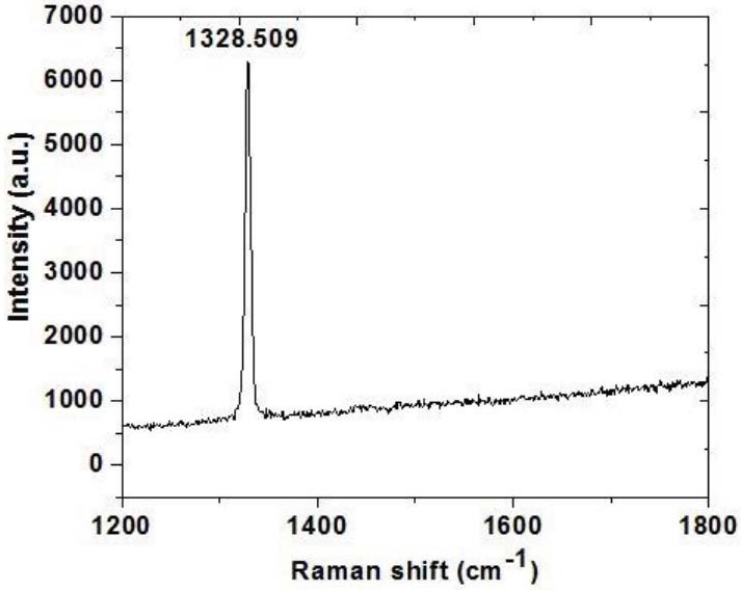

Figure 2: Raman spectroscopy of white diamond (W11) after chemo-mechanical polishing with distinct sp3 peak and little undulations/humps due to non-diamond (1450-1600 cm^{-1}) phases.

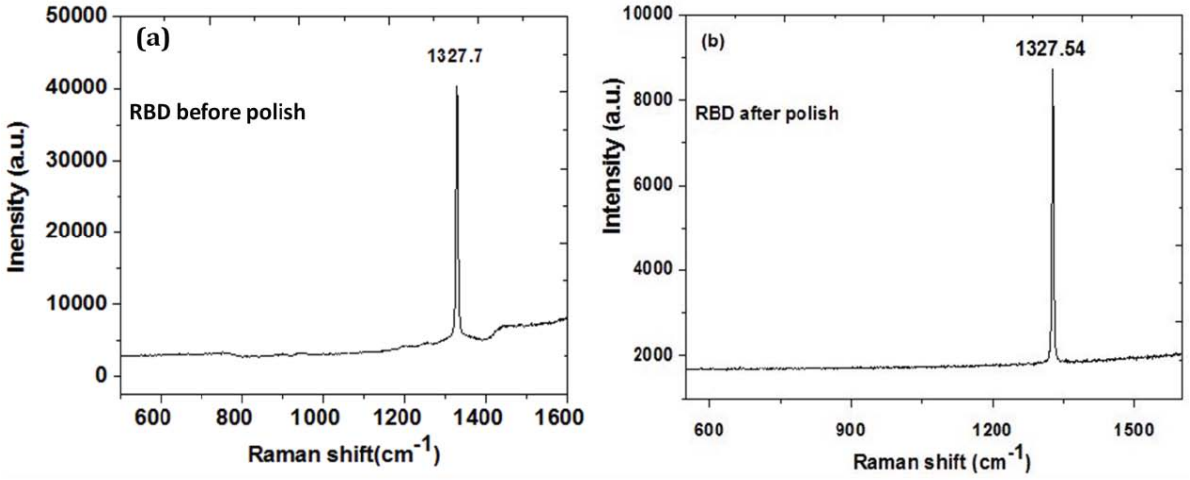

Figure 3: Raman Spectroscopy of black diamond (RBD) sample (**a**) before polishing and (**b**) after polishing.

Table 4: Roughness and Hardness of Polished Diamonds

SAMPLE	INITIAL ROUGHNESS (µm)	ROUGHNESS (AFTER MP) (µm)	MP REMOVAL RATE (µm/h)	FINAL ROUGHNESS (AFTER CMP) (µm)	CMP REMOVAL RATE (µm/h)	TOTAL TIME (hrs)	FINAL POLISHED SURFACE NANOHARDNESS (GPa)
Black	11.29	0.68	0.19	0.546	0.004	85 (55 + 30)	32.5±1
White	2.217	0.66	0.04	0.175	0.013	70 (35 + 35)	28.5±2

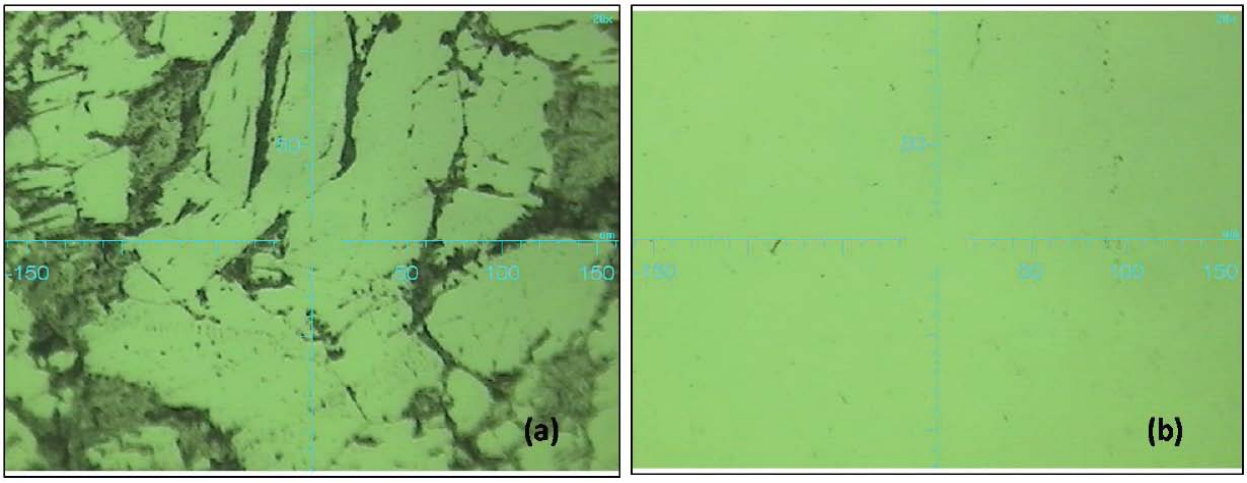

Figure 4: Optical images of the indented surfaces, **a**) black and, **b**) white diamond, showing no indentation mark by Berkovich indenter.

treated. White sample was polished altogether for 70 hours, whereas, the black sample was altogether polished for 85 hours. The initial roughness of 2.21µm was brought down to 0.661µm after 35 hours polishing (MP) for white diamond, whereas, it took 55 hours of mechanical polishing with water as cooling medium for the initial roughness of 11.29µm of black diamond to bring down to 0.68µm. The process of abrasive action could remove inferior quality black diamond more easily. The material removal rate for black PCD was 0.19µm/hr, in comparison to one order of magnitude slower 0.04µm/hr rate of polishing, for superior quality white diamond. The quality of diamond may be the determining factor in controlling the diamond removal rate by abrasive action only. Further mechanical polishing could not achieve better surface roughness. So it was then changed to chemical assisted mechanical polishing. Such chemo-mechanical polishing was carried out again for another 35 hours on the white sample which could bring down the roughness to 175 nm, acceptable for many technological applications. It may be argued that above 100nm surface R_a roughness value is quite high for such long polishing time, but it is to be noted that CSI scan was performed over

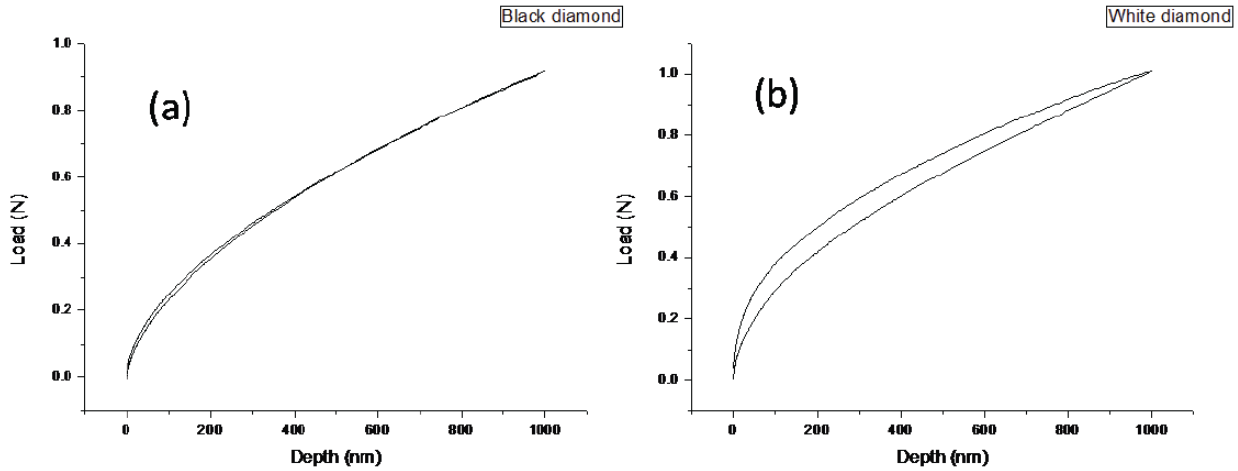

Figure 5: Typical nanoindentation loading-unloading curves of **a**) black and, **b**) white diamonds, showing perfect elastic behavior.

880μm×660μm area, which would include several diamond grains with intergranular pits as evidenced in Figure **1b**. Whereas, 2 nm roughness was reported earlier [44], when smaller 5μm×5μm area AFM scan was performed. After planarization individual grain sizes become 20-30μm (Figure **1c**). So area [45] over which the roughness is measured would ultimately determine the absolute roughness values. It might be so that further AFM scan on individual grain would reveal much smaller Ra value of roughness.

Surprisingly, the black variety diamond even after chemo-mechanical treatment for 30 hours could not bring down the roughness below 0.5μm level. Apparently the roughness value did not change much after successive mechanical and chemo mechanical polishing for black diamond. If the CMP rate for white diamond was 13nm/hr, it was three times slower (4nm/hr) for mechanically polished black diamond. Such slow rate of CMP is unexpected and is not technologically significant. The striking difference, in rates of polishing between white and black diamonds, during both the mechanical and chemo-mechanical polishing, is opposite. Mechanical abrasive action during MP could remove poorer quality as-deposited black diamond more easily whereas, afterwards when such new polished diamond surfaces were additionally treated with chemo-mechanical action, it was noticed that polishing was faster for white variety than the black one – which should not be the case.

4.2. Variable Polishing Rates for Black and White PCDs: Nanoindentation and Raman Analysis

In order to understand unusual nature of the polished black diamond surface (Figure **4a**) comparison to the white variety (Figure **4b**), their hardness were measured. 1000mN load was gradually applied with 30 sec of cyclic indentation time period. Line and array indents were carried out to get the average value of multiple indents with internal spacing of 30μm. Figure **4** does not reveal any indentation mark of Berkovich, which implies the material was elastic (almost coinciding loading-unloading curve). We could notice some hysteresis in Figure **5b** of white diamond, but for black variety the loading unloading curves were exactly overlapping (Figure **5a**). It was found that black diamond polished surface is harder than white diamond by almost 4 GPa (Table **4**). Such higher hardness of black variety was responsible for slower polishing rates during CMP. It may be so that after removal of top layers of CVD deposited diamond, there are more harder carbon phases present on the top polished surface of black diamond than its white variety. The hysteresis present in the white diamond indicates expenditure of energy in carrying out nanoindentation of the white surface. In other words, the black mechanically polished surface has harder sp3 phases, whereas, the white diamond has relatively more proportions of softer compliant amorphous carbon phases (Figures **2** and **5b**), conducive for effective polishing. It is also to be noted that after polishing Raman signals from black diamonds are rather downshifted than the white variety diamond (comparing Figures **3b** and **2**, although humps corresponding to non-diamond phases disappear from Figures **3a** to **3b**), which implies that the black surfaces are under more tensile stress due to creation of polishing defects.

The above measured hardness values are in commensurate with earlier reported results of nanoindentation hardness for CVD polycrystalline diamonds. Fazio *et al*. reported load control average nanohardness of 53±24 GPa. In their reported table of hardness [46] there are values as low as 11 or 18 GPa, which shows that present measurements are well above such wide variance of hardness data. On the other hand, an earlier paper [47] with depth control nanoindentation of hot filament CVD diamond coatings, reported nanohardness (~30 GPa) with much less variation of values, similar to the results presented in this work. Again, such comparatively lower values of nanohardness may raise question about testing conditions. But, it can be argued that even if there might be tip blunting effect on the indentation experiments, but here it was tried “to compare” the nanohardness between black and white polished diamonds - not “to measure” their absolute hardness value.

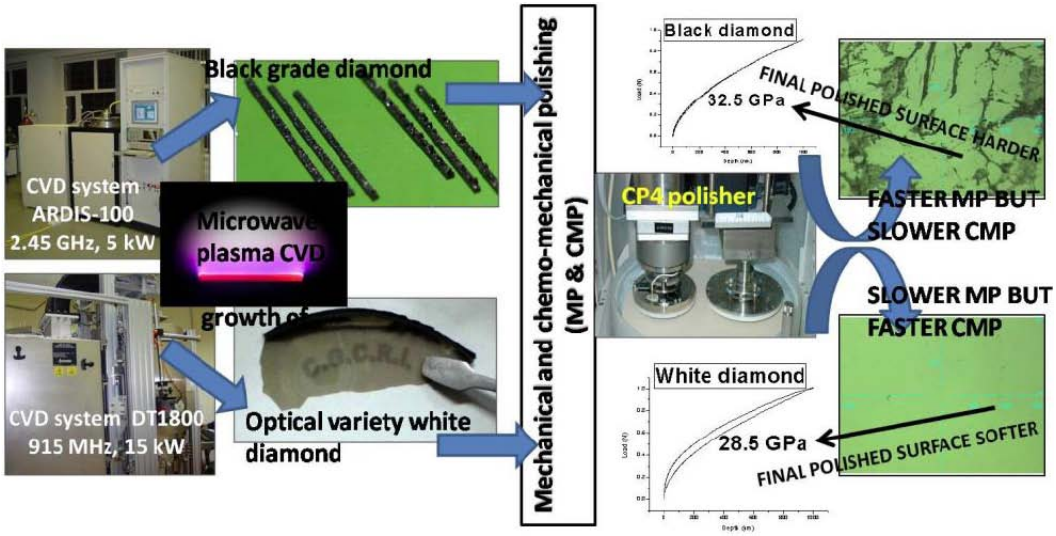

Figure 6: Summary of the results after polishing of black and white CVD grown PCDs is represented with a schematic flow diagram.

It is known from the literature [43-45] that diamond has anisotropic polishing tendency, i.e., it has favourable directions of soft and hard material removal directions inside their lattices. The relative proportion of sp^2 carbon to amorphous sp^3 carbon determines the soft or hard directions. More is the ratio, it is easier to polish the diamond. From hardness data, it appears that for white diamond, this ratio is higher than black diamond. Moreover Raman signals, after polishing, shows more downshift of sp3 peak in black than white variety – proving black diamonds are more stressed, another reason to make them harder. Although black diamond polishing rate was higher during MP but it became slower than white diamond during CMP. This sudden drop in polishing rate for black variety may suggest that during CMP, it was difficult for the process to create softer sp2 phases for easy polishing. Whereas, the white diamond still could be polished (i.e. surface material can be converted from sp3 to sp2 phases – small undulations 1450-1600 cm^{-1} in Figure **2** may be an indication) during CMP. In other words favourable transformation of surface diamond into soft graphitic phases helped polishing of white diamond. But the surface of the black diamond was not easily converted into softer graphitic phases for easy polishing, may be due to the presence of high proportion of harder defect structure like amorphous sp3 with more internal stress, as indicated by Raman and nanoindentation data.

5. CONCLUSIONS

Figure **6** represents summary of the present work. Black and white variety diamonds were polished successively by MP and CMP. It was found that MP rate was higher for black diamond than white, due to its inherent high as-grown roughness and poorer quality. But once the initial top layers were removed by MP, further reduction in roughness was not possible even after long hours of mechanical polishing. So the process was then shifted to chemically assisted mechanical polishing. But to our surprise, inferior black diamond was difficult to polish compare to white variety during CMP. It was found that polished black diamond surface had higher hardness than white variety with more internal tensile stress. Moreover, load-displacement curve for white diamond shows some hysteresis, indicating the presence of some soft graphitic phases which might have helped during CMP polishing. On the other hand, hard black diamond surface could not be chemo-mechanically polished may be due to the presence of hard amorphous sp3 phases. It may be inferred that the mechanically polished black diamond surface was defective enough to stop producing any further soft graphitic phases, which is necessary for diamond polishing.

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