Chapter 2
Understanding the Material Removal Mechanisms

Abstract This chapter explains various material removal pathways in relation to diamond polishing. The discussion here highlights the fact that the mechanisms for the removal of carbon atoms from a diamond substrate, which include the conversion of diamond to non-diamond carbon, chemical/mechanical removal, micro-cleavage, diffusion of carbon into soluble metals, chemical reactions, and removal of surface carbon atoms through evaporation, ablation or sputtering, are complicated. Individual diamond polishing processes can involve various mechanisms. Temperature rise plays an important role in the activation of almost all the mechanisms, as thermo-chemical reactions occur only at high temperatures. The fundamental understanding described in this chapter is of primary importance to an appropriate selection, design, and development of a sensible diamond polishing process.

Understanding the material removal mechanism in diamond polishing is important to the selection of processing conditions or development of a technique for a specific application. The surface roughness of diamond can be reduced by a number of different ways. They consist in the removal of carbon atoms from the diamond substrate via the following mechanisms:

- Conversion of diamond to non-diamond carbon, followed by chemical/mechanical removal
- Micro-cleavage
- Diffusion of carbon into soluble metals
- Chemical reactions
- Removal of carbon atoms from the diamond surface by evaporation, ablation, or sputtering

2.1 Conversion of Diamond to Non-Diamond Carbon

There are several types of carbon lattice structures including graphite, amorphous carbon, diamond, and fullerene (Pierson 1993). Carbon atoms can connect to each other by forming several kinds of chemical bonds. The different bonding schemes among the carbon atoms can form different crystalline structures.

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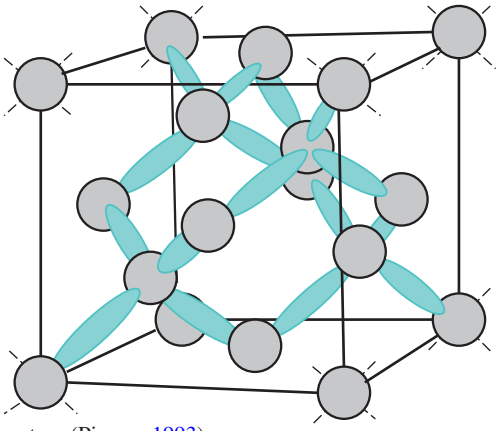

Fig. 2.1 Diamond structure (Pierson 1993)

Diamonds typically crystallize in the cubic crystal system and consist of tetrahedrally bonded carbon atoms. Figure 2.1 shows the unit cell of a diamond crystal. Each cell has eight atoms located as follows: 1/8 × 8 at the corners, 1/2 × 6 at the faces, and 4 inside the unit cube. The cubic structure of diamond can be visualized as a two face-centered interpenetrating cubic lattice. The edges for this cube are 3.5670 Angstroms long. Each carbon atom in diamond is surrounded by four nearest neighbors. They are connected together by covalent bonds that separate them by a small distance of 1.5445 Angstroms with a high bond energy of 711 kJ/mol. The angles between these bonds are 109 degrees. As a result, the central atom and its neighbors form a tetrahedron. The interlocking network of covalent bonds makes the diamond structure very rigid. The density of diamond is 3.5155 g/cm^3.

Crystalline graphite consists of parallel sheets of carbon atoms, each sheet containing hexagonal arrays of carbon atoms, as shown in Fig 2.2. Each atom is connected to three nearest neighbors, within the sheets, by covalent bonds that separate them by a distance of 1.415 Angstroms. This bonding arrangement results from the sp^2 hybridization of carbon‘s electronic orbitals. Another intriguing aspect of the bonding scheme within the graphite sheets is the distributed pi bonding between the carbon atoms. This distributed pi bonding gives rise to delocalized electrons that make graphite electrically conducting. The sheets are held together by weak Van der Waals forces and are separated from each other by a distance of 3.35 Angstroms. The density of graphite is 2.2670 g/cm^3.

Thermodynamically, graphite has a stable structure under atmospheric conditions, while the other lattice structures are metastable. Figure 2.3 shows the pressure-temperature (P–T) phase and transformation diagram for carbon (Bundy et al 1996, Asmussen and Reinhard 2002). In the figure, the straight solid line, so-called Berman-Simon line (B–S line), represents equilibrium phase boundaries: above the line diamond is the stable carbon allotrope, while graphite is stable below the line. Region A shows the commercial synthesis of diamond from graphite by catalysis (Bundy et al. 1996). Region B on the dashed line marks the

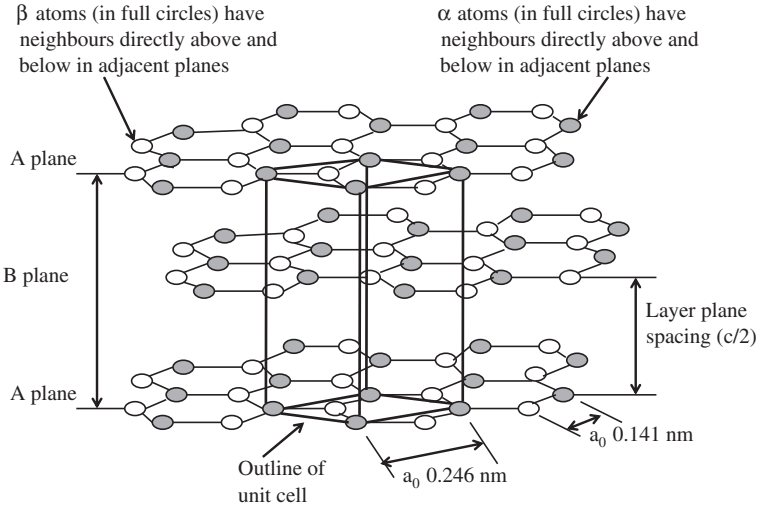

Fig. 2.2 Crystal structure of graphite

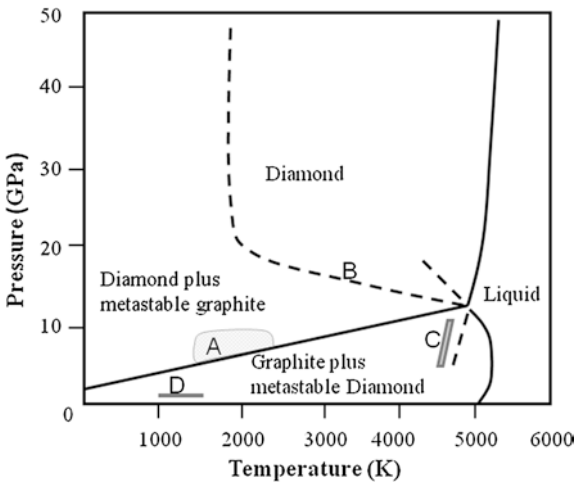

Fig. 2.3 P-T phase and transformation diagram for carbon (Bundy et al. 1996, Asmussen and Reinhard 2002) recreated with permission

temperature–pressure threshold of very fast solid–solid transformation of graphite to diamond. Region C shows the pressure/temperature threshold of very fast transformation of diamond to graphite. Region D represents the chemical vapour deposition (CVD) synthesis of diamond (Asmussen and Reinhard 2002).

From the P–T phase diagram of carbon, thermodynamics indicates that diamond is a metastable phase of carbon at room temperature and pressures, and should convert to graphite. However, the reaction rate (kinetics) is so slow that the change is normally undetectable. Increased temperatures will accelerate its conversion to

graphite (Howes 1962; Hall 1961). This fast conversion occurs only at elevated temperatures because of a high activation barrier of this phase transition.

The activation barrier of the phase transition can be depicted clearly by an energy diagram. Figure 2.4 shows an energy diagram for the conversion between diamond and graphite. Generally, more stable materials have lower potential energies. Graphite that is more stable is placed lower on the diagram (at lower potential energy) while the less stable diamond (higher potential energy compounds) is higher on the diagram. The binding energy between atoms of carbon is very large (e.g. the cohesive energy of diamond is 717 kJ/mol) (Bundy et al. 1996). Once carbon atoms are locked into a given phase configuration, typically a large amount of activation energy is required to produce a different stable phase. In order to transfer diamond to graphite, a high-energy barrier must be passed over. The top of this barrier is called the transition state, which represents the highest energy structure involved in the reaction. At the transition state, some of the C–C bonds are stretched and the carbon atoms are beginning to arrange into the planes of graphite (1996). These planes will be buckled due to stretched bonds between the ultimately non-bonded planes of carbon atoms of graphite. As a result the stabilization afforded by four single C–C bonds at each C atom has begun to be lost, but the resonance stabilization of graphite has not been fully realized. As a result the C–C bonds in the transition state on average are weaker than the bonds of either diamond or graphite. Thus, the potential energy of the transition state is greater than those of reactant and product.

The energy difference between the starting material (diamond) and the transition state is the activation energy. The value of the activation energy depends on the direction of the reaction; the activation energy for the diamond to graphite transformation E_1 is lower than that for the graphite to diamond E_{-1}. If the activation energy for a reaction is high, the reaction will occur slowly. The rate of reaction is determined by how frequently the atoms collect enough energy to 'climb' over the hill, so the higher the barrier the slower the reaction. In addition, increasing the temperature, or kinetic energy of the atoms, will increase the reaction rate because the atoms will collect the energy needed to 'climb' the barrier more frequently. Much energy in the form of high temperature is needed to convert diamond to a transition state so that graphite can be formed.

Moreover, catalytic metal can decrease the activation energy required to transform diamond into graphite and increase the kinetics of the reaction, though the catalysts do not affect the thermodynamics (in Fig. 2.4). These catalytic metals

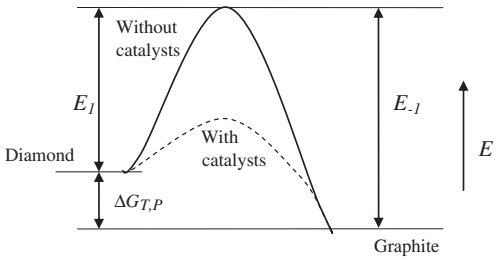

Fig. 2.4 Energy diagram for conversion of diamond to graphite

must have unpaired d electrons which may chemically bond with carbon, but not too reactive to form carbide (Sung and Tai 1997; Butenko 2000; Ikawa 1971). Such as, transition metals with intermediate reactivity Fe, Ni, Co, Mn, and Cr can catalyze the conversion of diamond to graphite at low pressure and at temperatures above 700 °C. The catalytic reaction can be in two directions. An effective catalyst can not only catalyze the graphite → diamond transition in the stability field of diamond under high pressure, but also the diamond → graphite transition (back conversion of diamond) in the stability field of graphite at low pressure.

Thus, diamond that comes into contact with these catalytic materials at elevated temperature loses its lattice structure and is transformed into graphite or amorphous carbon. Protruding crystals on the surface of a diamond that come initially into contact with a catalytic material are changed to graphite or other non-diamond carbon. After the transformation, the graphite is easily removed due to the weaker type of binding force.

2.2 Micro-Cleavage

Tolkowsky (1920) who performed the first systematic investigations to study the material removal mechanism of diamond during polishing suggested that the material is removed by processes of micro-cleavage. During polishing, two moving surfaces come into contact with each other and friction arises between them. When the friction force is higher than the atomic binding energy of the material, atoms on the surface layer are deformed or removed away from the surface, depending on the brittleness of the material (Bhushan 1991). Since diamond is very brittle, it is removed by a process of brittle fracture on the microscopic scale. Protruding parts of the surface experience higher friction forces and are easily chipped away to form a smooth surface. The process is named micro-cleavage. If a soft material and a hard material come into contact, the removal rate of the soft material is higher than that of the hard material.

As diamond is the hardest known material, the only effective way of polishing a diamond surface by the micro-cleavage mechanism is to use diamond abrasive, which has been used in the form of powder or is embedded in different kinds of binders. The material removal rate and the ultimate roughness are related to the size of the diamond abrasive used. In the processing, the material removal rate can be increased through adding abrasive and using bigger size of abrasive. However, at the same time, surface roughness will be impacted by the size of the abrasive used; the grooves produced by coarse abrasive are deeper than those created by fine abrasive. Therefore, coarse abrasives are used in rough polishing for higher material removal rate and fine abrasives are used for final polishing.

In the Hertz test, when a diamond surface is over-loaded by a diamond indenter, cracks appear in diamond (Yuan et al. 2003). All the cracks are around the outer circle of the contact area and parallel to the (111) plane of diamond. It means that the micro-cleavage of diamond is caused by the cleavage failure of

(111) plane. In diamond polishing, the surface layer of diamond is abraded off due to the micro-cleavage along (111) plane. Both the polishing line and cleavage lines of (111) plane are often visible on the polishing diamond surface. The direction of the cleavage lines does not change with the polishing direction. The inclination angle between the cleavage planes (111) and polishing surface has significant effects on the polishing rate. The difference of the inclination angle between (111) cleavage plane and the polishing surface causes a difference of friction coefficient and removal rate in diamond polishing.

The abrasion resistance of diamond is a function of the polishing direction relative to the surface being polished. According to Fig. 2.1, in each diamond unit cell on {100} planes, there are two atoms along < 100 > direction, while there are three atoms along < 110 > direction, therefore it is relatively easier to polish diamond along < 100 > than along < 110 > direction. On {110}, there are three atoms in < 110 > direction and two atoms in < 100 > direction, therefore the removal rate along < 100 > is higher than < 110 > direction. However, on < 111 > plane, there are three atoms in all three edges, therefore grains oriented near {111} are the most resistant to polishing wear since {111} planes have the highest atomic density. Consequently, it is very important to choose an easy direction for effective polishing of diamond.

2.3 Diffusion of Carbon to Soluble Metals

Carbon atoms easily diffuse into carbon-soluble metals such as Fe, Ni, Mo, and rare-earth metals/alloys. These metals are ready to react with any source of free carbon and absorb this carbon into their surface. Such a reaction is easily triggered under the temperature and pressure conditions occurring in the diamond polishing process. When a diamond surface comes into contact with the metal disk at elevated temperature, carbon atoms in the diamond diffuse into the metal disk until it is saturated. The diffusion path for atoms from protruding parts of the specimen is shorter and these areas are attacked at a greater rate. One of the main factors involved in diamond polishing is estimated to be the diffusion of carbon into the steel disk that forms iron austenite. The carbon concentration depends on the distance from the interface, the diffusion coefficient, and time. As the carbon diffusion coefficient and the carbon solubility of the mating material increase, the polishing rate also increases. A thick piece of metal can accommodate more carbon atoms than a thin piece.

Carbon atoms easily diffuse into high carbon-soluble metals. These metals include rare-earth metals/alloys and Fe, Ni, and Mo. They have a chemical characteristic known as carbon solubility potential, they are ready to react with any source of free carbon and absorb this carbon into their surface. The carbon concentration of a metal in contact with a carbon source is given by (Malshe et al. 1999):

$$C(y) = C_1 \left[erfc \left(y/2\sqrt{Dt} \right) \right] \tag{2.1}$$

where $C(y)$ is the carbon concentration at y, C_1 is the interface carbon concentration, *erfc* is the error function, y is the distance from the interface, D is the diffusion coefficient, and t is time.

One of the metal disk elements, iron, possesses one of the highest diffusion coefficients for carbon among the group VIII B transition metals (Zaitsev et al. 1998), though its value drops with increasing carbon concentration. Iron also has a high solubility of carbon reaching 170 mg/cm^3 at the eutectic temperature. In the polishing disk material, the initial carbon concentration is lower than 0.08 %, and the disk is very big and thick compared to the diamond specimen. Moreover, if the disk material is in austenite structure, the maximum carbon solubility in austenite is as high as 2.03 % C at 1147 °C. Therefore, such diffusion reaction is easily triggered under the temperature and pressure conditions occurring in the thermo-chemical and dynamical polishing process.

When a diamond surface comes into contact with a metal disk at high temperatures, converted carbon atoms in diamond diffuse into the metal disk until the metal is saturated. The diffusion path for atoms from protruding parts of the specimen is shorter and these areas are attacked at a greater rate, and the diffusion rate of graphite is much greater than that of diamond (Vishnevskii et al. 1975).

2.4 Chemical Reactions

Chemical reaction of diamond may be accomplished with gas, liquid, or solid metal/metal oxides and they are involved in different diamond polishing techniques.

There would be gas-surface reactions when diamond is exposed to a reactive atmosphere, such as oxygen or hydrogen in elevated temperature. Carbon in diamond would convert into COx or CHx gas and then evaporate. In addition, there are channels wherein O, O_2, O_3, OH, H, etc., interact and remove carbon from the diamond surface via heterogeneous chemical reactions. These reactions mainly happen in laser and ion beam polishing.

Diamond will also react with oxidizing reagents such as KOH or KNO_3 under pressure and elevated temperatures. The temperatures are slightly above their melting points (Ollison 1999); the melting temperatures of KNO_3 and KOH are 324 and 360 °C. The supplied heat and pressure decompose the liquid into oxygen and other constituents near the sample surface. The oxygen generated reacts with the diamond and forms CO or CO_2. This reaction occurs in chemo-mechanical polishing. In oxygenic ambient or direct contact with oxy-acid, e.g. H_3PO_4 or $NaNO_3$, diamond would oxygenate (be oxygenized) before being graphitized.

In addition, diamond reacts with some metals to form carbide such as Ti, Fe, V, Mn, and Cr under pressure and elevated temperatures. Furthermore, during thermo-chemical or dynamical friction polishing, metal such as iron would be oxidized and then have a reductive reaction with carbon or hydrogen. In these reactions, metal oxides such as Fe_2O_3 will reduce the level of carbon in metal disk and

convert into free iron and form CO or CO_2. It is thought that polishing takes place due to evaporation of converted carbon of the diamond into CO or CO_2 gas. Non-diamond carbon then diffuses into the free Fe formed from the above reactions along with the evaporation of carbon in the form of CO or CO_2. These chemical reactions are involved in most reactive contact polishing which includes thermo-chemical polishing, dynamic friction polishing and even chemo-mechanical polishing.

When carbon (including diamond and graphite) is exposed to oxygen bearing environments at elevated temperatures, it would react with oxygen to form CO and/or CO_2 by the following reactions:

$$C + 1/2\,O_2 = CO \tag{2.2}$$

$$C + O_2 = CO_2 \tag{2.3}$$

According to reaction kinetic theory, the reaction rate is

$$R_c = k_r\,(P_{O2})^n\,(C)^n \tag{2.4}$$

where R_c is the rate of carbon (diamond) consumption (kg/s), Po_2 is the partial pressure of oxygen, C is the concentration of carbon, n and k_r are reaction order and constant, respectively. Since diamond is solid, the concentration of carbon can be taken as constant. The reaction rate can be written as

$$R_c = k\ (P_{O2})^n \tag{2.5}$$

where k is the rate constant and obeys the Arrhenius equation:

$$k = Ae^{-E/RT} \tag{2.6}$$

where T is the absolute temperature, A is the pre-exponential factor of the Arrhenius equation, E is activation energy, and R is the gas constant.

According to equations 2.5 and 2.6, and Alam and Sun's experimental kinetic data (Alam and Sun 1993), at a given oxygen pressure, the oxidation rate increases with increasing temperature. On the other hand, at a given temperature, diamond oxidizes faster with increasing oxygen partial pressure. Moreover, the oxidation rate also depends on activation energy which varies at different conditions. The apparent activation energy values, which were determined by different research groups, are presented in Table 2.1.

The discrepancy among the activation energy values may be attributed to differences in the diamond structure, particularly in terms of film morphology, crystallite size, amount of non-diamond carbon, hydrogen, etc., and crystal defects (Alam and Sun 1993).

It has been shown that non-diamond carbon oxidizes more rapidly than diamond (Alam and Sun 1993; Joshi et al. 1990). Also sp^2 bonded carbon is generally known to oxidize faster than sp^3 bonded carbon (Joshi et al. 1990). The activation energy for graphite varies from 155 to 184 kJ/mol (Johnson et al. 1990a; Joshi et al. 1990), while that for a diamond film is from 213 to 240 kJ/mol (Johnson et al 1990a; Nimmagadda et al. 1990; Alam and Sun 1993).

Table 2.1 Activation energies for oxidation of diamond published in the literature

Source	Temperature range (K)	Other conditions	Activation energy (kJ/mol)
(Tankala et al. 1990)	873–1073	Air, atmospheric pressure, 0.5 L/min	151
(Johnson et al. 1990b)	873–1023	Air, atmospheric pressure, no flow. Increasing humidity inhibit oxidation, CVD film	213
(Alam and Sun 1993)	973–1073	25–100 vol. % O_2 (balance Ar), 1 atm pressure, 1 L/min. HF-CVD polycrystalline	232
(Sun and Alam 1992)	973–1173	Pure O_2, atm. Pressure,1 L/min	
		Natural (111)	260
		Natural (100)	199
		CVD polycrystalline	229

In addition, the oxidation of carbon can be accelerated by catalysts in a number of ways. Some research found that elements that catalyze carbon-oxygen reactions include Na, K, Ca, Cu, Ti, Fe, Mo, Cr, Co, Ni, and V (Luo et al. 2004). The heterogeneous distribution of impurity leads to the dispersibility of oxidation results. Inui et al. (1982) ranked the oxidation activities of single metal component catalysts at a temperature of about 500 °C at atmospheric pressure as Cu > Co > Fe > Ni > Pt. Many investigations show that the metals or metal oxides of Pb, Co, V, Mo, Fe, La, Mn, Ca, Cu, Ni, and Cr are very active to moderately active carbon oxidation catalysts (Mul et al. 1998; Neeft et al. 1997). The nature of catalysis involves either activation of carbon atoms or acting as a source of activated oxygen. During the oxidation of carbon, the catalyst metal also gets oxidized and reduced. Before the catalyst metal is completely oxidized, oxygen is rapidly transferred onto carbon to form CO_2. On the other hand, the oxidation rate of the metal is faster than the reduction rate of metal oxide by active carbon at 500 °C.

Inui et al. (1982) also found that the activity of the two or three component catalyst systems was greater than the sum of the activities of each catalyst system taken separately. This synergistic effect was observed in Fe-La_2O_3-Pt, Fe-Mn_2O_3-Cu catalyst, or when Co or Ni was used as the main component of the catalyst. The rate enhancement of carbon-oxidation in the composite catalyst system can be explained as: oxygen in the gas phase is rapidly taken up by one kind of metal particle; this adsorbed oxygen is easily transferred successively to the carbon via another partially reduced metal oxide. Since two different roles of each catalyst were combined in the composite catalyst, consequently, the oxidation reaction must be promoted with the synergistic effect (Inui et al. 1982).

As can be seen, metals or metal oxides of Co, Mo, Fe, Mn, Cu, Ni, and Cr are active carbon oxidation catalysts, and the combination of metal or metal oxide will notably increase the carbon oxidation rate. According to (Alam and Sun, 1993; Tankala et al. 1990), neither x-ray diffraction nor Raman spectroscopy data

revealed any phase transformation of diamond to either crystalline or amorphous non-diamond carbon phases after oxidation in air or oxygen gas. The oxidation behavior of diamond is consistent with two reaction paths (Tankala et al. 1990). (i) Diamond can oxidize directly to CO and CO_2. (ii) If diamond is to oxidize via an intermediate product such as graphite, as has been reported to occur under certain oxidation conditions for natural diamond, the oxidation of the intermediate product occurs at a rate which is not slower than its rate of formation.

Natural diamond is stable in inert environments up to temperatures of 1300–1500 °C and above this temperature it will transform to graphite (Johnston et al. 1992). In oxidizing atmospheres diamond is decomposed at temperatures as low as 700–800 °C via oxidation to gaseous products which are presumably CO or CO_2 or a mixture of them. At low oxygen pressures the diamond surface may contain graphite which is also converted to gaseous products at lower activation energy in this temperature regime. Diamond is reasonably stable in flowing oxygen at temperatures up to 600 °C. Above 600 °C oxidation of the diamond films has been observed to take place at a measurable rate. The oxidation rate increases with further temperature increases until at temperatures greater than 800 °C diamond only survives momentarily.

2.5 Evaporation and Sputtering

If sufficient heat is applied to a material surface, the surface will melt and/or evaporate (Malshe et al. 1999). Using this approach, the protruding crystals on the surface of a diamond can be evaporated to produce a smooth surface. Torches, electric arcs, and lasers can be used to heat a diamond surface. As the angle of incidence of applied heat to the substrate surface increases with respect to the normal, the illuminated area increases and the energy density decreases. However, non-normal incidence angles deliver more energy to the protruding portions of a substrate than to the rest of the substrate. This causes the protruding portions to be removed at a faster rate, and consequently, generates a flatter surface than when a normal angle is used.

Sputtering is a process whereby atoms are ejected from a solid target material due to bombardment of the target by energetic particles. When high-energy ions or atoms collide with a diamond surface, the diamond structure is broken and carbon atoms are detached from the surface, a physical process referred to as sputtering. The ion source must be stable and have a uniform current density to produce a good finish. The sputtering rate can be controlled by changing the collision energy and the number of colliding ions. The sputtering rate depends on the sputtering yield that, in turn, depends on the material, mainly the relative amount of graphite that it contains. Since the sputtering rate of graphite is higher than that of diamond, the more graphite that diamond contains, the higher the sputtering rate. This material remove mechanism takes place in ion beam polishing.

Sputtering, which is observed to occur below the threshold energy of physical sputtering, is also often called chemical sputtering. At elevated temperatures,

chemical sputtering of carbon can be understood to be due to the incoming ions, weakening bonds in the sample, which are then desorbed by thermal activation. The hydrogen-induced sputtering of carbon-based materials observed at low temperatures has been explained by H ions entering between C–C bonds and thus breaking them, a mechanism named swift chemical sputtering.

Sputtering can also play a role in reactive ion etching (RIE), a plasma process carried out with chemically active ions and radicals, for which the sputtering yield may be enhanced significantly compared to pure physical sputtering. Reactive ions are frequently used in secondary ion mass spectrometry (SIMS) equipment to enhance the sputter rates.

2.6 Summary of the Polishing Pathways

The above material removes mechanisms involved in different diamond polishing techniques and each technique consists of one or more of the above mechanisms. In addition, temperature plays an important role in all the above polishing mechanisms except micro-cleavage, as these reactions occur only at high temperatures. Figure 2.5 shows a schematic of polishing pathways relevant for diamond polishing.

Diamond can be directly polished via micro-cleavage by mechanical polishing; or via sputtering and evaporation by, ion beam and laser polishing, respectively.

Carbon in the diamond surface may convert into non-diamond carbon under pressure at elevated temperature and/or contact with catalytic metals, and the non-diamond carbon is detached from the surface by micro-cleavage, atomic diffusion into metal, or chemical reaction with gas and/or metal to form COx, CHx, or carbides. The transformation of diamond to non-diamond carbon occurs more or less in all the diamond polishing techniques.

Carbon atoms in protruding portions of the diamond surface may directly diffuse into molten earth metal or transition metal under pressure at elevated temperature through thermo-etching method. Also, a diamond oxidation process can occur at lower temperatures and atmospheric pressure in the presence of an

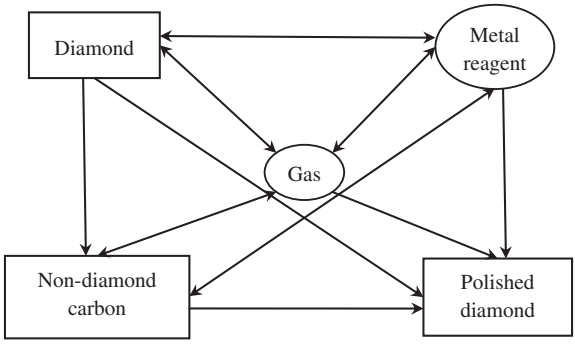

Fig. 2.5 Schematic of material removal pathways relevant for diamond polishing

oxidizing agent such as potassium nitrate (KNO_3) or potassium hydroxide (KOH). These processes happen in chemo-mechanical polishing.

Diamond might have a gas-surface reaction in different ambient atmospheres. These reactive gases such as oxygen and/or hydrogen gas might have a chemical reaction with carbon in the protruding of diamond surface to form CO_x/CH_x and then evaporate in EIR or laser polishing. Also ambient gas (such as hydrogen, oxygen, and water vapor) used during the thermo-chemical or dynamic friction polishing acts as an agent influencing the diamond-metal contact conditions and removing the non-diamond carbon formed on the polished diamond surface. Moreover, these gases will be inter-reactive with metals. Metal such as iron would be oxidized and then have a reductive reaction with carbon or hydrogen. The metal oxides such as Fe_2O_3 will reduce the level of carbon in a metal disk and convert into free iron and form CO or CO_2.

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