Claims

1. A method for manufacturing a plurality of synthetic single crystal diamonds, the method comprising:

forming a plurality of seed pads, each seed pad comprising a plurality of single crystal diamond seeds anchored to, or embedded in, an inert holder;

loading a carbon source, a metal catalyst, and the plurality of seed pads into a capsule;

loading the capsule into a high pressure high temperature (HPHT) press; and

subjecting the capsule to a HPHT growth cycle to grow single crystal diamond material on the plurality of single crystal diamond seeds, the HPHT growth cycle comprising:

initiating HPHT growth of single crystal diamond material on the plurality of single crystal diamond seeds by increasing pressure and temperature;

maintaining HPHT growth of single crystal diamond material on the plurality of single crystal diamond seeds via a pressure driven growth process by controlling and maintaining pressure and temperature; and

terminating HPHT growth of single crystal diamond material on the plurality of single crystal diamond seeds by reducing pressure and temperature,

wherein the plurality of single crystal diamond seeds remain anchored to, or embedded in, the inert holders during the HPHT growth cycle.

2. A method according to claim 1, wherein each seed pad comprises a number of single crystal diamond seeds in a range 8 to 3000, 30 to 1500, 50 to 800, or 80 to 650.

3. A method according to claim 1 or 2, wherein each seed pad comprises a density of single crystal diamond seeds in a range 0.3 to 45, 0.5 to 30, 0.8 to 20, or 1.0 to 10 seeds cm^{-2}.

4. A method according to any preceding claim, wherein the capsule comprises a number of seed pads in a range 4 to 30, 4 to 20, 6 to 15, or 8 to 10.

5. A method according to any preceding claim, wherein the seeds pads are spaced apart within the capsule such that a distance between the seeds pads is selected to be in a range 1 to 10, 1.2 to 5.0, 1.2 to 3.0, or 1.2 to 2.0 times a height of the single crystal diamond material after terminating HPHT growth.

6. A method according to any preceding claim, wherein the seeds pads are spaced apart within the capsule such that a distance between the seeds pads is selected to be in a range 1.0 to 12 mm, 1.5 to 10 mm, 2.0 to 8 mm, 2.5 to 7.0 mm, or 3.0 to 6.0 mm.

7. A method according to any preceding claim, wherein the capsule comprises a number of single crystal diamond seeds in a range 32 to 108000, 150 to 30000, 350 to 12000, or 450 to 6000.

8. A method according to any preceding claim, wherein the capsule comprises a density of single crystal diamond seeds in a range 0.3 to 45, 0.5 to 30, 0.8 to 20, or 1.0 to 10 seeds cm^{-3}.

9. A method according to any preceding claim, wherein the capsule has a volume no less than 100 cm^3, 500 cm^3, 1000 cm^3, 1500 cm^3, 2000 cm^3, or 2500 cm^3.

10. A method according to any preceding claim, where the single crystal diamond seeds have a longest dimension of at least 50 μm, 100 μm, 200 μm, 300 μm, 400 μm, 500 μm, 600 μm, 700 μm, 800 μm, 900 μm, 1 mm, 2 mm, 3 mm, or 4mm.

11. A method according to any preceding claim, where the single crystal diamond seeds have a longest dimension of no more than 10 mm, 5 mm, 3 mm, 2 mm, 1 mm, 900 μm, 800 μm, 700 μm, 600 μm, 500 μm, 400 μm, 300 μm, 200 μm, or 100 μm.

12. A method according to any preceding claim, where the plurality of single crystal diamond seeds within the capsule have a substantially equal longest dimension to within 30%, 20%, 10%, or 5% of a mean value.

13. A method according to any preceding claim, wherein the single crystal diamond seeds comprise an asymmetric growth surface having a length greater than a width by a factor of at least 1.5, 2, or 3.

14. A method according to any preceding claim, further comprising:

mixing the carbon source with the metal catalyst to form a reaction mixture prior to loading the reaction mixture and the plurality of seed pads into the capsule to form alternating layers of reaction mixture and seed pads.

15. A method according to claim 14, wherein the carbon source material is graphite in the form of a powder, grains, or flakes.

16. A method according to claim 14 or 15, wherein the carbon source material has a total impurity level no more than 0.1%, 0.05%, 0.02%, 0.015%, 0.01%, or 0.005% by weight.

17. A method according to any one of claims 14 to 16, wherein the carbon source material has a surface area per gram in a range 0.001 m^2/g to 10 m^2/g, 0.01 m^2/g to 4 m^2/g or 0.05 m^2/g to 1.9 m^2/g

18. A method according to any one of claims 14 to 17, wherein at least a portion of the carbon source material is located less than 0.1 mm, 0.05 mm, 0.02 mm, or 0.01 mm from the single crystal diamond seeds.

19. A method according to any one of claims 14 to 18, wherein the reaction mixture comprises graphite in an amount in a range 5% to 60%, 9% to 50%, 14% to 40%, 16% to 35%, 18% to 30%, or 20% to 30% by weight prior to the HPHT growth cycle.

20. A method according to any one of claims 14 to 19, wherein the reaction mixture comprises powdered metal catalyst in an amount in the range 40% to 95%, 60% to 90%, 65% to 85%, or 70% to 80% by weight prior to the HPHT growth cycle.

21. A method according to any one of claims 14 to 20, wherein the metal catalyst comprises one or more of nickel, cobalt, iron, or manganese, preferably in the following combinations: NiFe, CoFe, NiFeCo or NiFeCoMn.

22. A method according to any preceding claim, wherein the initiating step comprises raising the pressure to a target starting value P_s which is below the Berman-Simon graphite/diamond thermodynamic phase stability line, raising the temperature to a value T_g which exceeds a eutectic temperature for the carbon source and the metal catalyst, holding the temperature and pressure for a time t, and then raising the pressure to a target starting value P_g for the maintaining step to initiate HPHT growth.

23. A method according to claim 22, wherein the time t is in a range 1 to 36000 seconds, 20 to 24000 seconds, 40 to 15000 seconds, or 60 to 11000 seconds.

24. A method according to claim 22 or 23, wherein the temperature T_g is in a range 1070 Kelvin to 2470 Kelvin, 1370 Kelvin to 1970 Kelvin, 1520 Kelvin to 1770 Kelvin, or 1570 Kelvin to 1670 Kelvin.

25. A method according to any one of claims 22 to 24, wherein the pressure P_s is within 0.01 to 2.0 GPa, 0.05 to 1.5 GPa, 0.1 to 1 GPa, or 0.2 to 0.5 GPa of pressure P_g.

26. A method according to any one of claims 22 to 25, wherein the pressure P_g is in a range 4.0 to 8.0 GPa, 4.5 to 7.0 GPa, 5.0 to 6.0 GPa, or 5.2 to 5.7 GPa.

27. A method according to any one of claims 22 to 26, wherein the pressure is raised from P_s to P_g at a rate in a range 0.001 to 1.0 GPa per minute, 0.01 to 0.8 GPa per minute, 0.01 to 0.5 GPa per minute, or 0.05 to 0.3 GPa per minute.

28. A method according to any one of claims 22 to 27, wherein P_s, T_g, and t are selected and maintained to achieve re-crystallization of the carbon source material whereby at least 50%, 60%, 70%, 80%, or 90% by weight of the carbon source material is re-crystallized.

29. A method according to any preceding claim, wherein, during the maintain step, a temperature difference between a top side and a bottom side of the capsule is maintained to be no more than 100 Kelvin, 75 Kelvin, 50 Kelvin, 30 Kelvin, 20 Kelvin, 10 Kelvin, 5 Kelvin, or 1 Kelvin.

30. A method according to any preceding claim, wherein, during the maintain step, a temperature gradient between a top side and a bottom side of the capsule is maintained to be no more than 0.66 Kelvin mm^{-1}, 0.50 Kelvin mm^{-1}, 0.33 Kelvin mm^{-1}, 0.20 Kelvin mm^{-1}, 0.13 Kelvin mm^{-1}, 0.07 Kelvin mm^{-1}, 0.03 Kelvin mm^{-1}, or 0.01 Kelvin mm^{-1}.

31. A method according to any preceding claim, wherein, during the maintaining step, the temperature of the capsule is maintained to be in a range 1070 Kelvin to 2470 Kelvin, preferably in a range 1370 Kelvin to 1970 Kelvin., preferably in a range 1520 Kelvin to 1770 Kelvin, preferably in a range 1570 Kelvin to 1670 Kelvin.

32. A method according to any preceding claim, wherein during the maintaining step the temperature is maintained within 15%, 8%, or 5% of the temperature generated by the initiating step.

33. A method according to any preceding claim, wherein during the maintaining step the pressure within the capsule is maintained to be within a range 4.5 to 8.0 GPa, 5.0 to 6.5 GPa, 5.2 to 5.9 GPa, or 5.4 to 5.7 GPa.

34. A method according to any preceding claim, wherein during the maintaining step the pressure is maintained within 12%, 6%, or 3% of the pressure generated by the initiating step.

36. A method according to any preceding claim, wherein during the maintaining step the press moves anvils inwards whereby a volume of the capsule reduces by an amount in a range 0.5% to 50%, 0.5% to 30%, 1.0% to 25%, 2.0% to 20%, or 5% to 15%.

37. A method according to any preceding claim, wherein during the maintaining step the press moves anvils inwards by a combined distance in the range 1 mm to 100 mm, 5 mm to 75 mm, 10 mm to 60 mm, 20 mm to 50 mm, or 20 mm to 40 mm.

38. A method according to any preceding claim, wherein during the maintaining step the pressure and temperature are controlled and maintained to provide a rate of conversion of carbon source material to diamond per seed in a range 0.1 mg/hr/seed to 5 mg/hr/seed, 0.3 mg/hr/seed to 3 mg/hr/seed, 0.5 mg/hr/seed to 2 mg/hr/seed, 0.7 mg/hr/seed to 1.5 mg/hr/seed, or 0.9 mg/hr/seed to 1.2 mg/hr/seed.

39. A method according to any preceding claim, wherein during the maintaining step the pressure and temperature are controlled and maintained to provide a rate of conversion of carbon source material to diamond grown to having a longest dimension equal to or greater than 2.0 mm in a range 0.1 mg/hr/seed to 2 mg/hr/seed, 0.2 mg/hr/seed to 1.5 mg/hr/seed, 0.2 mg/hr/seed to 1.0 mg/hr/seed, 0.3 mg/hr/seed to 1.0 mg/hr/seed, or 0.4 mg/hr/seed to 0.8 mg/hr/seed.

40. A method according to any preceding claim, wherein during the maintaining step the temperature is decreased in a continuous or stepwise manner while maintaining growth of single crystal diamond material on the plurality of single crystal diamond seeds.

41. A method according to claim 40, wherein the temperature is decreased at a rate in a range 0.1 Kelvin/hour to 2 Kelvin/hour, 0.3 Kelvin/hour to 1.5 Kelvin/hour or 0.5 Kelvin/hour to 0.75 Kelvin/hour.

42. A method according to any preceding claim, wherein the maintaining step is performed for a time of at least 20 hours, 40 hours, 60 hours, 80 hours, 100 hours, 200 hours, 300 hours, 400 hours, or 500 hours.

43. A method according to any preceding claim, wherein over the whole HPHT growth cycle a volume of the capsule reduces by an amount in a range 10% to 60%, 20% to 50%, 30% to 50%, 35% to 45%, or 35% to 40%.

44. A method according to any preceding claim, wherein the pressure and temperature are controlled and maintained to grow single crystal diamond material on the seeds to have a longest dimension no less than 1.0 mm, 1.5 mm, 2.0 mm, 2.5 mm, 3.0 mm, 4.0 mm, 5.0 mm, 7.0 mm, or 10 mm.

45. A method according to any preceding claim, wherein the single crystal diamond material on the seeds accounts for at least 30%, 40%, 50% 60%, 70%, 80%, or 90% of the total mass of diamond material formed in the capsule during the HPHT growth cycle.

46. A method according to any preceding claim, wherein single crystal diamond material having a longest dimension equal to or greater than 2.0 mm accounts for at least 30%, 40%, 45%, or 50% of the total mass of diamond material formed in the capsule during the HPHT growth cycle.

47. A method according to any preceding claim, wherein an output mass of diamond material relative to a total mass of material loaded into the capsule is in a range 5 to 40%, 10 to 30%, or 15 to 20%.

48. An apparatus configured to perform the method according to any preceding claim, the apparatus comprising:

a capsule comprising a plurality of seed pads and reactants including a carbon source and a metal catalyst, wherein the reactants and seed pads are provided in alternating layers, and wherein each seed pad comprises a plurality of single crystal diamond seeds anchored to, or embedded in, an inert holder; and

a HPHT press comprising a heating circuit configured to maintain a substantially uniform temperature throughout the capsule whereby diamond growth on the single crystal seeds is achieved via a pressure driven growth process.

Claims

1. A method for manufacturing a plurality of synthetic single crystal diamonds, the method comprising:

forming a plurality of seed pads, each seed pad comprising a plurality of single crystal diamond seeds anchored to, or embedded in, an inert holder;

loading a carbon source, a metal catalyst, and the plurality of seed pads into a capsule;

loading the capsule into a high pressure high temperature (HPHT) press; and

subjecting the capsule to a HPHT growth cycle to grow single crystal diamond material on the plurality of single crystal diamond seeds, the HPHT growth cycle comprising:

initiating HPHT growth of single crystal diamond material on the plurality of single crystal diamond seeds by increasing pressure and temperature;

maintaining HPHT growth of single crystal diamond material on the plurality of single crystal diamond seeds via a pressure driven growth process by controlling and maintaining pressure and temperature; and

terminating HPHT growth of single crystal diamond material on the plurality of single crystal diamond seeds by reducing pressure and temperature,

wherein the plurality of single crystal diamond seeds remain anchored to, or embedded in, the inert holders during the HPHT growth cycle.

2. A method according to claim 1, wherein each seed pad comprises a number of single crystal diamond seeds in a range 8 to 3000, 30 to 1500, 50 to 800, or 80 to 650, and/or wherein each seed pad comprises a density of single crystal diamond seeds in a range 0.3 to 45, 0.5 to 30, 0.8 to 20, or 1.0 to 10 seeds cm^{-2}.

3. A method according to any preceding claim, wherein the capsule comprises a number of seed pads in a range 4 to 30, 4 to 20, 6 to 15, or 8 to 10.

4. A method according to any preceding claim, wherein the seeds pads are spaced apart within the capsule such that a distance between the seeds pads is selected to be in a range 1 to 10, 1.2 to 5.0, 1.2 to 3.0, or 1.2 to 2.0 times a height of the single crystal diamond material after terminating HPHT growth, and /or wherein the seeds pads are spaced apart within the capsule such that a distance between the seeds pads is selected to be in a range 1.0 to 12 mm, 1.5 to 10 mm, 2.0 to 8 mm, 2.5 to 7.0 mm, or 3.0 to 6.0 mm.

5. A method according to any preceding claim, further comprising:

mixing the carbon source with the metal catalyst to form a reaction mixture prior to loading the reaction mixture and the plurality of seed pads into the capsule to form alternating layers of reaction mixture and seed pads.

6. A method according to claim 5, wherein the carbon source material has a total impurity level no more than 0.1%, 0.05%, 0.02%, 0.015%, 0.01%, or 0.005% by weight.

7. A method according to any one of claims 5 or 6, wherein the carbon source material has a surface area per gram in a range 0.001 m^2/g to 10 m^2/g, 0.01 m^2/g to 4 m^2/g or 0.05 m^2/g to 1.9 m^2/g

8. A method according to any one of claims 5 to 7, wherein the reaction mixture comprises graphite in an amount in a range 5% to 60%, 9% to 50%, 14% to 40%, 16% to 35%, 18% to 30%, or 20% to 30% by weight prior to the HPHT growth cycle.

9. A method according to any one of claims 5 to 8, wherein the reaction mixture comprises powdered metal catalyst in an amount in the range 40% to 95%, 60% to 90%, 65% to 85%, or 70% to 80% by weight prior to the HPHT growth cycle.

10. A method according to any preceding claim, wherein the initiating step comprises raising the pressure to a target starting value P_s which is below the Berman-Simon graphite/diamond thermodynamic phase stability line, raising the temperature to a value T_g which exceeds a eutectic temperature for the carbon source and the metal catalyst, holding the temperature and pressure for a time t, and then raising the pressure to a target starting value P_g for the maintaining step to initiate HPHT growth.

11. A method according to claim 10, wherein the time t is in a range 1 to 36000 seconds, 20 to 24000 seconds, 40 to 15000 seconds, or 60 to 11000 seconds.

12. A method according to claim 10 or 11, wherein the temperature T_g is in a range 1070 Kelvin to 2470 Kelvin, 1370 Kelvin to 1970 Kelvin, 1520 Kelvin to 1770 Kelvin, or 1570 Kelvin to 1670 Kelvin.

13. A method according to any one of claims 10 to 12, wherein the pressure P_s is within 0.01 to 2.0 GPa, 0.05 to 1.5 GPa, 0.1 to 1 GPa, or 0.2 to 0.5 GPa of pressure P_g.

14. A method according to any one of claims 10 to 13, wherein the pressure P_g is in a range 4.0 to 8.0 GPa, 4.5 to 7.0 GPa, 5.0 to 6.0 GPa, or 5.2 to 5.7 GPa.

15. A method according to any one of claims 10 to 14, wherein the pressure is raised from P_s to P_g at a rate in a range 0.001 to 1.0 GPa per minute, 0.01 to 0.8 GPa per minute, 0.01 to 0.5 GPa per minute, or 0.05 to 0.3 GPa per minute.

16. A method according to any preceding claim, wherein, during the maintain step, a temperature difference between a top side and a bottom side of the capsule is maintained to be no more than 100 Kelvin, 75 Kelvin, 50 Kelvin, 30 Kelvin, 20 Kelvin, 10 Kelvin, 5 Kelvin, or 1 Kelvin.

17. A method according to any preceding claim, wherein, during the maintain step, a temperature gradient between a top side and a bottom side of the capsule is maintained to be no more than 0.66 Kelvin mm^{-1}, 0.50 Kelvin mm^{-1}, 0.33 Kelvin mm^{-1}, 0.20 Kelvin mm^{-1}, 0.13 Kelvin mm^{-1}, 0.07 Kelvin mm^{-1}, 0.03 Kelvin mm^{-1}, or 0.01 Kelvin mm^{-1}.

18. A method according to any preceding claim, wherein during the maintaining step the press moves anvils inwards whereby a volume of the capsule reduces by an amount in a range 0.5% to 50%, 0.5% to 30%, 1.0% to 25%, 2.0% to 20%, or 5% to 15%, and/or wherein during the maintaining step the press moves anvils inwards by a combined distance in the range 1 mm to 100 mm, 5 mm to 75 mm, 10 mm to 60 mm, 20 mm to 50 mm, or 20 mm to 40 mm.

19. A method according to any preceding claim, wherein during the maintaining step the temperature is decreased in a continuous or stepwise manner while maintaining growth of single crystal diamond material on the plurality of single crystal diamond seeds.

20. A method according to claim 19, wherein the temperature is decreased at a rate in a range 0.1 Kelvin/hour to 2 Kelvin/hour, 0.3 Kelvin/hour to 1.5 Kelvin/hour or 0.5 Kelvin/hour to 0.75 Kelvin/hour.

21. An apparatus configured to perform the method according to any preceding claim, the apparatus comprising:

a capsule comprising a plurality of seed pads and reactants including a carbon source and a metal catalyst, wherein the reactants and seed pads are provided in alternating layers, and wherein each seed pad comprises a plurality of single crystal diamond seeds anchored to, or embedded in, an inert holder; and

a HPHT press comprising a heating circuit configured to maintain a substantially uniform temperature throughout the capsule whereby diamond growth on the single crystal seeds is achieved via a pressure driven growth process.