

WHAT IS CLAIMED IS:

1. A method for exfoliation of graphene flakes from a graphite sample, the method
5 comprising:
compressing a graphite sample in an electrochemical reactor; and
applying a voltage between the graphite sample and an electrode in the electrochemical cell.
2. The method of claim 1, wherein compressing the graphite sample comprises:
10 pressing the graphite against an electrode member using a moveable ceramic membrane,
wherein the ceramic membrane is permeable to the electrolyte.
3. The method of claim 1, further comprising annealing hydrogenated graphene
flakes at 500 to 800°C to yield graphene flakes.
4. The method of claim 1, wherein the electrode member is a cathode.
- 15 5. The method of claim 4, wherein the graphite sample is in electrical contact with a
boron-doped diamond cathode member.
6. The method of claim 1, wherein the electrolyte comprises propylene carbonate
and 0.1 M tetrabutylammonium hexafluorophosphate.
7. The method of claim 1, wherein the applied voltage is -5 V to -100 V.
- 20 8. The method of claim 1, further comprising varying a force that compresses the
graphite sample.
9. The method of claim 8, wherein varying the force comprises reducing a pressure
pressing the graphite sample against the cathode member after 2-3 hours of applied voltage at -
60 V.
- 25 10. The method of claim 1, further comprising pelletizing the graphite sample.
11. The method of claim 1, wherein the voltage is applied for a total of 24 hours.
12. An apparatus for the exfoliation of graphite, the apparatus comprising:
an electrochemical reactor;
electrodes comprising

an anode;
a cathode member;
a graphite sample; and
a compression apparatus configured to compress the graphite sample during an
5 exfoliation reaction.

13. The apparatus according to claim 12, wherein the compression apparatus is configured to press the graphite sample against an electrode.

14. The apparatus according to claim 13, wherein the electrode is the cathode member.

10 15. The apparatus according to claim 12, wherein the graphite sample is free from binder.

16. The apparatus according to claim 12, further comprising at least two anodes.

17. The apparatus according to claim 14, wherein the cathode member comprises boron-doped diamond.

15 18. The apparatus according to any one of claims 14, wherein the cathode member comprises a metal film.

19. The apparatus according to claim 12, wherein the apparatus further comprises an electrolyte solution.

20 20. The apparatus according to claim 19, wherein the electrolyte solution comprises anhydrous propylene carbonate.

21. The apparatus according to claim 19, wherein the electrolyte solution further comprises 0.1 M tetrabutylammonium hexafluorophosphate.

22. The apparatus of any one of claim 12, wherein the compression apparatus comprises:
25 a ceramic membrane.

23. The apparatus of claim 22, wherein:
the ceramic membrane is disposed in a membrane press; and
the membrane press comprises

one or more rods, and
one or more force application mechanisms configured to apply force to the rods.