typically requires sonication, which results in small flake size. Cathodic exfoliation also requires a suitable graphite starting material. For example, highly oriented pyrolytic graphite (HOPG) is suitable for cathodic exfoliation, but HOPG can be costly.

Furthermore, cathodic exfoliation can occur at different layers distributed throughout different parts of the graphite, rather than layer-by-layer, starting from a surface. During cathodic exfoliation, large pieces of graphite can break away from the cathode. Once a piece breaks away from the cathode, electrical contact with the cathode is lost, and exfoliation within that piece stops.

This disclosure relates to the production of graphene, including an apparatus and a method for the expansion of graphite to graphene.

SUMMARY

An apparatus and a method for expansion of graphite to graphene, are described herein.

In one aspect, the present disclosure refers to a method for exfoliation of graphene flakes from a graphite sample, the method comprising:

compressing a graphite sample in an electrochemical reactor; and

applying a voltage between the graphite sample and an electrode in the electrochemical cell.

In a first ~~aspect~~example, a method for exfoliation of graphene flakes from a graphite sample includes compressing a graphite sample in an electrochemical reactor and applying a voltage between the graphite sample and an electrode in the electrochemical cell.

In a second ~~aspect~~example, combinable with any other ~~aspect~~examples, the method includes pressing the graphite against an electrode member using a moveable ceramic membrane, wherein the ceramic membrane is permeable to the electrolyte.

In a third ~~aspect~~example, combinable with any other ~~aspect~~examples, the method includes annealing hydrogenated graphene flakes at 500 to 800°C to yield graphene flakes.

In a fourth ~~aspect~~example, combinable with any other ~~aspect~~examples, the electrode member is a cathode. In a fifth ~~aspect~~example, combinable with any other ~~aspect~~examples, the graphite sample is in electrical contact with a boron-doped diamond cathode member.

In a sixth ~~aspect~~example, combinable with any other ~~aspect~~examples, the electrolyte includes propylene carbonate and 0.1 M tetrabutylammonium hexafluorophosphate.

In a seventh ~~aspect~~example, combinable with any other ~~aspect~~examples, the applied voltage is -5 V to -100 V.

In an eighth ~~aspect~~example, combinable with any other ~~aspect~~examples, the method includes varying a force that compresses the graphite sample.

In a ninth ~~aspect~~example, combinable with any other ~~aspect~~examples, varying the force includes reducing a pressure pressing the graphite sample against the cathode member after 2-3 hours of applied voltage at -60 V.

In a tenth ~~aspect~~example, combinable with any other ~~aspect~~examples, the method further includes pelletizing the graphite sample.

In an eleventh ~~aspect~~example, combinable with any other ~~aspect~~examples, the method further includes applying the voltage for a total of 24 hours.

In another aspect, the present disclosure refers to 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 exfoliation reaction.

In a twelfth ~~aspect~~example, combinable with any other ~~aspect~~examples, an apparatus for the exfoliation of graphite includes an electrochemical reactor, electrodes including an anode and a cathode member, a graphite sample, and a compression apparatus configured to compress the graphite sample during an exfoliation reaction.

In a thirteenth ~~aspect~~example, combinable with any other ~~aspect~~examples, the compression apparatus is configured to press the graphite sample against an electrode.

In a fourteenth ~~aspect~~example, combinable with any other ~~aspect~~examples, the electrode is the cathode member.

In a fifteenth ~~aspect~~example, combinable with any other ~~aspect~~examples, the graphite sample is free from binder.

In a sixteenth ~~aspect~~example, combinable with any other ~~aspect~~examples, the apparatus further includes at least two anodes.

In a seventeenth ~~aspect~~example, combinable with any other ~~aspect~~examples, the cathode member further includes boron-doped diamond.

In an eighteenth ~~aspect~~example, combinable with any other ~~aspect~~examples, the cathode member further includes a metal film.

In a nineteenth ~~aspect~~example, combinable with any other ~~aspect~~examples, the apparatus further includes an electrolyte solution.

In a twentieth ~~aspect~~example, combinable with any other ~~aspect~~examples, the electrolyte solution includes anhydrous propylene carbonate.

In a twenty-first ~~aspect~~example, combinable with any other ~~aspect~~examples, the electrolyte solution further includes 0.1 M tetrabutylammonium hexafluorophosphate.

In a twenty-second ~~aspect~~example, combinable with any other ~~aspect~~examples, the compression apparatus includes a ceramic membrane.

In a twenty-third ~~aspect~~example, combinable with any other ~~aspect~~examples, the ceramic membrane is disposed in a membrane press, and the membrane press includes one or more rods and one or more force application mechanisms configured to apply force to the rods.

The details of one or more implementations are set forth in the accompanying drawings and the description below. Other features, objects, and advantages will be apparent from the description and drawings, and from the claims.

DESCRIPTION OF DRAWINGS

FIG. 1 is a schematic representation of an apparatus for the expansion of graphite.
FIG. 2 is a flowchart illustrating an example method of expanding graphite.
FIG. 3 is an example X-ray diffraction spectrum of graphite and exfoliated graphite.
FIG. 4A is an example Raman spectrum of graphite before and after exfoliation in the range of 1400 to 2800 cm^{-1}.
FIG. 4B is an example Raman spectrum of graphite before and after exfoliation in the range of 2200 – 3200 cm^{-1}.
FIG. 5 is an example infrared (IR) spectrum of exfoliated graphite in the range of 4000-500 cm^{-1}.
FIG. 6 is an example Raman spectrum of hydrogenated graphene before and after annealing.
FIG. 7 is an example atomic force microscopy image of hydrogenated graphene flakes.
FIG. 8 is an example size evaluation of over 600 graphene flakes using optical microscope images.
FIG. 9 is an example of the electrical resistance of graphene flakes as a function of transparency, determined using the Van der Pauw method.

DETAILED DESCRIPTION

FIG. 1 shows an apparatus 1 that can be used to produce graphene using the methods described herein. The apparatus 1 includes an electrochemical chamber 100 and electrodes. The electrodes include at least one cathode 4 and at least one anode 2. The apparatus further includes an electrolyte 10 and a voltage or current source 12. The apparatus includes a graphite pellet 6.