

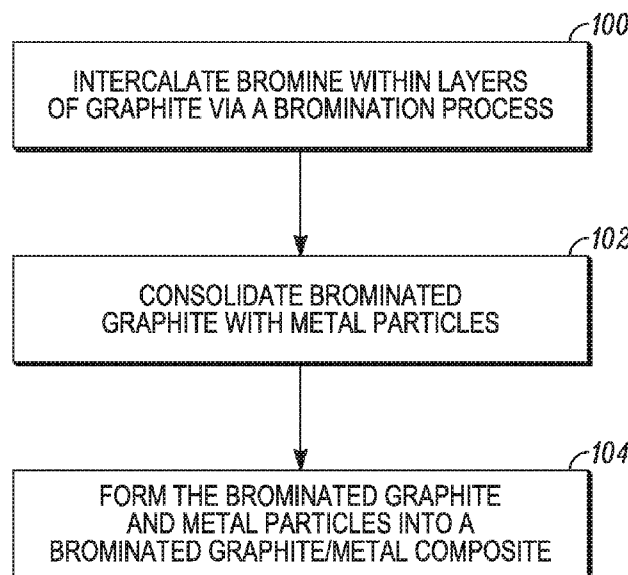


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[Continued on next page]

- (54) Title: BROMINE INTERCALATED GRAPHITE FOR LIGHTWEIGHT COMPOSITE CONDUCTORS



(57) Abstract: A method of fabricating a bromine-graphite/metal composite includes intercalating bromine within layers of graphite via liquid-phase bromination to create brominated-graphite and consolidating the brominated-graphite with a metal nanopowder via a mechanical pressing operation to generate a bromine-graphite/metal composite material.

FIG. 1



Declarations under Rule 4.17:

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BROMINE INTERCALATED GRAPHITE FOR LIGHTWEIGHT COMPOSITE CONDUCTORS

TECHNICAL FIELD

[0001] The present disclosure is related generally to a method of producing an electrically conductive composite.

BACKGROUND

[0002] Distribution of electrical power relies on conductive materials. For nearly every application that requires distributing electrical power, the conductive material relied upon is copper or some other metal.

[0003] However, although copper is highly conductive and low in cost, it has a relatively high mass density and therefore a high weight. In some applications, the weight added to the system by copper wires can become a significant factor. For example, in the aerospace industry, nearly 40% of the total aircraft weight resides in the electrical system, which may include tens of miles of copper wiring. Particularly with respect to the aerospace industry, weight reduction is one the driving forces in improving the fuel efficiency, range and operational costs of aircraft.

[0004] Therefore, it would be desirable to develop a light-weight replacement for the copper wire currently relied upon.

SUMMARY

[0005] Embodiments of the present disclosure describe a bromine-graphite/metal composite conductor and a method of fabricating the same. The method includes intercalating bromine within layers of graphite via liquid-phase bromination to create brominated-graphite. The brominated-graphite is consolidated with a metal nanopowder via a mechanical pressing operation to generate a bromine-graphite/metal composite material.

BRIEF DESCRIPTION OF THE DRAWINGS

[0006] Figure 1 is a flowchart illustrating fabrication of brominated graphite/metal composite for use as a conductor according to an embodiment of the present invention.

[0007] Figures 2a-2e are schematics that illustrate visually the fabrication of bromine intercalated graphite as described with respect to Figure 1, according to an embodiment of the present invention.

[0008] Figures 3a-3c are scanning electron microscope (SEM) images of graphite, brominated graphite, and brominated reduced graphite according to an embodiment of the present invention; Figure 3d is a chart illustrating elemental composition of carbon (C), Oxygen (O), and Bromine (BR) in graphite, Br-graphite, and Br-R-graphite obtained from energy dispersive spectroscopy (EDS) according to an embodiment of the present invention; Figure 3e is an elementary mapping images of brominated graphite, and Figures 3f and 3g are dark field images of Br-graphite, and dark field images of carbon (C) and bromine (Br), respectively, according to an embodiment of the present invention.

[0009] Figure 4a is an optical images of brominated graphite; Figures 4b-4d are Raman mapping image corresponding to G-peak, 2D-peak intensity and Br peak intensity, respectively, and Figures 4e-4g are Rama spectra scans for graphite and Br-graphite , the G-band, and 2D-band of graphite and Br-graphite, respectively, according to an embodiment of the present invention.

[0010] Figures 5a-5b are XRD and TGA analysis, respectively, of graphite and Br-graphite according to an embodiment of the present invention.

[0011] Figures 6a-6f are high-resolution transmission electron microscopy (HRTEM) images of pristine graphite at different scales, and brominated graphite at different scales according to embodiments of the present invention; Figures 6g-6i are images of pristine graphite and brominated graphite, respectively, and Figures 6h and 6j illustrate interlayer distance analysis of the pristine graphite and brominated graphite shown in Figures 6g and 6i, respectively, according to an embodiment of the present invention.

[0012] Figures 7a and 7b illustrate SEM and elemental analysis from EDS spectrum of pure copper powder and copper powder obtained from copper paste, respectively, according to an embodiment of the present invention.

[0013] Figures 8a-8e are SEM images of pure copper, 10% Br-graphite/copper, 20% Br-graphite/copper, 30% Br-graphite/copper, and 40% Br-graphite/copper, respectively; Figure 8f is a chart that illustrates elemental concentration of carbon, bromine and copper from EDS analysis.

[0014] Figure 9a is an SEM image of Br-graphite/Cu composite and Figures 9b-9d illustrate elemental mapping for elements carbon, copper and bromine, respectively, according to an embodiment of the present invention.

[0015] Figures 10a-10i are optical images of bulk copper, bulk silver and composites, with Raman spectra insets overlayed on top of the optical images according to an embodiment of the present invention.

[0016] Figures 11a and 11c illustrate the electrical conductivity of Br-graphite/copper and Br-graphite/silver composites, respectively; Figures 11b and 11d illustrate the density of Br-graphite/copper and Br-graphite/silver composites, respectively.

[0017] Figures 12a and 12b illustrate the specific conductivity of brominated graphite/copper and brominated graphite/silver composites, respectively, according to embodiments of the present invention.

[0018] Figure 13a illustrates the specific conductivity of graphite/copper composites – both with and without bromination; and Figure 13b illustrates specific conductivity of a graphite and brominated graphite (no metal) using different graphite powder size according to various embodiments of the present invention.

[0019] Figure 14 compares the specific conductivity of various metals as compared with composites that include graphite/copper, brominated graphite/copper, and brominated graphite/silver, and with a brominated graphite (no metal) according to various embodiments of the present invention.

[0020] Figures 15a and 15b illustrate the thermal stability of brominated graphite/copper and brominated graphite/silver composites, respectively according to embodiments of the present invention.

DETAILED DESCRIPTION

[0021] The present invention provides a method for fabricating lightweight, conducting metal-carbon composites. In one embodiment, the electrical conductivity of the composite is improved by using a liquid-phase brominated graphite. The liquid-phase brominated graphite may be intercalated with various conductors, such as copper or silver in a mechanical pressing operation to provide a brominated graphite/metal composite.

[0022] Figure 1 is a flowchart illustrating fabrication of a brominated graphite/metal composite for use as a conductor according to an embodiment of the present invention. The steps described with respect to Figure 1 are illustrated with respect to Figures 2A-2E.

[0023] At step 100, a brominated graphite is created by intercalating bromine within layers of graphite. In one embodiment, a liquid-phase bromination process is utilized to create the brominated graphite. At step 102, the brominated graphite is consolidated with metal particles, such as copper or silver, via a mechanical pressing operation to form a brominated graphite/metal composite material. At step 104, the brominated graphite is formed into a desired shape based on the application.

[0024] Figures 2a-2e illustrate visually the fabrication of a brominated graphite/metal composite as described with respect to Figure 1. Figure 2a illustrates the graphite layers 106 provided as an input to the fabrication process. In one embodiment, the graphite layers are comprised of pristine graphite, and in other embodiments the graphite layers are comprised of reduced graphite. A plurality of layers may be utilized.

[0025] Figure 2b illustrates the bromination process utilized to intercalate bromine particles within the graphite layers 106 (step 100, above). In the embodiment shown in Figure 2b, graphite layers 106 are placed into a container of liquid-phase bromine 108. Figure 2c illustrates the bromine intercalated graphite that results from the liquid-phase bromination process. In particular, bromine particles 112 are inserted between the plurality of graphite layers 106. In one embodiment, bromination of pristine graphite and/or reduced graphite results in percent mass of approximately 22% bromine, uniformly distributed between graphitic layers and on the graphite surfaces. In the embodiment shown in Figure 2c, the bromine particles 112 are distributed relatively uniformly between the layers of graphite 106.

[0026] Figure 2d illustrates consolidation of the brominated graphite 110 with a metal composition 114. In one embodiment, the metal composition is comprised of copper, but in other embodiments may include other conductive metal such as silver. In addition, as described in more detail below, the metal composition may be derived from a paste of a powder (e.g., pure copper powder, copper powder obtained from copper paste, silver powder obtained from silver paste).

[0027] Figure 2e illustrates a brominated graphite/metal composite 116 pressed into a desired form. In one embodiment, the brominated graphite/metal composite is formed via a mechanical pressing of the brominated graphite 110 with the metal composition 114. In one embodiment, a mechanical pressing operation is employed to intercalate the brominated graphite with the metal composition (e.g., copper nanopowder). Various mechanical pressing operations may be utilized, including both hot press and cold press operations, depending on the desired attributes of the finished composite. The result is a bromine-graphite/metal composite having a conductivity greater than that of copper (i.e., lower resistivity than copper, approximately 1.7×10^{-9} Ohm.m), while providing a density less than that of copper (e.g., less than approximately 8.96 g/cm^3). In one embodiment, a composite fabricated according to the method described in Figure 1 (and shown in Figures 2a-2e), the composite having a 40% weight of Br-graphite/pure copper displayed an electrical resistance of 6.9×10^{-9} Ohm.m (1.45×10^8 S/m) at a density of 3.5 g/cm^3 . In this way, the bromine intercalated graphite/metal conductor fabricated according to the present invention provides improved conductivity at a reduced weight.

[0028] Figures 3a-10f illustrate - using various type of imaging – the inputs and results of the various process steps described with respect to Figures 1 and 2a-2e. For example, Figures 3a-3g provide images and descriptions of graphite prior to the liquid-phase bromination and the resulting brominated graphite following the liquid-phase bromination. Figures 4a-4g provide additional elemental analysis of brominated graphite following the liquid-phase bromination step. Figures 6a-6j provide additional imagery of both pristine graphite prior to liquid-phase bromination and brominated graphite following the liquid-phase bromination step. Figures 7a-7b provide imagery of various types of metal (e.g., copper) nanopowder that may be consolidated with the brominated graphite to form the bromine intercalated graphite/metal composite. Figures 8a-8f are images that illustrate various elemental concentrations of graphite, bromine and copper. Figures 9a-9d similarly provide additional images of a bromine/graphite/copper composite that is generated as a result of the process shown in Figures 1 and 2a-2e.

[0029] Figures 3a-3c are scanning electron microscope (SEM) images of graphite, brominated graphite, and brominated reduced graphite, respectively, according to an embodiment of the present invention. The SEM images are illustrated at a scale of approximately 50 micrometers (μm). In addition, spectroscopy results are displayed on top of the SEM images in order to illustrate the composition of each image. For example, Figure 3a illustrates an SEM image of graphite, and spectroscopy confirms that carbon is the most prevalent element (indicated by the label “C”, for carbon, with respect to the peak of the spectroscopy results). Figure 3b is an SEM image of brominated graphite (Br-graphite) intercalated between the graphite layers. The spectroscopy confirms the presence of carbon and bromine (as indicated by the first peak labeled “C” and the second peak labeled “Br” for bromine). Figure 3c illustrates brominated reduced graphite (Br-R-graphite). Once again, spectroscopy confirms the presence of carbon “C” and bromine “Br”.

[0030] Figure 3d is a chart illustrating elemental composition of carbon (C), Oxygen (O), and Bromine (BR) in pristine graphite, Br-graphite, and Br-R-graphite obtained from energy dispersive spectroscopy (EDS) according to an embodiment of the present invention. As shown in Figure 3d, both graphite and reduced graphite are composed nearly entirely of carbon (two left-most bars associated with C). No oxygen is present in reduced graphite (R-graphite), but trace amounts are detected in the pristine graphite. The brominated graphite (Br-graphite) and brominated reduced graphite (Br-R-graphite) both are comprised of approximately 70-80% carbon (C), with the brominated reduced graphite being comprised of slightly more carbon than

the brominated graphite. In addition, both the brominated graphite and brominated reduced graphite are comprised of approximately 20-25% bromine (Br).

[0031] Figure 3e is an elementary mapping image of brominated graphite, and Figures 3f and 3g are dark field images that illustrate the elemental distribution of carbon (C) and bromine (Br), respectively, from the brominated graphite shown in Figure 3e.

[0032] Figure 4a is an optical images of brominated graphite. Figures 4b-4d are Raman mapping image corresponding to G-peak, 2D-peak intensity and Br peak intensity, respectively, at a scale approximately half of that shown in Figure 4a. Figures 4e is a full Raman spectra scan for graphite and brominated graphite, with graphite illustrated with the solid line 400 and the brominated graphite illustrated with dashed line 402. Figure 4f is a magnified view of a G-band Raman scan (focusing on wavenumbers in the range of $1560\text{-}1650\text{ cm}^{-1}$), with graphite once again illustrated with a solid line 400 and brominated graphite illustrated with a dashed line 402. Finally, Figure 4g is a magnified view of a 2D-band Raman scan (focusing on wavenumbers in the range of $2600\text{-}2900\text{ cm}^{-1}$), with graphite illustrated with a solid line 400 and brominated graphite illustrated with a dashed line 402.

[0033] Figures 5a-5b are X-ray diffraction (XRD) and thermogravimetric analysis (TGA), respectively, of graphite and Br-graphite according to an embodiment of the present invention. The XRD analysis provides phase identification of the material being analyzed – in particular the spacing of the lattice planes - and provides peak positions at 2θ (deg). The XRD analysis shown in Figure 5a, a zoomed-in view of the peaks (shown in the inset, in the range of 25-28 degrees) illustrates that the peak position of brominated graphite (dashed line 502) is slightly shifted from the peak position of graphite (solid line 500).

[0034] The TGA analysis shown in Figure 5b illustrates changes in physical and chemical properties of material – expressed as weight percentage - as measured as a function of increasing temperature. As illustrated, the TGA analysis of graphite (solid line 504) illustrates very little change in percentage weight over the given temperature range (approximately $50\text{-}600^{\circ}$ Celsius). In contrast, the TGA analysis of brominated graphite (dashed line 506) illustrates a substantial change in percentage weight (approximately 75% of original weight) over the given temperature range.

[0035] Figures 6a-6f are high-resolution transmission electron microscopy (HRTEM) images of pristine graphite at different scales, and brominated graphite at different scales according to embodiments of the present invention. Figure 6a illustrates pristine graphite at a scale of $0.5\text{ }\mu\text{m}$, while Figure 6b illustrates pristine graphite at a scale of 100 nm , and Figure 6c illustrates pristine graphite at a scale of 5 nm . Figures 6d-6f illustrate brominated graphite at the same

scales (0.5 μm , 100 nm, and 5 nm). In particular, Figures 6d-6f illustrate bromine intercalated into the graphite layers.

[0036] Figures 6g-6i are images of pristine graphite and brominated graphite, respectively. Figures 6h and 6j provide interlayer distance analysis of the pristine graphite and brominated graphite shown in Figures 6g and 6i, respectively, via inverse fast fourier transform (FFT) of the magnified images shown in Figures 6h and 6j. As illustrated in the comparison of Figures 6h and 6j, the interlayer distance of the brominated graphite is greater than the interlayer distance of the pristine graphite. In the embodiment shown in Figures 6h and 6j, the interlayer distance of pristine graphite is approximately 0.33 nm, while the interlayer distance of brominated graphite is 0.4 nm.

[0037] Figures 7a and 7b illustrate scanning electron microscope (SEM) images and elemental analysis from EDS spectrum (inset within Figures 7a and 7b) of pure copper powder and copper powder obtained from copper paste, respectively, according to an embodiment of the present invention. The elemental analysis of the pure copper powder shown in Figure 7a indicates that the copper powder is comprised almost entirely of copper iodide (e.g., elemental analysis indicated Wt% of 100%). In contrast, the elemental analysis of the copper powder obtained from a copper paste shown in Figure 7b indicates that the copper powder is comprised of a combination of carbon, oxygen and copper per the percentage weights given.

[0038] Figures 8a-8e are SEM images of pure copper, 10% Br-graphite/copper, 20% Br-graphite/copper, 30% Br-graphite/copper, and 40% Br-graphite/copper, respectively; Figure 8f is a chart that illustrates elemental concentration of carbon, bromine and copper from EDS analysis, with respect to the percentage of bromine-graphite filler by percentage. Figure 8a provides an image of pure copper, without the presence of any composites. Figures 8b-8e provide images of bromine-graphite/copper composites generated as a result of the mechanical pressing operation described with respect to Figures 1 and 2, with various combinations of elemental concentrations. As illustrated in Figure 8f, as the bromine-graphite filler material increases in percentage, the elemental content of carbon (line 800) increases in percentage weight while the elemental content of copper (line 802) decreases in percentage weight. The elemental content of bromine (line 804) increases as the bromine-graphite filler material percentage increases, but at a slower rate.

[0039] Figure 9a is an SEM image of Br-graphite/Cu composite and Figures 9b-9d illustrate elemental mapping for elements carbon, copper and bromine, respectively, according to an embodiment of the present invention. In particular, Figures 9b-9d illustrate elemental mapping for the region indicated by the box 900 shown in Figure 9a. Figure 9b illustrates elemental

mapping of carbon elements in the Br-graphite/Cu composite. Figure 9c illustrates elemental mapping of copper elements in the Br-graphite/Cu composite, and Figure 9d illustrates mapping of bromine elements in the Br-graphite/Cu composite. As indicated in this embodiment, copper (shown in Figure 9c) is the most prevalent element, with the bromine-graphite filler material being less prevalent, and in particular with bromine (shown in Figure 9d) being less prevalent than carbon (shown in Figure 9b).

[0040] Figures 10a-10i are optical images of bulk copper, bulk silver and composites, with Raman spectra insets overlayed on top of the optical images according to an embodiment of the present invention. In particular, Figures 10a-10c illustrate images resulting from the combination of copper powder derived from a bulk copper paste with brominated graphite. Figure 10a is an image of the bulk copper paste utilized to form the bromine-graphite/copper composite illustrated in Figures 10b and 10c. In particular, circle 1000 shown in Figure 10b is focused on a graphite rich region of a bromine-graphite/copper composite, while circle 1002 shown in Figure 10c is focused on a copper rich region of the same bromine-graphite/copper composite.

[0041] Similarly, Figures 10d-10f illustrate images resulting from the combination of pure copper powder with brominated graphite. Figure 10d is an image of the pure copper powder utilized to form the bromine-graphite/copper composite illustrated in Figures 10e and 10f. In particular, circle 1004 in Figure 10e is focused on a graphite rich region of a bromine-graphite/copper composite, while circle 1006 in Figure 9f is focused on a copper rich region of the same bromine-graphite/copper composite.

[0042] Figures 10g-10i illustrate images resulting from the combination of silver paste with brominated graphite. Figure 10g is an image of the silver paste utilized to form a bromine-graphite/silver composite illustrated in Figures 10h and 10i. In particular, Figure 10h is an image focused on a graphite rich region of a bromine-graphite/silver composite, while Figure 10i is an image focused on a silver rich region of the same bromine-graphite/silver composite. The inset shown in Figure 10h shows splitting of the G-peak, indicating Bromine intercalated graphite in copper matrix.

[0043] Figures 11a and 11c illustrate the electrical conductivity of Br-graphite/copper and Br-graphite/silver composites, respectively, while figures 11b and 11d illustrate the density of Br-graphite/copper and Br-graphite/silver composites, respectively. Figures 11f and 11d are digital images of Br-graphite/copper composites and Br-graphite/silver composites fabricated according to various embodiments of the present invention.

[0044] In particular, Figure 11a illustrates the electrical conductivity of bromine-graphite/copper (Br-graphite/Cu) formed according to a mechanical hot press operation (line 1100), according to

a mechanical cold pressure operation (line 1102) as well as the conductivity of bromine-graphite/pure copper (Br-graphite/Pure Cu) (line 1104) according to a mechanical cold press operation at various percentages of bromine-graphite filler materials (e.g., 0%, 10%, 20%, 30%, and 40%). As indicated in Figure 11a, the bromine-graphite/pure copper (Br-graphite/Pure Cu) shown by line 1104 – with 40% bromine-graphite filler – had the highest electrical conductivity (approximately 1.45×10^8 S/m). For both the bromine-graphite/pure copper (cold press, line 1104) and bromine-graphite/copper (cold press, line 1102) embodiments, conductivity increased with an increase in the bromine-graphite filler percentage. In contrast, for the bromine-graphite/copper (hot press, line 1100), the conductivity peaked with a bromine-graphite percentage of 10% and 20%, and decreased when the bromine-graphite percentage was increased to 40%.

[0045] Figure 11b illustrates the density of the materials described with respect to Figure 11a, including Br-graphite/Cu (hot press, line 1110), Br-graphite/Cu (cold press, line 1112) and Br-graphite/Pure Cu (cold press, line 1114), measured at various percentages of bromine-graphite filler materials (e.g., 0%, 10%, 20%, 30%, and 40%). For each sample, the density of the material decreased as the percentage of bromine-graphite filler increased. Thus, for each embodiment the lowest density (and thus lowest weight) occurred at 40% percentage weight of bromine-graphite. In general, the bromine-graphite/copper (cold press, line 1112) materials displayed the lowest density, although the difference was less pronounced at higher percentage weights of bromine-graphite.

[0046] Figures 11c and 11d provide information similar to that shown in Figures 11a and 11b, but rather than bromine-graphite/copper, Figures 11c and 11d show conductivity and density measurements taken for bromine-graphite/silver (Ag) consolidated via a hot press operation. Once again, various percentage weights of bromine-graphite filler material were studied (e.g., 10%, 20%, 30% and 40%). The best conductivity was measured with respect to the bromine-graphite/Ag composite having a bromine-graphite percentage weight of 30%. Figure 11d illustrates once again that density (in general) decreases as the percentage of bromine-graphite is increased – although in the embodiment shown the density did increase slightly when the bromine-graphite percentage was increased from 20% to 30%.

[0047] Figures 12a and 12b illustrate the specific conductivity of brominated graphite/copper and brominated graphite/silver composites, respectively, according to embodiments of the present invention. In particular, Figure 11a illustrates the specific conductivity of bulk copper (labeled “1202”) as compared with the specific conductivity of brominated graphite/copper with various percentage weights of brominated graphite (labeled by

line “1200”). In particular, brominated graphite/copper composites comprised of a percentage weight of brominated graphite of approximately 10-20% provides the highest level of specific conductivity. As the percentage weight of brominated graphite continues to increase, the specific conductivity decreases accordingly. The upper right hand corner of the chart illustrates the same information shown in the larger graph, but on a logarithmic scale, which illustrates the decrease in specific conductivity as the percentage weight of brominated graphite increases to 50-60%.

[0048] Similarly, Figure 12b illustrates the specific conductivity of bulk silver (labeled “1206”) as compared with the specific conductivity of brominated graphite/silver with various percentage weights of brominated graphite (labeled by line “1204”). In particular, brominated graphite/silver composites comprised of a percentage weight of brominated graphite of approximately 120% provides the highest level of specific conductivity. As the percentage weight of brominated graphite continues to increase, the specific conductivity of the brominated graphite/silver composite decreases accordingly. The upper right hand corner of the chart illustrates the same information shown in the larger graph, but on a logarithmic scale, which illustrates the decrease in specific conductivity as the percentage weight of brominated graphite increases to 50-60%.

[0049] Figure 13a illustrates the specific conductivity of graphite/copper composites – both with and without bromination; and Figure 12b illustrates specific conductivity of a bromine/graphite solid (without metal) using different graphite powder size and with and without bromination according to various embodiments of the present invention. In particular, Figure 12a illustrates the specific conductivity of a graphite/copper (Gr/Cu) composite (indicated by line “1302”) as compared with the specific conductivity of a brominated graphite/copper (Br-Gr/Cu) composite (indicated by line “1300) over various percentage weights of graphite (e.g., 0%, 10%, 20%, 30%, 40%, 50% and 60%). As illustrated, the brominated graphite/copper (Br-Gr/Cu) composite (line “1300”) provides higher specific conductivity than the graphite/copper (Gr/Cu) composite (line “1302”).

[0050] In contrast, Figure 13b illustrates the specific conductivity of a plurality of graphite solids (i.e., no metal such as copper) and a plurality of brominated graphite solids (again, no metal such as copper) using large graphite flakes, small graphite flakes, and a mix or combination of large and small graphite flakes. The embodiment shown in Figure 12b illustrates that graphite solid (labeled “Gr-S”, “Gr-L”, and “Gr-Mix”) – without bromination – provides a lower specific conductivity than brominated graphite (in each case less than 3000 S.m²/kg) (labeled “Br-Gr-S”, “Br-Gr-L”, and “Br-Gr-Mix”). In contrast, brominated graphite formed

from large graphite flakes (i.e., graphite powder size) (labeled “Br-Gr-L”) provided the highest specific conductivity (approximately 15,000 S.m²/kg). Brominated graphite formed from small graphite (labeled “Br-Gr-S”) provided a lower specific conductivity, while brominated graphite formed from a mix of small and large flakes (labeled “Br-Gr-Mix”) increased the specific conductivity from that illustrated by the small flakes.

[0051] Figure 14 compares the specific conductivity of various metals as compared with composites that include graphite/copper, brominated graphite/copper, and brominated graphite/silver, and a brominated graphite solid according to various embodiments of the present invention. The various metals include iron (Fe), gold (Au), silver (Ag), copper (Cu) and aluminum (Al), and are illustrated along the x-axis starting from the left-most side of the chart. As illustrated, aluminum (Al) has the highest specific conductivity at approximately 12,960 S.m²/kg, with copper (Cu) next at approximately 6,650 S.m²/kg, and silver (Ag) at approximately 6000 S.m²/kg. Both iron (Fe) and gold (Au) have lower specific conductivities.

[0052] To the right of the plurality of metals illustrated are a plurality of composites, including a graphite/copper composite (Cu/Gr), a brominated graphite/silver (Ag/Br-Gr) composite, and a brominated graphite/copper (Cu/Br-Gr) composite. In addition, a solid comprised of brominated graphite (Br-Gr) is illustrated which does not include any metal. Of the various composites and solid, the brominated graphite/copper (Cu/Br-Gr) composite demonstrated the highest specific conductivity with a value of approximately 29,400 S.m²/kg (according to this embodiment). The brominated graphite/silver composite (Ag/Br-Gr) demonstrates the next highest level of specific conductivity with a value of approximately 24,000 S.m²/kg (according to this embodiment). The brominated graphite solid (Br-Gr) demonstrates the next highest level of specific conductivity with a value of approximately 14,700 S.m²/kg, which represents a two-fold increase in conductivity over copper (Cu). The non-brominated copper-graphite (Gr/Cu) demonstrates a specific conductivity of approximately 11,500 S.m²/kg, less than that of aluminum.

[0053] Figures 15a and 15b illustrate the thermal stability of brominated graphite/copper and brominated graphite/silver composites, respectively according to embodiments of the present invention. In particular, Figure 15a illustrates via line 1500 initial specific conductivities of brominated graphite/copper (Br-graphite/Cu) composites at various percentage weights of brominated-graphite as illustrated along the x-axis (e.g., 0%, 10%, 20%, 30%, 40%, 50%, and 60%). The brominated graphite/copper composites were then heated at a temperature of 500 degrees Celsius for a period of one hour and the specific conductivity of each was re-measured, illustrated by line 1502. As illustrated in Figure 15a, the brominated graphite/copper composites

show very good thermal stability, with only slight decreases in specific conductivity illustrated at higher percentage weights (e.g., 40%, 50%) of brominated-graphite.

[0054] Figure 15b similarly illustrates the thermal stability of brominated graphite/silver (Br-graphite/Ag) composites. In particular, Figure 15b illustrates via line 1504 initial specific conductivities of brominated graphite/silver (Br-graphite/Ag) composites at various percentage weights of brominated-graphite as illustrated along the x-axis (e.g., 0%, 10%, 20%, 30%, 40%, 50%, and 60%). The brominated graphite/silver composites were then heated at a temperature of 500 degrees Celsius for a period of one hour and the specific conductivity of each was re-measured, illustrated by line 1506. As illustrated in Figure 15b, the brominated graphite/silver composites show very good thermal stability, with very little decrease in specific conductivity. In addition, Figure 15b illustrates heating the brominated graphite/silver composites at a temperature of 700 degrees Celsius for a period of one hour, and then again measuring the specific conductivity of brominated/graphite/silver composite as illustrated by the triangular data points (line 1508) shown at 0%, 10%, and 20% percentage weights. Once again, the brominated-graphite/silver composite illustrates good thermal stability, with very little change in the specific conductivity of the composite following the heating cycle.

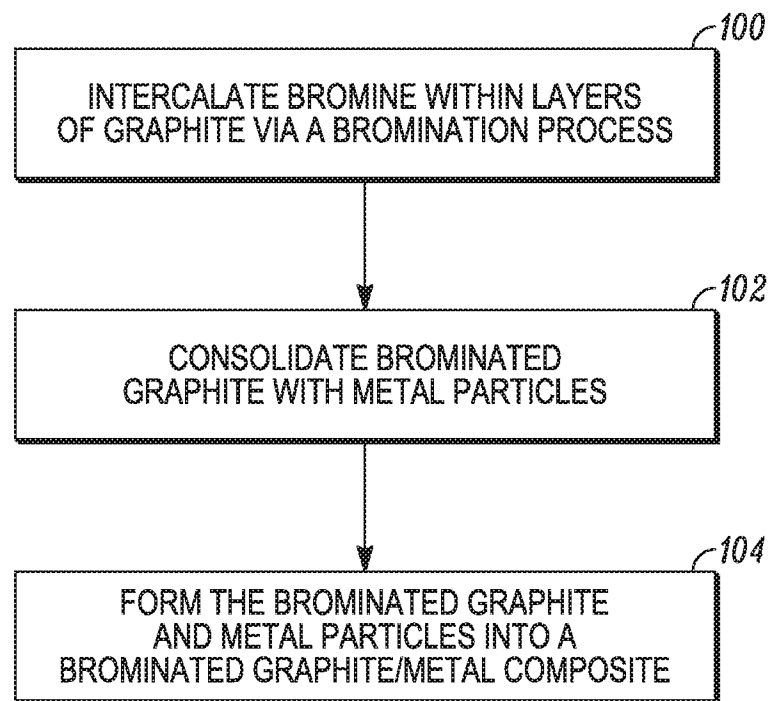
[0055] While the invention has been described with reference to an exemplary embodiment(s), it will be understood by those skilled in the art that various changes may be made and equivalents may be substituted for elements thereof without departing from the scope of the invention. In addition, many modifications may be made to adapt a particular situation or material to the teachings of the invention without departing from the essential scope thereof. Therefore, it is intended that the invention not be limited to the particular embodiment(s) disclosed, but that the invention will include all embodiments falling within the scope of the appended claims.

CLAIMS:

1. A method of fabricating a bromine-graphite/metal composite, the method comprising:
intercalating bromine within layers of graphite via liquid-phase bromination to create
brominated-graphite; and
consolidating the brominated-graphite with a metal nanopowder via a mechanical
pressing operation to generate a bromine-graphite/metal composite material.
2. The method of claim 1, wherein the mechanical pressing operation is a hot press
operation.
3. The method of claim 1, wherein the mechanical pressing operation is a cold press
operation.
4. The method of claim 1, wherein the bromine-graphite/metal composite material has a
percentage weight of bromine-graphite of greater than 10%.
5. The method of claim 1, wherein the bromine-graphite/metal composite material has a
percentage weight of bromine-graphite greater than 20%.
6. The method of claim 1, wherein the bromine-graphite/metal composite material has a
percentage weight of bromine-graphite greater than 30%.
7. The method of claim 1, wherein the bromine-graphite/metal composite material has a
percentage weight of bromine-graphite greater than 40%.
8. The method of claim 1, wherein the metal nanopowder is a pure copper nanopowder.
9. The method of claim 1, wherein the metal nanopowder is a copper powder obtained from
a copper paste.
10. The method of claim 1, wherein the metal is a silver nanopowder obtained from a silver
paste.
11. The method of claim 1, wherein the bromine-graphite/metal composite material has an
electrical conductivity greater than 6.0×10^7 S/m.

12. The method of claim 1, wherein the bromine-graphite/metal composite material has an electrical conductivity of approximately 1.45×10^8 S/m.
13. The method of claim 1, wherein the bromine-graphite/metal composite material has a density of less than 8.9 g/cm^3 .
14. The method of claim 1, wherein the bromine-graphite/metal composite material has a density of approximately 3.5 g/cm^3 .
15. A bromine-graphite/metal composite prepared by a process comprising the steps of:
 - intercalating bromine within layers of graphite via liquid-phase bromination to create brominated-graphite; and
 - consolidating the brominated-graphite with a metal nanopowder via a mechanical pressing operation to generate a bromine-graphite/metal composite material.
16. The bromine-graphite/metal composite of claim 15 having a percentage weight of bromine-graphite of more than 10%.
17. The bromine-graphite/metal composite of claim 15 having a percentage weight of bromine-graphite of more than 20%.
18. The bromine-graphite/metal composite of claim 15 having a percentage weight of bromine-graphite of more than 30%.
19. The bromine-graphite/metal composite of claim 15 having a percentage weight of bromine-graphite of more than 40%.
20. The bromine-graphite/metal composite of claim 15, wherein the metal is a pure copper nanopowder.
21. The bromine-graphite/metal composite of claim 15, wherein the metal is a copper powder obtained from a copper paste.
22. The bromine-graphite/metal composite of claim 15, wherein the bromine-graphite/metal composite material has an electrical conductivity greater than 6.0×10^7 S/m.

23. The bromine-graphite/metal composite of claim 15, wherein the bromine-graphite/metal composite material has an electrical conductivity of approximately 1.45×10^8 S/m.
24. The bromine-graphite/metal composite of claim 15, wherein the bromine-graphite/metal composite material has a density of less than 8.9 g/cm^3 .
25. The bromine-graphite/metal composite of claim 15, wherein the bromine-graphite/metal composite material has a density of approximately 3.5 g/cm^3 .

*FIG. 1*

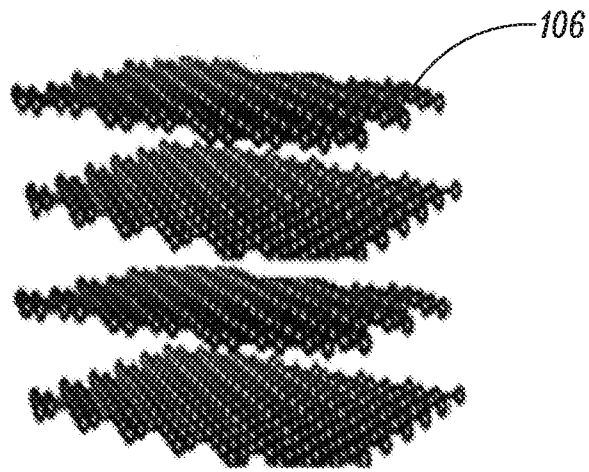


FIG. 2A

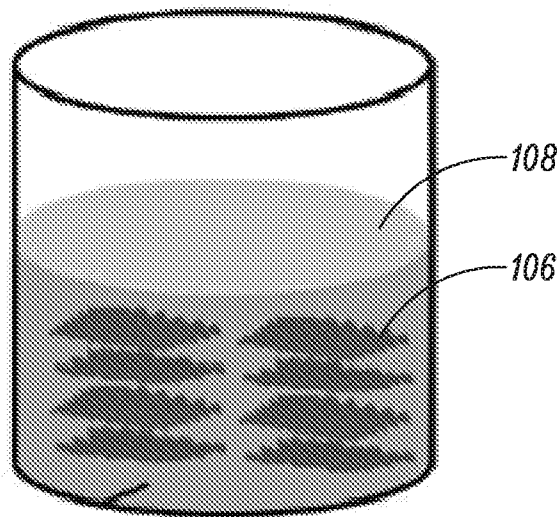


FIG. 2B

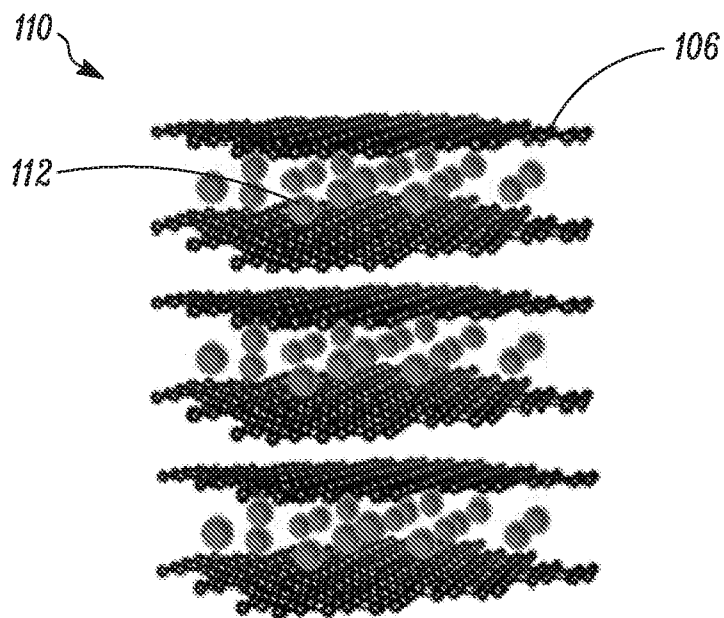


FIG. 2C

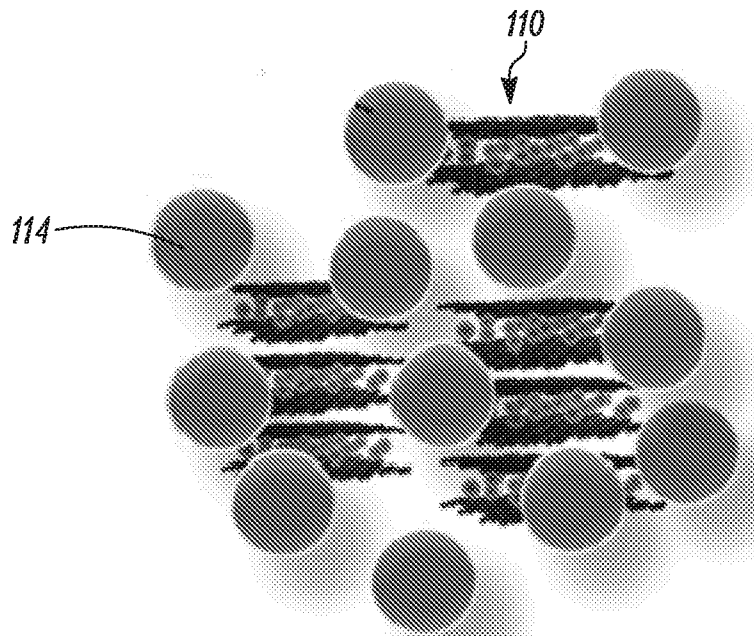


FIG. 2D

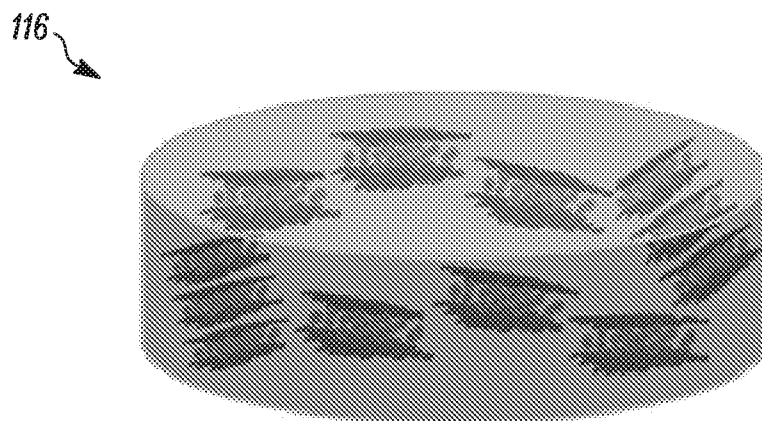
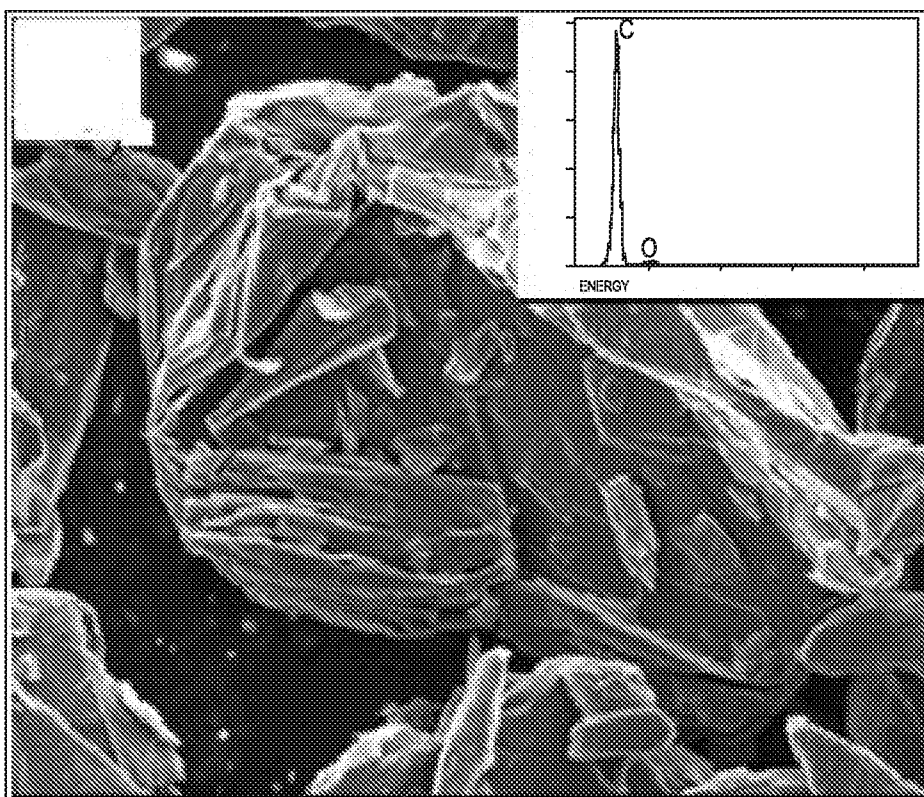
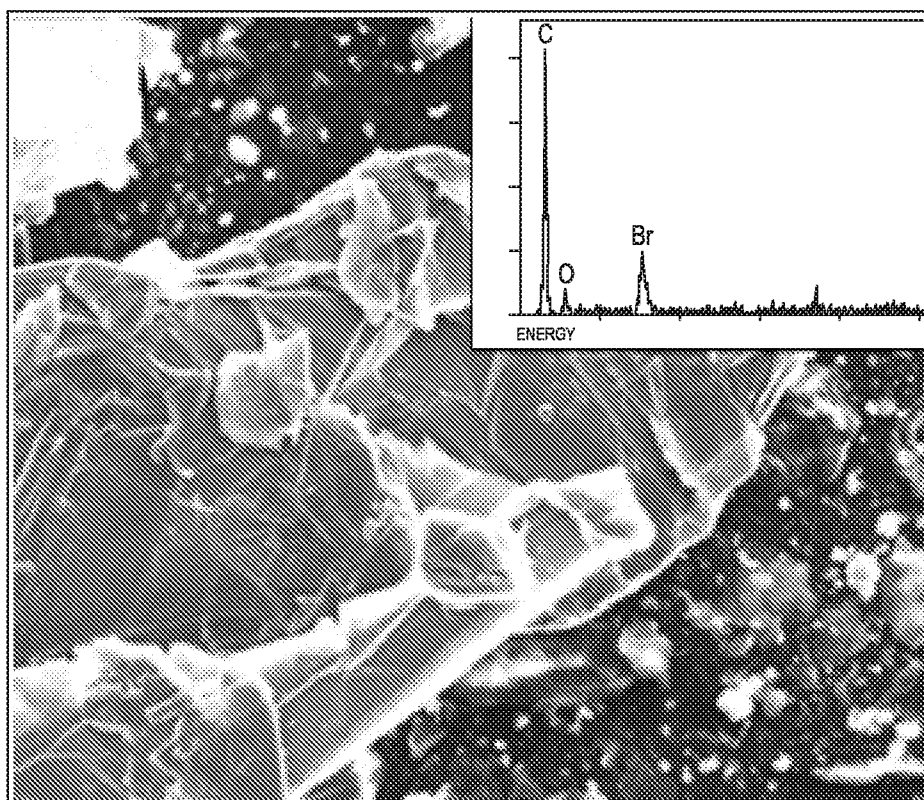
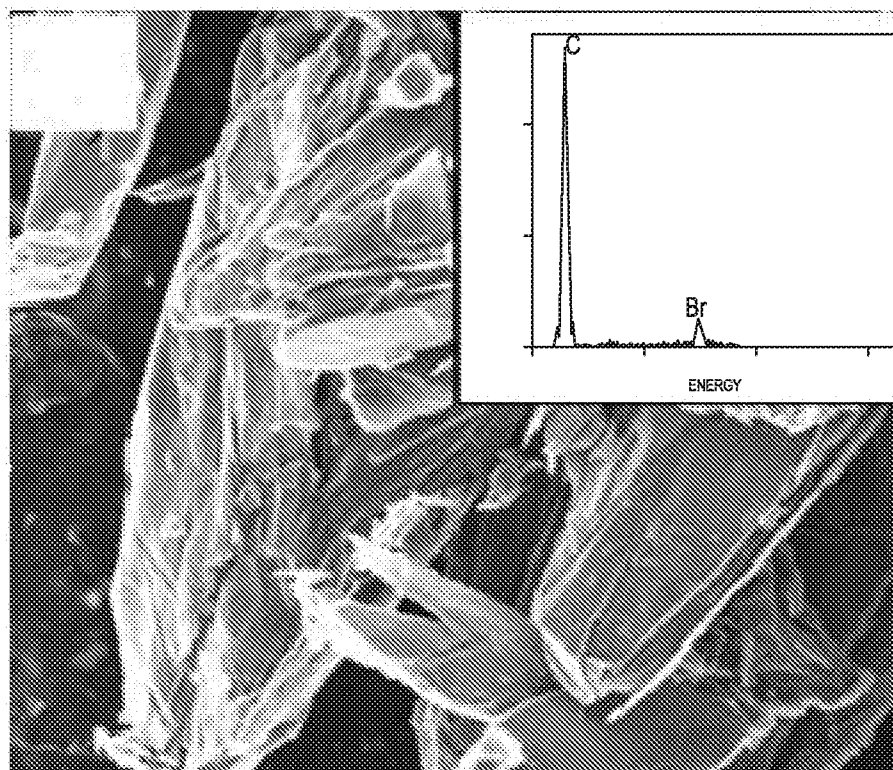
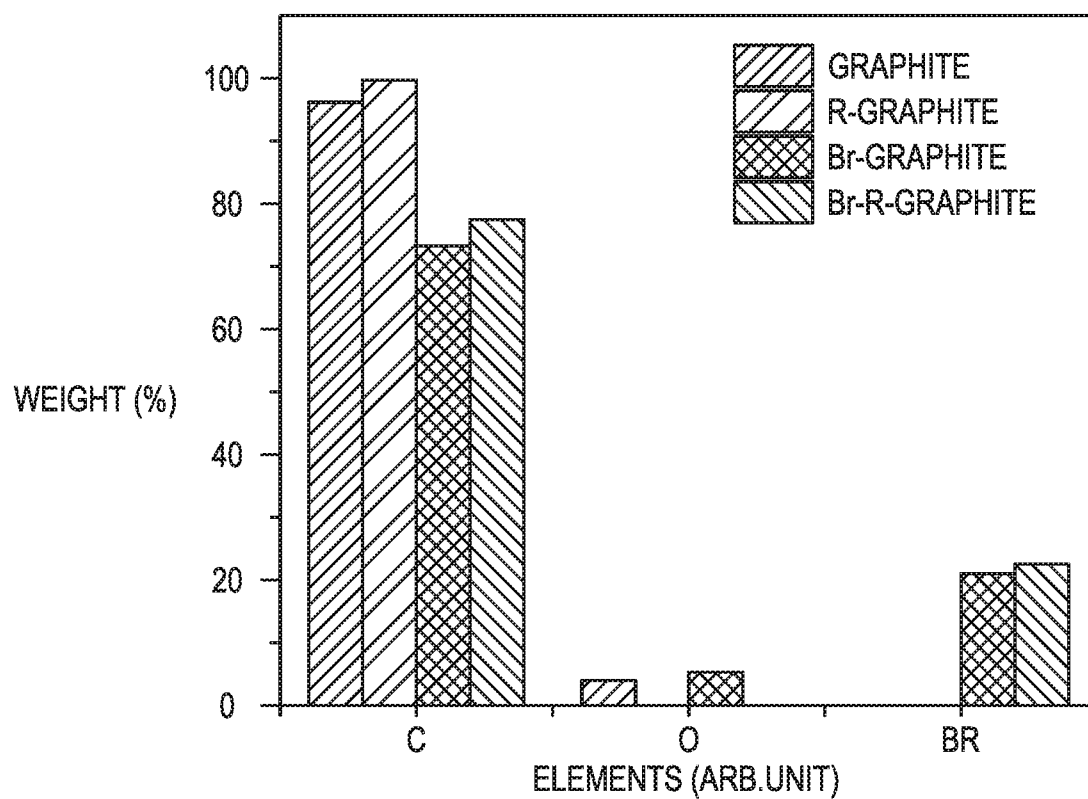


FIG. 2E

*FIG. 3A**FIG. 3B*

*FIG. 3C**FIG. 3D*

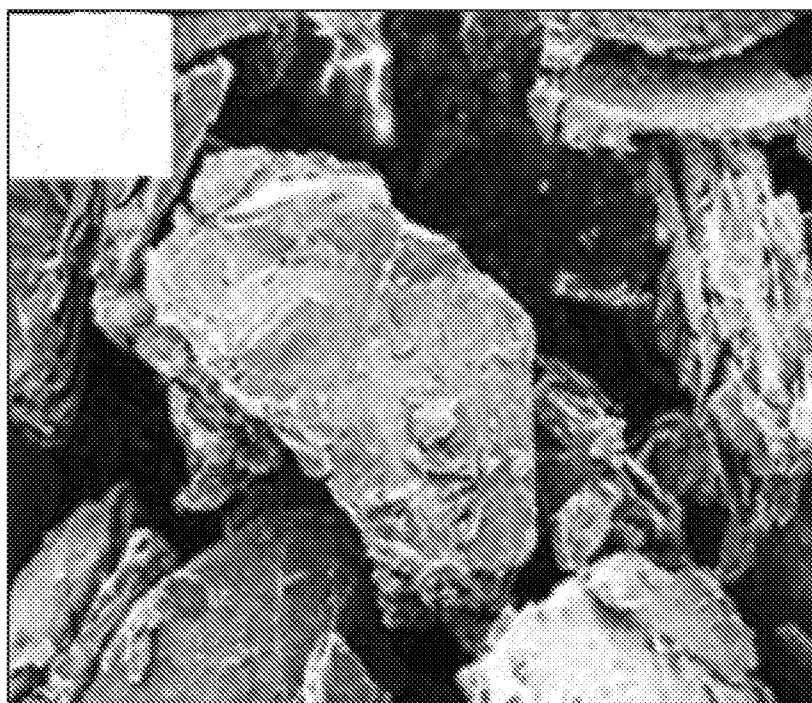


FIG. 3E

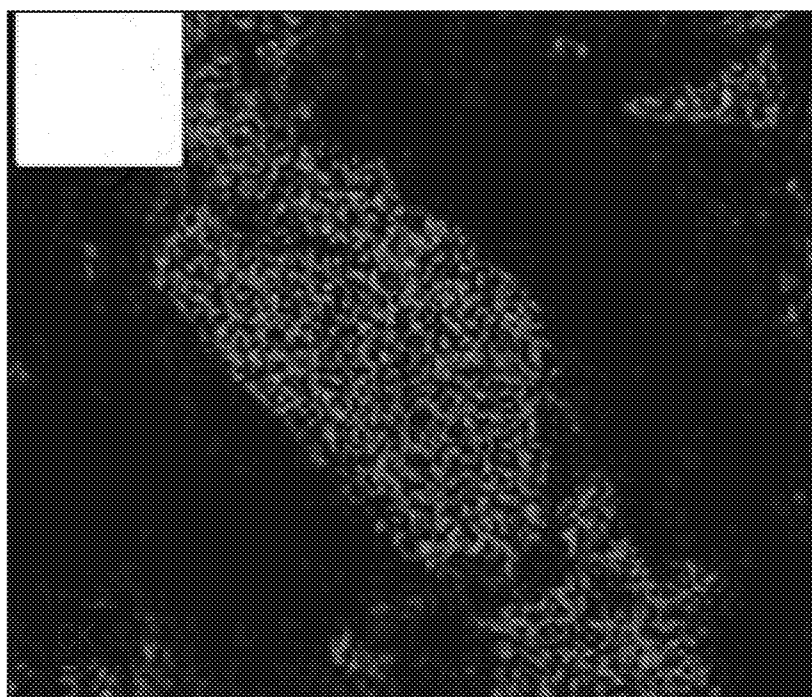


FIG. 3F

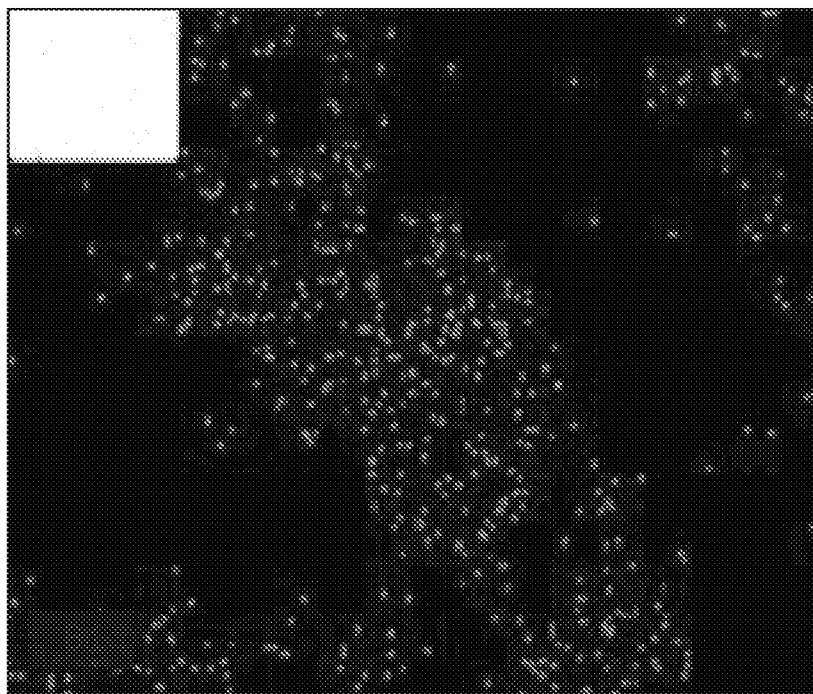


FIG. 3G

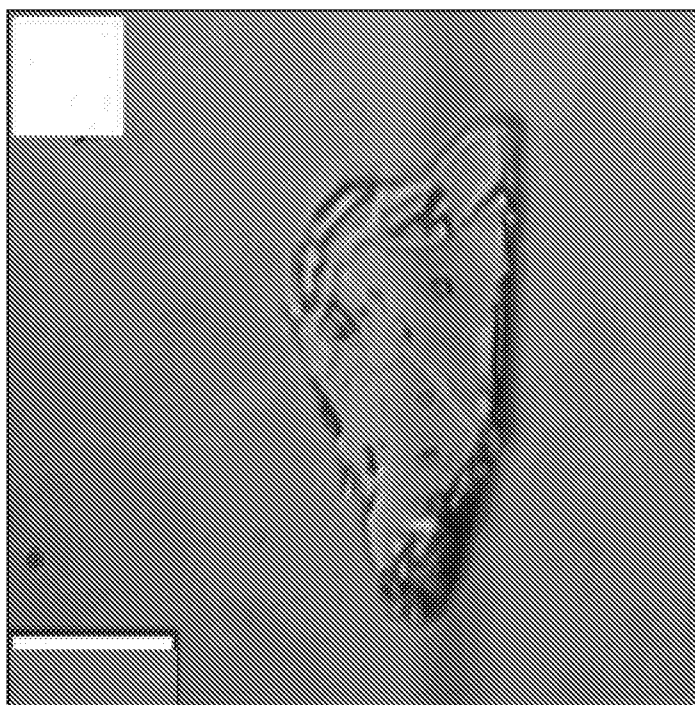


FIG. 4A

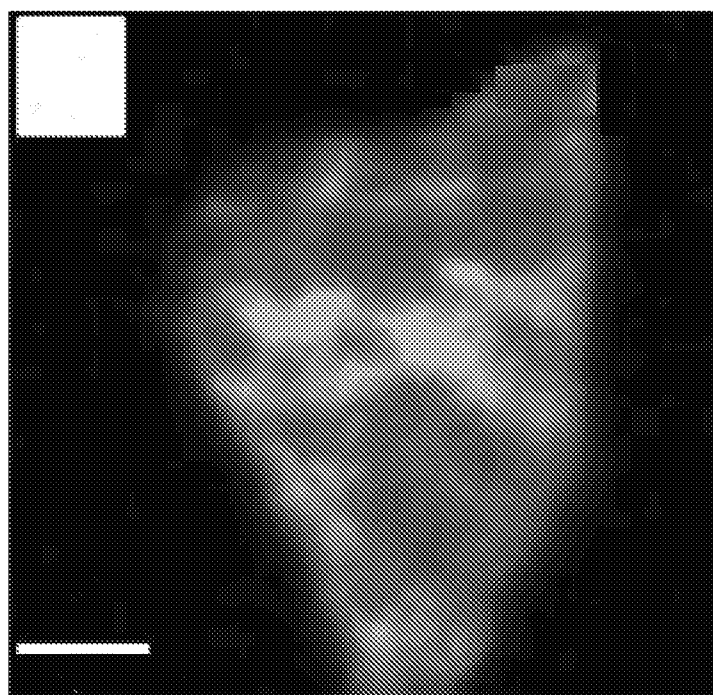


FIG. 4B

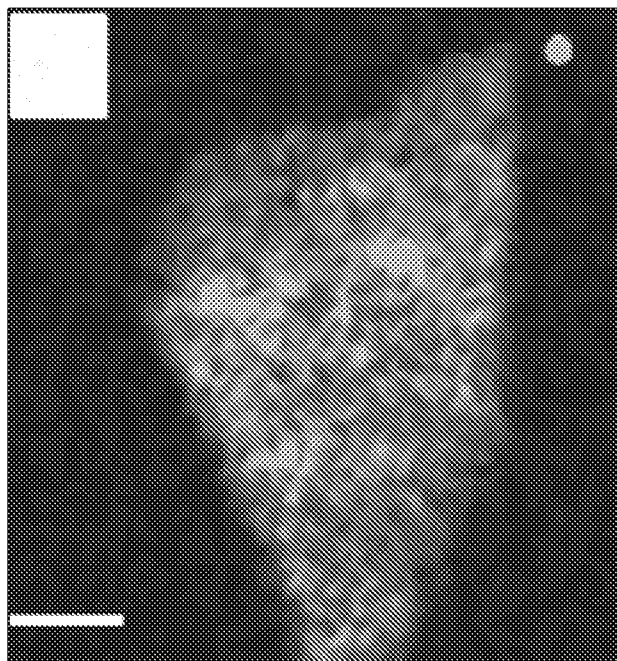


FIG. 4C

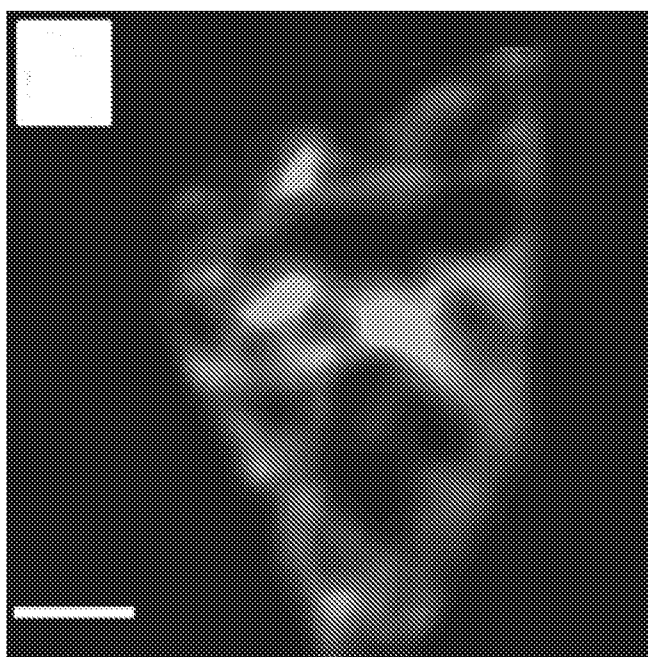
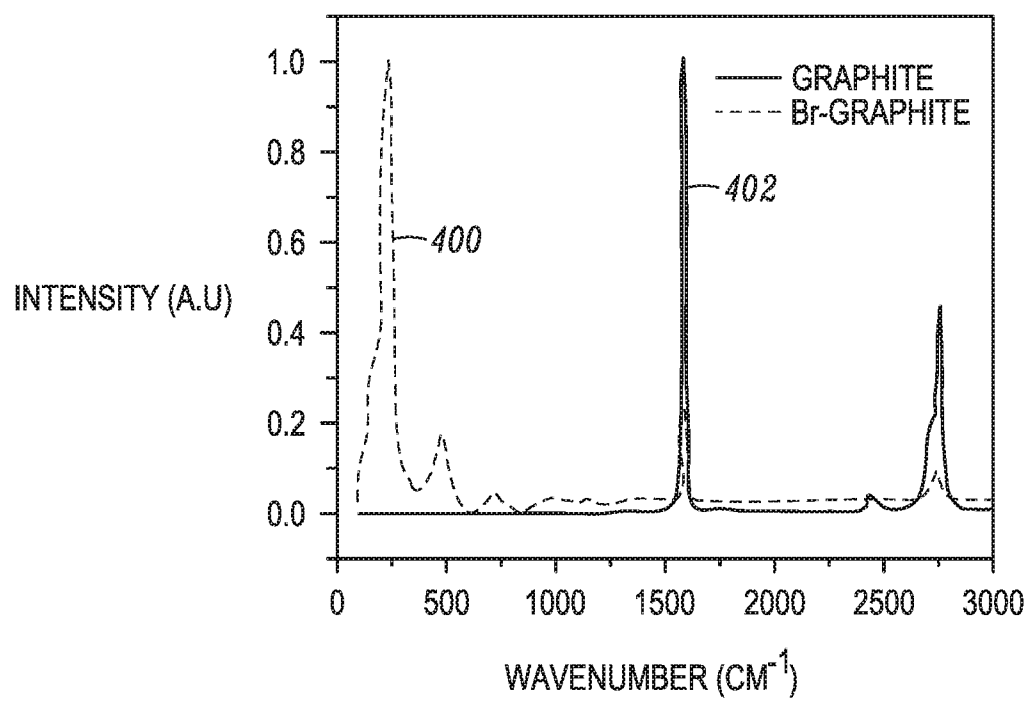
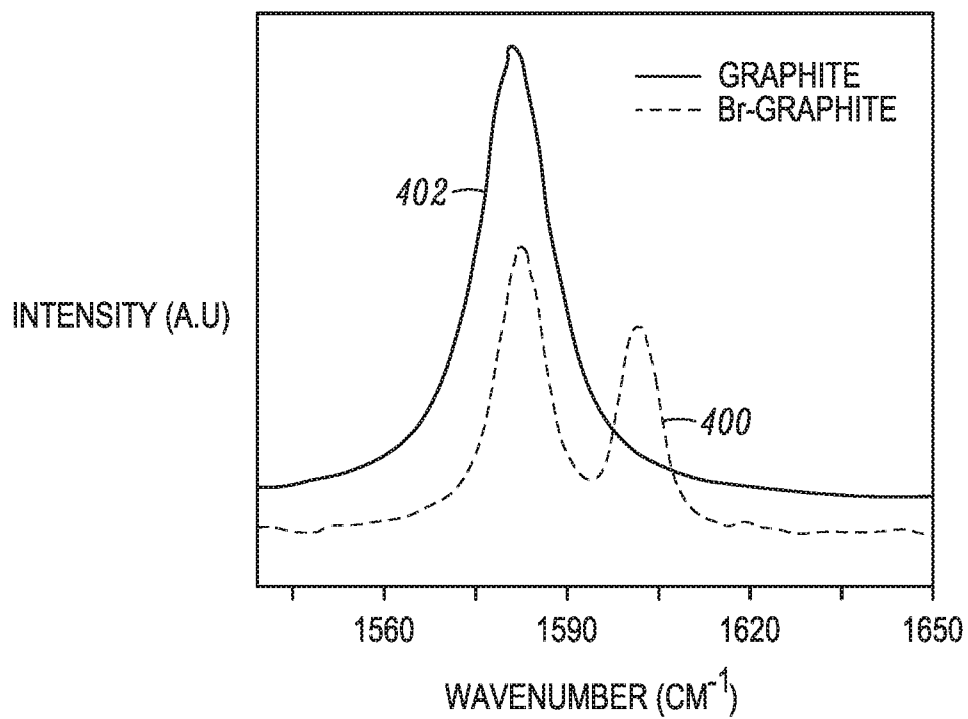
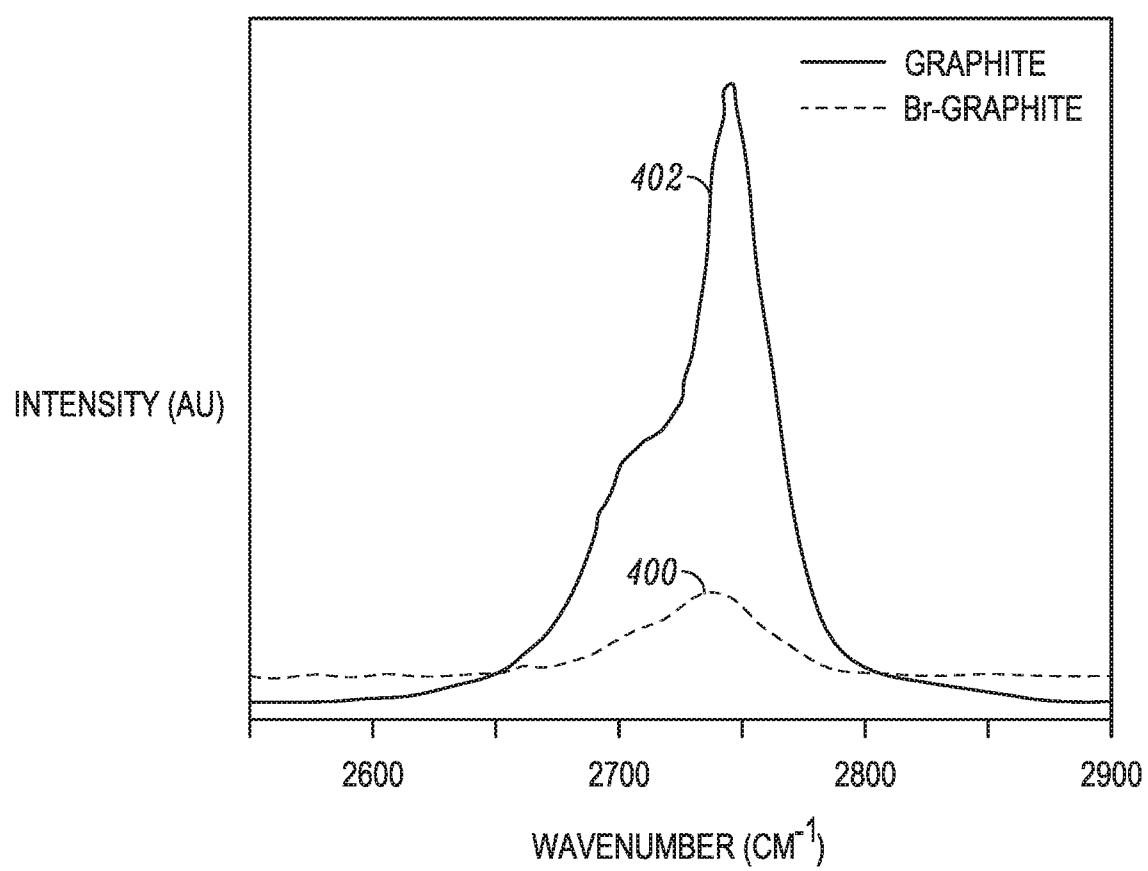
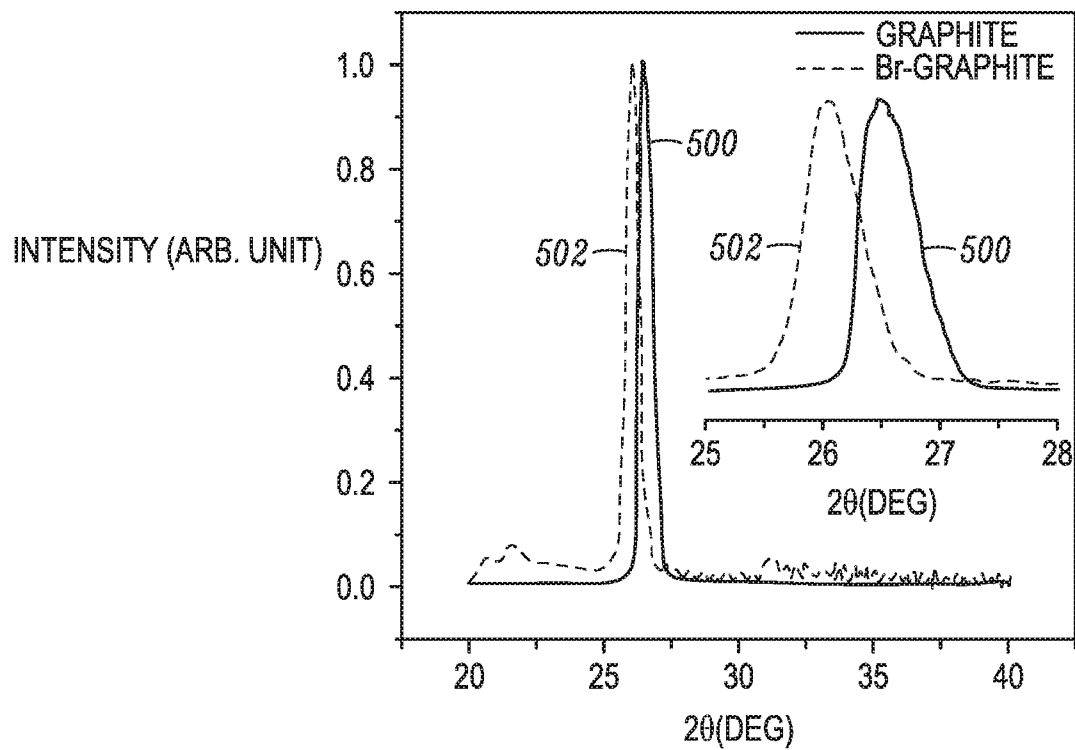
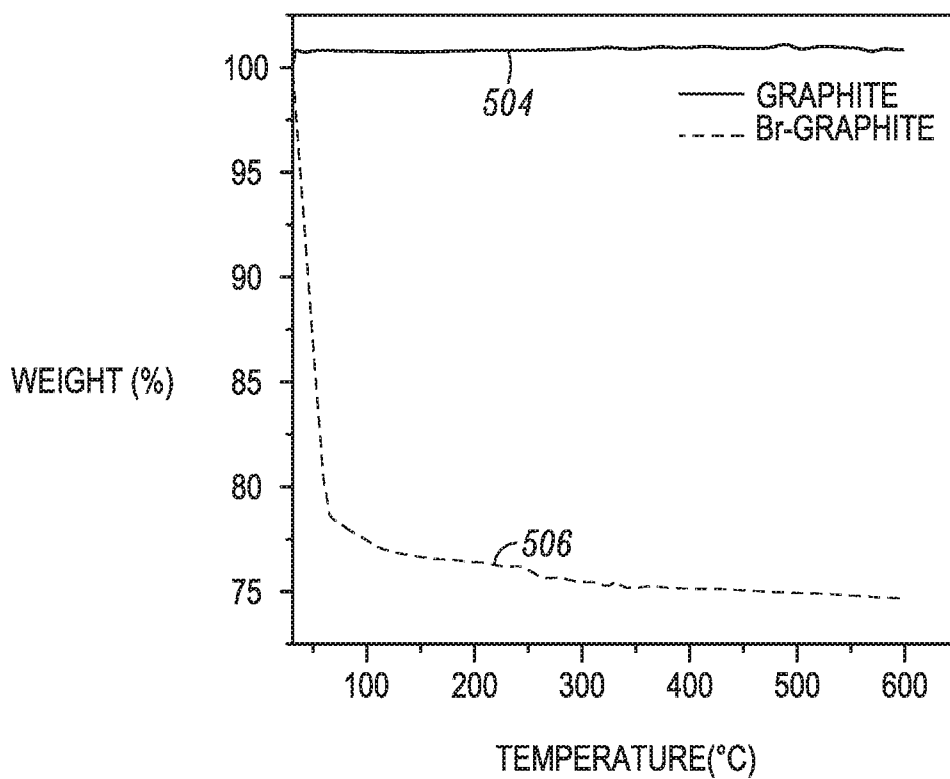


FIG. 4D

*FIG. 4E**FIG. 4F*

*FIG. 4G*

*FIG. 5A**FIG. 5B*

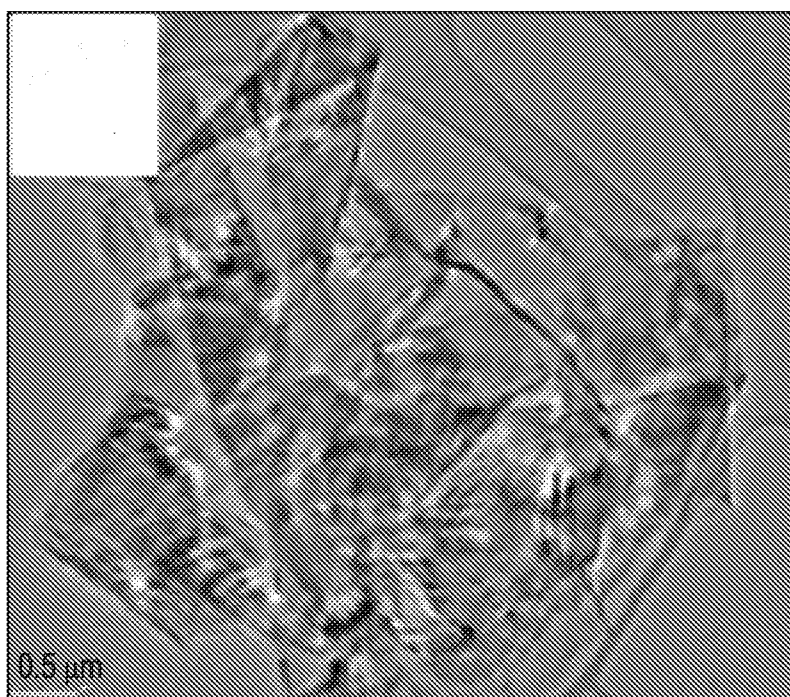


FIG. 6A

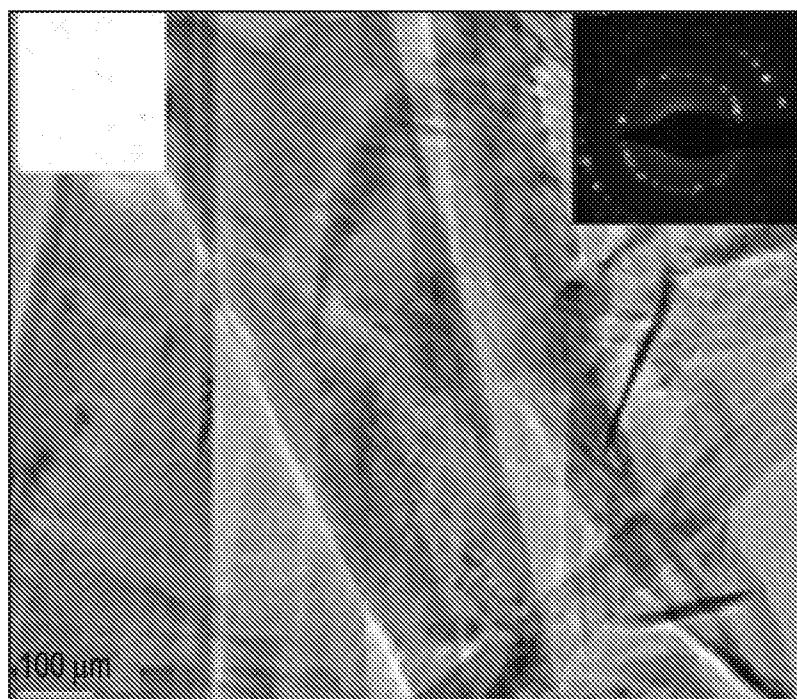
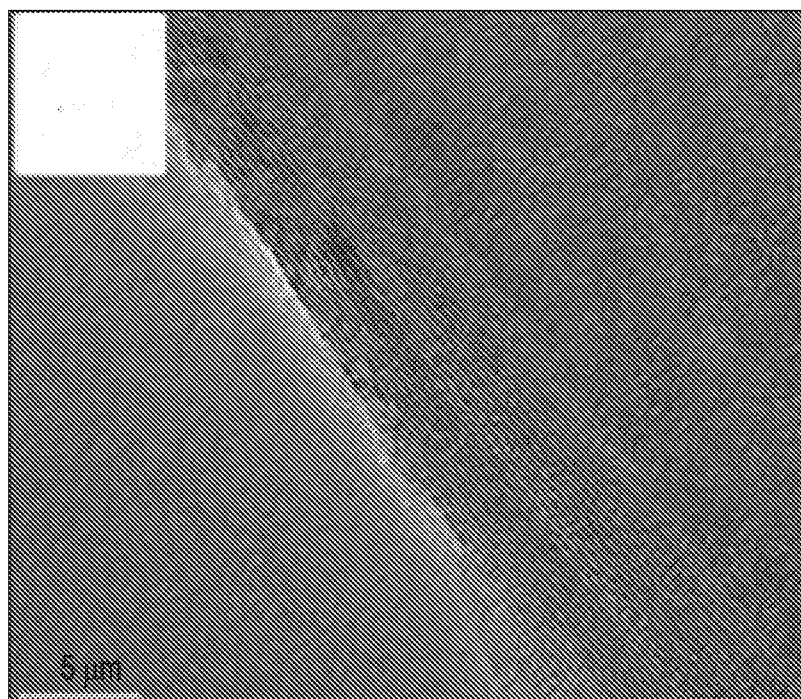
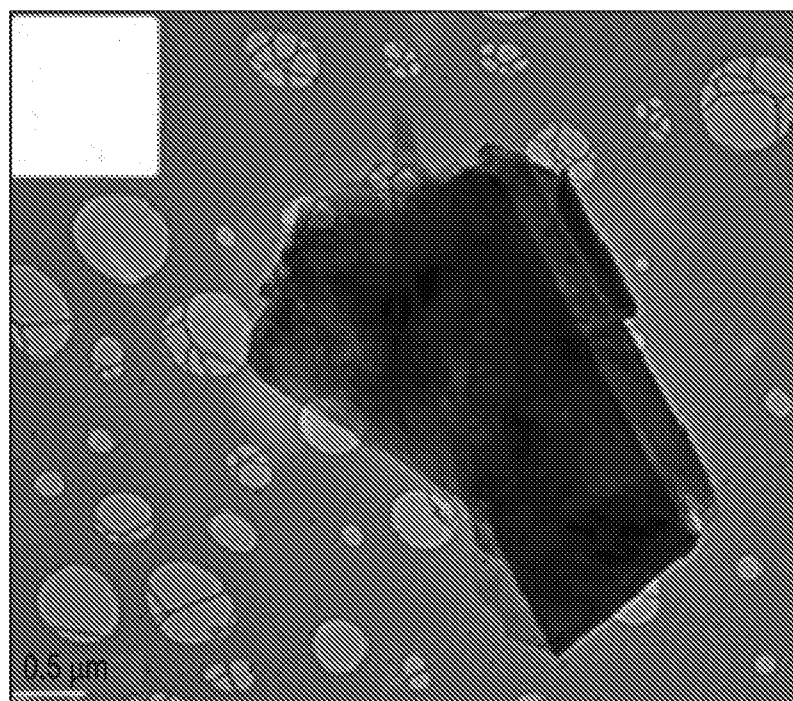
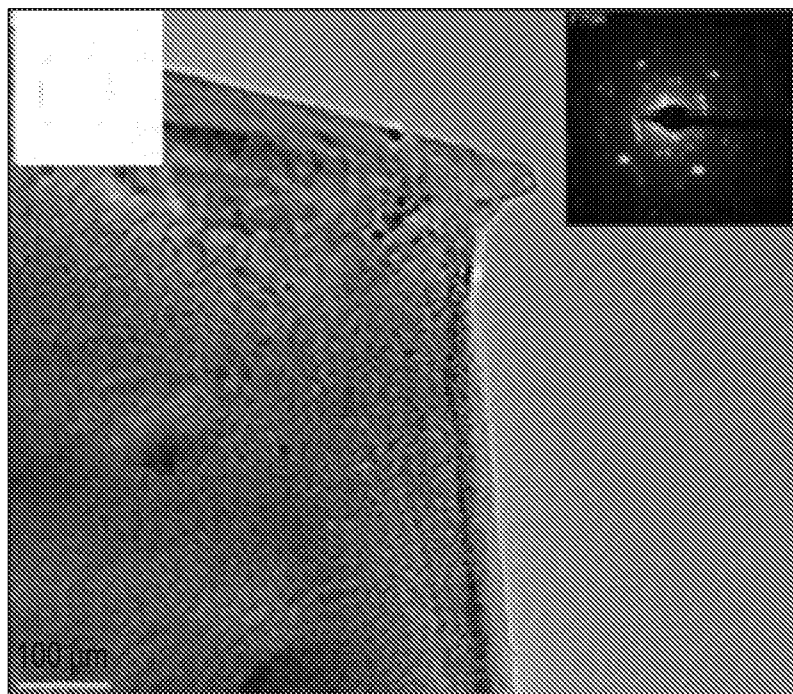
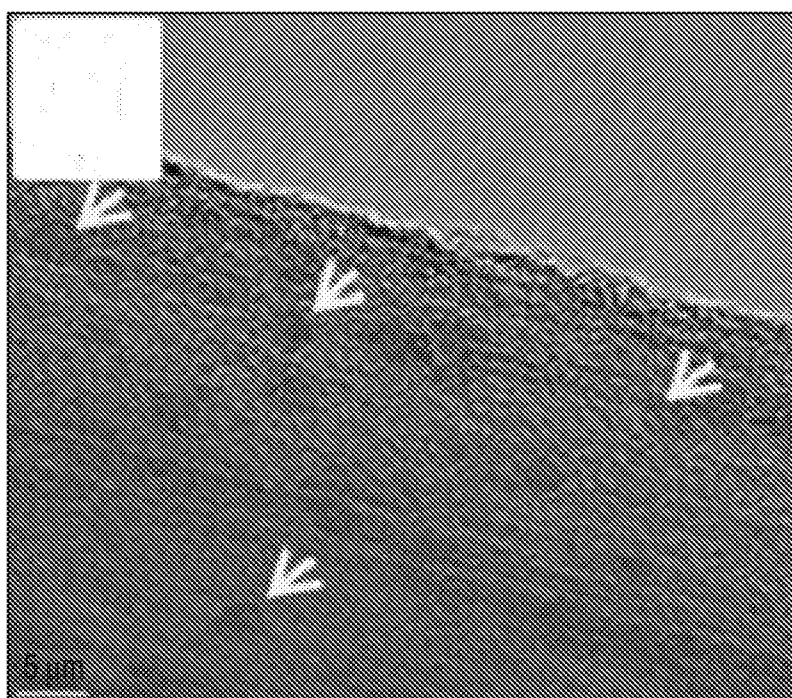


FIG. 6B

*FIG. 6C**FIG. 6D*

*FIG. 6E**FIG. 6F*

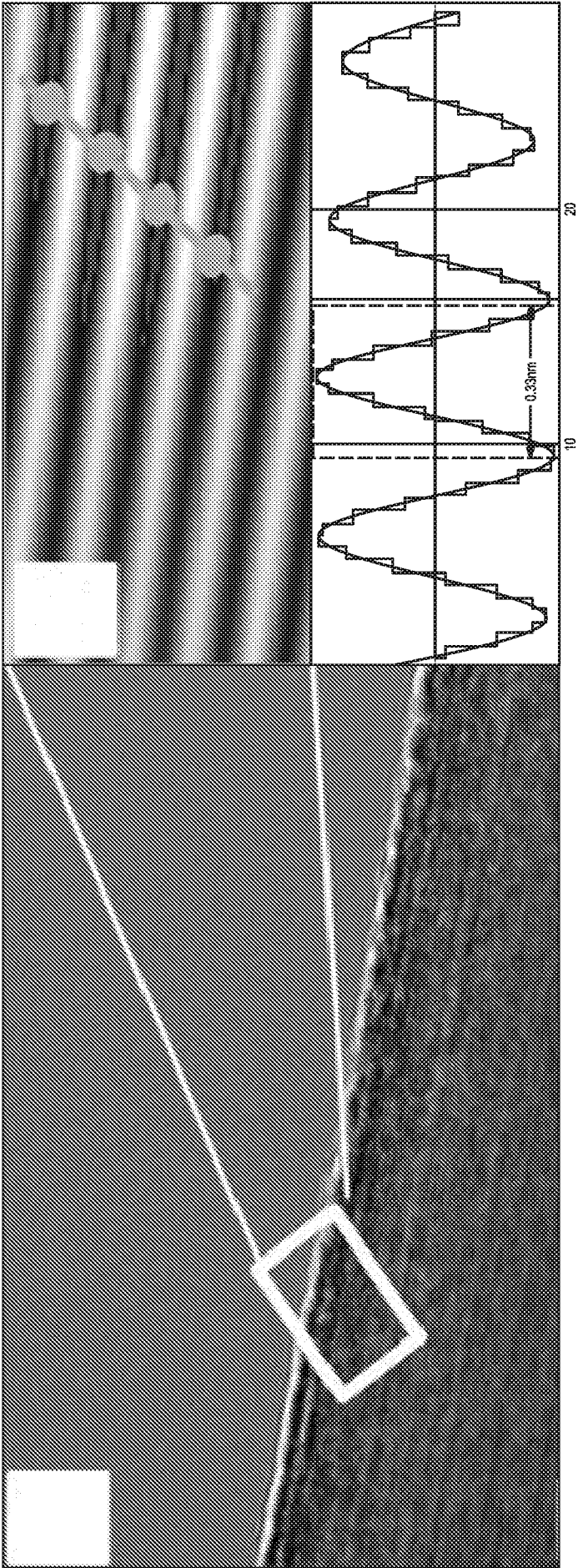


FIG. 6H

FIG. 6G

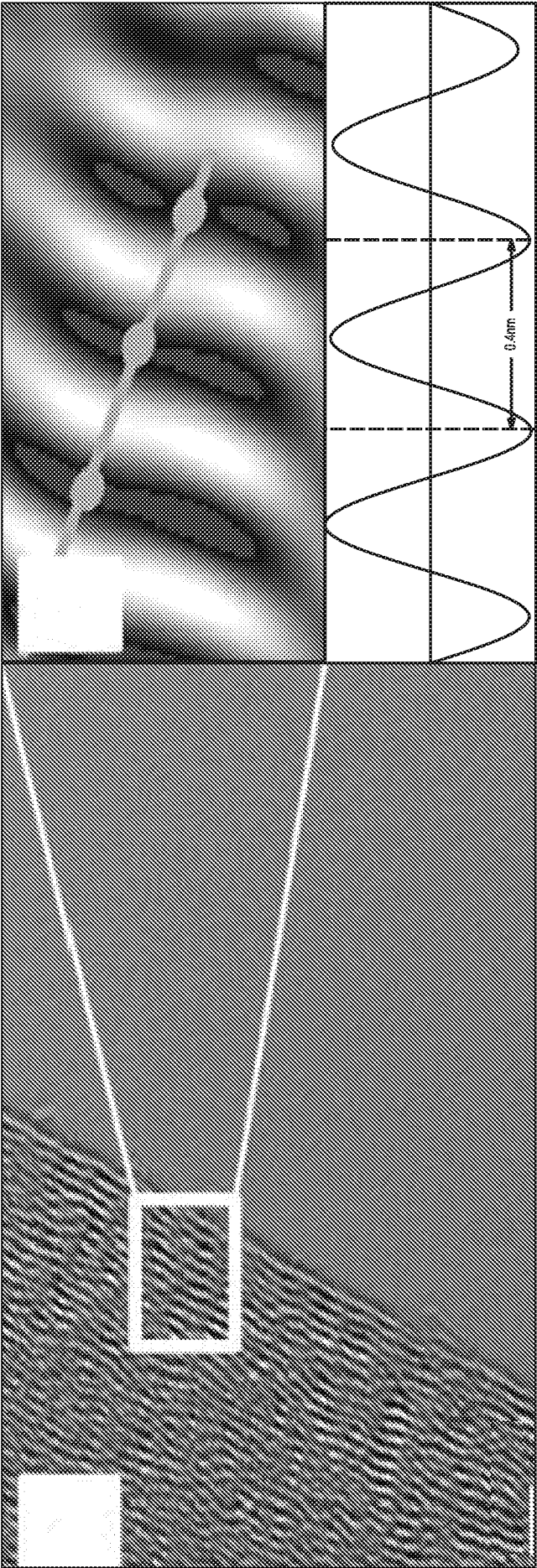


FIG. 6J

FIG. 6I

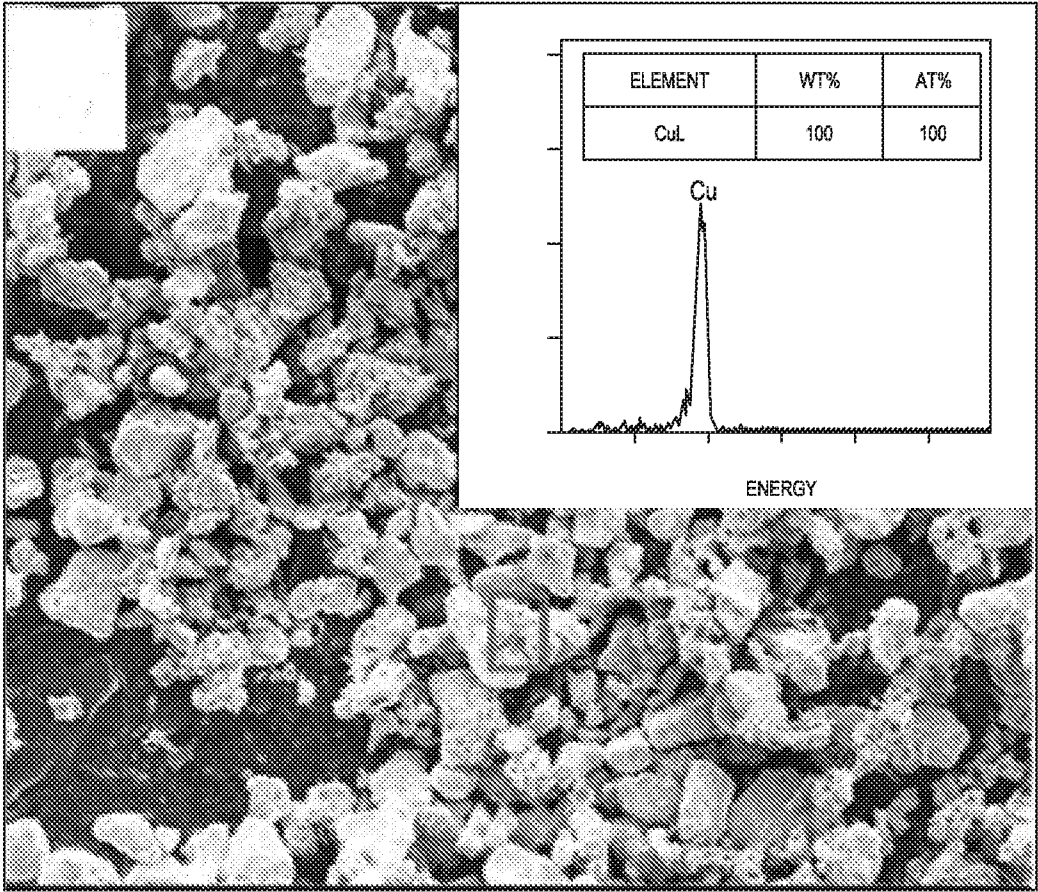


FIG. 7A

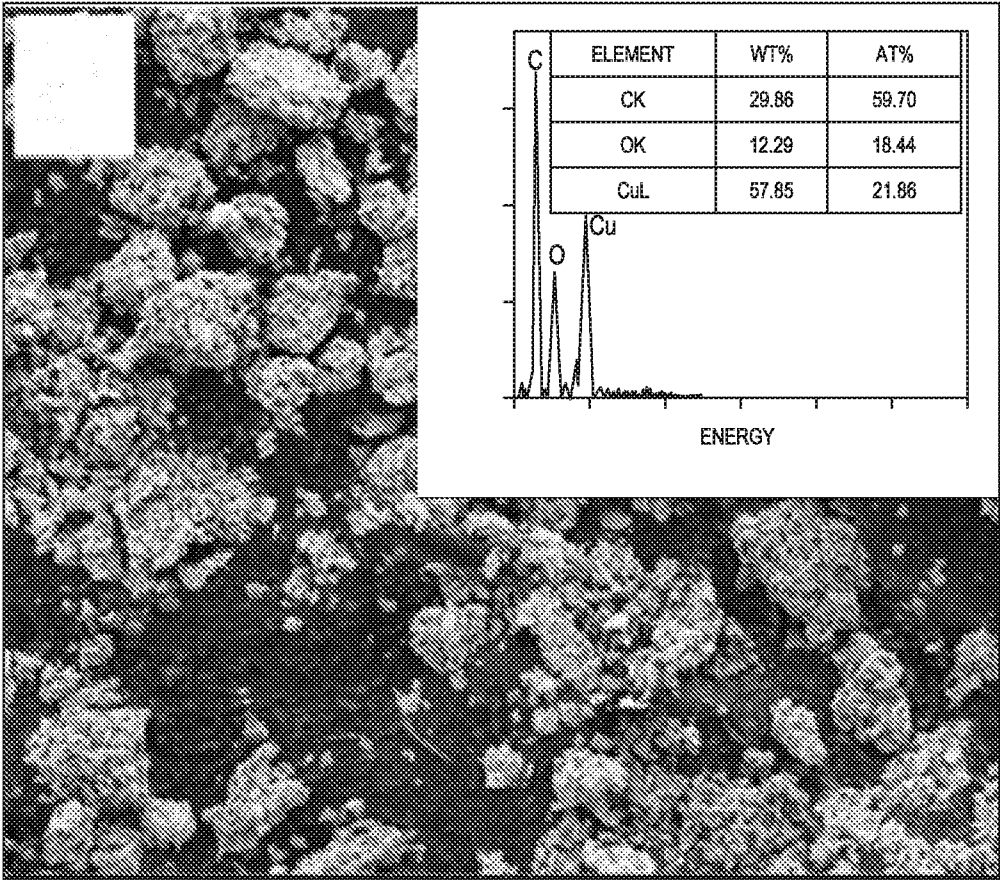


FIG. 7B

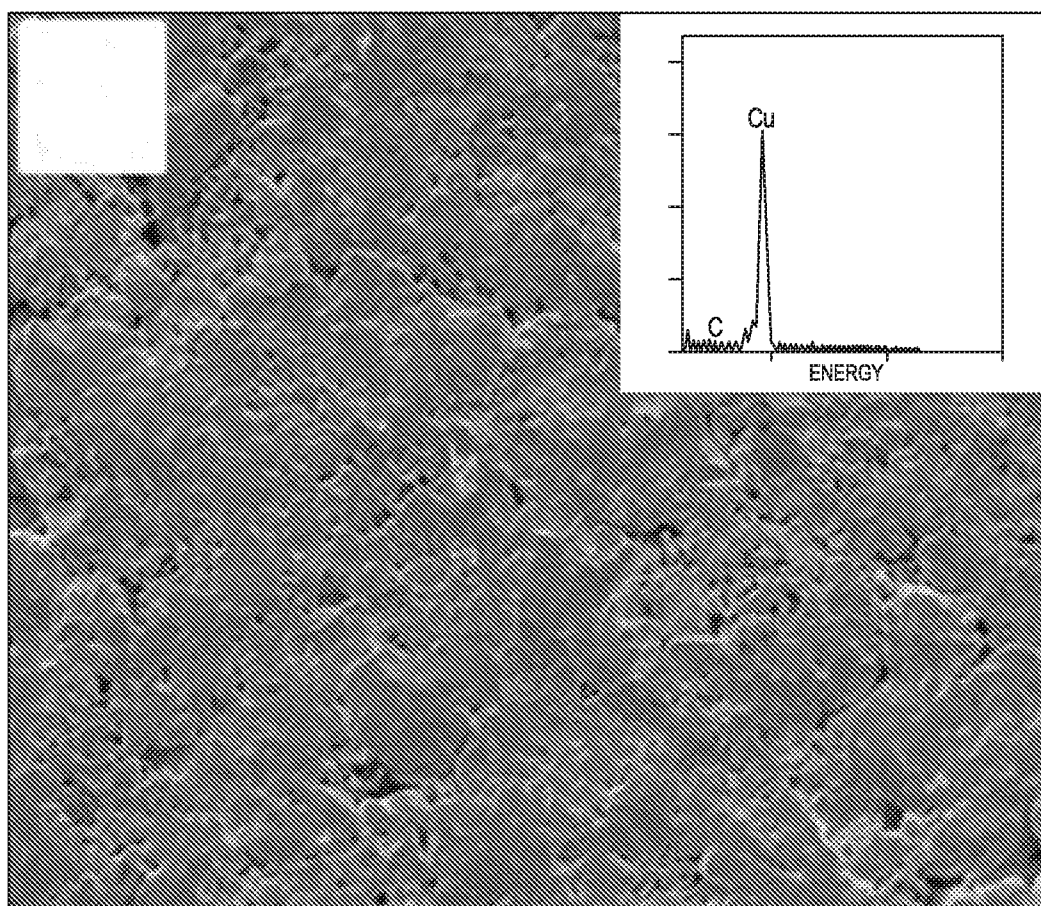
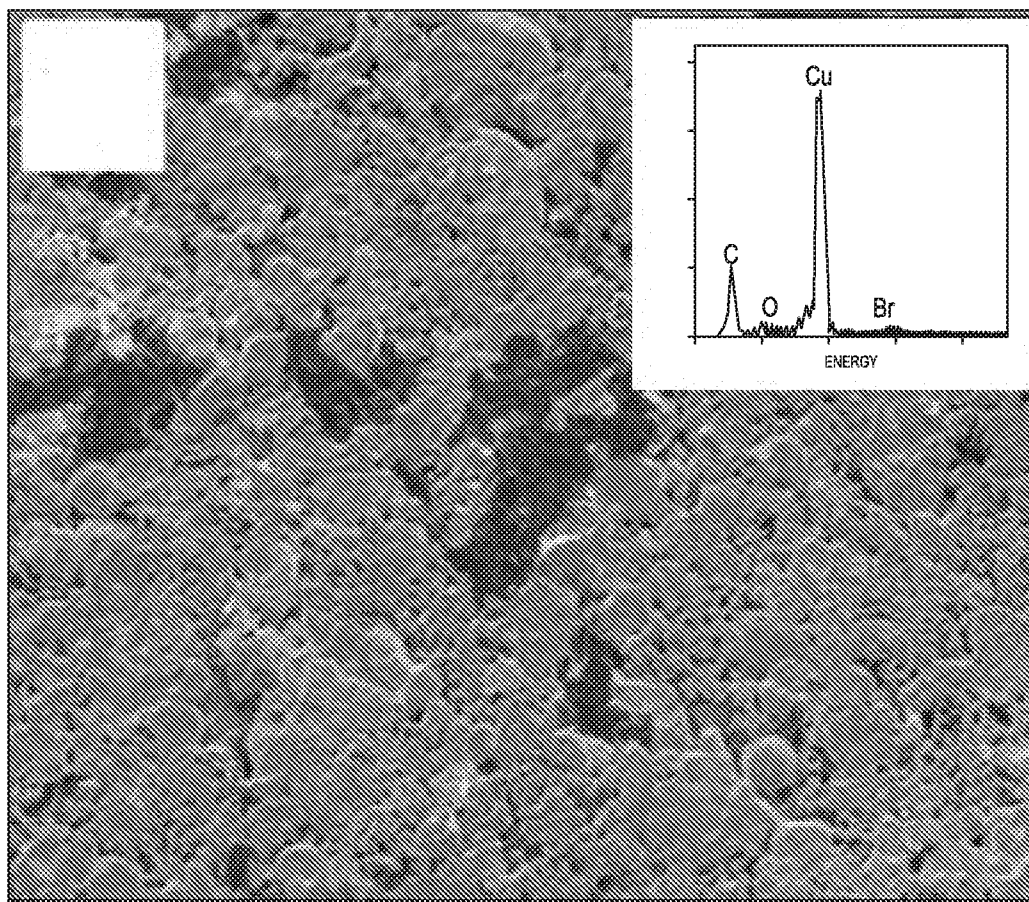


FIG. 8A

*FIG. 8B*

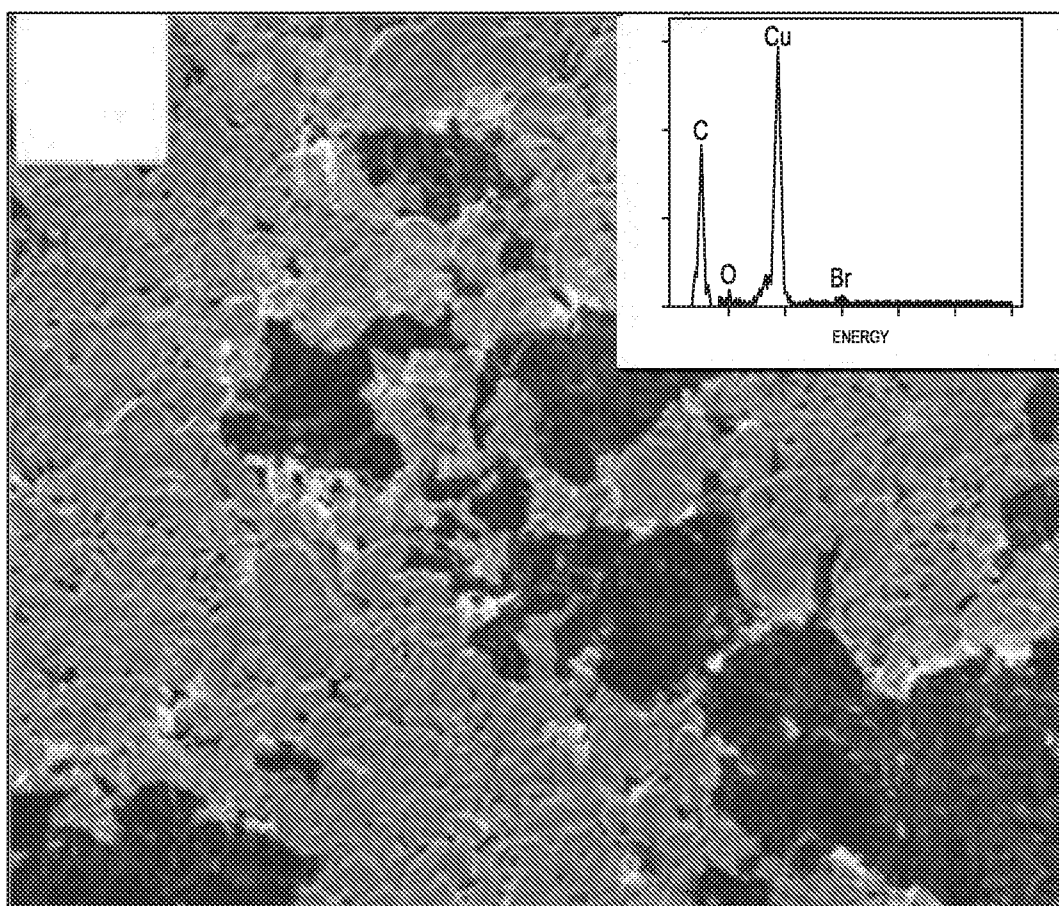


FIG. 8C

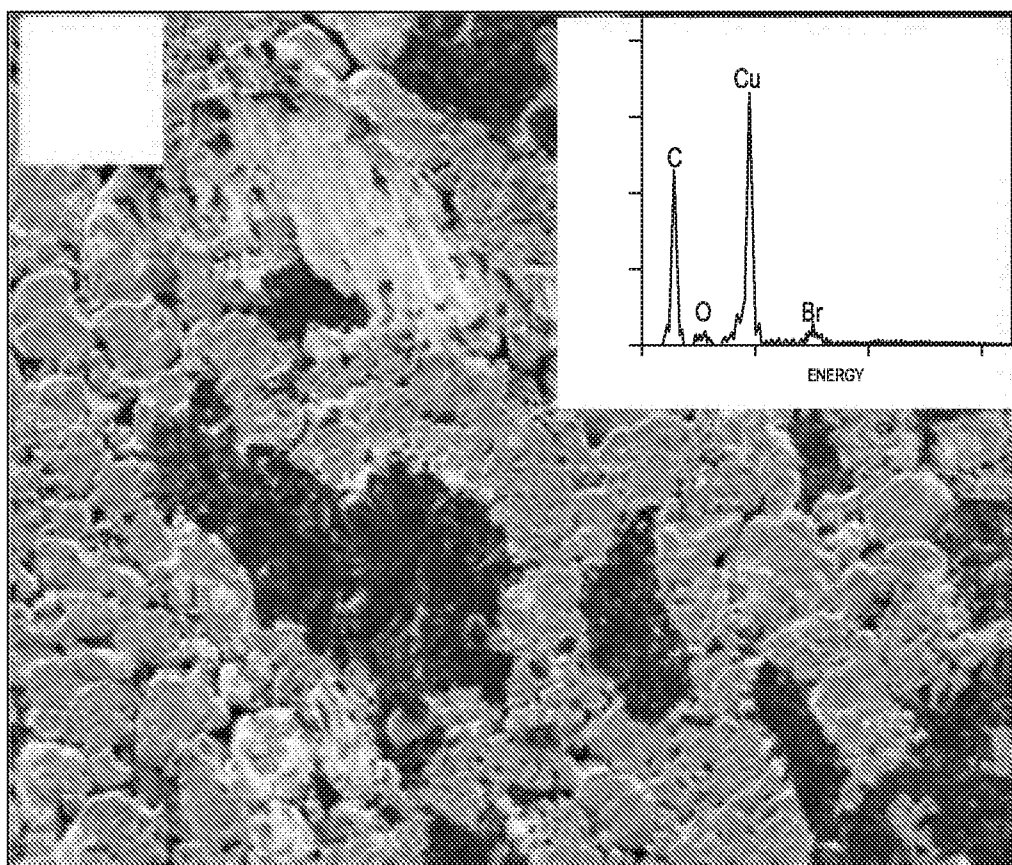
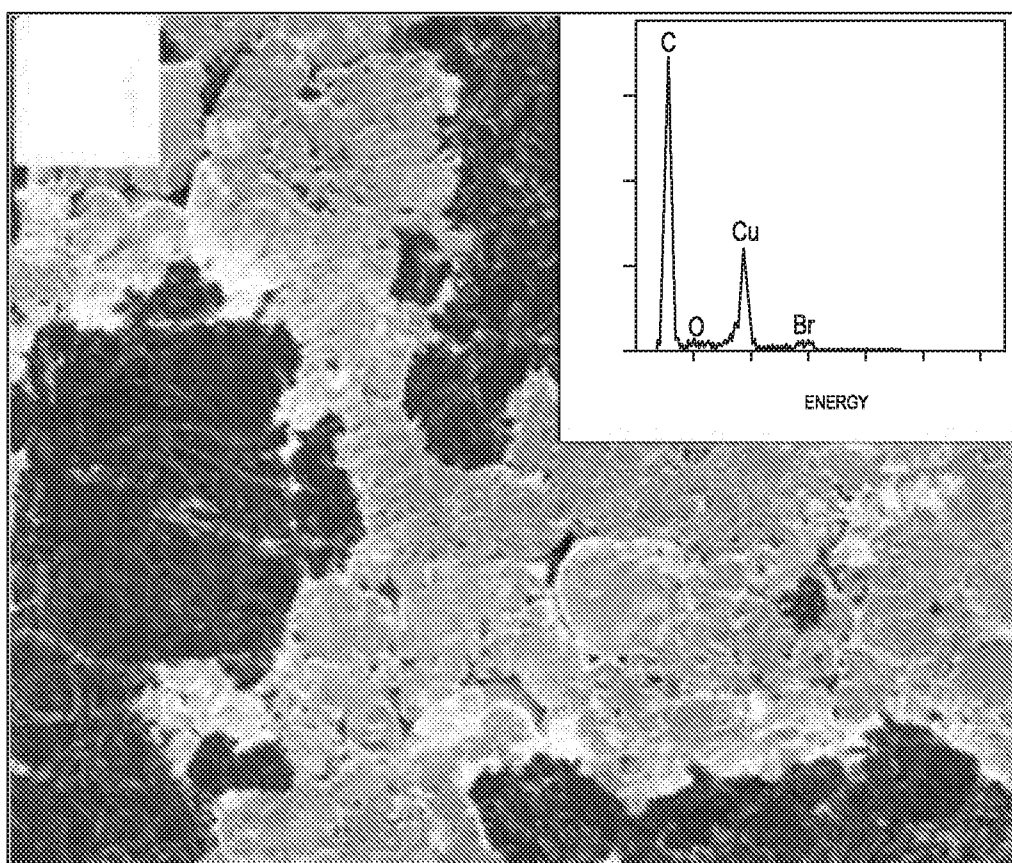
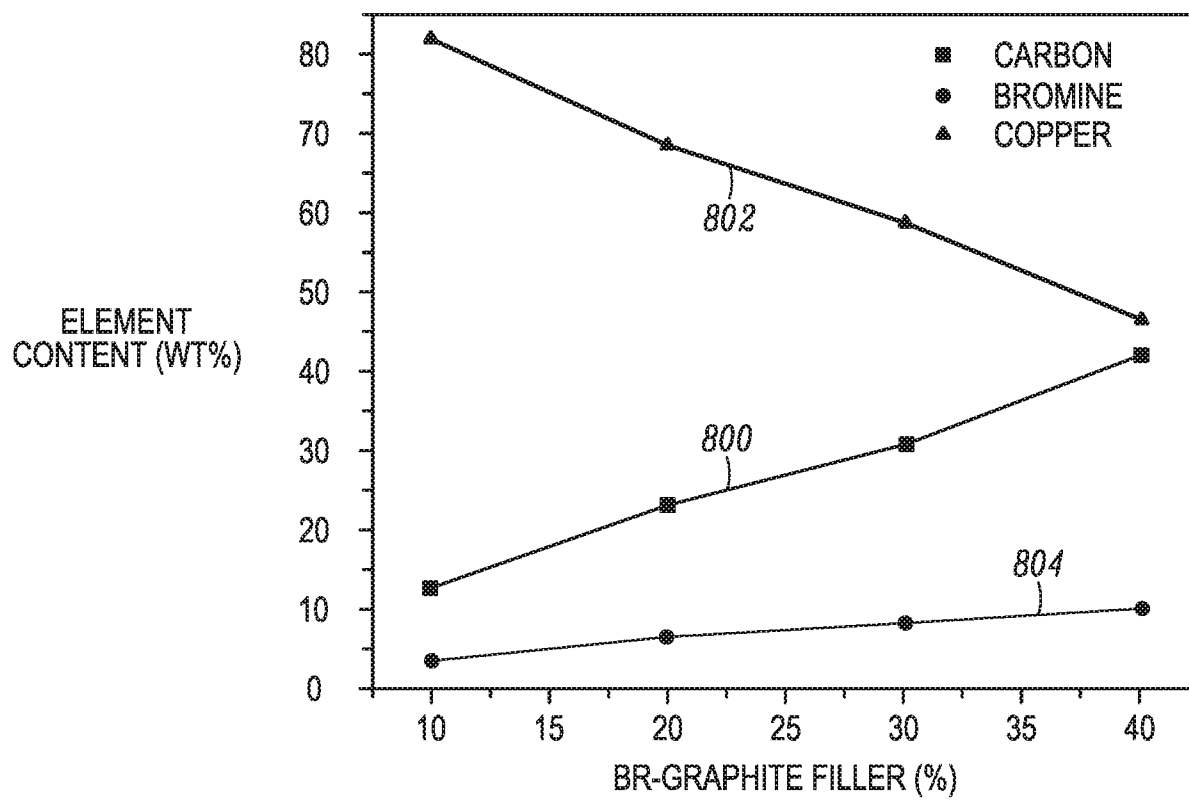


FIG. 8D

*FIG. 8E*

*FIG. 8F*

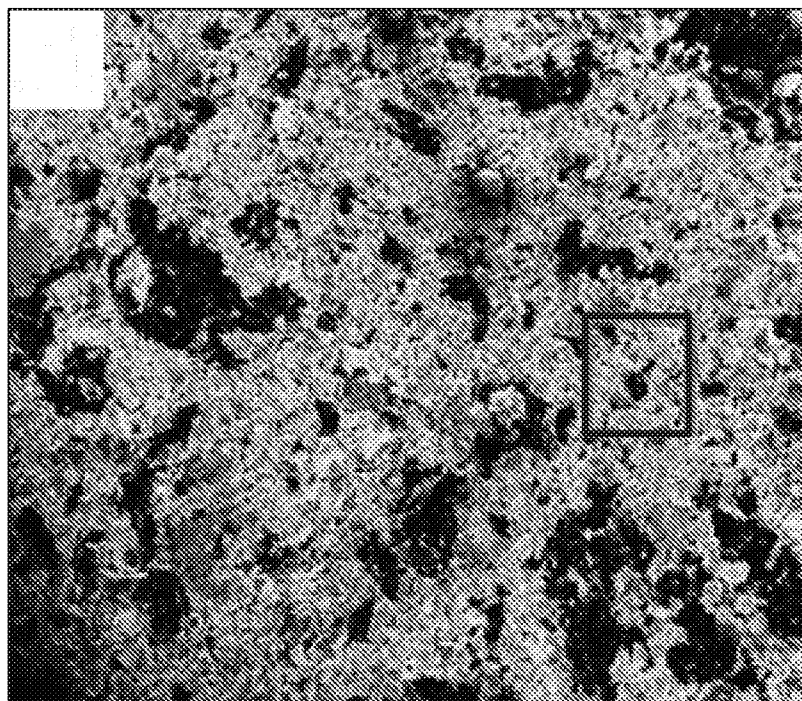


FIG. 9A

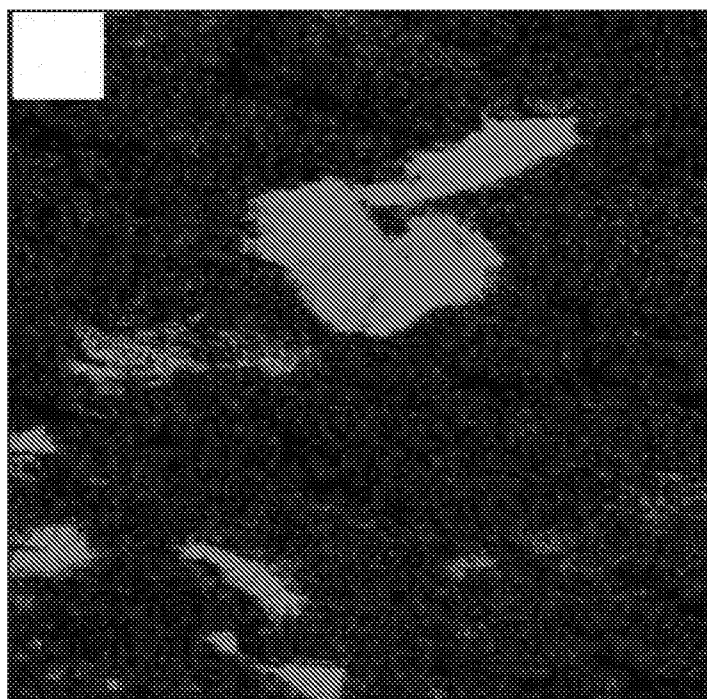


FIG. 9B

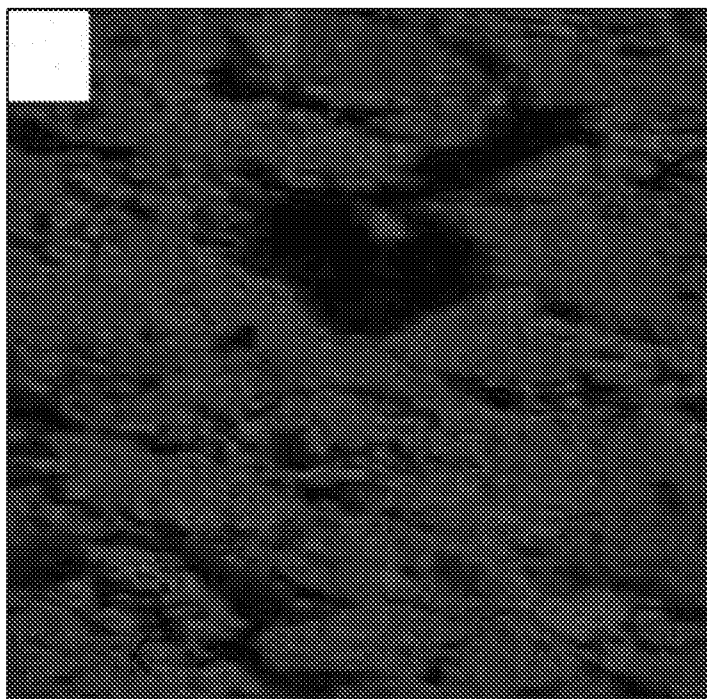


FIG. 9C

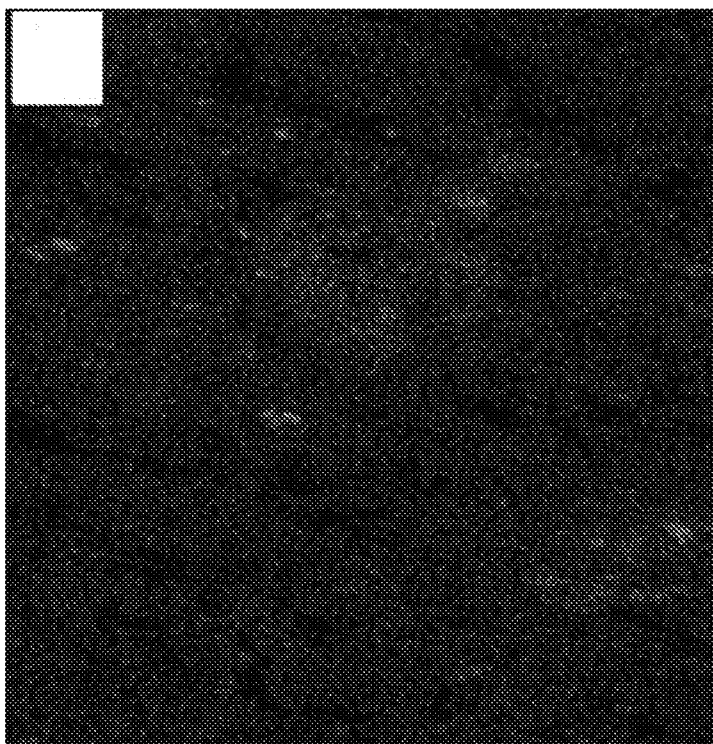


FIG. 9D

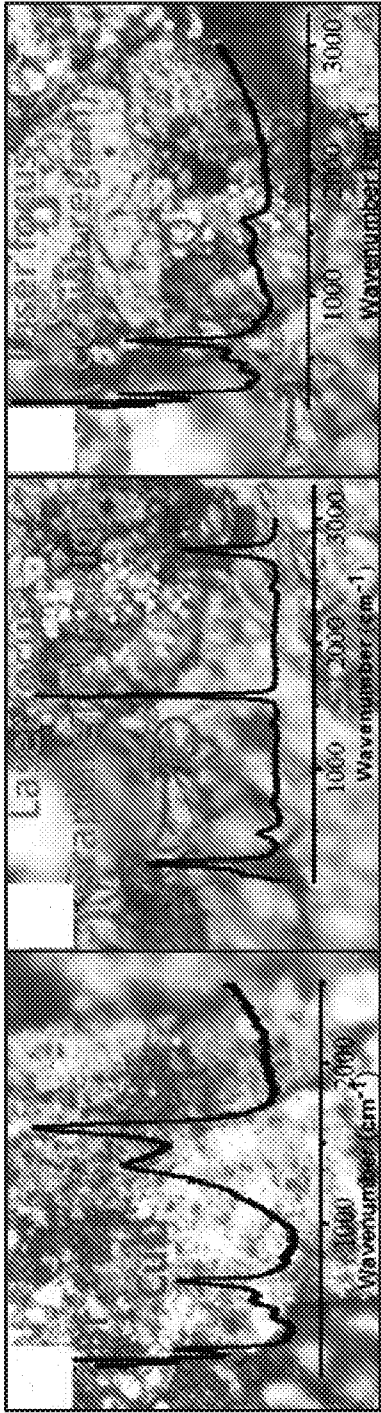


FIG. 10A FIG. 10B FIG. 10C

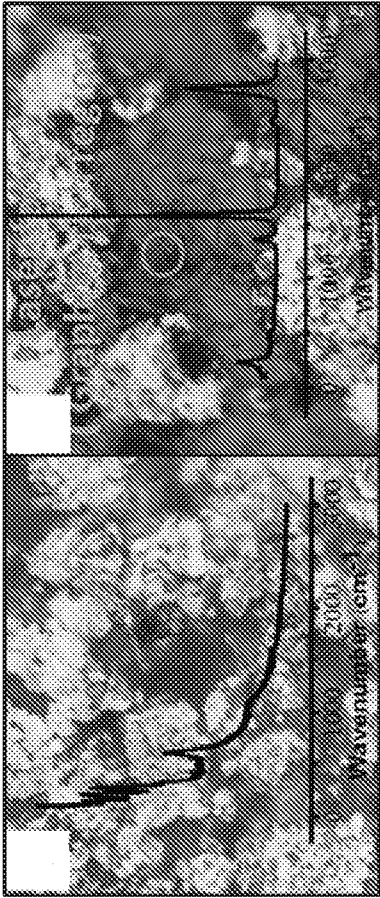


FIG. 10D FIG. 10E

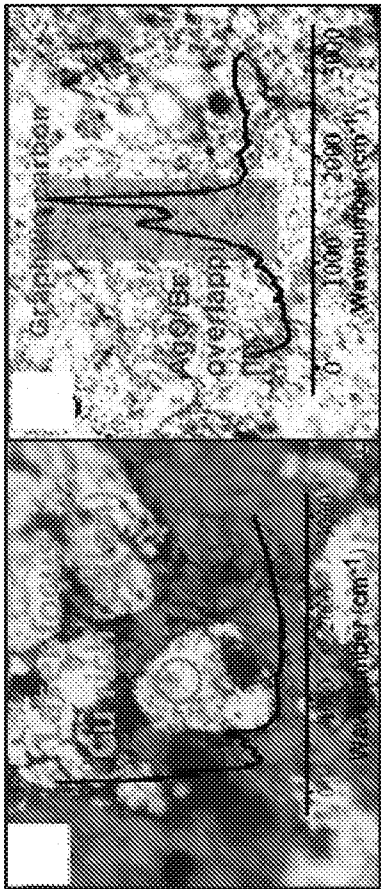


FIG. 10F FIG. 10G

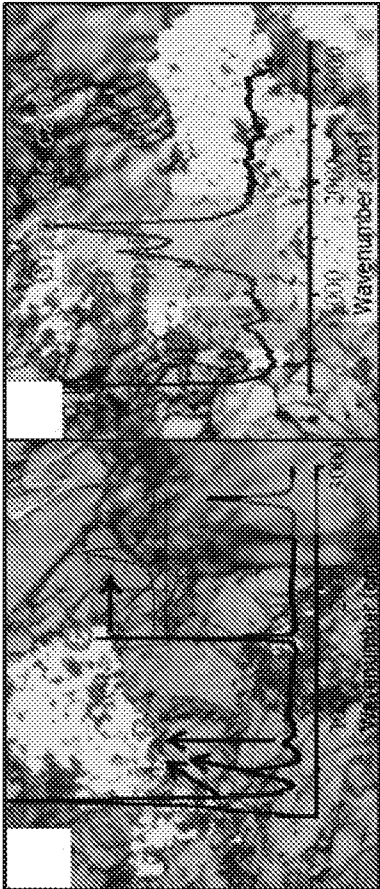
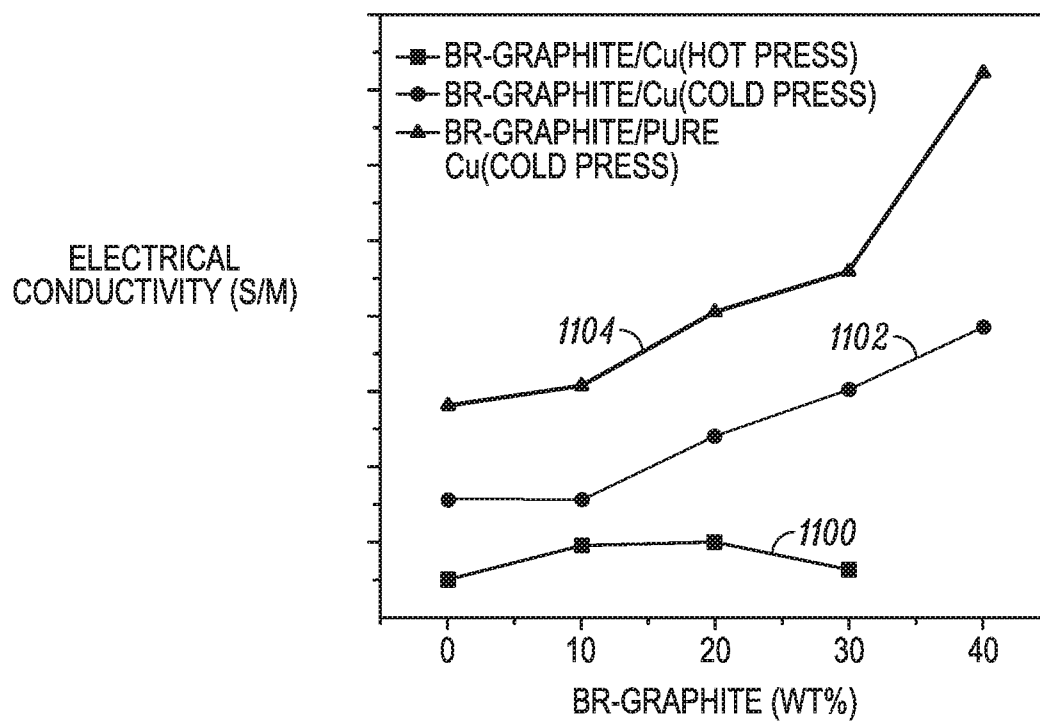
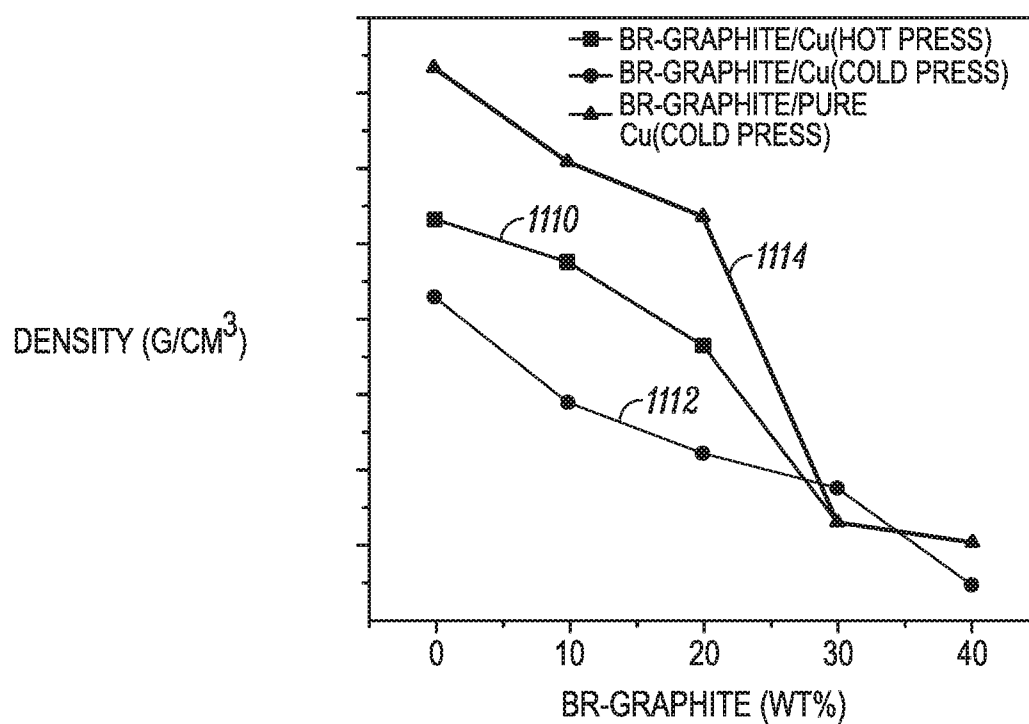
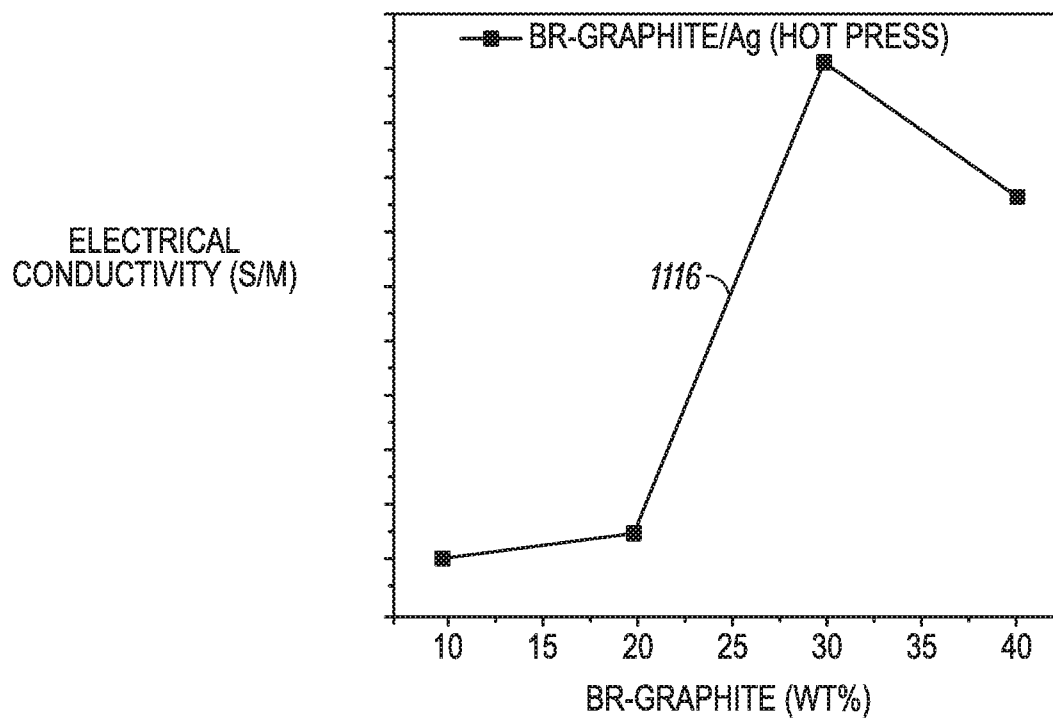
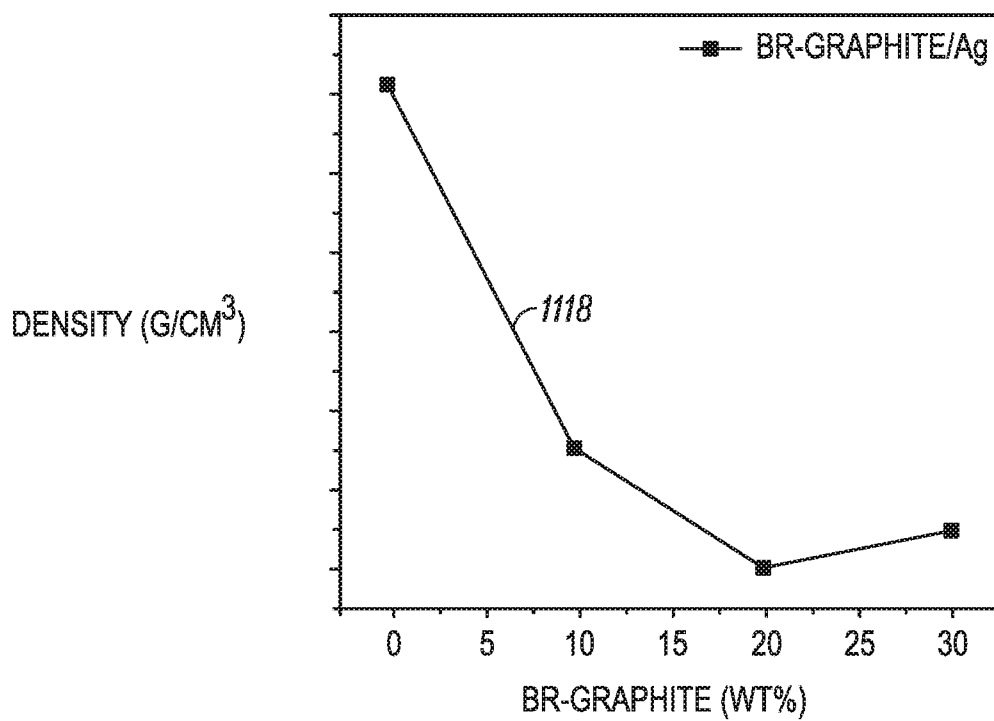
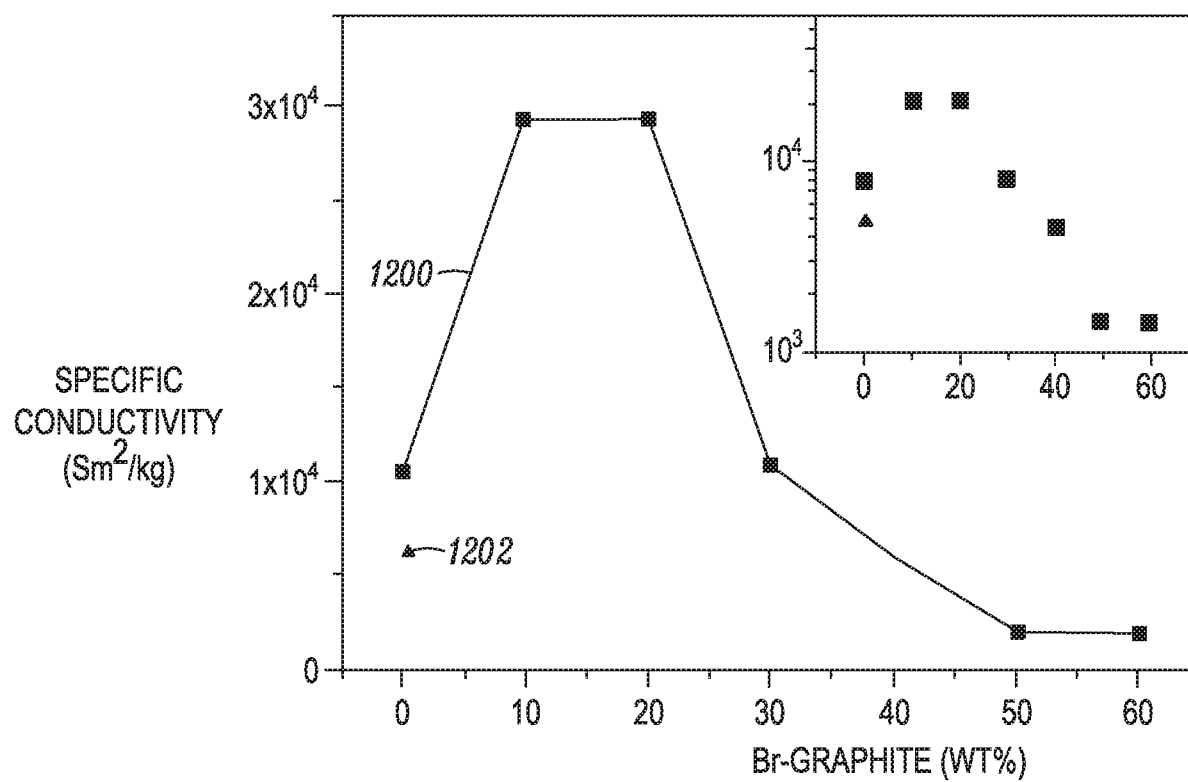
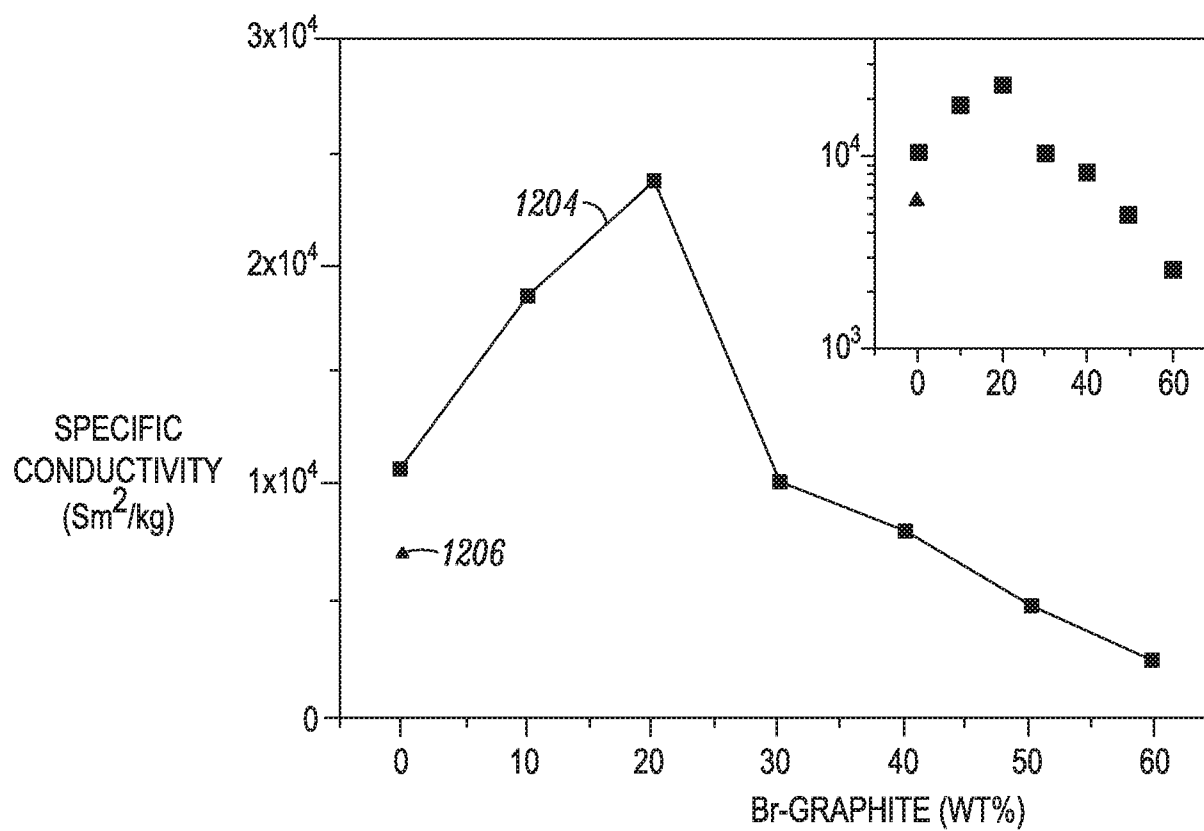
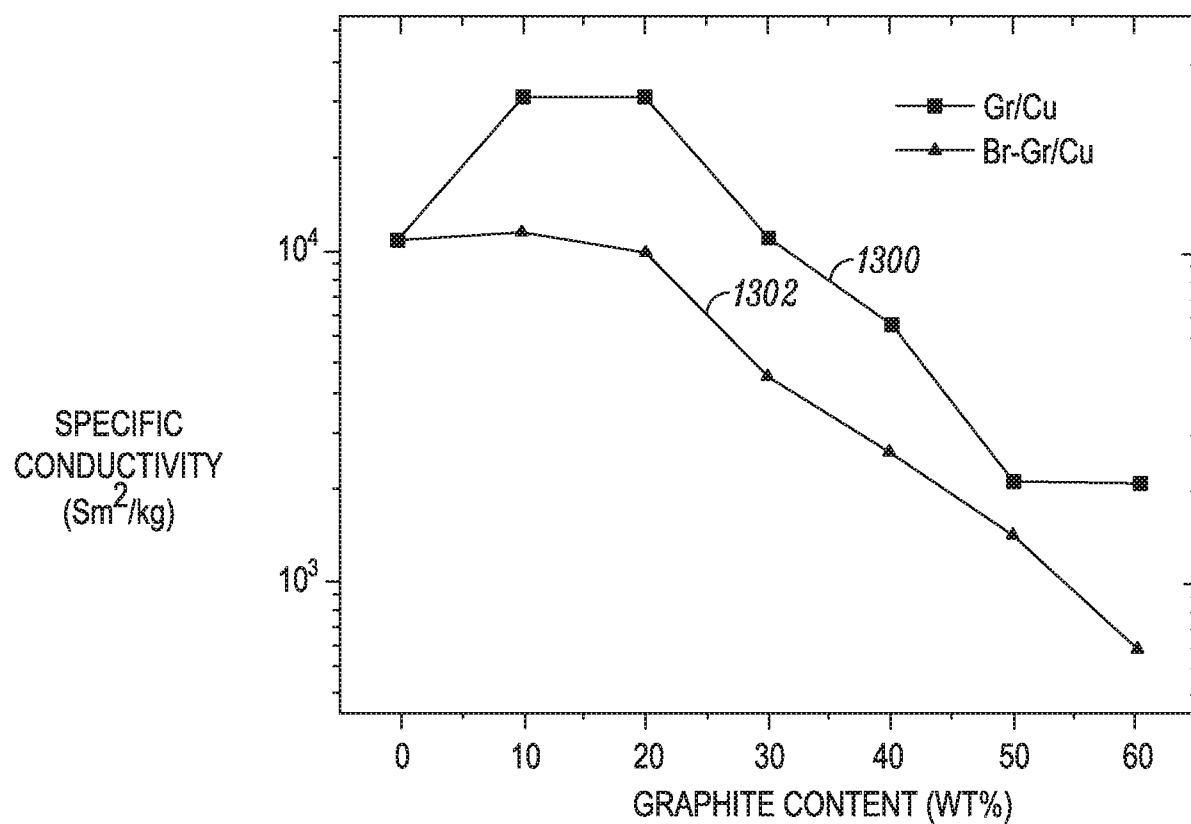
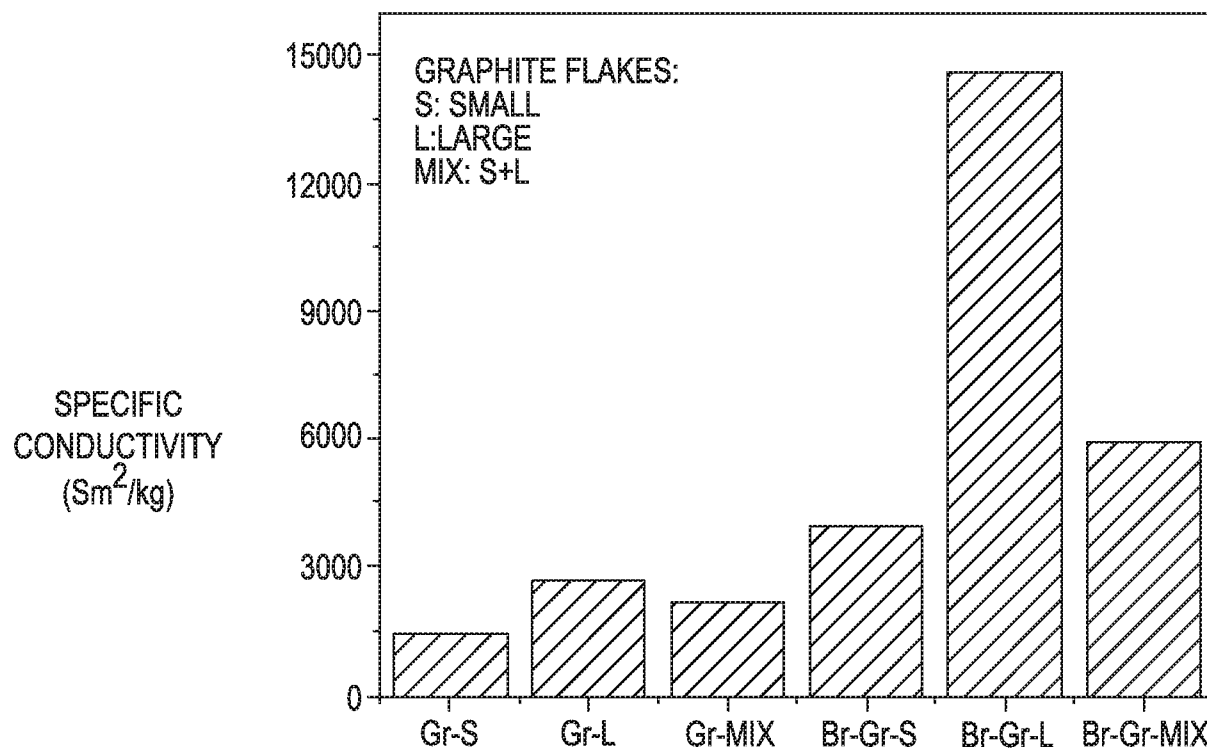


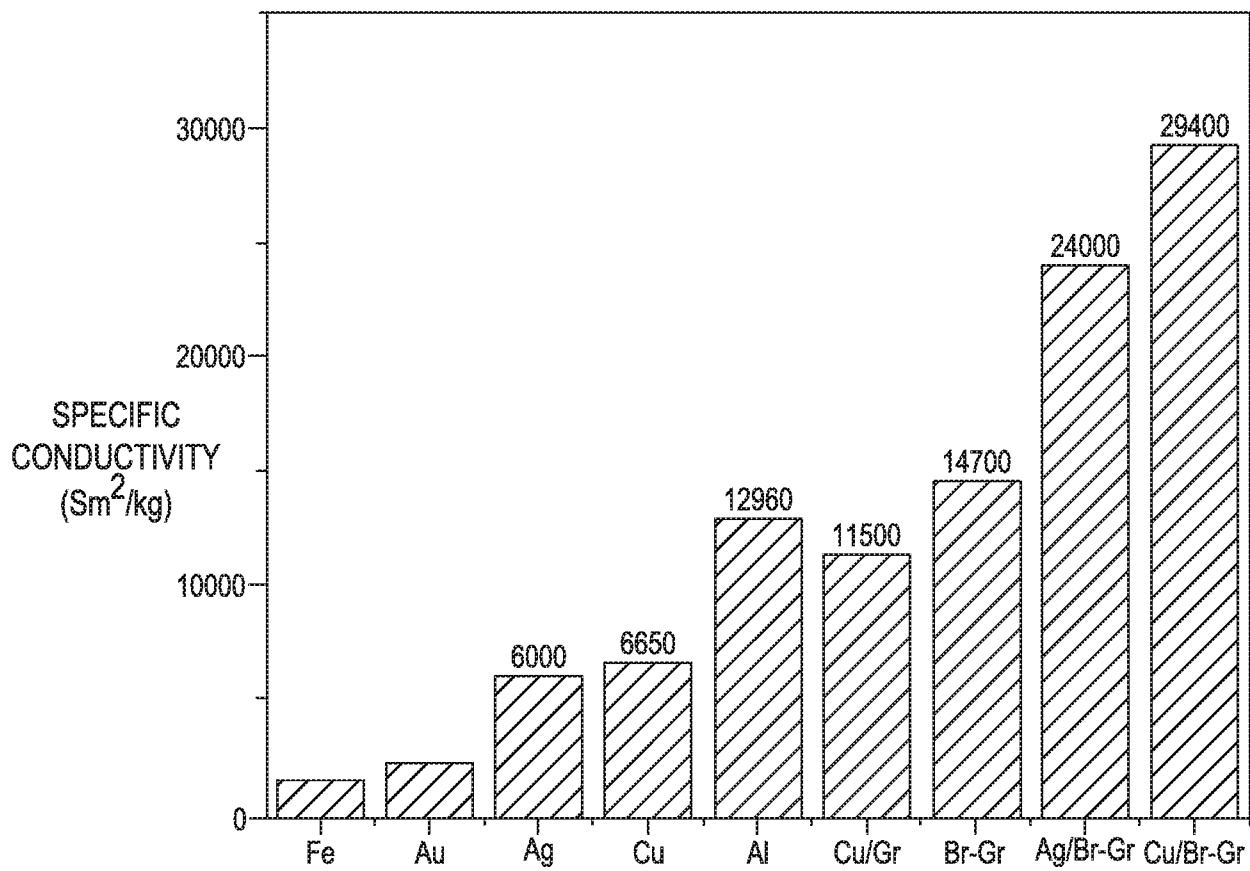
FIG. 10H FIG. 10I

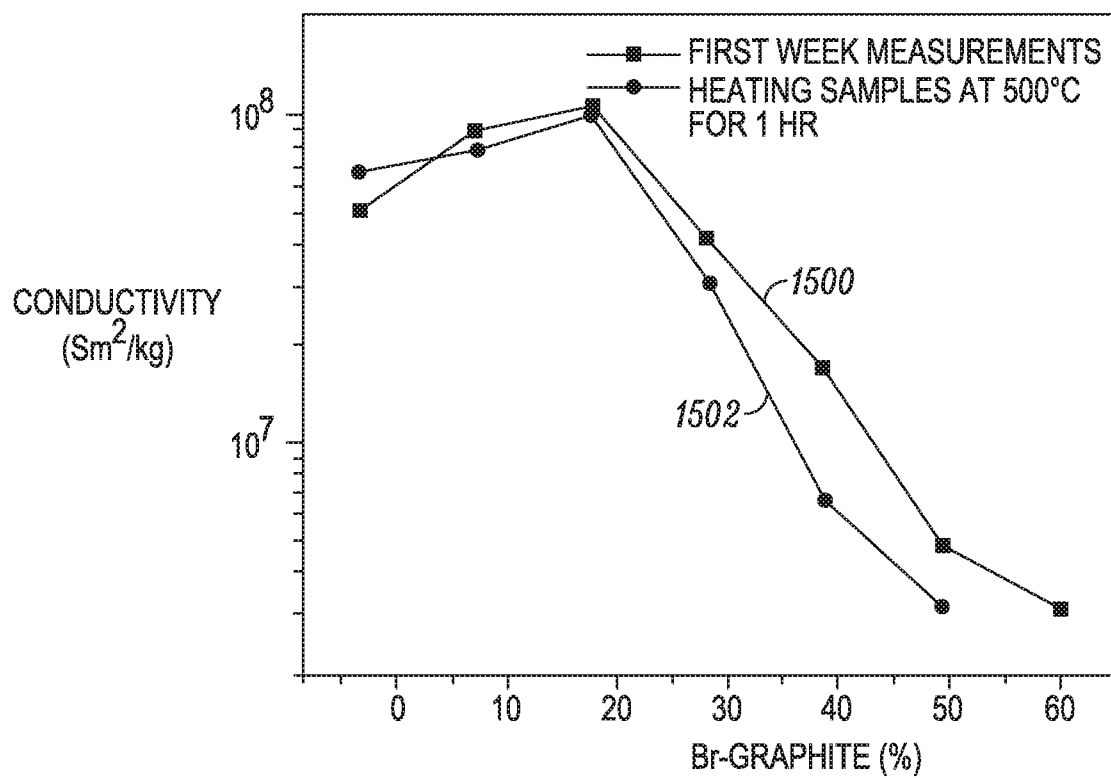
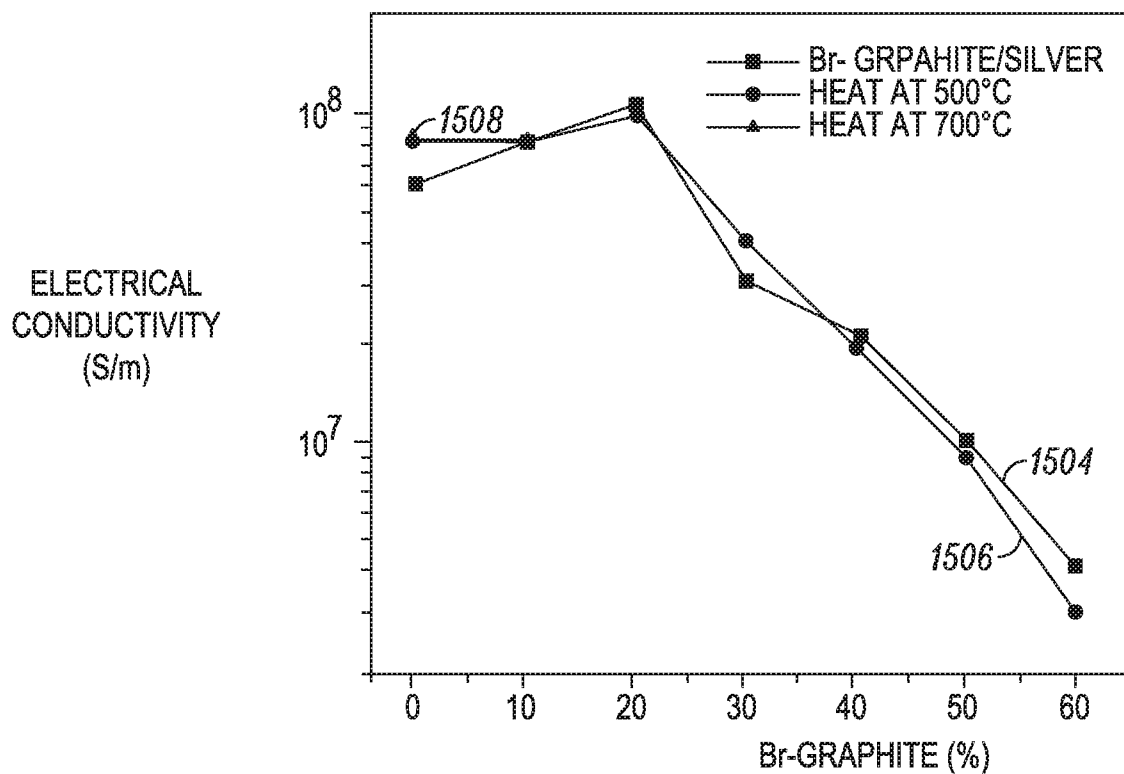
*FIG. 11A**FIG. 11B*

*FIG. 11C**FIG. 11D*

*FIG. 12A**FIG. 12B*

*FIG. 13A**FIG. 13B*

*FIG. 14*

*FIG. 15A**FIG. 15B*

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2017/050137

A. CLASSIFICATION OF SUBJECT MATTER
INV. H01B1/04 H01B1/02
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
H01B C01B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EP0-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 565 649 A (VOGEL F LINCOLN [US]) 21 January 1986 (1986-01-21) column 3, lines 43-46 column 4, line 62 - column 5, line 16; claim 4 example 6	1-14
X	----- US 3 409 563 A (FRANCISZEK OLSTOWSKI) 5 November 1968 (1968-11-05) column 2, lines 46-56; example 8	1
X	----- US 2010/206363 A1 (CHOI JAE-YOUNG [KR]) 19 August 2010 (2010-08-19) paragraph [0109]; claims 5,7; example 4 ----- -/--	1



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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"E" earlier application or patent but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

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Date of the actual completion of the international search

4 April 2017

Date of mailing of the international search report

12/04/2017

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Fax: (+31-70) 340-3016

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Vanier, Cécile

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2017/050137

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2015/194241 A1 (TANIELIAN MINAS H [US]) 9 July 2015 (2015-07-09) paragraphs [0019], [0024], [0025], [0021]; figure 4	15-25
A	----- US 2014/231718 A1 (LIN YI-JUN [TW] ET AL) 21 August 2014 (2014-08-21) paragraphs [0069], [0071], [0072], [0080]; figure 1(b) -----	1-25

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/IB2017/050137

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 4565649	A	21-01-1986	NONE	
US 3409563	A	05-11-1968	NONE	
US 2010206363	A1	19-08-2010	KR 20100093965 A US 2010206363 A1	26-08-2010 19-08-2010
US 2015194241	A1	09-07-2015	CN 105706179 A EP 3092652 A1 US 2015194241 A1 WO 2015105537 A1	22-06-2016 16-11-2016 09-07-2015 16-07-2015
US 2014231718	A1	21-08-2014	US 2014231718 A1 US 2017076833 A1	21-08-2014 16-03-2017