Fabrication of Metal/Carbon Nanotube Composites by Electrochemical Deposition

Definition

Metal/carbon nanotube (CNT) composites are promising functional materials due to the various superior properties of CNTs in addition to the characteristics of metals. Electrochemical deposition can be classified into three types: (1) composite plating by electrodeposition or electroless deposition, (2) metal coating on CNT by electroless deposition, and (3) electrodeposition using CNT templates, such as CNT sheets and CNT yarns.

1. Introduction

Carbon nanotubes (CNTs) have excellent mechanical characteristics such as high tensile strength and high elastic modulus, and also possess high thermal and electrical conductivity. Therefore, research into the practical applications of carbon nanotubes has been expanding into wide field, and composite materials of such nano-sized filler materials, such as polymer/CNT composites, have been studied expecting their innovative functions. Metal/CNT composites also have been investigated to enhance properties of metals and/or to give new innovative functions to metals. However, in general, the wettability of molten metals against CNTs is poor, resulting in difficulties of controlling the interface between the filler and matrix. In addition, since CNTs are nanosized fibrous materials and easily form aggregates, it is very difficult to form a metal/CNT composite with well-distributed CNTs in the metal matrix.

Electrochemical deposition is roughly classified into electrodeposition and electroless deposition, and the fabrication processes of metal/CNT composites by the electrochemical deposition can be categorized into three types: (1) composite plating by electrodeposition or electroless deposition, (2) CNT coating by electroless deposition, and (3) electrodeposition using CNT templates (Figure 1). “Composite plating” is one of the electrochemical deposition techniques. CNTs are incorporated in deposited metal matrix during plating. In the case of "metal coating on CNTs by electroless deposition", the prepared metal-coated CNTs are mainly used as filler of composites, such as resin composites. In the case of “electrodeposition on CNT templates”, CNT yarns or CNT sheets are used as CNT templates. The electrochemical deposition is a nano-scale or atomic scale process to fabricate metal materials and hence is effective to form atomic scale boundary between metals and CNTs. Moreover, this method is a wet process and consequently is likely advantageous to form metal/CNT composites with well-distributed CNTs in the metal matrix, especially in the case of the composite plating.

![Figure 1](image_url). Classification of fabrication process for metal/CNT composites by electrochemical deposition.

2. Fabrication of Metal/CNT Composites Using Composite Plating by Electrodeposition or Electroless Deposition

2.1. Composite Plating

Rough schematics of composite plating by electrodeposition and electroless deposition are displayed in
In the case of electrodeposition, inert particles are dispersed homogeneously in a plating bath. When a voltage is applied, metal is electrodeposited on a cathode and the particles adsorb on the surface of the deposited metal. Then, the particles are embedded in depositing metal, resulting in a metal composite (Figure 2). In the case of CNT composite plating by electrodeposition, inert particles are dispersed homogeneously in a plating bath containing a reducing agent. When a substrate is soaked in the bath, metal is reductively deposited on the substrate accepting electrons from the reducing agent and, at the same time, the particles adsorb on the surface of the deposited metal. The particles are then embedded in depositing metal, resulting in a metal composite (Figure 3). In general, the substrate is pre-treated and catalyst particles, such as Pd particles, are fixed on the surface of the substrate before soaking into the plating bath. As far as was searched, the first article of the composite plating is on Cu/graphite composites by electrodeposition and was reported in 1928 [3]. Regarding the mechanism of the composite plating, several models have been proposed [4][5][6][7][8].

2.2. Preparation of Plating Bath for Metal/CNT Composite Plating

To fabricate metal/CNT composites with uniform distribution of CNTs, the preparation of plating baths with homogeneous dispersion of CNTs is important. In general, plating baths are aqueous solutions, while CNTs are hydrophobic. Therefore, hydrophilization of CNTs have been examined by the addition of surfactants or the direct introduction of hydrophilic groups on the surfaces of CNTs (Figure 4). The addition of surfactants in plating baths is a common method. Various kinds of surfactants such as sodium dodecylbenzene sulfonate and sodium deoxycholate, have been examined for the homogeneous dispersion of CNTs in a pure water. However, effective surfactants for the dispersion in a pure water are not always effective in plating baths which contain great amounts of ions. Moreover, even if the surfactant is effective for the dispersion of CNTs in a plating bath, CNTs are not always co-deposited by electrochemical deposition. Therefore, the selection of appropriate surfactants is essential. Since the surfactants are likely incorporated in deposited metal matrix during electrochemical deposition, the concentration of surfactants should be examined. On the contrary, the direct introduction of hydrophilic groups, such as -COOH, onto the surfaces of CNTs has been examined using a chemical treatment [27], a plasma treatment [28], a heat treatment [29], and so on. These methods destroy the sp² carbon bonding of the surfaces of CNTs. Therefore, the conditions of the treatments should be examined.
On the contrary, CNTs are nanosized fibrous material and consequently tend to aggregate. In particular, SWCNTs have the thinnest (ca. 1-4 nm in diameter) among the various types of CNTs and can thus easily form aggregates referred to as bundles (Figure 5).

2.3. Unique Feature of Composite Plating Using CNTs as Inert Particles

Since a single CNT, especially multi-walled CNT (MWCNT) has a fibrous shape with large aspect ratio in addition to a high electrical conductivity in the axis direction. Therefore, composite plating using CNTs as inert particles often shows a unique feature unlike other composite plating using insulation particles such as Al₂O₃ particles. The schematic of the unique feature is showed in Figure 6 [30]. When a part of a MWCNT is incorporated in the deposited metal matrix during electrodeposition, the metal can be electrodeposited not only on the deposited metal but also on the protruding edge (a defect site) of the MWCNT. If the defect sites exist on the sidewall of the MWCNT, the metal can also be electrodeposited on the defect sites.
Using this unique phenomena, powder Cu/MWCNT composites could be obtained \[31\]. Figure 7a displays the surface morphology of Cu/MWCNT composites just after the electrodeposition. Many Cu/MWCNT composites particles are seen. These particles are fixed loosely on the cathode substrate and can be separated easily by ultrasonification. Figure 7b displays the morphology of the Cu/MWCNT composite powder after the separation from the substrate by ultrasonification. A large number of MWCNTs stick out from the Cu particles, resulting in a sea urchin shape. The size of the Cu spheres is 2–15 μm.

### Figure 7. SEM images of (a) Cu/MWCNT composite immediately after electrodeposition and (b) Cu/MWCNT composite powder separated by ultrasonification. (Figure 8 is adapted from reference \[31\]).

### 2.4. Fabrication of Metal/CNT Composites Using Composite Plating by Electrodeposition

Fabrication conditions in these articles are listed in Table 1.

#### Table 1. Fabrication conditions of metal/CNT composites using composite plating by electrodeposition.

| Metal | CNT   | Treatment of CNT       | Base Plating Bath   | Surfactant             | Remarks                  | Year | Ref. |
|-------|-------|------------------------|---------------------|------------------------|--------------------------|------|------|
| Ni    | MWCNT | Chemical treatment     | Dull Watts bath     | Sodium lauryl sulfate  | Corrosion behavior       | 2020 | \[32\] |
| Ni    | MWCNT | Chemical treatment     | Dull Watts bath     | Sodium lauryl sulfate  | Corrosion protection     | 2020 | \[33\] |
| Metal  | CNT  | Treatment of CNT                  | Base Plating Bath     | Surfactant                                                                 | Remarks                              | Year  | Ref. |
|-------|------|-----------------------------------|-----------------------|---------------------------------------------------------------------------|--------------------------------------|-------|------|
| Ni    | MWCNT| Wrapped by polydopamine           | Dull Watts bath       | Non                                                                       | Wear and corrosion resistance       | 2019  | [34]|
| Ni    | MWCNT| Non                               | Ionic liquid (choline chloride/carbamide) | Non | Non-aqueous solvent                                                      | 2017  | [35]|
| Ni    | MWCNT| Non                               | Sulfamate bath        | Cationic surfactant, compound name is unknown                             | Improvement in tool life             | 2014  | [36]|
| Ni    | MWCNT| Ball milling                       | Bright Watts bath     | Sodium lauryl sulfate and Hydroxypropylcellulose                          | Corrosion behavior                  | 2011  | [37]|
| Ni    | MWCNT| Chemical treatment                | Choline chloride/urea | Non                                                                       | Non-aqueous solvent                 | 2010  | [38]|
| Ni    | MWCNT| Non                               | Bright Watts bath     | Sodium lauryl sulfate, Cetyltrimethylammonium bromide                     | Solid lubrication                   | 2008  | [39]|
| Ni    | MWCNT| Ball milling                       | Watts type bath       | Cetyltrimethylammonium bromide                                            | Effects of surfactants              | 2008  | [40]|
| Ni    | MWCNT| Chemical treatment                | Dull Watts bath       | Non                                                                       | Effects of current density          | 2008  | [41]|
| Ni    | MWCNT| Ball milling                       | Bright Watts bath     | Sodium lauryl sulfate and Hydroxypropylcellulose                          | Mechanical properties               | 2008  | [42]|
| Ni    | MWCNT| Non                               | Bright Sulfamate bath | Polyacrylic acid                                                          | Low internal stress                 | 2007  | [43]|
| Ni    | MWCNT| Non                               | Dull Watts bath       | Polyacrylic acid                                                          | Pulse-reverse parameter            | 2007  | [44]|
| Ni    | MWCNT| Non                               | Bright Watts bath     | Polyacrylic acid                                                          | Thermal conductivity               | 2006  | [45]|
| Ni    | MWCNT| Non                               | Dull Watts bath       | Poly(diallyldimethylammonium chloride)                                    | Pulse-reverse electrodeposition     | 2005  | [46]|
| Ni    | MWCNT| Chemical treatment                | Dull Watts bath       | Cetyltrimethylammonium bromide                                            | Corrosion behavior                 | 2005  | [47]|
| Ni    | MWCNT| Non                               | Dull Watts bath       | Polyacrylic acid                                                          | Ni deposition on incorporated CNT  | 2004  | [48]|
| Ni    | MWCNT| Ball milling                       | Dull Watts bath       | Non                                                                       | CNT content                        | 2002  | [49]|
| Ni    | MWCNT| Ball milling                       | Dull Watts bath       | Non                                                                       | Tribological property              | 2001  | [50]|
| Ni-Co | MWCNT| Chemical treatment                | Dull Watts bath + Co salt | Non | Corrosion behavior                                                      | 2019  | [51]|
| Ni-P  | MWCNT| Non                               | Dull Watts bath + citric acid + P compound | Polyacrylic acid | Tribological properties                                                | 2010  | [52]|
| Ni-Co | MWCNT| Non                               | Dull Watts bath + Co salt | Compound name is unknown | Mechanical and tribological properties | 2006  | [53]|

**Ref.** indicates references to supporting materials or further reading.
| Metal | CNT     | Treatment of CNT | Base Plating Bath | Surfactant                                                | Remarks                                      | Year   | Ref.  |
|-------|---------|------------------|-------------------|----------------------------------------------------------|----------------------------------------------|--------|-------|
| Ni-P  | MWCNT   | Non              | Ni salts + citric acid + P compounds | Compound name is unknown                               | Corrosion properties                         | 2004   | [55]  |
| Cu    | MWCNT   | Chemical treatment | Citric bath      | Non                                                       | Corrosion behavior                           | 2021   | [56]  |
| Cu    | MWCNT   | Chemical treatment | Sulfate bath     | Non                                                       | Pulse reverse, electrical conductivity       | 2020   | [57]  |
| Cu    | MWCNT   | Chemical treatment? | Sulfate bath     | Non-ionic surfactants, Compound name is unknown          | Mechanical properties, Microlaminated structure | 2020   | [58]  |
| Cu    | SWCNT   | Non              | Sulfate bath     | Stearyltrimethylammonium chloride                        | Mechanical properties                        | 2020   | [59]  |
| Cu    | SWCNT   | Non              | Sulfate bath     | Non                                                       | Microstructure                               | 2019   | [60]  |
| Cu    | MWCNT   | Non              | Sulfate bath     | Sodium lauryl sulfate                                     | Jet electrodeposition, Tribological properties | 2019   | [61]  |
| Cu    | MWCNT   | Non              | Sulfate bath     | Polyacrylic acid                                         | Current collector for LIB anode             | 2019   | [62]  |
| Cu    | MWCNT   | Chemical treatment | Sulfate bath     | Stearyltrimethylammonium bromide                        | Electrical conductivity, Corrosion resistance | 2018   | [63]  |
| Cu    | MWCNT   | Non              | Sulfate bath     | Non-ionic surfactants, Compound name is unknown          | Mechanical properties, Laminated structure   | 2018   | [64]  |
| Cu    | MWCNT   | Chemical treatment | Sulfate bath     | Non                                                       | Cu/CNT powder + powder metallurgy           | 2018   | [65]  |
| Cu    | MWCNT   | Chemical treatment | Sulfate bath     | Non                                                       | Cu/CNT powder + powder metallurgy           | 2018   | [66]  |
| Cu    | MWCNT   | Chemical treatment | Sulfate bath     | Non                                                       | Cu/CNT powder + powder metallurgy           | 2017   | [67]  |
| Cu    | MWCNT   | Chemical treatment | Commercially available | Nano diamond                              | Periodic pulse reverse electrodeposition     | 2016   | [68]  |
| Cu    | MWCNT   | Non              | Sulfate bath     | Polyacrylic acid                                         | Current collector for LIB anode             | 2016   | [69]  |
| Cu    | MWCNT   | Non              | Sulfate bath     | Polyacrylic acid                                         | Co-deposition mechanism of CNT              | 2013   | [70]  |
| Cu    | MWCNT   | Non              | Sulfate bath     | Non                                                       | Electrochemical reduction behavior          | 2011   | [71]  |
| Cu    | MWCNT   | Non              | Sulfate bath     | Polyacrylic acid                                         | Pulse-reverse                               | 2011   | [72]  |
| Metal | CNT       | Treatment of CNT | Base Plating Bath | Surfactant                  | Remarks                                      | Year  | Ref. |
|-------|-----------|------------------|-------------------|-----------------------------|----------------------------------------------|-------|------|
| Cu    | MWCNT     | Non              | Sulfate bath      | Polyacrylic acid            | Surface morphology, Hardness, Internal stress | 2010  | [73] |
| Cu    | MWCNT     | Non              | Sulfate bath      | Polyacrylic acid            | Patterned field emitter                      | 2008  | [74] |
| Cu    | SWCNT     | Non              | Sulfate bath      | Commercial products         | Mechanical properties                        | 2008  | [75] |
| Cu    | SWCNT     | Chemical treatment | Sulfate bath      | Cetyltrimethylammonium chloride | Mechanical properties                      | 2008  | [76] |
| Cu    | Cup-stacked CNT | Non       | Sulfate bath      | Polyacrylic acid            | Various CNTs                                 | 2005  | [77] |
| Cu    | MWCNT     | Non              | Sulfate bath      | Polyacrylic acid            | Microstructure                               | 2004  | [78] |
| Cu    | MWCNT     | Non              | Sulfate bath      | Polyacrylic acid            | Cu/MWCNT composite powder                   | 2003  | [79] |
| Zn    | MWCNT     | Chemical treatment | Sulfate bath      | Cetyltrimethylammonium bromide | Corrosion resistance                       | 2021  | [80] |
| Zn    | MWCNT     | Non              | Zincate bath      | Unknown                     | Pulse electrodeposition, Corrosion resistance | 2020  | [81] |
| Zn    | MWCNT     | Chemical treatment | Sulfate bath      | Cetyltrimethylammonium bromide | Corrosion resistance                       | 2007  | [82] |
| Zn-Ni | MWCNT     | Non              | Chloride bath     | Non                         | Pulse reverse, Tribological and Corrosion properties | 2016  | [83] |
| Cr    | MWCNT     | Non              | Trivalent Cr bath | Sodium lauryl sulfate       | Tribological properties, Corrosion resistance | 2020  | [84] |
| Cr    | MWCNT     | Non              | Trivalent Cr bath | Sodium lauryl sulfate       | Tribological properties                      | 2018  | [85] |
| Cr    | MWCNT     | Non              | Trivalent Cr bath | Non                         | Mechanical properties                        | 2009  | [86] |
| Co    | MWCNT     | Non              | Choline chloride/urea | Non                       | Non-aqueous solvent                            | 2017  | [87] |
| Co    | MWCNT     | Non              | Sulfate bath      | Polyacrylic acid            | Field emission properties                     | 2013  | [88] |
| Co    | MWCNT     | Non              | Sulfate bath      | Polyacrylic acid            | Tribological properties                      | 2013  | [89] |
| Co    | MWCNT     | Acid-treatment   | Sulfate bath + citrate | Sodium lauryl sulfate     | Tribological properties, Corrosion properties | 2013  | [90] |
| Co-W  | MWCNT     | Non              | Co salt + Tungstate + Citrate | Polyacrylic acid         | Tribological properties, Corrosion properties | 2015  | [91] |
### Table 2. Fabrication conditions of metal/CNT composites by electroless deposition.

| Metal | CNT    | Treatment of CNT | Base Plating Bath | Surfactant                | Remarks                                | Year  | Ref. |
|-------|--------|-------------------|-------------------|---------------------------|----------------------------------------|-------|------|
| Co-W  | MWCNT  | Non               | Co salt + Tungstate + Citrate | Polyacrylic acid          | Tribological properties                | 2013  | [91] |
| Au    | MWCNT  | Non               | Sulfite bath      | Stearyltrimethylammonium chloride | Electrical conductivity, Tribological properties | 2009  | [92] |
| Ag    | MWCNT  | Non               | Choline chloride + glycerol | Poly (N-vinyl pyrrolidone) | Pulse reverse electrodeposition          | 2021  | [93] |
| Ag    | MWCNT  | Non               | Iodide bath       | Non                       | Electrical contact resistance against H₂S gas | 2021  | [94] |
| Ag    | MWCNT  | Non               | Iodide bath       | Non                       | Hardness, Electrical and Tribological properties | 2020  | [95] |
| Ag    | MWCNT  | Non               | Cyanide bath      | Unknown                   | Electrical contact resistance against H₂S gas | 2010  | [96] |
| Al    | MWCNT  | Acid treatment    | Diethylene glycol dimethyl ether | Non                     | Hardness                               | 2020  | [97] |
| Al    | MWCNT  | Non               | 1-ethyl-3-methylimidazolium chloride | Non                     | Hardness                               | 2006  | [98] |
| Sn    | MWCNT  | Non               | Choline chloride + ethylene glycol | Non                     | Nucleation study                       | 2019  | [99] |
| Pb-Sn | MWCNT  | Acid treatment    | Fluoroborate bath | Polyacrylic acid          | Corrosion resistance                    | 2010  | [100]|

### 2.5. Fabrication of Metal/CNT Composites Using Composite Plating by Electroless Deposition

Regarding the number of published articles on metal/CNT composite plating using electroless deposition, those on the Ni-P alloy/CNT is large. In the case of electroless deposition of Ni, phosphorous compounds such as sodium hypophosphite (NaH₂PO₂) are usually used as the reducing agent and the P derived from the NaH₂PO₂ is co-deposited with Ni, resulting in Ni-P alloy deposit. Most of the purpose of the fabrication of Ni-P alloy/CNT composites is the improvement of tribological properties. Fabrication conditions in these articles are listed in Table 2.
3. Metal-Coated CNTs by Electroless Deposition

3.1. Fabrication Process

A fabrication process of metal-coated CNTs by an autocatalytic electroless deposition is schematically showed in Figure 13. Even in the case of electroless deposition, homogeneous dispersion of CNTs in the plating bath is important. The introduction of functional groups on the surface of CNTs likely effective to increase deposition sites, resulting in CNTs coated by metal films and not metal particles.

3.2. Metal-Coated CNTs

Fabrication conditions in these articles are listed in Table 3.

Table 3. Fabrication conditions of metal-coated CNTs by electroless deposition.

| Metal | CNT    | Pre-Treatment of CNT | Reducing Agent | Surfactant                        | Remarks                              | Year  | Ref.  |
|-------|--------|----------------------|----------------|-----------------------------------|--------------------------------------|-------|-------|
| Ni-P  | MWCNT  | Chemical treatment Ball milling | NaH₂PO₂        | Sodium lauryl sulfate             | Mechanical attrition, Tribological properties | 2012  | [104]|
| Ni-P  | MWCNT  | HNO₃                 | Commercial product | Commercial product             | Substrate: Mg powder                    | 2011  | [105]|
| Ni-P  | MWCNT  | Non                  | NaH₂PO₂        | Stearyltrimethylammonium chloride | Substrate: ABS resin, Tribological properties | 2011  | [106]|
| Ni-P  | MWCNT  | Non                  | NaH₂PO₂        | Stearyltrimethylammonium chloride | Various P content, Tribological properties | 2010  | [107]|
| Ni-P  | MWCNT  | Chemical treatment | NaH₂PO₂        | Unknown                           | Effects on solder joint                | 2009  | [108]|
| Ni-P  | MWCNT  | Chemical treatment | NaH₂PO₂        | Cetyltrimethylammonium bromide    | Tribological properties                | 2009  | [109]|
| Ni-P  | MWCNT  | Chemical treatment | NaH₂PO₂        | unknown                           | Tribological properties                | 2006  | [110]|
| Ni-P  | MWCNT  | Ball milling         | NaH₂PO₂        | Compound name is unknown          | Hardness, Corrosion resistance          | 2005  | [111]|
| Ni-P  | SWCNT  | Heat treatment       | NaH₂PO₂        | Compound name is unknown          | Tribological properties                | 2004  | [112]|
| Ni-P  | MWCNT  | Ball milling         | NaH₂PO₂        | Cetyltrimethylammonium bromide    | Tribological properties                | 2003  | [113]|
| Ni-P  | MWCNT  | Ball milling         | NaH₂PO₂        | Cetyltrimethylammonium bromide    | Tribological properties                | 2003  | [114]|
| Ni-P  | MWCNT  | Ball milling         | NaH₂PO₂        | Cetyltrimethylammonium bromide    | Tribological properties                | 2002  | [115]|
| Cu    | SWCNT  | Non                  | CHOCOOH        | Sodium lauryl sulfate Hydroxypropylcellulose | Mechanical disintegration,             | 2016  | [116]|
| Cu    | MWCNT  | Non                  | CHOCOOH        | Sodium lauryl sulfate Hydroxypropylcellulose | Various CNTs, Tribological properties | 2014  | [117]|
| Co-P  | MWCNT  | Non                  | NaH₂PO₂        | Non                               | Magnetic properties                    | 2016  | [118]|

3. Metal-Coated CNTs by Electroless Deposition

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3.2. Metal-Coated CNTs

Fabrication conditions in these articles are listed in Table 3.

Table 3. Fabrication conditions of metal-coated CNTs by electroless deposition.

| Metal | CNT    | Pre-Treatment of CNT | Reducing Agent   | Surfactant                        | Remarks                              | Year  | Ref.  |
|-------|--------|----------------------|------------------|-----------------------------------|--------------------------------------|-------|-------|
| Ni-P  | MWCNT  | Sn²⁺ sensitization + Pd²⁺ activation | NaH₂PO₂        | Non                               | Microstructure, Co-coated CNTs       | 2020  | [119]|

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Fabrication conditions in these articles are listed in Table 3.
4. Metal/CNT Composites by Electrodeposition Using CNT Templates (Sheet, Yarn)

CNT templates, such as CNT sheets \[139\][140][141][142] and CNT yarns or fibers \[143\][144][145][146], have been developed and their various practical applications have been researched. Although a single CNT has a high electrical conductivity, electrical conductivities of those templates are far less than metals such as Cu, due to

| Metal | CNT   | Pre-Treatment of CNT | Reducing Agent | Surfactant | Remarks                                      | Year | Ref. |
|-------|-------|----------------------|----------------|------------|---------------------------------------------|------|------|
| Ni-P  | MWCNT | Introduction of -COOH on CNT + Pd²⁺ | NaH₂PO₂        | Non        | EMI properties, Cotton fabric substrate     | 2020 | 1320 |
| Ni-P  | MWCNT | Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂        | Non        | Arc discharge synthesized CNTs              | 2015 | 1321 |
| Ni-P  | MWCNT | Sn²⁺/Pd²⁺ commercial product | NaH₂PO₂        | Non        | Fe-50Co composites, magnetic properties      | 2014 | 1323 |
| Au/Ni-P | MWCNT | Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂, KB₄    | Non        | Improved wettability with molten Al         | 2012 | 1323 |
| Fe-B/Ni-P | MWCNT | Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂, KB₄    | Polyaclryic acid (Pre-treatment)            | Microwave absorbing properties   | 2011 | 1324 |
| Ni-P  | SWCNT | Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂        | Non        | Microstructure of Ni-layer                  | 2011 | 1325 |
| Ni-B  | MWCNT | Sn²⁺sensitization + Pd²⁺activation | (CH₃)₂NH·BH₃    | Polyaclryic acid (Pre-treatment)            | Graphitized MWCNTs Heat treatment | 2011 | 1326 |
| Ni    | MWCNT | Sn²⁺sensitization + Pd²⁺activation | N₂H₄           | Polyaclryic acid (Pre-treatment)            | Graphitized MWCNTs Magnetic properties | 2010 | 1327 |
| Ni-P  | MWCNT | K₂Cr₂O₇+H₂SO₄ Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂ | Non | Microwave absorbing properties, Ni-N alloy | 2008 | 1328 |
| Ni-P  | MWCNT | HNO₂ Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂        | Diallyl-dimethylammonium chloride            | Graphitized MWCNTs              | 2005 | 1329 |
| Ni-P  | MWCNT | Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂        | Polyaclryic acid (Pre-treatment)            | Graphitized MWCNTs              | 2004 | 1330 |
| Ni-P  | MWCNT | Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂        | Non       | Continuous Ni-layer                          | 2002 | 1331 |
| Ni-P  | MWCNT | Mixed Pd²⁺/Sn²⁺ | NaH₂PO₂ | Non | Pd-coated CNTs | 1999 | 1332 |
| Ni-P  | MWCNT | Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂        | Non       | Magnetic property                            | 1997 | 1333 |
| Al    | MWCNT | Sn²⁺/Pd²⁺ commercial product | LiAlH₄        | Non       | Non-aqueous bath: AlCl₃-urea                 | 2020 | 1334 |
| Ag    | MWCNT | H₂SO₄ + HNO₃ Sn²⁺sensitization + Pd²⁺activation | HCHO        | Non       | Interfacial adhesion of composites           | 2004 | 1335 |
| Cu    | MWCNT | Sulphoric acid + HNO₃ Sn²⁺sensitization + Cu²⁺activation | HCHO        | Non       | Electrical and mechanical properties         | 2009 | 1336 |
| Cu    | MWCNT | HNO₃ Sn²⁺sensitization + Pd²⁺activation HNO₃ | CHOClO₂      | Diallyl-dimethylammonium chloride            | Graphitized MWCNTs              | 2004 | 1337 |
| Co-P  | MWCNT | K₂Cr₂O₇+H₂SO₄ Sn²⁺sensitization + Pd²⁺activation | NaH₂PO₂ | Non | Heat-treatment | 2000 | 1338 |
the contact resistance between each CNT of which they consist. Therefore, metallization of the CNT templates is a promising process to give them enough electrical conductivity. On the contrary, CNTs have strong anisotropy in electrical and thermal properties. Therefore, the orientation of CNTs which make up the templates is also important in order to achieve the expected properties of metal/CNT composites. Fabrication conditions in these articles are listed in Table 4.

Table 4. Fabrication conditions of Metal/CNT Composites by Electrodeposition using CNT Template.

| CNT Template | Feature of CNT Template | Metal | Plating Bath | Remarks | Year | Ref. |
|--------------|-------------------------|-------|--------------|---------|------|------|
| MWCNT film   | Super-aligned           | Cu, Ni| Acid sulfonic bath + glucose Dull Watts Bath | Improved mechanical and electrical properties | 2019 | [148]|
| MWCNT film   | Super-aligned           | Ni    | Dull Watts Bath | Improved mechanical properties | 2019 | [149]|
| SWCNT paper (Bucky paper) | Orientation: in-plane direction | Cu | Acid sulfonic bath + polyethylene glycol + Cl⁻ + bis(3-sulfopropyl) disulfide + Janus green B | One-step electrodeposition by a combination of additives | 2017 | [150]|
| MWCNT paper  | Super-aligned           | Cu    | Acid sulfonic bath + glucose + polyethylene glycol + Cl⁻ Alkaline bath (EDTA, Citrate) | Electrical conductivity | 2017 | [151]|
| MWCNT film   | Super-aligned           | Cu    | Acid sulfonic bath + glucose | Improved mechanical properties | 2016 | [152]|
| MWCNT film   | Super-aligned           | Cu    | Acid sulfonic bath + glucose | Improved mechanical properties | 2015 | [153]|
| SWCNT yarn   | Straight                | Cu    | Acid sulfonic bath | Graphen growth on the surface of electrodeposited Cu | 2021 | [154]|
| MWCNT yarn   | Twisted                 | Cu    | Acid sulfonic bath + polyethylene glycol + Cl⁻ + bis(3-sulfopropyl) disulfide + Janus green B | One-step electrodeposition by a combination of additives | 2020 | [155]|
| CNT yarn     | Straight                | Cu    | Acid sulfonic bath | Superior current carrying capacity | 2018 | [156]|
| MWCNT yarn   | Twisted                 | Cu    | (CH₃COO)₂ + CH₃CN Acid sulfonic bath | Effect of CNT yarn density | 2018 | [157]|
| MWCNT yarn   | Twisted                 | Cu    | Cu (CH₃COO)₂ + CH₃CN Acid sulfonic bath | Two-step electrodeposition Uniform composite wire | 2017 | [158]|
| MWCNT yarn   | Twisted                 | Cu    | (CH₃COO)₂ + CH₃CN Acid sulfonic bath | Two-step electrodeposition Electrical properties, Solderability, | 2017 | [159]|
| MWCNT yarn   | Straight                | Cu    | Acid sulfonic bath | Electrodeposition of Cu interior of CNT yarn | 2016 | [160]|
| MWCNT yarn   | Twisted                 | Ag, Pt| KNO₃+AgNO₃ H₂SO₄ + H₂Pt₆Cl₆ | Improved tensile strength and electrical conductivity | 2013 | [161]|
| MWCNT yarn   | Twisted                 | Cu    | Acid sulfonic bath + octyl phenyl poly (ethylene glycol) ether | Continuous process: fiber spinning, anodization, electrodeposition | 2011 | [162]|
| MWCNT yarn   | Twisted                 | Au, Pd, Pt, Cu, Ag, Ni | Metal salt solution | Self-fueled electrodeposition Improved electrical conductivity | 2010 | [163]|

5. Conclusions
The fabrication process can be classified into three types: (1) composite plating by electrodeposition and electroless deposition, (2) metal coating on CNTs by electroless deposition, and (3) electrodeposition using CNT templates. In the composite plating, homogeneous dispersion of CNTs in plating baths is essential and, consequently, various processes, such as the addition of dispersants and introduction of hydrophilic groups on CNTs, have been studied. Numerous articles on Ni/CNT or Ni-P alloy/CNT composites by composite plating have been published and their excellent tribological properties and improved corrosion resistances have been reported. Many papers on Cu/CNT composites have also been published and their properties, such as electrical conductivity, have been investigated. The further elucidation of the mechanism of CNT composite plating process is expected. In the metal coating on CNTs by electroless deposition, the pre-treatments, such as sensitization and activation, are important. Oxidation of CNTs is useful for coating CNTs perfectly. A lot of articles on Ni-P alloy-coated CNTs have been published. In the electrodeposition using CNT templates, many papers on Cu/CNT composites using CNT sheets and CNT yarns have been published and their electrical properties have been reported. The preparation process to deposit Cu not only on the surfaces but also on the interior of CNT templates is likely the key technical point.

The practical applications of these technologies are expected in future work.

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Keywords
metal/carbon nanotube composite;electrochemical deposition;electrodeposition;electroless deposition;composite plating;carbon nanotube sheet;carbon nanotube yarn

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