Research Article

Nanostructured Multifilamentary Carbon-Copper Composites: Fabrication, Microstructural Characterization, and Properties

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This work is part of research on the emerging techniques to produce bulk nanostructured composites materials by severe plastic deformation and their characterization. Based on the Levi work, we present a new method to synthesize a composite wire-containing carbon-nanosized filaments (graphite and C60 fullerenes) embedded in a copper matrix. The originality of this process is using powder media as fiber. Microstructures and electrical, mechanical, and thermal properties are presented.

1. Introduction

Plastic deformation processing is a “top-down” approach to nanostructuring metals and alloys based on the accumulation of ultralarge plastic strain to achieve microstructure refinement [1]. Various plastic-deformation techniques have been developed to produce nanostructured materials over the last two decades [2, 3]. The more common techniques are Equal-Channel Angular Pressing (ECAP) [4], High-Pressure Torsion (HPT) [5, 6], and Accumulative Roll Bonding (ARB) [7, 8]. However, several variants and combination [9] of plastic deformation techniques, including accumulative repetitive deformation [3], were also developed Accumulative Spin-Bonding (ASB) [10], Equal Channel Angular Rolling (ECAR) [11, 12], Cyclic Extrusion Compression (CCE) [13], Cyclic Closed-Die Forging (CCDF) [14, 15], Linear Flow Splitting (LFS) [16], Severe Torsion Straining (STS) [17], Torsion Extrusion Process (TEP) [18], Cylinder Covered Compression (CCC) [19], Asymmetric Rolling (AR) [20, 21], and Accumulative Deep Wire-Drawing and bonding (WD) [22–24]. In this paper, we present a modified wire-drawing process.

Wire drawing is an ancient manufacturing technique of metallic profiles, bars, rods, and wires. The best known example is cold drawing of eutectoid carbon steels (0.77 wt% C): plastic deformation close to \( \varepsilon = 4 \) leads to a considerable refinement of the pearlitic microstructure down to the nanoscale [25–27]. Codrawing and coextrusion have also enabled the rise of a new class of nanostructured metal matrix composite materials whose industrial development is not mature but got intensive research. The fabrication process is based on the Levi’s technique [28]. It consists of a series of steps involving first co-drawing of bimetallic monofilamentary macrocomposite, bundling and re-stacking of the drawn elements followed by another drawing operation. Accumulative drawing and re-stacking result in to multifilamentary materials with a priori incompatible properties, such a high yield strength and good electrical conductivity [29, 30]. The archetypes of these materials are the Cu-Nb micro- or nanocomposites, used to manufacture coils generating very high magnetic fields [31, 32]. In these systems, the mechanical strength of composite in the plastic domain exceeds the “linear rule of mixtures”. Except Cu-Nb, other bimetallic couples such as Cu-Fe [28, 33, 34], Cu-Ag [35, 36], Cu-Cr [37], and Cu-Ta [38, 39] are also studied. In Cu-X couples, X-fibers are used as reinforcement phase, and conducting phase is assumed by the copper matrix. The choice of the couple Cu-X is generally dictated partly by a weak mutual solubility of Cu and X to reduce phase interactions but also by the reinforcing capacity of the X element.
Under these conditions, the copper-carbon (Cu-C) pair is interesting to study. Indeed these two elements are extremely immiscible [40], and therefore the composite could combine the high electrical conductivity of copper with the interesting characteristics of carbon, namely, a low coefficient of thermal expansion (CTE) and good tribological properties [41, 42].

These alloys are used in various applications such as electrical contact materials with friction [44, 45] and as heat sink for electronic components which require a material with a low CTE. Their use is also proposed to replace silver in electronic circuits [46] and as materials to confine plasma in nuclear fusion reactors [47, 48].

However, manufacturing these composites is difficult because the mutual solubility of carbon and copper [49] and the wettability of carbon by liquid copper [48, 49] are extremely low. The main manufacturing processes are the infiltration of a network of carbon fibers or graphite with copper, the deposition of copper on carbon fibers by hollow cathode and powder metallurgy. Furthermore, to our best knowledge, no process is available to manufacture nanostructured copper-carbon multifilamentary wire.

Therefore, the objective of this research is to obtain copper matrix composite wires containing millions of nano- or micro-sized carbon filaments. We have already demonstrated the ability to make such multifilamentary composite using the Levi’s technique [50]. The originality of this process [50, 51] is that carbon powder is used as a fiber unlike most of multifilament composites which are made from two bulk metallic materials. The microstructures obtained were studied at different scales and we present and discuss the resulting electrical, mechanical, and thermal properties.

## 2. Materials and Experimental Techniques

### 2.1. Materials

#### 2.1.1. Copper

Copper tubes used in processing route are Cu-b2 engineering materials with 99.95 wt% purity (deoxidized low phosphorous grade). We employed tubes of different diameters: tubes A (O_{in} = 4 mm/O_{out} = 8 mm) and tubes B (O_{in} = 10 mm/O_{out} = 12 mm). In order to recrystallize the microstructure, they were heat-treated at 500 °C during 3 hours under medium vacuum, followed by pickling with diluted nitric acid solution to remove oxide layers.

#### 2.1.2. Carbon

Two allotropes of carbon powders were used: natural graphite and C_{60} fullerenes. Graphite consists of agglomerated particles whose average size is 2 μm [43, 50]. C_{60} fullerenes powder has a purity of 98 wt%. SEM observation of C_{60} powder reveals agglomerates of equiaxed grains with a size ranging from tens to hundreds micrometers and faceted rods structure whose width is between 1 and 10 μm and length is about 10 μm [43, 52, 53]. We assume that the equiaxed grains of C_{60} result from the fragmentation of rods [54]. Before any use, and to remove organic impurities from powder purification process, 3-hour heat treatment at 150 °C under medium vacuum is applied [55]. X-ray analysis reveals that it does not change the structure of the truncated icosahedron C_{60} molecules and FCC solid structure [56–59].

### 2.2. Fabrication Technique

The first step (i.e., stage 0, monofilamentary composite) was synthesized by filling tube A with graphite or C_{60} fullerenes powders. The powder is manually compacted with a copper rod so it is uniformly distributed in the tube. The carbon-filled tube is then drawn up to 0.7 mm diameter. It is thereupon cut into pieces of same length which are bundled and introduced into tube B. The assembly is drawn again up to 0.7 mm diameter (stage 1) as shown schematically in Figure 1. A recrystallization heat treatment (500°C, 3 h, under medium vacuum) is applied at the end of each stage or when fractures occur during the drawing operations.

The evolution of graphite and C_{60} fullerenes fibers number embedded in the copper matrix according to re-stacking stages is given in Table 1. At the end of the processing Cu-C_{Gr} (final diameter: 0.8 mm with about $3.1 \times 10^7$ filaments of graphite and Cu-C_{C_{60}} wires (final diameter: 3 mm with about $1.33 \times 10^8$ filaments of C_{60}) are obtained.

### 2.3. Microstructural Observations and Mechanical, Electrical, and Thermal Characterizations Techniques

The samples microstructures were characterized by optical microscopy (OM) and scanning electron microscopy (SEM) following a metallographic preparation: mounting in a bakelite resin, mechanical polishing ending with 3 μm diamond paste and chemical-mechanical polishing with a colloidal suspension of silica to which few drops of hydrogen peroxide were added. The microstructure of the copper matrix is revealed by etching few seconds with an iron chloride alcoholic solution. The OM observations were made with an Olympus microscope coupled to a CCD camera to acquire digital images enabling image analysis (AnalyzeSIS software from the Olympus Soft Imaging System Company). The SEM images were obtained from a LEO FE1530 equipped with a field emission gun.

The deformation and recrystallized texture in metals with FCC lattices have been studied for a long time [60, 61]. The main method to study crystallographic orientation is X-ray diffraction texture analysis. The texture measurements were performed using X-ray four-circle diffractometer. The samples have coin-like shape between 4 mm to 5 mm in diameter and a thickness of less than 6 mm. One side is polished to equalize the height of the carbon and copper. Then the samples were etched to remove the hardening owed by polishing operations. Due to small carbon filaments cross-section, we have obtained only copper texture measurements. The signal from the carbon was too weak to produce satisfactory pattern. The acquisition has required from 10 to 20 hours counting time.

The mechanical tests were carried out at the initial strain rates of $\dot{\varepsilon} = 3.3 \cdot 10^{-4}$ s⁻¹ (cross-head speed of 2 mm/min) at room temperature with an Instron 4483 tensile testing machine with a 2 kN load cell. Samples for tensile have been annealed 3 hours at 500 °C, to obtain the same thermomechanical state to compare each stage. To measure electrical resistance, we used the four probes method using an OM21 microhmeter from AOIP Company on annealed samples (3 h @ 500 °C), to improve electrical conductivity. The resolution is 0.1 μΩ and the accuracy is about 0.03%.
The coefficient of linear thermal expansion of several composites was measured between 25 and 800°C with a differential dilatometer NETZSCH DIL 402C. The experimental conditions are as follows:

(i) heating rate: 300°C/h;
(ii) atmosphere: argon at 0.3 bar;
(iii) specimen dimensions: \( \Omega = 4 \text{ mm}, L = 20 \text{ mm} \);
(iv) number of test: 3 per measure;
(v) measurement uncertainty: ±5%.

### 3. Microstructures

Figure 2 presents Cu-C\textsubscript{Gr} sample micrographs from “stage 1” for different strains. The interstices between “stage 0” wires are still visible, but they disappear gradually with increasing strain. Figure 3 shows the multiscale features characteristic of these materials.

This process therefore allows a considerable refinement of carbon filaments diameter. Indeed, starting from an initial 4 mm fiber diameter and after several stages of restacking, transverse filaments dimensions became nanoscale 200 nm and 350 nm for Cu-C\textsubscript{Gr} and Cu-C\textsubscript{60}, respectively. However, the morphological evolution of both Cu-C\textsubscript{Gr} and Cu-C\textsubscript{60} composites is different, especially at the highest stages. For the Cu-C\textsubscript{Gr} composite, three structures of graphite filaments are observed as shown in Figure 4.

The first graphite structure results from the agglomeration of several filaments which form large particles of several microns in diameter. The second structure consists of a network of very fine filaments of a few hundred nanometers thickness. This structure is reminiscent of the phenomenon of “curling” observed during the codeformation of bimetallic composite with different crystal structure and Young’s modulus [62] (Figure 4(b)). The third category is represented by filaments whose cross-section is almost circular with a diameter of several tens of nanometers.
For Cu-C_{60} composites, filaments are individually identifiable; however, unlike Cu-C_Gr, no agglomeration occurs, though that C_{60} filaments do not have a circular cross-section but rather an elliptical or curvilinear.

The filaments diameter is also submicrometric. The difference between both composites morphology is directly connected to the crystalline structure and therefore mechanical behavior of each carbon phase. Indeed, C_{60} is a molecular solid (truncated icosahedron molecules) crystallizing in FCC structure and the intermolecular bonds are only Van der Waals type, hence sliding is easy. The graphite structure is hexagonal with \(\sigma\) bonds between the carbon atoms of the hexagonal graphene planes and \(\pi\) bonds between planes. Although sliding of graphene sheets with respect to another is possible, this cannot occur through the plane because the \(\sigma\) bonds are very strong. The molecular solid C_{60} therefore allows a priori an easier accommodation of mechanical stress than graphite.

In addition, etching of the composite Cu-C_{60} with an iron chloride solution highlights C_{60} filaments. Indeed, after dissolution of the copper matrix, we can observe naked filaments in “bundle-like” form (Figure 5(a)). These filaments are individually identifiable and keep their longitudinal appearance (Figure 5(b)).

This was also attempted on the Cu-C_Gr composite but without success; during the dissolution of the copper matrix, and contrary to those of fullerenes, graphite does not retain a filamentary structure; it disperses into the etching solution. This reveals a loss of mechanical strength compared to C_{60} fullerenes filaments.

Composite materials were heated to a temperature where recrystallization can occur (3 h @ 500°C), thereby strongly decreasing the number of defects caused by plastic deformation. However, as can be noted in Figure 6, the grain size of copper is not homogeneous.

Indeed, depending on the processing, it varies very significantly as shown in Table 2. Grain growth is inhibited by the carbon filaments and the interfaces between the copper tubes [43].

### Table 2: Mean grain size of copper matrix depending on the location of grains (Cu-C_{60} and Cu-C_Gr composites, stage 3, \(\varnothing = 5.2\,\text{mm}\)).

<table>
<thead>
<tr>
<th>Location of grain</th>
<th>Average grain size of copper ((\mu m)) Cu-C_{60} stage 3</th>
<th>Cu-C_Gr stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area 3</td>
<td>19 ± 6</td>
<td>19 ± 6</td>
</tr>
<tr>
<td>Area 2</td>
<td>19 ± 5</td>
<td>19 ± 5</td>
</tr>
<tr>
<td>Area 1</td>
<td>8.4 ± 2</td>
<td>8.4 ± 2</td>
</tr>
<tr>
<td>Area 0</td>
<td>2.0 ± 0.5</td>
<td>5.0 ± 1.8</td>
</tr>
</tbody>
</table>

Figure 2: Optical micrographs of the Cu-C_Gr composite cross-sections at “stage 1” at different strains [43].

Figure 3: Optical cross-section micrographs of Cu-C_Gr composite at “stage 3”, highlighting the multiscale structure (704 000 filaments) [43].
Figure 4: SEM micrographs to compare Cu-C$_{60}$ at stage 3 (a) and Cu-C$_{Gr}$ at stage 4 (b) cross-section microstructure [43].

Figure 5: SEM images of etched Cu-C$_{60}$ composite. The submicrometer thickness and continuity of C$_{60}$ filaments are underlined. Although they are in "bundle-like" form, they are, however, individually identifiable.

Microstructure study was supplemented by X-ray diffraction analysis to extract pole figures presented in Figure 6. The literature shows that wire-drawn FCC metals such as copper have a double-directions texture $\langle 111 \rangle$ and $\langle 200 \rangle$ [63–66] along drawing axis. As it be seen in Figures 7(a) and 7(b), the $\langle 111 \rangle$ texture component dominates in cold drawn and annealed matrix of Cu-C$_{60}$ and Cu-C$_{Gr}$ composites. However, the intensity varies from one composite to another and from one stage to another. The highest degree of texturing is observed in steps 1 and 3 for Cu-C$_{Gr}$ composite materials and at step 1 for Cu-C$_{60}$ composite materials. The $\langle 100 \rangle$ component comes in lower intensity except for the composite Cu-C$_{Gr}$ in step 1.

According to several studies [67–69] intermediate recrystallization annealing and severe deformation ($\varepsilon < 9$) may affect and change the crystallographic orientations of deformed copper matrix. On the one hand, they showed that copper, which has undergone a deformation of $\varepsilon < 9$, develops a $\langle 111 \rangle$ structural component in majority and more stable [70, 71]. On the other hand, intermediate recrystallization annealing and the application of very high deformations causing dynamic recrystallization of the Cu matrix [72] will promote the development of $\langle 100 \rangle$ component. The presence of these two types of texture in our samples shows that there has been deformation and then recrystallization of the copper matrix, although textures associated with intermediate annealed composites are low except for the composite Cu-C$_{Gr}$ in step 1. We see globally on both types of composite that an increase of the number of stages (Cu-C$_{60}$ step 2 and Cu-C$_{Gr}$ stage 4) causes a decrease of the intensity of the (111) texture component. Indeed, Dubois et al. [73] show that this regime is specific to nanocomposites metal and corresponds to a frustration of the normal recovery and recrystallization processes, impeded by the increased thermal resistance of Cu nanochannels.

4. Results and Discussion

4.1. Electrical Resistivity. Cu-C$_{Gr}$ and Cu-C$_{60}$ composites electrical resistivity is around 2 $\mu\Omega \cdot$ cm, close to Cu-b2 copper used for processing (Table 3). The presence of graphite or C$_{60}$ fullerenes, the first being a poor conductor compared to copper, the second being an insulator [43], weakly influences the electrical conductivity of the composite. Electrical resistivities of both composite materials, Cu-C$_{Gr}$ stage 4 (89% IACS) and Cu-C$_{60}$ stage 3 (85% IACS), are lower than most metals and copper alloys. Only silver (106% IACS) and pure copper (101% IACS) have the lowest electrical conductivity.

4.2. Mechanical Properties. The stress-strain curves of Cu-C$_{60}$ and Cu-C$_{Gr}$ are given in Figures 8(a) and 8(b), respectively. The tests were performed on materials annealed at 500°C during 3 hours. We deliberately choose to compare the materials properties after a recrystallization annealing, because the resistivity decreases, and because in the deformed state the material is often fragile. Furthermore, comparing the mechanical properties of the as-fabricated
Figure 6: SEM micrographs highlighting copper matrix grains (Cu-C_{60}, stage 3 Ø = 5.2 mm) [43].

Figure 7: X-ray pole figures of Cu matrix after deformation and recrystallization showing (111) and (100) texture components measured from cross-section of rod sample. (a) Pole figures of samples Cu-C_{Gr} in stages 1, 3, and 4. (b) Cu-C_{60} in stages 1 and 2 pole figures. All samples were annealed 3 hours at 500°C [43].

Table 3: Electrical resistivities (conductivities) of Cu-C_{60} and Cu-C_{Gr} composites and Cu after recrystallization heat treatment (500°C, 3 h).

<table>
<thead>
<tr>
<th></th>
<th>Cu</th>
<th>Cu-C_{Gr} (stage 4)</th>
<th>Cu-C_{60} (stage 3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface fraction $f_s$</td>
<td>—</td>
<td>0.061</td>
<td>0.096 ± 0.006</td>
</tr>
<tr>
<td>Nb. filaments ($\times 10^6$)</td>
<td>—</td>
<td>31.7</td>
<td>1.33</td>
</tr>
<tr>
<td>$\rho$ ($\mu \Omega \cdot \text{cm}$) (%IACS)</td>
<td>1.87 ± 0.06 (92.2 ± 3.0)</td>
<td>1.94 ± 0.15 (88.9 ± 6.9)</td>
<td>2.03 ± 0.05 (84.9 ± 2.1)</td>
</tr>
</tbody>
</table>
Table 4: Mechanical properties of Cu-C_{60} and Cu-C_{Gr} composites (stage 3) and comparison with copper.

<table>
<thead>
<tr>
<th></th>
<th>Copper</th>
<th>Cu-C_{60} stage 3</th>
<th>Cu-C_{Gr} stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface fraction f_{S}</td>
<td>—</td>
<td>0.095</td>
<td>0.096 ± 0.006</td>
</tr>
<tr>
<td>Nb. of filaments</td>
<td>—</td>
<td>704 000</td>
<td>1.33 million</td>
</tr>
<tr>
<td>R_{0.2%} (MPa)</td>
<td>81 ± 11</td>
<td>110 ± 3</td>
<td>238 ± 36</td>
</tr>
<tr>
<td>A_{f} (%/%)(%)</td>
<td>41 ± 3</td>
<td>10 ± 4</td>
<td>10 ± 5</td>
</tr>
<tr>
<td>R_{m} (MPa)</td>
<td>248 ± 11</td>
<td>166 ± 3</td>
<td>248 ± 26</td>
</tr>
</tbody>
</table>

The stress-strain curves for the different re-stacking stages are shown in Figure 8. Compared to pure copper, the yield strength of Cu-C_{60} composites increases significantly while the elongation decreases.

Meanwhile the stress/strain curves for Cu-C_{Gr} composites are always below the copper one in the plastic regime. We have no certain explanation for this behaviour. In the present state of our knowledge, the comparison with the hardening of “pure” copper is difficult because even if the process of forming and heat treatments are the same, the resulting microstructures are very different. The recrystallized microstructure of copper is obviously homogeneous in terms of grain size (30 to 40 microns) [43]. That of Cu-C_{Gr} is biphasic and the grain size is heterogeneous as shown in Table 2: it varies between 2 and 19 microns, and regardless of the presence of graphite filaments, we ignore the work hardening behavior of such microstructure during tensile testing. Furthermore, the presence of an irregular and heterogeneous distribution of graphite filaments with increasing re-stacking does not allow yet a prediction of the mechanical behavior of the composites. This may explain why the stress/strain curves for Cu-C_{Gr} composites at stage 1, 2, and 3 are below the one at stage 0.

The mechanical properties determined from these stress-strain curves are shown in Table 4 but only for stage 3.

The presence of carbon filaments (graphite or C_{60}) increases significantly the yield stress compared to pure copper, but decreases elongation at fracture. However, Cu-C_{60} has the most remarkable increase since yield stress reached (238 ± 36) MPa compared to (81 ± 11) MPa for copper. The variation of yield stress depends on the mean grain size (d); the strengthening results from grain refinement according to Hall-Petch relation [74–76]:

\[
\sigma(\varepsilon) = \sigma_0 + k \cdot d^{-1/2},
\]

where \(k\), which falls between 0.104 and 0.25 MPa/mm\(^{1/2}\) [72, 77–79], is the so-called Hall-Petch “constant,” despite the fact that it is strain, temperature, and strain rate dependent. \(\sigma_0\) is the intrinsic stress (36 MPa [79]). The Hall-Petch model is based on the hypothesis that grain boundaries act as obstacles in dislocation movements. That is to say, as the grain size is reduced, dislocation movements are impeded by grain boundaries, so the yield stress of specimen increases. We have made two assumptions. First, we consider that only copper matrix contributes to the strength because carbon (graphite, C_{60}) filaments have a small mechanical strength compared to copper. Second, we introduced a modified Hall-Petch relation by taking into account the surface fraction of each area (see Figure 6) which has different mean grain size. Then the Hall-Petch equation becomes

\[
\sigma = \sigma_0 + k \cdot \sum_{i=1}^{n} \frac{f_i}{\sqrt{d_i}},
\]

where \(f_i\) is the fraction surface of grains with \(d_i\) size.

After calculation, we obtain a yield in the range of 63–100 MPa according on the values of \(k\). However, this result,
The number of filaments and carbon fraction surface. plastic behavior of Cu matrix [80]. Also a beginning of poly-
matrix texture. It was demonstrated that even a
crease [43].

Table 5: Linear coefficient of thermal expansion of Cu-CGr and Cu-C_{60} composites in the temperature range (25°C–100°C) depending on

<table>
<thead>
<tr>
<th></th>
<th>Stage 1</th>
<th>Stage 2</th>
<th>Stage 3</th>
<th>Stage 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-C_{Gr}</td>
<td>Nb of filaments of C_{Gr}</td>
<td>110</td>
<td>8800</td>
<td>704000</td>
</tr>
<tr>
<td></td>
<td>Surface fraction of C_{Gr}</td>
<td>0.092</td>
<td>0.074</td>
<td>0.095</td>
</tr>
<tr>
<td></td>
<td>CTE_{mes} (×10^{-6} C⁻¹)</td>
<td>13.7</td>
<td>16.8</td>
<td>15.1</td>
</tr>
<tr>
<td>Cu-C_{60}</td>
<td>Nb of filaments of C_{60}</td>
<td>—</td>
<td>12100</td>
<td>1.33 millions</td>
</tr>
<tr>
<td></td>
<td>Surface fraction of C_{60}</td>
<td>—</td>
<td>0.11 ± 0.05</td>
<td>0.096 ± 0.005</td>
</tr>
<tr>
<td></td>
<td>CDT_{mes} (×10^{-6} C⁻¹)</td>
<td>—</td>
<td>12.4</td>
<td>13.3</td>
</tr>
</tbody>
</table>

comparable to that of the Cu-C_{Gr} composite at stage 3, is
below that experimental value which is 110 ± 3 MPa. In the
case of Cu-C_{60} composite, the values of the theoretical yield
strengths range from 67 MPa to 112 MPa (with an average of
86 ± 23 MPa). The experimental value is nearly three times
greater than calculated one.

The application of this modified Hall-Petch relationship
shows that grain refinement does not fully explain this in-
crease [43].

Other contributions should be considered especially the
influence of matrix texture. It was demonstrated that even a
moderate texture can result in significant anisotropy in the
plastic behavior of Cu matrix [80]. Also a beginning of poly-
merization of filaments C_{60} [81, 82] and structure change
can slightly contribute to mechanical reinforcement. A con-
tribution from the Cu-C interface is unlikely given the low
solubility of carbon and the absence of copper-carbon com-
pounds [40].

4.3. Thermal Properties. The average coefficient of thermal
expansion between 25 and 100°C (CTE) of Cu-C_{60} and Cu-
C_{Gr} composite is given in Table 5. With the exception of the
composite Cu-C_{Gr} stage 2, the presence of graphite or full-
erenes decreases significantly CTE compared to the pure
copper (CTE = 17 × 10^{-6} C^{-1}) [83].
The decrease of CTE is the largest for Cu-C_{Gr} at stage
4, (11 × 10^{-6} C^{-1}). It is also surprising that the CTE of
Cu-C_{60} is well below the CTE of the components, namely,
17 × 10^{-6} C^{-1} for copper and 20 × 10^{-6} C^{-1} for C_{60}. There-
fore, we may assume that successive annealing and cumulate
deformation can lead to a polymerization of C_{60} with a
change of structure and properties [81, 82].

5. Conclusions and Perspectives

In this work we demonstrate that the manufacturing method
of bimetallic composite multifilamentary wire by codrawing
and re-stacking is applicable to the development of Copper-
Carbon micro- or nanocomposite wires where the matrix is
the metallic phase and the second phase is a carbon powder.
The fabrication process combines simultaneously plastic
deformation and powder compaction. The successive wire-
drawing and re-stacking lead to multi-scale microstructure
of the metal matrix. The diameter of carbon filaments is
greatly reduced. Thus, the Cu-C_{Gr} wire has more than 3.1 ×
10^7 graphite filaments with a diameter of 200 nm, while that
of Cu-C_{60} contains more than a million of filaments with
350 nm in diameter.

This process enables development of materials with pro-
properties better or close to copper. Indeed, the yield stress is
significantly increased compared with that of copper, while
the elongation decreases dramatically. The electrical conduc-
tivity is weakly influenced by the presence of carbon (graph-
ite or fullerenes), but the coefficient of linear thermal expan-
sion decreases notably.

Cumulative co-drawing and bundling can be extended to
other metal-carbon systems like aluminum-carbon nano-
tubes and nonmetallic, such as metal-polymer [84]. We also
demonstrated that after drawing and etching, this process
enables to obtain naked C_{60} fullerenes filaments with nano
size section. These filaments, thus highlighted, can be used as
fibers which could be coated by metal, polymeric, or ceramic
matrix.

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