Preparation of Ni-C Ultrafine Composite from Waste Material

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**A R T I C L E  I N F O**

Article history:
Received: 02 April, 2017
Accepted: 15 May, 2017
Online: 09 June, 2017

Keywords:
Carbon Ultrafine Scale
Carbon-Nickel Ultrafine Composite
Plasma Technology
Spent Engine Oil
Plasma Heat Decomposition

**A B S T R A C T**

This work depicts the preparation of Ni-C ultrafine composite from used engine oil. The used oil was emulsified with detergent loaded with Ni (OH)2. The loaded emulsion was sprayed on electric plasma generated between two C electrodes to a DC main 28 V and 70-80 A. The purged Ni-doped carbon fume was trapped on a polymer film moistened with synthetic adhesive to fix the trapped smoke. Characterization of the deposit was made using SEM, XRD examined the crystal morphology. Carbon density in the cloud was calculated. The average size and thickness of the deposited composite is 120-160 nm. Aliphatic hydrocarbons readily decompose to gaseous products. Solid carbon smoke originates from aromatic compounds. Plasma heat blasts the oil in short time to decompose in one step.

1. Introduction

Teng [1] reported the use of plasma arc technology to produce fluid of carbon ultrafine particles dispersed in distilled water. Carbon is simultaneously heated and vaporized in a chamber furnace. Carbon vapor was cooled to prepare a carbon/water ultrafine slurry. The problem of heat dissipation from electrical appliances became a significant issue. To ameliorate this problem, there are four approaches commonly taken [2] enlarged the heat exchanger area and structure, [3] fabricated the heat exchanger using materials with higher thermal conductivity, [4] increased the working fluid flow rate to the heat exchanger, and [5] improved the heat transfer performance of the heat exchange working fluid. Akoh [6] prepared superfine fluids using the VEROS method. Teng [7] improved the VEROS technique and developed successful and effective preparation of Ag, Fe fine fluids. Bo [8] showed that plasma-enhanced chemical vapour deposition (PEVCD) has emerged as a key method to generate vapor-gas; and determine the development of VG with required properties. The paper summarized the state-of-the-art research on PECVD growth of VG ultrafine sheets. It provided guidelines on the design of plasma sources and operation parameters. Also to offer a future good challenges that would help commercial applications of VG. The authors studied the influence of feedstock gas temperature and pressure on VG growth. The effect of pretreatment and the growth of VG patterns on cylindrical and carbon ultrafine tube (CNT) substrates was shown. After discussion conclusion showed challenges and future directions for PEVCD growth of VG.

In the year 2014, defects-free graphene and with highest electron mobility was successfully prepared. Mayer [9] used adhesive tape to separate graphene sheets from graphite. It was reported that hydrazine followed by annealing in argon/hydrogen gases was successful to reduce graphite oxide. It was shown that almost intact carbon framework allowed efficient removal of functional groups. Some molten salts attack graphite particles to form a variety of carbon ultrafine structures including graphene [10]. Hydrogen cations, dissolved in molten lithium chloride, were discharged and intercalated and peeled to graphene sheets [11]. The graphene ultrafine sheets produced displayed a single-crystalline structure with a lateral size of several hundred ultrafine meters and a high degree of crystalline and thermal stability [12]. In 2014 defect-free, not oxidized graphene-containing liquids were made from graphite using mixers that produce local shear rates greater than 10x103 [13]. Mubarak [14] reported that the chemical vapor deposition (CVD) had been found a potential way to produce high quality and at high yield of CNTs. El Mel et al., [15]
Industrial nitrogen gas was used as a propellant to spray the emulsified oil on the plasma. The gas was supplied in cylinder with the following specifications:

- Nitrogen and trace inserts: 99.998%
- Oxygen: < 0.001%
- Density (g cm\(^{-3}\)): 1.2506
- Sp. Gravity (g cm\(^{-3}\)): 0.9737
- Viscosity (g cm\(^{-1}\)s\(^{-1}\)): 1.809x10\(^{-4}\)

The chemicals used are technical pure grade. Nickel hydroxide salt was synthetically prepared by precipitation by adding 10% ammonium hydroxide to nickel sulfate solution. The precipitated Ni(OH)\(_2\) was filtered, washed and dried.

2. Emulsification of the used engine oil

About 100 g of the used engine oil was placed in a conical flask of a thermostated agitator maintained at 80°C. The flask was fitted with an electrical mechanical stirrer. After the oil has acquired the required temperature, sodium sulphonate detergent containing nickel hydroxide suspended in distilled water was added step wise while stirring for 4 hours. The emulsion was then transferred to a separating funnel and left over night.

3. Method of preparation of the Ni-doped carbon ultrafine particles.

Determination of the extent of engine oil emulsification was determined by weighing the separated portion of the not emulsified oil. Fig. 2 shows partial (2a) and full (2b) emulsification of the used oil. Fig. 2 here.

4. Methods of measurement of the physic-chemical properties

Determination of nickel in the oil suspension or in the prepared composite was carried out by dissolving 1 g of the oil or 250 mg of the catalyst in 1 N nitric acid. Nickel content was determined using spectrophotometer Unicom 7400. Fig. 3 shows a schematic diagram of the Electric Plasma device used for thermal decomposition of the engine oil. Particle size and shape of the product were determined using the light-scattering size analyzer, SEM. Crystal morphology was examined by X-ray diffraction (XRD).

5. Results

The specifications of the engine oil are given in Table 1. It can be seen that the spent oil has lower viscosity, foam and gelation index and higher phosphorus content.
Fig. 4 shows the extent of emulsification of the spent engine oil (SEO) as affected by the weight of commercial anionic detergents containing 10% Ni as Ni(OH)_2. It is seen that the extent of emulsification of the spent engine oil increases linearly with the corresponding increase of the detergent. Anionic detergent is far efficient to emulsify engine oil as compared to the cationic detergent to emulsify the spent engine oil.

![Graph showing emulsification extent](image)

**Table 1.** The specifications of the engine oil used in this study

| Viscosity Grade | API. SN | Multigrade variations of 0W, 5W and 10W |
|-----------------|--------|------------------------------------------|
| 0W20, 0W30, 5W20, 5W30, 10W30 | Others |  |
| (spent) | | |
| Sculpture, max % | 0.5 | 0.45 |
| High temp. deposits, max mg | 35 | 46 |
| Foam | 1 min settling | <1 min settling | 10 min. settling | 1 min. settling |
| Gelation Index | 12 | 6 | NR | 12 |
| Emulsion Retention | NA | NA | NA | No Water Separation |
| P, max% / min% | 0.08/0.06 | 0.06 | NA / 0.06 | 0.08 / 0.06 |

Table 2 shows a comparative data on the effect of mass of anionic and cationic polymers as a detergent for emulsifying the used engine oil. It is shown that anionic polymer is more power

![Graph showing comparative data](image)

**Table 2** The effect of mass of the polymer on the emulsification extent of Em. at room temperature

| % emulsification | Anionic polymer | Cationic polymer |
|------------------|-----------------|-----------------|
| 20 | 18 | 20 |
| 40 | 30 | 30 |
| 60 | 38 | 50 |
| 80 | 50 | 72 |
| 100 | 60 | 100 |

Fig. 6 shows the rate of discharge of oil from the spraying nozzle as affected by the pressure of propellant nitrogen gas. It is seen that convenient rate of discharge amounting to 2.5-3.0 g/min is achieved with 3–3.5 KPa. Such rate was found suitable to generate and decompose the Em. together with the Ns solutio

![Graph showing rate of discharge](image)

**Fig. 5** Effect of detergent type on the extent of emulsification of spent engine oil.

**Fig. 6** The rate of the oil discharge from the spraying nozzle as affected by the propellant nitrogen gas

Fig. 7 shows a photograph of the electric plasma generated between the carbon electrodes.

![Photograph of electric plasma](image)

**Fig. 7** A photograph of the electric plasma exerted between two carbon electrodes
Table 3 The carbon density in the plasma hood

| No. | OIlg/ min. | ∑C, mg/min | H, mm | r, mm at H, mm | i² mm² | V, cm³ | Cc, mg/cm³/min | Cc, ug/cm²/min |
|-----|------------|-------------|-------|----------------|--------|--------|----------------|----------------|
| 1   | 1          | 275         | 100   | 10 50 30       | 900    | 94.247 | 2.917          | 297            |
|     |            |             |       | 20 60 40       | 1600   | 167.5  | 1.642          | 164            |
|     |            |             |       | 20 70 45       | 2025   | 212.0  | 1.297          | 129            |
|     |            |             |       | 25 80 55       | 3025   | 318.2  | 0.864          | 86.4           |
|     |            |             |       | 30 90 60       | 3600   | 375.04 | 0.7332         | 73.3           |
| 2   | 1.5        | 412.5       | 175   | 10 50 30       | 900    | 164.0  | 2.515          | 251            |
|     |            |             |       | 20 70 45       | 2025   | 369.2  | 1.1172         | 111.7          |
|     |            |             |       | 30 90 60       | 3600   | 656.3  | 0.6285         | 62.85          |
| 3   | 2          | 550         | 200   | 10 50 30       | 900    | 187.5  | 2.933          | 293.3          |
|     |            |             |       | 20 70 45       | 2025   | 421.9  | 1.303          | 130.3          |
|     |            |             |       | 30 90 60       | 3600   | 750.0  | 0.7333         | 73.33          |
| 4   | 2.5        | 687.5       | 250   | 10 50 30       | 900    | 234.4  | 2.933          | 293.3          |
|     |            |             |       | 20 70 45       | 2025   | 527.4  | 1.3035         | 130.35         |
|     |            |             |       | 30 90 60       | 3600   | 937.6  | 0.7339         | 73.39          |
| 5   | 3          | 825         | 300   | 10 50 30       | 900    | 281.3  | 2.933          | 293.3          |
|     |            |             |       | 20 70 45       | 2025   | 632.9  | 130.35         | 130.3          |
|     |            |             |       | 30 90 60       | 3600   | 1125.13| 0.7332         | 73.32          |

Table 3 shows the mean value of the weight of carbon particles disseminated in the cone of a plasma heat as a function of the weight of inserted oil and the dimensions of the cone.

Fig. 8 shows the XRD diffraction patterns of nickel-carbon composite species. It is seen that the diffraction peaks of Ni confirm the existence of well crystalline Ni metal. Raman spectrum of Ni doping C appeared with \( I_D/I_G = 0.63 \).

Fig. 9 shows the weight of the deposited carbon-Ni composite on the polymer film as a function of the weight of the sprayed engine oil as a function of the weight of the sprayed emulsified engine oil.

WCNIO is attained (45.5 ug/cm²). Fig. 10 shows the SEM of the deposited C-Ni composite material.

![Fig. 8 The XRD diffraction patterns of nickel-carbon composite species](image)

It can be seen that the weight of the deposited C-Ni amounts to 10 ug/cm² and increases gradually with the increase of the weight of sprayed engine oil up to 350 mg whereby the maximum weight of the deposited C-NiO (WCNIO) amounts to 100 ug/cm².

![Fig. 9 The weight of the deposited C-NiO (WCNIO)](image)

![Fig. 10 SEM photograph of the deposited C—Ni composite](image)
6. Mathematical Model

A mathematical model has been proposed to simulate the formation of C-Ni particles in the hood of the plasma heat vicinity based on the following assumptions:

1. The chemical analysis of the used engine oil (O_u) and the projected plasma heat at the hood vicinity (h_v) are kept constant throughout the experiments.
2. Solid carbon particles generate at a constant weight percentage of 27.5% of the weight of the oil as determined experimentally.
3. The weight of carbon generates in nearly no time; \( W_{tc} = 0.275 W_{to} \).
4. Ejection of the emulsified engine oil takes place in the plasma core at a constant discharge rate for a given driving pressure.
5. No extra air was fed into the hood vicinity of the plasma arc.
6. The carbon particles dissemination is distributed symmetrically at a given horizontal level in the hood.

\[ \dot{Q}_c = kA (T'_{H_x}) \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldot
The output results conclude the following.

8. Conclusion

The authors are thankful to Dr. Morsi Ameen and Eng. Usama Khedr, Welding Department, CMRDI, for their great help and support.

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