

The Seventeenth CIRP Conference on Electro Physical and Chemical Machining (ISEM)

Electrical discharge machining of ceramic/carbon nanostructure composites

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Abstract

The miniaturization of mechanical components with complex shapes is a great challenge in emerging applications. Silicon nitride (Si_3N_4) ceramics are excellent candidates for such applications due to their outstanding mechanical, thermal, and tribological properties. However, they are difficult to machine using normal mechanical machining methods. If the material were electrically conductive, electrical discharge machining (EDM) could be applied to produce precise and complex shapes. In this paper, in order to investigate the effects of electrical conductivity on the EDM characteristics, several carbon nanostructure composite materials are fabricated and EDMed using the assisting electrode method proposed by the current authors. The performance of the process is evaluated as a function of the carbon nanostructure content and type. The former is separately selected to be close to the electrical percolation threshold (0.9 vol.% and 5.3 vol.% for carbon nanotube (CNT) and graphene Nano platelet (GNP) composites, respectively), and well above that limit (5.3 vol.% and 20.6 vol.%), where electrical conductivities on the order of 10 and 100 $\text{S}\cdot\text{m}^{-1}$ are attained for CNTs and GNPs-based nanocomposites, respectively. In addition, bare Si_3N_4 specimens are also tested. Material removal rate, electrode wear ratio, and surface roughness of the machined pieces are analyzed for all testing conditions.

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Selection and/or peer-review under responsibility of Professor Bert Lauwers

Keywords: Sinking EDM; insulator Si_3N_4 ceramics; nanocomposite of Si_3N_4

1. Introduction

The miniaturization of mechanical components with complex shapes is a great challenge in emerging applications linked to the energy and communications sectors, such as in micro turbines or micro electro mechanical systems (MEMS). Silicon nitride (Si_3N_4) ceramics are excellent candidates for such applications owing to their outstanding mechanical, thermal, and tribological properties [1]. However, due to the hardness of these ceramics, alternative machining methods to diamond grinding are required for producing complex micro components. One promising technique is electrical discharge machining (EDM), which uses a spark of electricity to blast away the unwanted material in order to create complex shapes [2]. However, EDM commonly requires the use of materials with sufficient electrical conductivity ($> 0.3\text{-}1 \text{ S}\cdot\text{m}^{-1}$) [3], which limit its use on

insulating ceramics such as Si_3N_4 , SiC, and ZrO_2 . To overcome these difficulties, different approaches have been attempted by many researchers. Muttamara et al. [4] machined Si_3N_4 specimens by EDM using an assisting electrode (AEM) in the form of an electrically conductive coating, although the material removal rate (MRR) was low and the electrode was considerably worn. Other researchers were focused on the addition of large amounts of electrically conductive secondary phases, mainly 40 vol.% of TiN [5], but the surface of the machined material was high in roughness and yielded a foamy structure. Malek et al. [6] have recently reported the manufacture of a Si_3N_4 micro gear with a remarkably high MRR, low surface roughness (Ra), and low tool wear compared to $\text{Si}_3\text{N}_4/\text{TiN}$ materials, introducing just 5.3 vol.% of carbon nanotubes (CNT) into the ceramic matrix. Thanks to the outstanding electrical properties of CNT, a conductive percolating network was created in the composite at low CNT

contents, extraordinarily enhancing the electrical performance of Si_3N_4 [7]. Other carbon nanostructures (CNs) such as graphene present similar or even better electrical properties, and consequently, Si_3N_4 composites containing graphene nanoplatelets (GNP) have been developed with electrical conductivities as high as $4000 \text{ S}\cdot\text{m}^{-1}$ [8]. In this study, to investigate the difference in the EDM properties between the insulating and conductive Si_3N_4 composites containing CNs, two different fillers (CNT and GNP) and concentrations were employed: one close to the electrical percolation threshold (pc) and one significantly greater than that limit. These were EDMed using a homemade machine, and the performance of the EDM process was evaluated as a function of the content and type of CNs. Bare Si_3N_4 specimens, which are an insulating material, were also tested for comparative purposes.

2. Machining mechanism of AEM

The mechanism of the assisting electrode method has been assumed in previous literature, but without enough theory explaining the adhesion phenomena of carbonized products [9] [10]. A schematic of the assisting electrode method is given in Fig. 1. The surface of the insulating workpiece is covered by, or attached to, the electrical discharge material and is set up in the machine under a working oil. (1) At the start of EDM, discharge occurs between the tool electrode and the covered layer, similar to EDM of conductors. (2) Discharge occurs between the electrode and the covered material at deeper levels. In this region, the electrically conductive products, which are composed of carbonized machined material, adhere to the insulating surface. (3) The carbide products come in contact with the insulating material itself. (4) The carbide products generated from the dissociation of the working oil under the discharge maintain the electrical conductivity at the discharged area, even after the assisting electrode material is removed. Many machining trials have been carried out for Si_3N_4 , SiC, AlN, and ZrO_2 [9] [10].

This indicates that the electrical machining conditions depend on the physical characteristics of the workpiece. Three-dimensional precise complex shapes have been successfully machined with sinking and wire EDM methods for insulating ceramic materials such as Si_3N_4 , SiC, AlN, ZrO_2 , and diamond. Fig. 2 shows a typical machined sample for Si_3N_4 . In this sample, a star shape was machined on a concave surface. The tip of the star was kept with a sharp corner edge. A micro-hole was also machined on the insulating Si_3N_4 using this method.

The machinable limit of electrical conductivity for normal EDM, and the effects of the carbon layer creation phenomenon on the boundary of the insulating area and

electrically conductive area still require further discussion.

3. Experimental procedure

The fabrication of Si_3N_4 monolithic materials and composites containing CNs is described elsewhere [11]. In short, chemical vapor deposition (CVD) synthesized multi-walled CNTs (MWCNTs) of 9.5 nm average diameter and 1.5 μm average length (NC3100 Nanocyl S.A., Belgium) in concentrations of 0.9 and 5.3 vol.%, and GNP of nominally 200 nm diameter and 1 nm thickness (Angstrom Materials LLC, USA) in concentrations of 11.3 and 20.6 vol.%, were selected as CNs. Ceramic matrix compositions containing $\alpha\text{-Si}_3\text{N}_4$ powders (SN-E10, UBE Industries, Japan) plus Y_2O_3 (HC-Starck) and Al_2O_3 (SM8, Baikowski Chimie, France), were used as sintering aids. Both CNs and the ceramic powders were separately dispersed in alcohol by sonication and attrition milling, respectively, and then mixed in an ultrasonic bath under continuous stirring. The material compositions are displayed in Table 1. Powder compositions, maximum SPS temperature (TSPS), and electrical conductivity (σ) of the used materials are also shown in Table 1. The different composition powders were sintered using the spark plasma sintering apparatus (Dr. Sinter, SPS-510CE, and Japan). The sintered conditions are as follows: 5 min in vacuum (4 Pa), applying a pressure of 50 MPa, at temperatures in the range of 1585-1625 °C depending on the powder composition (Table 1). Sintered specimens were discs of 20 mm diameter and about 3 mm thickness. Fractured specimens containing CNs were observed using a field emission scanning electron microscope (FESEM, Hitachi S-4700, Japan). EDMed surfaces were observed using a laser microscope. The DC electrical conductivity (σ) was measured with an Agilent E3646A DC source and an Agilent 34401A voltmeter, in the DC voltage range of 6-60 mV. A four-probe method was employed using silver paste (Agar 6302) to improve the electrical contacts between the copper wires and the sample. The conductivity of the samples was determined in the direction parallel to the SPS pressing axis. The insulating materials were EDMed using the assisting electrode method [12] as shown in Fig. 3.

Table 2 shows the fundamental EDM conditions. The EDM experiments were carried out using the assisting electrode method. The conditions were determined from previous experimental results of the insulating Si_3N_4 material using the assisting electrode method. The machining properties were estimated by the MRR, electrode wear ratio, and Ra, of the EDMed surface. The surface roughness was observed on the removed surface of the adhered, electrically conductive layer using a laser optical microscope.

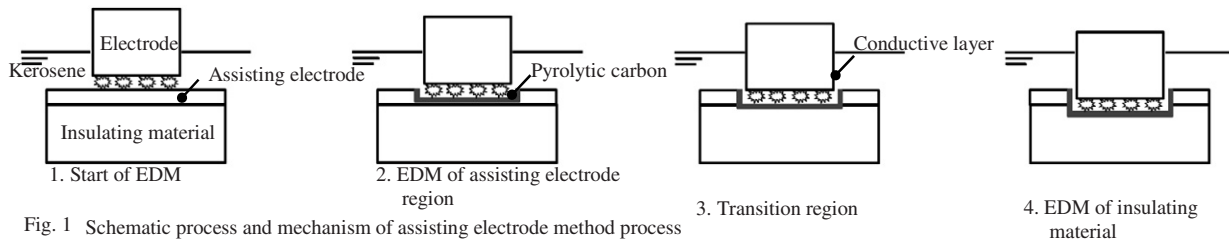
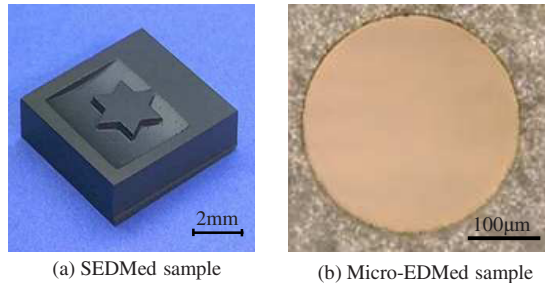


Fig. 1 Schematic process and mechanism of assisting electrode method process

Fig. 2 EDMed samples of insulating Si_3N_4 ceramics

4. Results and discussion

The densities of the sintered specimens were measured by water immersion. They were above 99% of the theoretical values, with the final α -phase content of the Si_3N_4 matrix $\sim 50\%$. Furthermore, the composites exhibited an excellent CN dispersion within the matrix, avoiding CN degradation after the sintering process, as was confirmed by the micro-Raman spectroscopy shown in Fig. 4 [10]. For the monolithic Si_3N_4 material, the electrical conductivity value (Table 1) corresponded to the detection limit of our experimental set-up, $10\text{--}13 \text{ S}\cdot\text{m}^{-1}$. Therefore, the real conductivity must be even lower than this value. As has been previously mentioned, the CN concentrations were selected to be close to the electrical percolation threshold, and well above that limit. In the case of 0.9 vol. % CNT composites, the electrical conductivity of $\sigma = 4 \times 10^{-2} \text{ S}\cdot\text{m}^{-1}$ became more than 10 orders of magnitude larger than the monolithic Si_3N_4 . It was assumed that this value would attain the machinable limit for the normal EDM method. A higher amount of CNT (5.3 vol. %) led to composites with $\sigma = 14 \text{ S}\cdot\text{m}^{-1}$. These were considered machinable by normal EDM.

On the other hand, in the GNP-based materials, an σ value of $1 \text{ S}\cdot\text{m}^{-1}$ was attained for the $\text{Si}_3\text{N}_4/11.3 \text{ vol. \% GNP}$ composite. For $\text{Si}_3\text{N}_4/20.6 \text{ vol. \% GNP}$, a maximum σ value of $2002 \text{ S}\cdot\text{m}^{-1}$ was reached, which is 15 orders of magnitude higher than the Si_3N_4 specimen. To experimentally confirm the machinable limit of electrical conductivity, as well as the effects of the assisting electrode on the machining properties, the assisting electrode EDM and normal EDM methods were applied for all specimens. The discharge phenomenon was not observed on the monolithic Si_3N_4 and CNT 0.9 vol.%. This indicates that the minimum electrical conductive value was lower than $4 \times 10^{-2} \text{ S}\cdot\text{m}^{-1}$. To compare the machining properties of the insulating and conductive materials, EDM was carried out using the same conditions. These were selected by considering pre-machining data as the most available conditions for the Si_3N_4 . The assisting electrode method was carried out on all workpieces. The relationships between the electrical conductivity value σ and the MRR, electrode wear rate, and Ra (evaluated by a laser optical microscope) were analyzed. Results are shown in Figs. 5, 6, and 7, respectively. In these figures, the results of normal EDM data are also displayed for the machinable specimens. All data represents the average value of three experiments. Very small deviations (within 15%) were observed for each data point. With the assisting electrode method, lower discharge machining conditions of discharge open circuit voltage and current were selected than normal conditions for electrically conductive material, and a 1/10 order reduction of the MRR was observed for the AEM than for the electrically conductive materials. The same MRR values were obtained on GNP materials independent of electrical

Table 1. Powder compositions, maximum SPS temperature (TSPS), and electrical conductivity (σ) of the materials tested by EDM

Material	Si_3N_4 (wt.%)	Al_2O_3 (wt.%)	Y_2O_3 (wt.%)	CNs (wt.%)	CNs (vol.%)	TSPS ($^\circ\text{C}$)	σ ($\text{S}\cdot\text{m}^{-1}$)
SN	93.0	2.0	5.0	0.0	0.0	1600	10^{-13}
SN/0.9 CNT	92.5	2.0	5.0	0.5	0.9	1585	4×10^{-2}
SN/5.3 CNT	90.0	2.0	5.0	3.0	5.3	1585	14
SN/11.3 GNP	85.0	2.0	5.0	8.0	11.3	1625	1
SN/20.6 GNP	78.0	2.0	5.0	15.0	20.6	1625	2002

Table 2. EDM conditions

Electrode	Cu (Φ8)
Electrode Polarity	negative
Open circuit voltage (V)	100
Setting Current (A)	5.0
Discharge duration (μs)	1.0

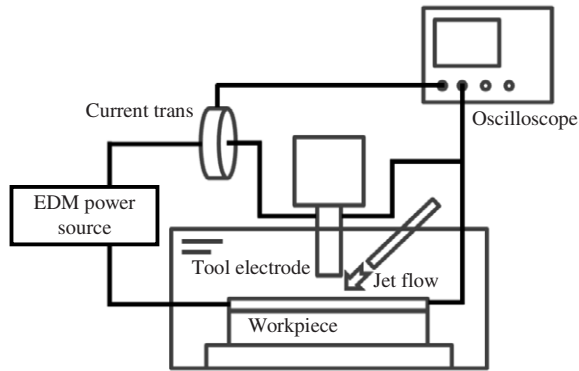


Fig. 3 Experimental schematic of the machining system

conductivity. In the CNT materials, the MRR decreased with increasing electrical conductivity. For the materials machinable by the normal EDM method, MRR values decreased with increasing electrical conductivity, and were lower than the assisting electrode case. The electrode wear rate values were larger on the insulating material than the other conductive materials, and decreased with increasing conductivity. Regarding the GNP materials, a lower wear rate was detected in the results of the assisting electrode method than the normal EDM method for the low conductivity composites Si₃N₄/11.3 vol.% GNP and CNT 5.3 vol.%. However, the opposite case was shown for Si₃N₄/20.6 vol.% GNP, which has higher conductivity. The Ra of the insulating materials was also larger than that of the conductive materials. The rougher surfaces were observed on the assisting electrode method than the normal method. Under these machining conditions, the MRR values of the monolithic Si₃N₄ and CNT 0.9 vol.%, which were regarded as insulating materials, were almost identical and were larger than those of the other conditions as observed using the laser optical microscope. Results are shown in Fig. 8 for (a) Si₃N₄, (b) CNT 0.9 vol.%, (c) CNT 5.3 vol.%, (d) GNP 11.3 vol.%, (e) GNP 20.5 vol.%, respectively. The other surface conditions observed between the insulating and conductive material as shown in Figs. 8(c) and 8(e). With regard to the roughness of the EDMed surface, many small size discharge craters were observed as in Fig.8. As mentioned in section 2, the electrically conductive layer, composed of carbonized products from the dissociation of the working oil under discharge, adhered to the

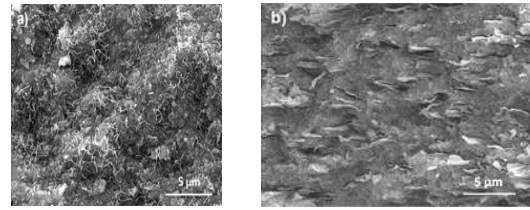


Fig. 4 FESEM micrographs of the fracture surfaces corresponding to: a) SN/5.3 CNT and b) SN/20.6 GNP specimens.

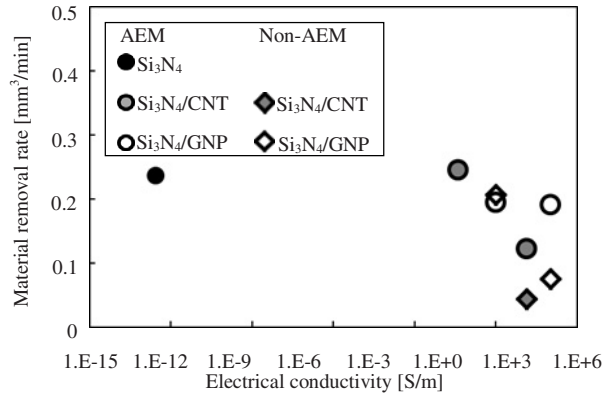


Fig. 5 Material removal rate as a function of the electrical conductivity

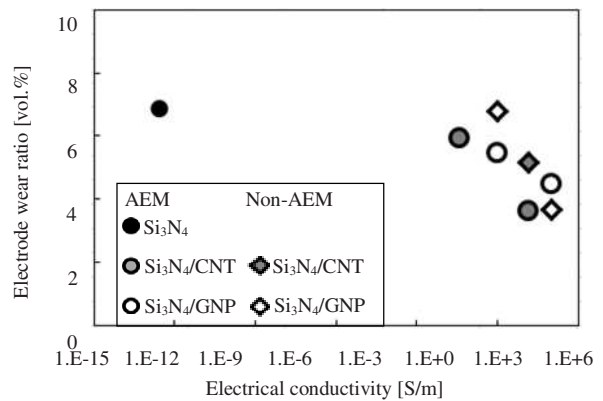


Fig. 6 Wear rate of electrode as a function of the electrical conductivity

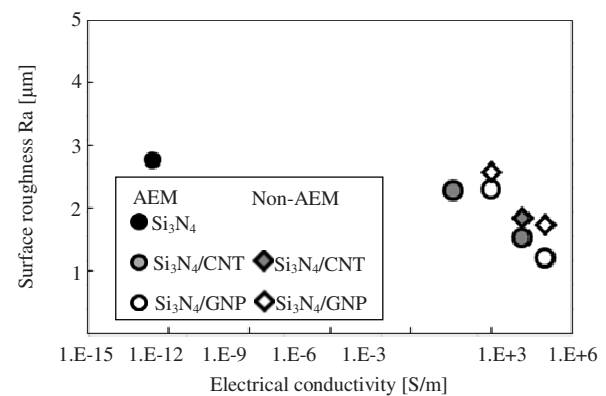


Fig. 7 Surface roughness, Ra, as a function of the electrical conductivity

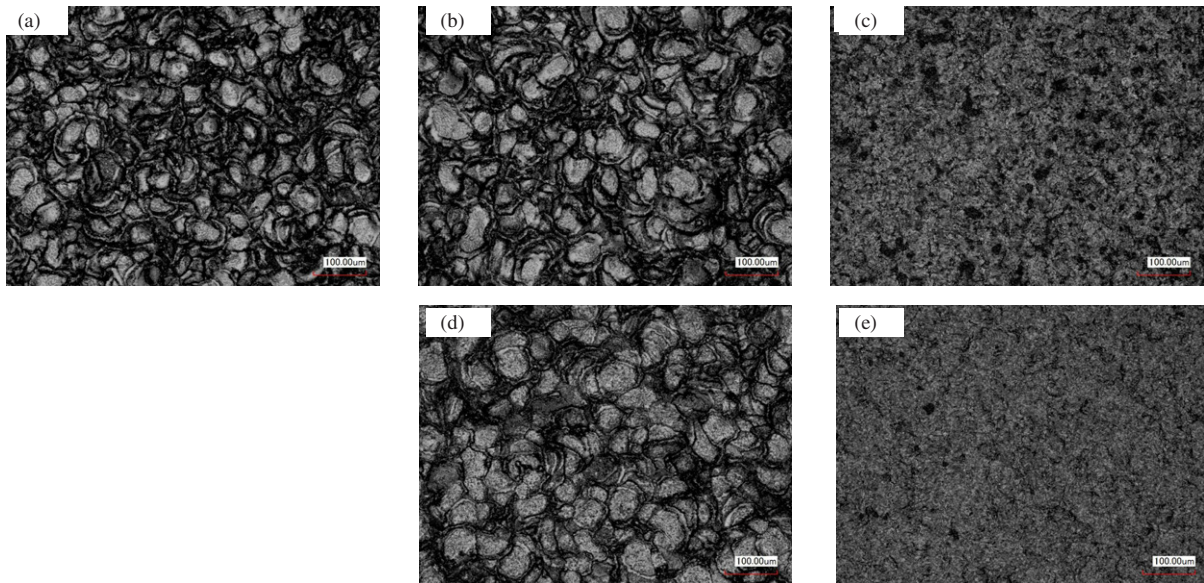


Fig. 8 Laser optical microscope images of AEMed surfaces: (a) Si₃N₄, (b) CNT 0.9 vol.%, (c) CNT 5.3 vol.%, (d) GNP 11.3 vol.%, and (e) GNP 20.6 vol.%

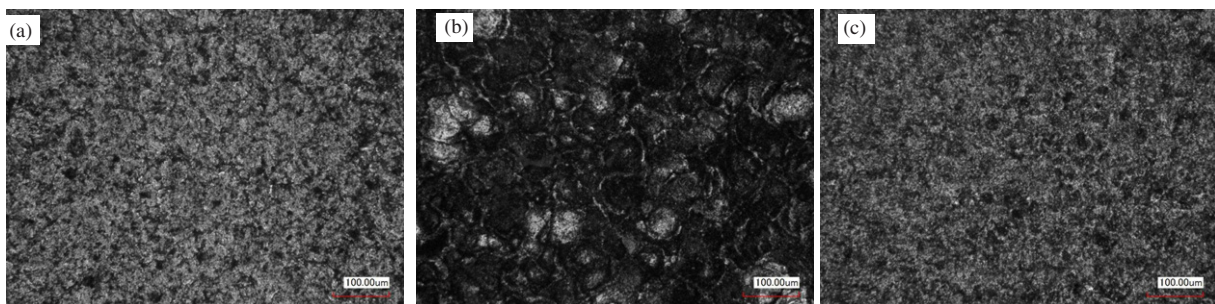


Fig. 9 Laser optical microscope images of normal EDMed surfaces: (a) CNT 5.3 vol.%, (b) GNP 11.3 vol.%, and (c) GNP 20.6 vol.%

surface continuously. It is assumed that the roughened surface produced the large carbonized craters. On the other hand, carbonized craters were not observed on the smooth surface. It appears that the assisting electrode material could not contribute to the carbonization phenomenon in the EDM process. However, these craters were also generated on the EDMed surface of GNP 11.3 vol.% composite (Fig. 8(d)), which could be EDMed using the normal method. If some carbonization occurred in the discharge process, carbide adhesion could occur on the electrically conductive workpiece. To confirm this phenomenon, the EDMed surfaces of electrically conductive materials were observed using a laser optical microscope. The observed results are displayed in Fig. 9, for (a) CNT 5.3 vol.%, (b) GNP 11.3 vol.%, and (c) GNP 20.5 vol.%, respectively. As seen in Fig. 9(b), some carbonized craters formed on the normal EDM method. We thus consider the carbonizing

phenomenon to occur at the electrically conductive area of GNPs. It indicated that when the amount of GNPs materials increased a little from the machinable limit for normal EDM, the carbon element adhered on the EDMed surface around the districted electrical conductive area. On the other hand, as shown in Figs. 9(a) and (c), carbonized craters are not observed on the machinable composite material. Considering the structures of CNT and GNP, the discharge phenomenon of carbonization would be different in the narrow electrically conductive area. The distribution of the conductive region in the insulating matrix was considered an important factor in controlling the carbonization phenomenon during discharge on the insulator and conductive mixed composite. To confirm the effects of the assisting electrode material on the EDM properties, the machined shape produced using the normal and assisting electrode EDM methods were compared for the GNP 11.3 vol.%

composite. The results are shown in Fig. 10 for (a) the assisting electrode method, and (b) the normal method. For the normal EDM method, a large chipping area could be observed, whereas for the assisting electrode method, a very smooth shape was detected. It appears that the continuous discharges occurred evenly on the distributed conductive element surface.

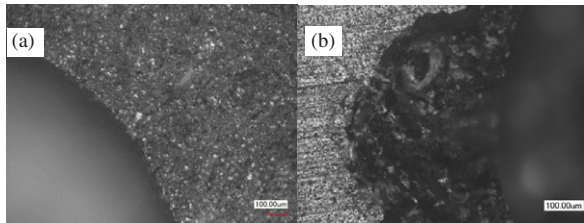


Fig. 10 EDMed surface shapes: (a) GNP 11.3 vol.%, and (b) GNP 11.3 vol.% without the assisting electrode material

5. Conclusions

To investigate the discharge behavior of Si_3N_4 ceramic/carbon nanostructure composites, EDM was carried out using the assisting electrode method. The results are summarized below:

- Insulating Si_3N_4 ceramics, and $\text{Si}_3\text{N}_4/\text{CNT}$ and $\text{Si}_3\text{N}_4/\text{GNP}$ nanocomposites, could be machined using the assisting electrode method on EDM.
- The machinable limit of electrical conductivity was below $4 \times 10^{-2} \text{ S} \cdot \text{m}^{-1}$.
- The EDM properties of electrode wear ratio and surface roughness were better on the conductive materials than insulating materials.
- Better MRRs were obtained on the insulating materials than the conductive materials.
- The same EDMed surface composed with discharge craters was detected on the electrically conductive material.
- The discharge craters were made by carbonized products and generation phenomena were controlled by the amount of the conductive element and its distribution.
- A better hole edge shape was obtained by the assisting electrode method than the normal method on a GNP composite with low conductivity.

Acknowledgements

This work was supported by the Spanish Government under the project MAT2009-09600. C. Ramirez thanks the financial support of the JAE (CSIC)

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