

## ACTUAL PROCESS PARAMETER DETERMINATION FOR MICRO-ENGRAVING OF FULLERENE FILM

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**Abstract:** Mechanical engineers are consistently challenged with the requirements posed by contemporary materials machining by using the existing equipment. In this case determining the cutting conditions becomes an actual problem. This paper offers a response to that request in the form of micro-machining of thin fullerene film deposited on a glass plate by using chemical vapor deposition method. Experimental verification of thin fullerene film machinability is conducted on computer numerical control engraving machine using a diamond scraper. Different values of process parameters are combined to determine adequate parameters set from groove edge quality aspect. During machining we noticed intensive wear, so one part of our research was directed towards determining the cause of tool wear. Modern equipment was used for qualitative analysis and near-optimal cutting condition selection and for analysis of wear debris. The results present a basis for further process optimization of thin fullerene film micro-engraving and for introduction of cutting conditions in the existing table for well-known materials.

**Keywords:** Fullerene film, Cutting condition setting, Micro-engraving.

### 1. INTRODUCTION

Processing parameters are very important during machining. The introduction of new contemporary materials has led to an increasing need for their machining. The real challenge here is to use the existing methods and equipment for machining these new materials. This paper presents optimal parameters for engraving one of these new materials, fullerene, on computer-aided engraving machine. Engraving was done on thin fullerene film. This film was applied on the transparent float glass using CVD method.

Fullerene, although a new material, has found the field of its application. The combination of micro-engraving and fullerenes can be applied in micro-fluidic, micro-structuring of the surface, micro-sensors, etc. Fullerene is the third allotropic modification of carbon [1]. It is characterized by a unique structure and excellent physical and chemical properties. Some features of fullerenes gave rise to a lot of ideas for research of its behavior under different processing methods.

The main goal of our research in the beginning was to establish the optimal parameters for machining fullerene film, deposited on glass, with engraving machine; however, intensive tool wear was noticed. This tool wear was noticeable with every combination of the parameters used. A later stage of our research went in the direction of determining the cause of the wear. To determine the cause we examined the wear debris more precisely, its origin and to do that we used spectral analysis.

Modern equipment was used for qualitative analysis and rough selection of optimal parameters. The results represent a basis for further optimization of micro processing of thin fullerene film.

### 2. METHODS AND MATERIALS

As already mentioned, fullerene thin film deposited onto a glass plate was used in the experiment. Fullerene is applied on glass (PGO, Germany) using chemical vapor deposition (CVD). Characteristics of glass and deposition methods can be taken from the literature [2]. Glass was 1.1 mm thick. In

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the range of 0 – 300 °C thermal expansion coefficient for glass is  $84 \cdot 10^{-7}$ , while the transparency, in the wavelength range of 380 – 2500 nm, is 92%. Vapor depositor JEE-400 (JEOL, Japan) was used for vapor deposition vacuum. The pressure in the depositor bell-jar, with 240 nm in diameter and with 270 nm of height, was  $10^{-5}$  Pa. There are two pairs of electrodes located in the jars, one of them is

equipped with carriers, while the second pair of electrodes is fitted with fullerene holders.

After applying a thin film of fullerene, the plate was recorded with the AFM (Atomic Force Microscopy) in order to determine the thickness of fullerene film. The measurements were performed in contact mode, and we used the MicroMasch CSC37 cantilever. Based on Figure 2.1 we determined that the thickness of fullerene film was 1.3 µm.

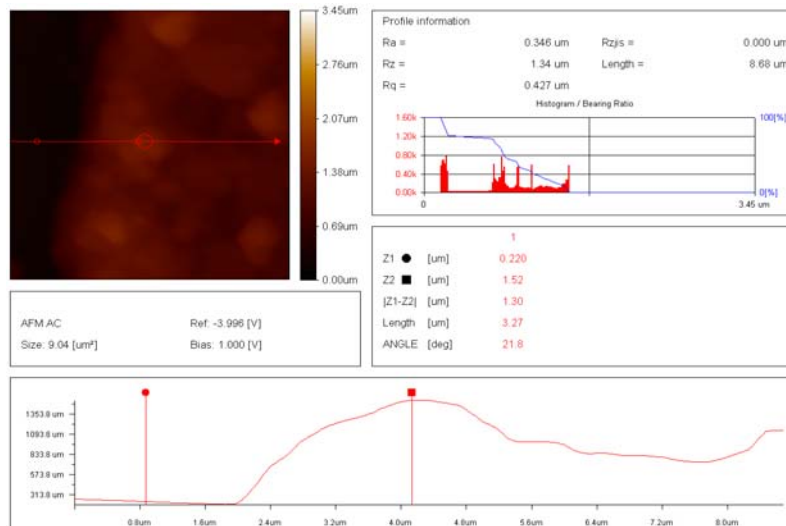


Figure 2.1. AFM analysis of plate with fullerene films in order to determine fullerene film thickness

After determining the thickness of fullerene, the plate was subjected to engraving. Engraving was performed on the Roland PNC-2300 CAMM engraving machine. Thanks to its engraving speed, high torque motors, enclosed working space, safety switch and improved mechanical precision, this machine is one of the more advanced engraving systems.

We decided to use a tool with a diamond tip, because diamond has the best qualities that are required for a cutting tool [3]. This is a cutting tool type SCD (Single crystal diamond), based on monocrystalline diamond. Monocrystalline diamond provides a wide range of extreme features, including hardness, friction coefficient and high thermal conductivity. Diamond has a high elasticity modulus, 1000 GPa, compared with e.g. steel with elasticity modulus of 206 GPa [3]. A consequence of the large elastic modulus is high specific strength, which enables a tool to withstand high mechanical forces, resulting in more accurate processing.

Pictures of a tool tip and engraved grooves were recorded with an optical system consisting of Canon SD1000 digital camera, with 7.1 MP resolution that was placed on KEYENCE VHX-100 magnifying system which gives 1000 times more magnification. Chip that has emerged as the wear product

was examined with the spectrometer Spotlight 400 FTIR Imaging System. This system is based on FTIR spectroscopy. Fourier transform infrared spectroscopy (FTIR) is a technique used to obtain an infrared spectrum of absorption of various liquid, solid or gas samples. FTIR spectrometer simultaneously collects spectral data in a wide spectral range. The Spectrum Spotlight Imaging System allows collection of images from extremely small samples. The mirror system has a wide collection angle (high numerical aperture) and is highly efficient in collecting infrared radiation for microspectroscopy. The Imager includes a camera and a viewing system that magnifies the visible-light image of the sample so that you can see, position, and isolate a point of interest.

## 2.1. Fullerene features

The term fullerene encompasses a whole family of carbon clusters molecules consisting of 12 pentagons and a variable number of hexagons. This family can be represented by a general formula  $C_{2n+20}$ , where  $n = 0, 2, 4, 6, \dots$  is a number of hexagons [1,2]. The most interesting molecule of the family is fullerene C<sub>60</sub>, which consists of 60 carbon atoms arranged in a cage made up of 12 pentagons and 20

hexagons. These materials have opened a new chapter in the science of materials and thus made room for further research, especially in the field of biomedicine. What makes them attractive are their characteristics based on physical chemistry, such as phase transformations, photoconductivity, ferromagnetism, superconductivity and electrical doped forms of alkaline metals or doped films of carbon C<sub>60</sub> and C<sub>70</sub> at room temperature [1].

In this molecule, there are two typical bonds between the carbon atoms. One type of the bond that appears is a single bond length of 0.146 nm, and it occurs within the pentagon, while the other type of the bond, a double bond length of 0.139 nm, occurs between the pentagon and hexagon.

The problem with the fullerene is in very low solubility in most solvents and the pursuit of aggregation in solution. Solubility of fullerenes can be achieved by their functionalizing with polar groups -OH and -COOH, where the most tested are fullerols and fullerlenols with a couple of attached functional-OH groups.

## 2.2. Diamond tool

Diamond tools are increasingly used in micro-nano processing [4]. Tool wear is a very important factor that affects the surface quality and the economic picture of the entire treatment process [5]. Seen from the microscopic scale, tool wear is a result of the interaction of atoms of the material that has been processed and atoms from the cutting tools. It has been shown [4] that there is a very pronounced friction between these atoms.

Reference [4] states that the highest observed local temperature, located at the top of the diamond, is 813 K. This temperature was recorded at 1.43 nm

from the tool tip. Energy of sublimation of the carbon and silicon atoms decreases with increasing temperature. Since the energy of sublimation of carbon atoms decreases much faster than silicon atoms, a resulting weakening of C-C bond and therefore these connections are easier to tear.

The basic mechanism of diamond tool wear is thermo-chemical wear or tear caused by thermal and chemical effects. In terms of chemical wear, the atoms separation from tightly bound diamond lattice [6] is an important step. When leaving the lattice, the atom can diffuse into the material which is processed, it can also be carbonated by combining with other laid-off carbon atoms or react with oxygen and form CO or CO<sub>2</sub>. Also, a free atom may react with the atoms of the workpiece, thus forming carbides. Presence of unpaired d electrons in the workpiece will cause the tool wear [6]. These electrons allow disconnecting carbon-carbon relations in diamond, which is further followed by the formation of metal-carbon complex. Thus formed complex can lead to chemical wear of diamond tools. There is a fundamental relationship between the number of non-paired electrons, melting point and the crystal structure.

## 2.3. Processing modes

To determine the optimal treatment regime of the fullerene film with a diamond tipped tool, we varied three parameters, the auxiliary cutting speed *xy*, the speed of rotating the main movements *z*, and the tool rpm. Tables 1-3 show the processing modes that were tested. We engraved the total of 27 grooves so that each groove is engraved in a different mode.

Table 1. Round per minute: 500

Processing mode	1	2	3	4	5	6	7	8	9
<i>xy</i> [mm/s]	1	1	1	15	15	15	29	29	29
<i>z</i> [mm/s]	1	30	59	1	30	59	1	30	59

Table 2. Round per minute: 1000

Processing mode	10	11	12	13	14	15	16	17	18
<i>xy</i> [mm/s]	1	1	1	15	15	15	29	29	29
<i>z</i> [mm/s]	1	30	59	1	30	59	1	30	59

Table 3. Round per minute: 10000

Processing mode	19	20	21	22	23	24	25	26	27
<i>xy</i> [mm/s]	1	1	1	15	15	15	29	29	29
<i>z</i> [mm/s]	1	30	59	1	30	59	1	30	59

After engraving, 27 grooves were examined under KEYENCE VHX-100 magnifying system (Series Digital Microscope) to determine the optimal processing parameters of machining with regards to the quality of the grooves edge.

### 3. RESULTS

During the experiment we noticed radial tossing of the machine making it impossible to cut a straight line. This tossing represents a strong perturbing factor during micro machining. Also, it is important to notice that the tossing occurs no matter which combination of parameter values is used. We noticed that this tossing occurs only when the main movement of the tool is rotational, which leads to a conclusion: if we want to avoid radial tossing we must avoid rotational movement of the tool.

Figure 3.1 shows two grooves made with optimal cutting mode. Here we engraved grooves in glass, without a thin fullerene film. The left one was made with one pass, while the right one is made with three passes. This figure helped us discover radial tossing of the machine. Pictures of these grooves were taken with Spotlight 400 FTIR Imaging System. From this picture we can conclude that the tool is moving in the coil pattern, which is different than we expected. This movement may be caused by the

radial tossing of the machine. This groove was not taken in count for determining the values of optimal cutting mode because it did not involve the fullerene film.

After examining all 27 grooves, we concluded that the optimal values for cutting fullerene film with diamond tool was 10000 rpm, the optimal speeds were 30 mm/s for the main movement of the tool and 15 mm/s for additional movement of the tool. Figure 3.2 shows the groove engraved with optimal cutting mode in fullerene film deposited on glass.



Figure 3.1. Groove made with optimal regime (glass without fullerene)

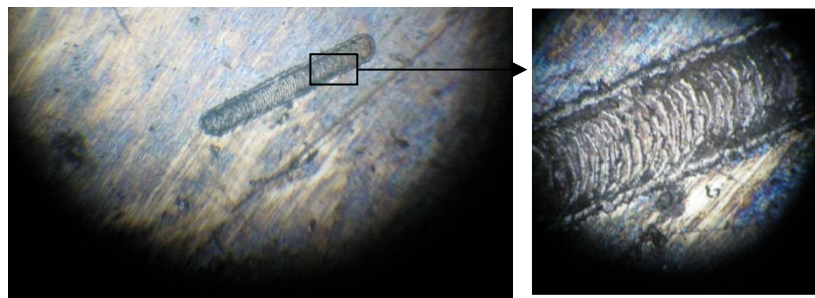


Figure 3.2. Groove made with optimal regime (glass with fullerene)

#### 3.2. Wear of diamond tool

Figure 3.3 shows a tip of the diamond tool ZDC-A2000 (Diamond scraper) which we used in our experiments. Figure 3.4 shows the same tool tip after completing engraving, and we can see that something led to wear of tool tip. This brings up a question, what caused this wear?

Temperature that occurs during machining, as well as the length of the grooves [7], were most certainly the factors causing the wear. The length of the grooves directly affects the time spent for machining. The longer the machining, the greater the chances for wear to appear because a long contact

between the tool and the workpiece leads to higher temperatures.

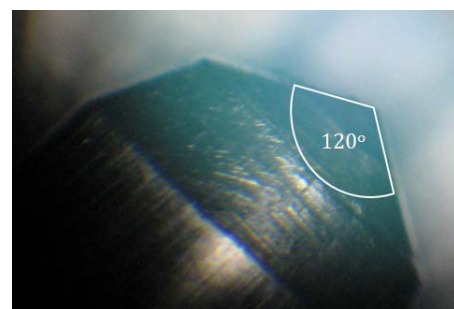


Figure 3.3. Tip of a diamond tool ZDC-A2000 before engraving



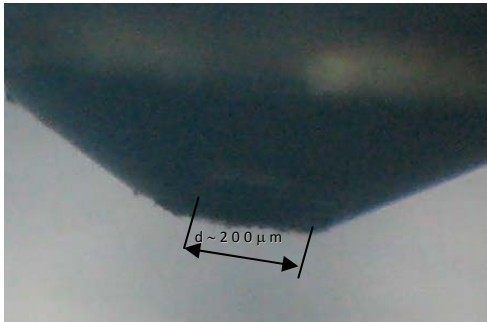


Figure 3.4. Tip of a diamond tool ZDC-A2000 after engraving

Paper [6] quotes that because silicon does not contain unpaired d electrons, silicon cannot be responsible for tool wear caused by the mechanism of forming electron complexes. However, silicon is

very reactive with carbon atoms at high temperatures and under high pressures [7]. Products of these reactions are silicon carbides. During machining of glass with diamond tool, paper [5] quotes that one can notice continuous chip and also plastic deformation in glass.

### 3.1. Spectral analysis of chip

We used spectral analysis to examine the chip that we noticed. Figure 3.5 shows this chip; the picture was taken by KEYENCE VHX-100 magnifying system (Series Digital Microscope).

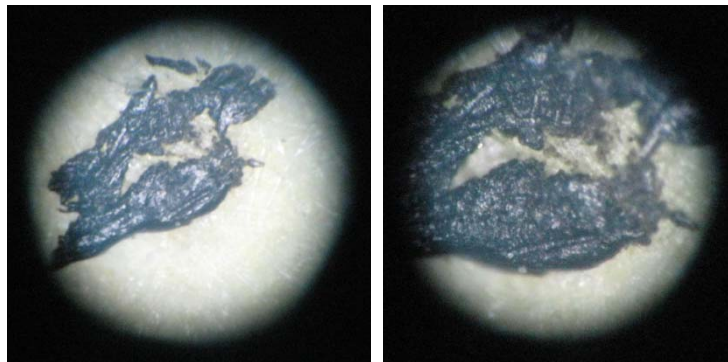


Figure 3.5. Chip, left - magnifying 700 x, right - magnifying 1000 x

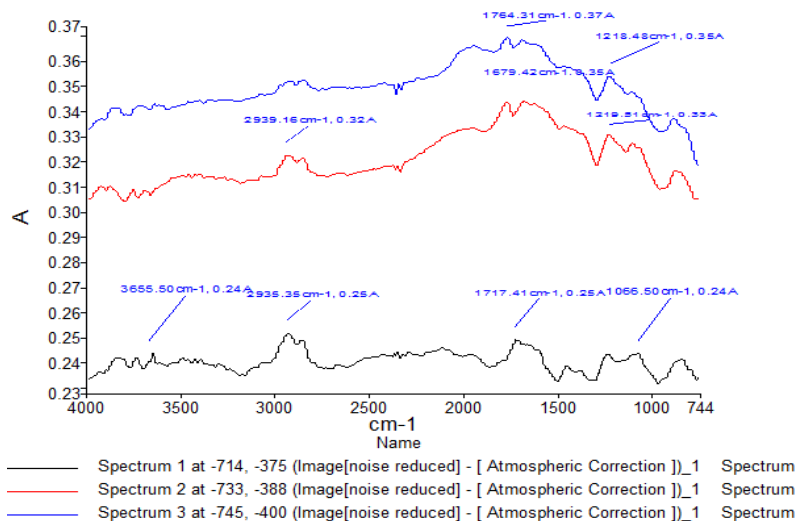


Figure 3.6. Chip spectrum

Figure 3.6 shows a spectrum of the examined chip. Characteristic peaks are marked on the figure. We conducted three measurements and they are shown in different colors. By examining the characteristic peaks we can determine composition, and then the origin of the chip. For the analysis database SDBS, Spectral Database for Organic Compounds were used. Based on [8–13] and the data from the

SDBS we could determine the bonds that occur at the same wavelengths as the peaks.

After spectral analysis we can conclude that our chip includes carbon, silicon and oxygen atoms. Bonds that occur between these atoms are shown in Table 4. The wavelengths and their characteristic bonds taken from [8–13] are also shown in the table.

Table 4. Bonds between atoms

Wavelength on which peaks occurs [cm <sup>-1</sup> ]	Characteristic bond	Wavelength on which characteristic bond occurs [cm <sup>-1</sup> ]
3655.5	OH OH C-OH Si-OH	3600-3100 3400 3300 3650
2935.35	C-H >CH- >CH <sub>2</sub>	2936 2900-2950 2935
1717.41	>C=O carboxyl and/or ester	1750-1700 1720
1066.5	OH >CO Si-O-Si	1250-1000 1150-1050 1065
2939.16	C-H >CH- >CH <sub>2</sub>	2936 2900-2950 2935
1679.42	C=O >C=O	1630 1650-1550
1219.51	OH	1250-100
1764.31	C=O >C=O anhydrides	1740 1750-1700 1750-1790
1218.48	OH	1250-100

#### 4. CONCLUSION

Because fullerene is a new material, machinability of this material is still unexplored, which makes it a good candidate for further investigation. The wear of the diamond tool is also under-examined [5]. This has to do with a small number of published papers on quantitative data. In this paper we investigated machinability of thin fullerene film deposited on glass with diamond cutting tool on engraving machine and showed that no matter which cutting mode is chosen the wear always occurs. This wear is not negligible. Wear debris consists of combination of carbon, oxygen and silicon atoms. Some further investigation may be directed towards machinability of fullerene deposited on some other material than glass.

Long contacts between tool and workpiece should be avoided, because it may be a cause of radial tossing of the machine. This tossing directly affects the quality of engraved grooves, and there is a lot more room for improvements in the field of micro engraving that will lead to a better quality of micro engraving.

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#### АКТУЕЛНО ОДРЕЂИВАЊЕ ПАРАМЕТАРА ПРОЦЕСА ЗА МИКРОГРАВИРАЊЕ ФУЛЕРЕНСКОГ ФИЛМА

**Сажетак:** Машински инжењери се редовно сусрећу са захтјевима за обрадом савремених материјала коришћењем постојеће опреме. Одређивање параметара обраде, у том случају, постаје актуелан проблем. Овај рад даје одговор на један такав захтјев. У питању је обрада танког фулеренског филма нанесеног на стаклену плочицу методом хемијског напаравања. На компјутерски управљаној машини за гравирање, примјеном алата са дијамантским врхом спроведена је експериментална верификација могућности обрађивања фулеренског филма. Различите вриједности параметара обраде су комбиноване у циљу утврђивања адекватног скупа параметара са аспекта квалитета израде ивице жлијеба. Приликом обраде плочице примјеђено је интензивно хабање и наше истраживање се једним дијелом усмјерило и ка одређивању узрока хабања. Савремена опрема је коришћена за квалитативну анализу и грубу селекцију оптималних параметара и за анализу продуката хабања. Резултати су основа за даљу оптимизацију процеса микрообраде танког фулеренског филма и увођења препоручених вриједности параметара у већ постојећу табелу за добро познате материјале.

**Кључне ријечи:** фулеренски филм, параметри обраде, микрогравирање.

