

## NANOSTRUCTURED METAL-FULLERENE FIELD EMISSION CATHODE

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### ABSTRACT

We developed new type of metal-nanocarbon composite field emission cathodes by hot sintering of ball-milled nanostructured mixture of a copper with C<sub>60</sub> fullerene. A mixture of copper and C<sub>60</sub> was subjected to co-grinding of raw materials made in the form of about 1 μm size granules in a planetary mill. The purity of raw materials was not less than 99.5%. Using the grinding we reduced the size of the copper particles down to about 10-30 nm. The content of the fullerene compound in the initial mixture was 10% (by weight). TEM analysis showed 2 nm thick nanocarbon film covering 10-30 nm size copper nanocrystals. The metal provides a high electrical conductivity, high thermal conductivity and mechanical strength of the cathode and nanocarbon material provides its high electron emission properties. Further, the resulting material was subjected to hot pressing at a pressure of 6.5 GPa and temperatures of 850°C, 1 minute. The finally obtained material can be easily mechanically processed for shaping of a cathode for any particular design. To improve the efficiency of a field emission we used chemical and plasma etching of its working surface. The turn-on electrical field of the cathode is 10-15 V/μm. The Fowler-Nordheim plot of the emission current shows linear function, indicating electron field emission effect. The resulting cathode emission current density was about 20 mA/cm<sup>2</sup> with high stability and homogeneity. Such cathodes can be used as electron sources in various electronic devices - electron guns, X-ray tubes, the amplifier and generator devices in microwave electronics, light sources, etc.

**Key words:** nanocomposite, fullerene, hot sintering, field emission cathode.

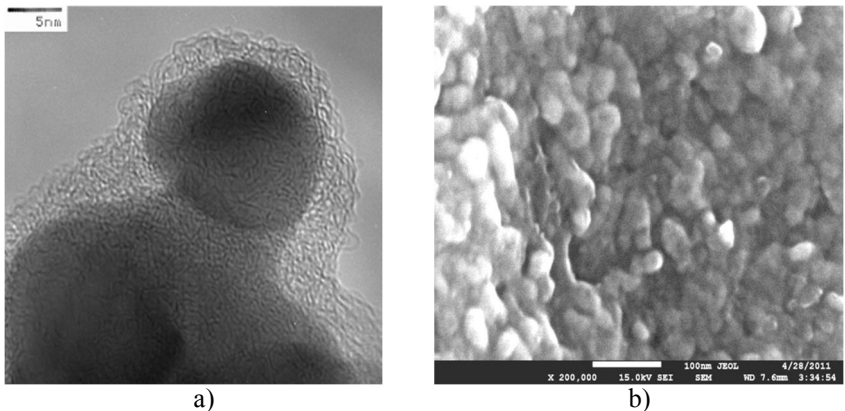
### INTRODUCTION

One of the important properties of carbon nanostructures is their cold electron emission ability. Carbon nanotubes and other nanostructures are capable of emitting high currents at relatively low electrical fields [1-4]. They are already used in functional devices such as field emitters. The conventional method of carbon nanostructured cathodes manufacturing is thin film nanocarbon deposition using CVD process on electrically conducting substrate like metal or doped silicon plates. The alternative way of manufacturing of carbon field emission cathodes is based on a special processing of carbon microfibers or composite

materials in metal holders [5]. We used the similar approach to produce composite metal-nanocarbon material which may be easily processed and shaped to produce an effective field emission cathode which can be easily fixed in any environment.

### **METHODS OF SAMPLE MANUFACTURING AND ANALYSIS**

We employed the method of hot sintering of mixed metal-nanocarbon powder to make nanocomposite field emission cathode. Copper was used as the metal compound due to its elastic and electrical properties and availability. The next (nanocarbon) component was  $C_{60}$  fullerene. A mixture of copper and  $C_{60}$  was subjected to co-grinding of raw materials made in the form of about  $1\ \mu\text{m}$  size granules in a planetary mill. The purity of raw materials was not less than 99.5%. The processing time was about 30 min. Using the grinding we reduced the size of the copper particles down to about 10-30 nm. The content of the fullerene compound in the initial mixture was 10% (by weight). The treatment was carried out in 99.999% argon atmosphere (the oxygen content of less than 0.1 ppm) in a glove box. After the processing in a planetary mill the material was studied by transmission electron microscope. The TEM images (*Fig. 1a*) showed that the copper nanoparticles are covered with  $\sim 2\ \text{nm}$  thick nanocarbon layer.



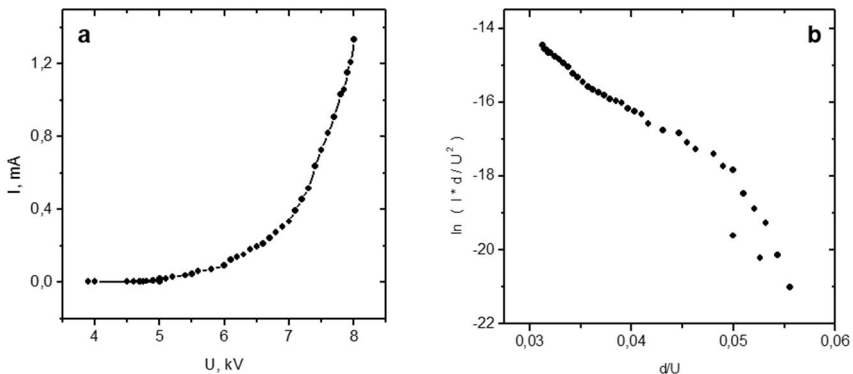
**Fig. 1** – a) TEM image of Cu nanoparticle covered by nanocarbon layer;  
b) SEM image of the surface of Cu- $C_{60}$  nanocomposite field emitter

Further, the resulting material was subjected to hot pressing at a pressure of 6.5 GPa and temperatures of  $850^\circ\text{C}$ , 1 minute. The result was a composite metal-nanocarbon material whose surface after additional treatment is highly effective field-emitter. Finally we obtained  $\varnothing 3 \times 2\ \text{mm}^3$  cylindrical samples as electron emitters. One of the plane faces (the working surface of the cathode)

was mechanically polished to roughness of about 50-20 microns. It was then followed by the chemical etching. We used an aqueous solution of the ferric chloride  $\text{FeCl}_3$  as it reacts with copper and completely inert with respect to carbon compound. The chemical treatment cleaned an activate surface of the nanostructured carbon content. An additional activation of the field emission properties of the cathode was performed by processing in a hydrogen plasma at a pressure of 35 Pa with a power density of  $0.5 \text{ kW/cm}^2$  and a substrate temperature  $150^\circ\text{C}$ . *Figure 1b* shows the SEM image of the surface of the obtained cathode.

### RESULTS AND DISCUSSION

*Figure 2a* shows the voltage-current characteristic of the cathode in a vacuum diode scheme with the cathode - anode distance of 300 microns. The value of the current  $I$  increases approximately exponentially with the increase of bias  $U$ . In these measurements the field emission cathode showed high stability. *Figure 2b* shows the same data in a Fowler-Nordheim coordinates. It has distinctly linear part at  $U > 15 \text{ V}/\mu\text{m}$  (the upper part of the plot in *Fig. 2b*). The field emission current density was up to  $20 \text{ mA/cm}^2$ .



**Fig. 2** a) - A voltage-current characteristic of  $\text{Cu-C}_{60}$  nanocomposite field emitter at 300  $\mu\text{m}$  anode-cathode distance; b) - the Fowler-Nordheim plot of a voltage-current characteristic of  $\text{Cu-C}_{60}$  nanocomposite field emitter.

When using a composite with a different content of nanocarbon components results deteriorated. For example, by decreasing the content of  $\text{C}_{60}$  to 5% by weight the maximum field emission current reduced by about 20%, while increasing to 20% significantly decreased the mechanical strength and the machining of the material becomes impossible.

### **CONCLUSIONS**

We developed new type of metal-nanocarbon composite field emission cathodes by hot sintering of ball-milled nanostructured mixture of a copper with C<sub>60</sub> fullerene. The metal provides a high electrical conductivity, high thermal conductivity and mechanical strength of the cathode and nanocarbon material provides its high electron emission properties. Such cathodes can be used as electron sources in various electronic devices - electron guns, X-ray tubes, the amplifier and generator devices in microwave electronics, light sources, etc. The material can be easily mechanically processed for shaping of a cathode for any particular design. To improve the efficiency of a field emission we used chemical and plasma etching of its working surface. The resulting cathode emission current density was about 20 mA/cm<sup>2</sup> with high stability and homogeneity.

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