Applied Surface Science 306 (2014) 94–97

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

The influence of ion bombardment on emission properties of carbon materials

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ARTICLE INFO

Article history:
Received 15 November 2013
Received in revised form 28 March 2014
Accepted 30 March 2014
Available online 6 April 2014

Keywords:
Carbon
Field emission
Ion bombardment
XPS
Surface modification
Emission properties recovery

ABSTRACT

When electric-vacuum device works its cathode surface experiences bombardment with ions of residual gases. Effects of ion bombardment impact on surface of field emission cathodes made of carbon materials may essentially change emission properties of such cathodes. It changes emission start electric field strength, voltage vs. current characteristic of material, its relief and electron structure of the surface layer. Field emission cathode operating mode, variation of radiation doses allow to obtain both good effects: maximal electric current, surface recovery – and negative ones: the worst emission properties and surface destruction, amorphization.

1. Introduction

Every vacuum device works in residual gases atmosphere. Gases molecules can be ionized with electron impact during exploitation. Ions interaction with electrodes results in cathode material sputtering and other electrodes pollution. This leads to device characteristics derating and deterioration (for example, decrease of X-ray spectrum purity in X-ray tube), cathode destruction and degradation etc. Thus manufacturers try to create ultrahigh vacuum to avoid device damage and extend its lifetime. The applying of field emission cathodes in electric vacuum devices allows reducing of requirements for the vacuum level. The researchers from the laboratory of pulsed radiation sources of the Institute of Electrophysics UD RAS develop X-ray tube with the field emission cathode made of carbon material. Experiments on work of field emission cathodes at the technical vacuum conditions are carried out. Studied samples are massive specimens, but during the experiments ion bombardment results in nanoclusters formation on the surface. And these new nanostructures make contribution to improvement of the samples emission properties.

2. Materials and methods

Researchers studied various carbon specimens: from pure carbon up to commercial graphite. Carbon materials such as highly oriented pyrolytic graphite (HOPG), coke and pitch compositions with different synthesis temperature (B-1000, B-1300, B-1500, B-1700, B-1900, B-2100, B-2700) and, finally, commercial carbon materials (GS-1800, ARV, GMZ, GE, MG, MPG-7) are considered.

HOPG refers to form of high-purity pyrolytic graphite with an angular spread between the graphite sheets of less than 1°. This highest-quality synthetic form is used in scientific research, in particular, as a standard for scanner calibration of scanning probe microscopes [1]. Chemically it is pure carbon.

Coke and pitch compositions are synthesized from coke and pitch materials. Then it is annealed at the high temperature what is reflected in specimen name. These forms of carbon are used as bonding medium for structural materials producing.

Commercial carbons are graphites for metallurgical industry: electrodes, special materials etc.:

– *GS-1800*: fine grain graphite with high density obtained under high pressure and temperature conditions by means of isostatic pressing; characterized with low gas permeability, porosity, homogeneity and high strength;

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http://dx.doi.org/10.1016/j.apsusc.2014.03.188
0169-4332/

- *ARV*: fine-grained material obtained with calcined pitch-based coke, natural graphite, coal tar pitch; characterized by a high mechanical strength and low ash;
- *GMZ*: low-ash graphite with medium grain size;
- *GE*: electrode graphite, is made of petroleum coke and coal tar pitch; physical and mechanical properties of the material are similar to graphite electrodes; designed for the manufacture of molded products;
- *MG*: fine-grained material made of calcined pitch coke and natural graphite;
- *MPG-7*: high-strength and heat-resistant materials of coke-pitch compositions.

Experiments are carried out using installation described in source [2]. Samples set in experimental cell. Strong electric field is generated between electrodes by means of high voltage supply. Measuring circuits allow detecting values of emission current and anode voltage. Vacuum system has inlet valve what gives opportunity to change pressure and gas composition of volume.

The first stage of our experiments is sample heating in vacuum. It is necessary to clean cathode surface by means of desorption. Then experiments on the current–voltage characteristics (CVC) acquisition follows. It provides primary information about material emission properties and facilitates specimen training. After obtaining of sustained cathode operation, stable CVC the sample work during the long time is studied. Different regimes of time dependant characteristics are explored: emission current and pressure ranges are 0.1–0.3 mA and 2×10^{-4} to 3×10^{-2} Pa consequently, injected gas – argon. The field emission properties depend on surface condition therefore samples electron structure of the surface layer is studied. The most appropriate methods for surface analysis are X-ray photoelectron and auger-electron spectroscopy (XPS and AES). Such data obtained with ES IFM-5 and PHI 5000 VersaProbe Scanning ESCA Microprobe (Physical Electronics) spectrometers. Spectra of active cathode side and flip one before and after ion bombardment are processed, compared and analyzed.

3. Results and discussion

3.1. Time dependant characteristics

Time-dependent characteristic helps to directly observe influence of ion bombardment effects on field emission properties of carbon materials. It shows parameters changes under specific conditions (emission current and gas pressure) during the long time. Two processes compete on the specimen surface when experimental cell works. They are emission centers destruction (because of current and ion impact) and new emission points formation as a result of ion radiation. Domination of one of processes in a certain mode results in degradation or self-recovery of the cathode. The recovery of emission properties of the sample surface is possible due to numerous factors: relief changes, electron structure modification, chemical transformations, etc.

Time dependent characteristics give 2 data arrays what describe current and voltage behavior. During experiments emission current is fixed and measuring system registers the data. It is comfortable to use cell resistance R value to interpret results. This resistance R is determined by the ratio of voltage to current: $R = U/I$. Cell resistance depends on cathode emission properties thus it can be used for field emission processes description. Fig. 1 shows several cases of field emission cathode made of carbon materials work under ion bombardment.

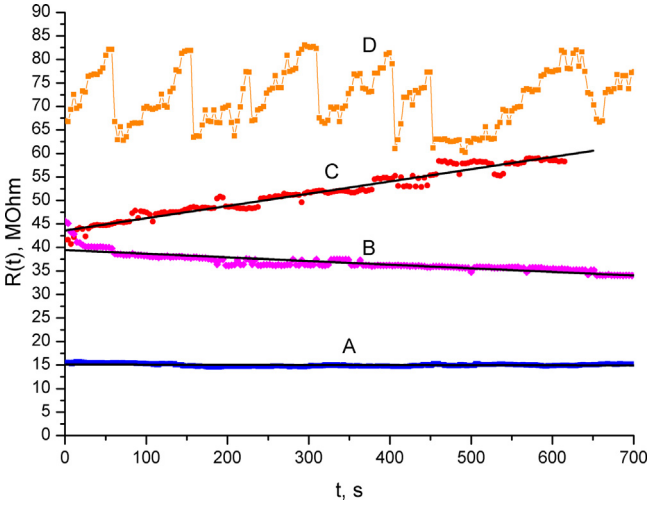

Fig. 1. Resistance versus time for experimental cell: (A) stable work of B-1700 at 0.3 mA current and at the pressure of 6.4×10^{-4} Pa (B-1700, 0.3 mA, 6.4×10^{-4} Pa); (B) resistance decrease mode for ARV (ARV, 0.1 mA, 3×10^{-4} Pa); (C) resistance increase regime for MG (MG, 0.1 mA, 9×10^{-4} Pa); (D) unstable characteristic of GMZ (GMZ, 0.1 mA, 7×10^{-3} Pa).

Fig. 1A shows stable operation mode of cathode made of B-1700 at the current 0.3 mA and pressure 6.4×10^{-4} Pa. Inner resistance of experimental cell fluctuates around 15 MΩ. Relatively stable work for our samples is obtained at small argon pressure and small emission current, i.e. under insufficient fluence. It is this fact that can explain cathode stable operation. The specimen does not suffer neither from ion impact nor Joule heating.

At Fig. 1B the mode of resistance decrease for ARV sample (at 0.1 mA, 3×10^{-4} Pa) is considered. The effect of small current and low residual gases pressure level create favorable conditions for emission centers generation. Better cathode operation is connected with material properties, features and slight ion radiation treatment.

Fig. 1C demonstrates destruction, or degradation, of field emission cathode. It is made of MG graphite and works under the pressure of 9×10^{-4} Pa and at 0.1 mA emission current. The growth rate of the resistance for this situation is about 26 kΩ/s. Likely, increased argon pressure results in sufficient ion bombardment what destructs specimen surface. One of the possible processes, what is characteristic of such regime, is carbon surface layer amorphization.

Fig. 1D demonstrates unstable mode of the experimental cell operation. It was observed for GMZ sample at 7×10^{-3} Pa argon pressure and 0.1 mA emission current. Considerable ion bombardment corresponding to increased pressure results in sufficient carbon material properties modification during the experiments. We can see both cathode degradation effects (rise of $R(t)$-curve) and abrupt emission properties renovations. Perhaps, the specimen surface accumulates ineffective material layer what can be removed by following ion impacts.

These cases are characterized by the following parameters: sample, emission current and vacuum level (pressure). The specimen has its own properties, but emission electron current and pressure in volume affect ion bombardment intensity and change emission properties of samples. So Fig. 1 demonstrates different types of cathode properties behavior during the ion bombardment.

Obtained data demonstrate important advantages of massive carbon cathode over carbon nanotubes: it is able work steady and even recovery its properties under certain conditions. So it is the first step to creating of reliable, durable field emission cathode.