Nanocomposite of polycrystalline silicon and carbon nanotubes for micro- and nanomechanical systems

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Abstract. A thick proof mass is desired for inertial sensors obtained by surface micromachining that could improve its output characteristics. Alternative to thick layers additional layers are used. We consider nanocomposite of carbon and silicon as a material and high aspect ratio structures to increase layer thickness. Carbon nanotube arrays were grown and covered with silicon filling the space between the tubes by plasma enhanced chemical vapor deposition. The goal of the experimental research is to qualitatively establish the role of parameters of carbon nanotube arrays on the composition and phase distribution of carbon, silicon and voids. The nanocomposite has been obtained. The formation conditions of carbon nanotube arrays with intertube distance from 50 to 200 nm are determined. Silicon deposition parameters to ensure filling of the intertubular space, and to obtain nanocomposite layers with an acceptable density of voids at the base have been established. The estimation of geometric parameters of the nanocomposite was carried out.

1. Introduction
Micro- and nanoelectromechanical systems (MEMS) are the rapidly developing scientific and industrial field [1]. New products are being developed, manufactured and used in many commercial electronic applications [2]. The output characteristics of inertial sensors can be further improved by increasing of proof mass [3]. The latter is achieved by depositing thicker layer or locally depositing of additional layer [4]. In particular, increasing the thickness of the structural layer in capacitive sensors improves sensitivity by raising the total capacitance of the electromechanical converter. It allows to reduce the overall dimensions, which are in demand for navigation applications. Specified approach is limited by the material strength characteristics and the features and rules of industrial processes.

The basic and the main structural material of the devices fabricated by surface micromachining is polycrystalline silicon or polysilicon [5]. From our point of view, it is interesting to use nanocomposite of silicon and carbon nanotubes (CNTs) as part of the structural layer or as additional layer. At present, CNTs are becoming increasingly popular, including in MEMS [6-9]. However, there are limited numbers of papers devoted to carbon nanotubes-silicon nanocomposite using [10-12].

An accurately control the composition and uniformity over the entire surface of the substrate is important and possible by using plasma enhanced chemical vapor deposition (PECVD) from the gas phase during the formation of carbon and then silicon components [13-14]. The nanocomposite of polycrystalline silicon and CNTs can be an alternative to the epitaxial layer of polycrystalline silicon used by THELMA© (Thick Epipoly Layer for Microactuators and Accelerometers) process [15].
This paper is devoted to study conditions for obtaining of polycrystalline silicon and CNTs nanocomposite, and role of the carbon nanotube array parameters on the filling of the intertubular space, composition and phase distribution of carbon, silicon and voids.

2. Experimental details
Carbon nanotubes were grown by plasma enhanced chemical vapor deposition at 675°C in a flow of ammonia and acetylene in the presence of 10 nm thick nickel catalyst film. CNTs with a diameter of 20-60 nm, a length up to 2 μm and a distance between the nanotubes in arrays from 50 to 200 nm have been obtained. Polycrystalline silicon was deposited on CNTs (Oxford Instruments, PlasmaLab 100 PECVD) at 650°C, flow rate of 500 sccm (Ar:SiH₄) and 10 W. Deposition conditions were selected to obtain low deposition rate and stresses [14].

The study of the initial CNTs, silicon thickness and the obtained nanocomposite CNT-silicon was carried out by scanning electron microscopy (SEM) and local patterning by focused ion beams (FEI, Nova NanoLab 600). Silicon film thicknesses measured on the satellite CNT-free sample were 0.25 μm and 0.91 μm for the first (figure 1) and second (figure 2) depositions, respectively.

3. Results and discussion
The structures of the CNT arrays formed prior to silicon deposition are shown in figure 1a and figure 2a,b. When silicon was deposited on a dense array of nanotubes with insufficient distance between each other and/or the case of non-zero slope of the tubes relative to surface normal, the film was fused and a large number of pores were formed at the bottom (figure 1b). This can lead to a small thickness and poor adhesion of the nanocomposite, which are insufficient for inertial application. The deposition on the tubes with a considerable spread of height led to the formation of a more roughly surface and uncompleted film. So, it could be noticed that to form a qualitative nanocomposite special techniques should be used that allow the growing of carbon nanotube arrays with given geometric parameters. The main parameters are height, slope and distance between nanotubes. However, this is a separate technological challenge.

Considering simultaneous formation of a nanocomposite on samples with different CNT arrays (figure 2), it becomes evident that the geometric parameters of the CNT have a decisive influence on the degree of filling of the intertubular space (figure 2). Increasing the distance between the tubes, its uniformity and slope (to normal) leads to obtain thicker nanocomposite with a smaller number of pores at the bottom and a lower roughness (figure 2d).

![Figure 1. SEM images of (a) CNTs and (b) the nanocomposite](image_url)
However, the obtained results do not allow one to draw a conclusion about the increase in the thickness. Estimates show that for the formation of a dense layer of a nanocomposite with a thickness of at least 10 μm, such polycrystalline silicon layer (about 1 μm thick) should be deposited on tubes not less than 8-9 μm in height. This would increase the thickness by almost 10 times. The number of unfilled areas or pores can be minimized by placing nanotubes at a distance not less than 0.5-0.8 μm. As the distance between tubes decreases, the requirements for their normality become tougher.

In view of the aforesaid obtained nanocomposite can be optimized and used in the fabrication processes of micro- and nanomechanical inertial sensors [16, 17]. In the case of surface micromachining, the nanocomposite layer has to be formed on a structural layer of polycrystalline silicon or locally in the required places.

4. Conclusion
Silicon deposition parameters to ensure filling of the intertubular space, and to obtain nanocomposite layers with an acceptable density of voids at the base have been established. The estimation of geometric parameters of the nanocomposite was carried out. The influence of the distance between nanotubes is revealed. It has been noted that as the distance between nanotubes decreases, the requirements for their slope become more rigid.

Figure 2. SEM images of (a,b) CNTs and (c,d) nanocomposites
Analysis of the results showed that the obtained nanocomposite is suitable for inertial applications. It using will increase proof mass, and the local thickness of critical elements. Optimization and mechanical parameters of the obtained nanocomposites will be investigated further. The described approach will allow polysilicon sensors to achieve the parameters of bulk micromachined devices.

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