New system for manipulation of nanoobjects based on composite Ti$_2$NiCu/Pt nanotweezers with shape memory effect

A M Zhikharev$^1$, A V Irzhak$^{2,3}$, M Y Beresin$^{1,3}$, P V Lega$^1$, V V Koledov$^1$, N N Kasyanov$^{1,3}$ and G S Martynov$^{1,3}$

$^1$ Laboratory magnetic phenomena in microelectronics, Kotelnikov Institute of Radioengineering and Electronics RAS, Moscow 125009, Russia.
$^2$ X-Ray acoustooptics laboratory, Institute of Microelectronics Technology and High Purity Materials RAS, Moscow Region, Noginsk, Chernogolovka 142432, Russia
$^3$ Department of the Material Science of Semiconductors and Dielectrics, National University of Science and Technology “MISiS”, Moscow 119049, Russia

Abstract. We report the new system for manipulation of nanoobjects based on composite Ti$_2$NiCu/Pt nanotweezers with shape memory effect. The design consists of the bimetallic Ti$_2$NiCu/Pt shape memory nanotweezers placed on a tip of electrochemically etched tungsten needle. The semiconductor diode placed on the tip of the needle plays both role of resistive element of the heater and temperature sensor for feedback control loop closing. The device is compatible with existing positioning systems like OmniProbe®, Kleindiek®, etc. and may find numerous practical applications in various tasks of nanotechnology connected with 3D manipulation.

1. Introduction

Nowadays the manipulation of the individual nanoscale objects in the vacuum chamber of scanning electron microscope (SEM) using existing manipulators (probe-type like OmniProbe® or Kleindiek®) performs a difficult and exhausting task. Despite this, such manipulations are demanded in different fields of experimental physics, biology, medicine, and nanoelectronics. The application of tweezers-type devices can make this work easier. Sizes of modern actuators are fundamentally limited by the corresponding physical effects. For example, relative deformation of piezoelectric actuators are about $10^{-4}$, thus the sizes of device are greater than displacement by several orders of magnitude [1]. Alternatively, materials with shape memory effect (SME) demonstrate the large deformation (up to 10%) and strong force at shape recovery. For example, Ti$_2$NiCu shape memory alloy (SMA) can generate actuation stress up to 500 MPa at temperatures of phase transition between 30 °C and 70 °C [2-5] and withstands more than 2000 heating-cooling cycles [6]. High durability, small size and, respectively, small energy consumption makes SMA nanotweezers one of the best candidates to use in vacuum chambers of scanning electron and ion microscopes. Magnetic shape memory materials are known, but their use is hampered by the need to use strong magnetic fields (~ 8-10 T), which may induce interference on SEM systems [7-9]. Previous design of SME nanotweezers, presented in [10, 11], has proved itself as a useful device for performing 3D manipulation. In particular, the nanotweezers were applied for submicron and nanosized sample preparation for studying electrical properties of quasi-one dimensional conductors with a charge density waves – TaS$_3$ whiskers. Figure 1 shows the process of capturing TaS$_3$ whisker of about 500 nm thick. As a result the samples were
obtained with ohmic contacts, representing a thin bridge of TaS₃ whisker connected to two separate golden contacts by platinum layer, obtained by ion stimulated chemical vapour deposition (CVD) [12]. An image of one of the prepared sample is represented in Figure 2. A significant disadvantage of such design of the nanotweezers control system was the need for laser installation inside the vacuum chamber of SEM for nanotweezers control. In this work we present the robust SME nanotweezers system easy compatible with the existing manipulation systems, applicable in SEMs and FIB devices. The aim of the present paper is to describe the results of modelling, design and tests of the new nanomanipulation system.

2. Design and implementation of the control system.

2.1. Technical implementation

The new system of thermally controlled nanotweezers made of bimetal composite Ti₂NiCu/Pt with SME produced by FIB-milling and CVD. Nanotweezers were connected to tungsten needle with a heating element disposed. The tungsten needles are obtained from a wire by the method of cathode etching in 5% solution of KOH at a constant current. Manufactured tips of the needles have radii of curvature about 0.3÷1 µm. Typical size and form of the needles tip shown in the Figure 3. The SME nanotweezers attached to the end of the tungsten needle by the layers of CVD platinum as shown at Figure 4. The schematic representation of the installed in the SEM vacuum chamber system shown at Figure 5. The semiconductor diode mounted near the top of needle by thermally conductive glue. It plays both roles of resistive heating element and temperature sensor for feedback control loop closing. Brass cone used for mounting contact pads, is required for connecting of thin and fragile golden wires from semiconductor diode with the stout wires running to DAC-ADC module of control system, connected to a PC. To facilitate installation in vacuum chamber of SEM, tungsten needle were attached to a collet. The device is compatible with existing positioning systems like Zyvex®, Kleindiek®, etc. and may find applications in various tasks of nanotechnology connected with 3D manipulation of different kinds of micro- and nanoobjects. Automatically controlled heating system requires minimum modification to scanning electron microscope to be installed and can maintain the temperature of working body near martensitic-austenitic phase transition threshold, thus increasing the response speed of device. The new control system makes the nanotweezers much more robust and technological compared to existing ones [13].
2.2. Math modeling

As shown in [14], the nanotweezers on the tip of the tungsten needle can be treated as negligibly small. The heating/sensing diode is placed near the tip of the needle (Figure 5). One can write on the basis of energy balance the heat conduction equation for temperature $T$, as function of coordinate $x$ and time $t$, as follows:

$$C_p f(x) \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left( \lambda \frac{\partial T}{\partial x} f(x) \right) + q(x)$$  \hspace{1cm} (1)

$$f(x) = \pi \left( \frac{R - r}{L} x + R \right)^2$$  \hspace{1cm} (2)

where $C$ – heat capacity, $\rho$ – density, $\lambda$ – heat conductivity of the tungsten needle. $R$ and $r$ correspond to cross-sectional radii of the tungsten wire and needle tip, respectively. $L$ – distance between the diode centre and the tip of the needle, the coordinate of centre of the diode is set equal to zero. $f(x)$ refers to the tapered needle cross-section as a function of coordinate $x$, while $q(x)$ correspond to heating power density, produced by the diode. The boundary and initial conditions are defined, respectively, as:

$$\left. \frac{\partial T}{\partial x} \right|_{x=L} = 0, \quad T|_{x=0} = T_0$$  \hspace{1cm} (3)
\[ T_{t=0} = T_0 \]  \hspace{1cm} (4)

Using modeling software COMSOL Multiphysics® the analysis was done according to the described above mathematical model. Comparison of the results of math modeling and experimentally obtained data is shown in the Figure 6. As it can be seen the theoretical curve has good correlation with the experimental one. The successful modeling makes it possible to further carry out an analysis of an optimal structure of the installation setup, allowing to achieve a maximum operating speed of the SMA nanotweezers and minimal energy consumption.

3. Application of the SME nanotweezers.
To test the possibility of using nanotweezers with new control system in the SEM vacuum chamber and evaluation of the dislocation of its position, resulting from thermal expansion, a number of experiments on the manipulation of ZnO whiskers of different sizes have been carried out. For this purposes nanomanipulation system was installed instead of the Kleindiek® manipulator’s probe and was connected to its electrical contacts. The experiments have demonstrated the ability to perform basic manipulation tasks such as capture, secure retention and movement of individual samples extracted from the forest of ZnO nanowhiskers. Key stages of the experiments manipulation of micro- and nanoscale structures are shown in Figures 7 and 8, respectively.

4. Summary
We report the new system for nanomanipulation based on layered Ti₂NiCu/Pt composite nanotweezers with SME, controlled automatically by resistive heating element. The system has demonstrated its compatibility with existing nanopositioning systems such as Kliendiek™ and allows to perform its installation avoiding SEM modifications. Proposed theoretical model of nanotweezers results are in good correlation with the experimentally obtained data. Actual manipulation of micro- and nanoscale objects is demonstrated.
Acknowledgments
This work was supported by the Russian Science Foundation Grant No. 14-19-01644.

References
[1] Dikan V A, Mashirov A V, Zakharov D I, Mazaev P V, Zhikharev A M, Kalashnikov V S, Koledov V V, von Gratowsky S V, Sitnikov N N, Irzhak A V et al. 2016 J. Commun. Technol. Electron. 61(3) 302-10
[2] Irzhak A V, Tabuchkova N Y, Sagalova T B, Shelyakov A V and Koledov V V 2014 Bull. Russ. Acad. Sci., Phys. 78(12) 1379-81
[3] Irzhak A V, Zakharov D I, Kalashnikov V S, Koledov V V, Kuchin D S, Lebedev G A, Lega P V, Perov E P, Pikhtin N A, Pushin V G et al. 2010 J. Commun. Technol. Electron. 55(7) 818-830
[4] Lee A P, Ciarlo D R, Krulevitch P A, Lehew S, Trevino J and Northrup A 1996 Sens. Actuators, A 54 755
[5] Irzhak A V, Kalashnikov V S, Koledov V V, Kuchin D S, Lebedev G A, Lega P V, Pikhtin N A, Tarasov I S, Shavrov V G and Shelyakov A V 2010. Tech. Phys. L. 36(4) 329-32
[6] Krulevitch P, Lee A P, Ramsey P B, Trevino J C, Hamilton J and Northrup A 1996 J. Microelectromech. Syst. 5(4) 277
[7] Irzhak A, Koledov V, Zakharov D, Lebedev G, Mashirov A, Afonina V, Akatyeva K, Kalashnikov V, Sitnikov N, Tabuchkova et al. 2014 J. Alloy Compd. 586 (Suppl. 1) S464-8
[8] Kalimullina E, Kamantsev A, Koledov V, Shavrov V, Nizhankovskii V, Irzhak A, Albertini F, Fabbrici S, Ranzieri P and Ari-Gur P 2014 P. Status Solidi C 11(S5-6) 1023-25

Figure 7. Manipulating with micro-sized ZnO whisker. (a) Nanotweezers in cold state. (b) Tweezers in hot state.

Figure 8. Manipulating with nano-sized ZnO whisker. (a) Nanotweezers in cold state. (b) Tweezers with the part of ZnO whisker in arms.
[9] Akatyeva K, Afonina V, Albertini F, Von Gratowski S, Irzhak A, Fabbrici S, Khovaylo V, Koledov V, Krasnoperov E and Shavrov V 2012 Sol. St. Phen. 190 295-8
[10] Zakharov D, Lebedev G, Koledov et al. 2010 Phys. Procedia 10 58-64
[11] Zakharov D, Lebedev G, Irzhak A, Afonina V, Mashirov A, Kalashnikov V, Koledov V, Shelyakov A, Podgorny D, Tabachkova N and Shavrov V 2012 VTT Symp. 21(5) 052001
[12] Zhikharev A, Zybtsev S, Pokrovskii V, Irzhak A et al. 2013 3M-NANO 2013. Conf. Proc. 2013 (Suzhou) (Piscataway, NJ : IEEE) 196
[13] Zhao H, Chang M, Liu X, Gabayno J L and Chen H T 2014 J. Micromech. Microeng. 24 095012
[14] Lega P V, Koledov V V, Kuchin D S, Mazaev P V, Zhikharev A M, Mashirov A V, Kalashnikov V S, Zybtsev S A, Pokrovskii V Y, Shavrov V G et al. 2015 J. Commun. Technol. Electron. 60(10) 1124-33