

Ultrahigh vacuum holder-positioner for in situ studies of conductive nanostructures in a wide temperature range

D V Shevtsov^{1,2}, S A Lyaschenko^{1,2}, S N Varnakov^{1,2}

¹ Reshetnev Siberian State Aerospace University, Krasnoyarsk, 660037, Russia

² Kirensky Institute of Physics, Federal Research Center KSC SB RAS, Krasnoyarsk, 660036, Russia

E-mail: snowman-82@mail.ru

Abstract. A holder-positioner was developed in order to be used in high vacuum systems designed for synthesis and in situ samples investigation by optical methods in 77÷1171 K temperature range. During tests of holder-positioner the designed system demonstrated independence from other process chamber components, compactness, a large number of the sample degrees of freedom and the stability of maintaining temperatures in the range from 77 to 1171 K with a maximum sample cooling rate about 0.1 K/s.

1. Introduction

Preparation and research of new functional materials for the aerospace industry necessitates the creation of equipment to carry out controlled synthesis of such materials and in situ analysis of their physical properties. In particular, epitaxial ferromagnetic nanostructures on the monocrystalline silicon surface [1] are important materials for the development of the element base for aerospace appliances. In situ analysis methods have a great practical value for obtaining quantitative information about the structure and morphology of the samples [2, 3]. For non-contact examination of magnetic properties and electronic structure of the absorbing ferromagnets, the method based on the magneto-optical Kerr effect has already become conventional [4, 5].

Trends in the development of cryogenic and high-temperature equipment for optical studies reveal that the current challenges in this area is the implementation of temperature inserts, which allow us to study nanomaterials physical properties in a wide temperature range (2-1300 K) [6, 7]. Detailed analysis of the world's scientific and technical literature has not revealed information on systems combining in situ spectroscopic magneto-optic ellipsometry with a range of the sample operating temperatures 77-1200 K. However, the this analysis made it possible to establish the most common approaches for heating and cooling of semiconductor substrates, in a special medium or vacuum.

Technical devices and methods that combine heating up to 1300 K and cooling down to several K units, with the possibility of spectral measurements in the optical wavelength range, can be divided into two classes. The first one is universal systems of the sample displacement and attachment, including temperature control option in a wide range. Such systems are usually built into the ultra-high vacuum sputtering research systems. The second one is systems for conducting optical investigations in a wide temperature range [5, 8-12].

In this regard, the modernization of technological equipment, which permits us to carry out the in situ spectral magneto-optical ellipsometric measurements over a wide temperature range, is the current trend in the development of the means for the production and in situ investigation of ferromagnetic nanostructures.



2. Results and discussion

The analysis of the existing solutions revealed that the most efficient cooling system is a heat-conducting copper rod with the sample fixed on one side and having a refrigerant on the other side [13-14]. We created a sample holder design that showed high stability in maintaining a constant temperature in the range from 77 to 470 K. The specimen heating system based on the direct current passage was also added to the cooling system developed. A key element of the holder is a sapphire dielectric plate placed between the specimen and the copper base. Sapphire has a fast-falling dependence of the thermal conductivity coefficient [15, 16] under the temperature rising from 80 to 1300 K. That allows us to use sapphire as a heat insulator at high temperatures and efficient heat-removal at the boiling point of nitrogen. Figure 1 is a schematic representation of the holder-positioner created.

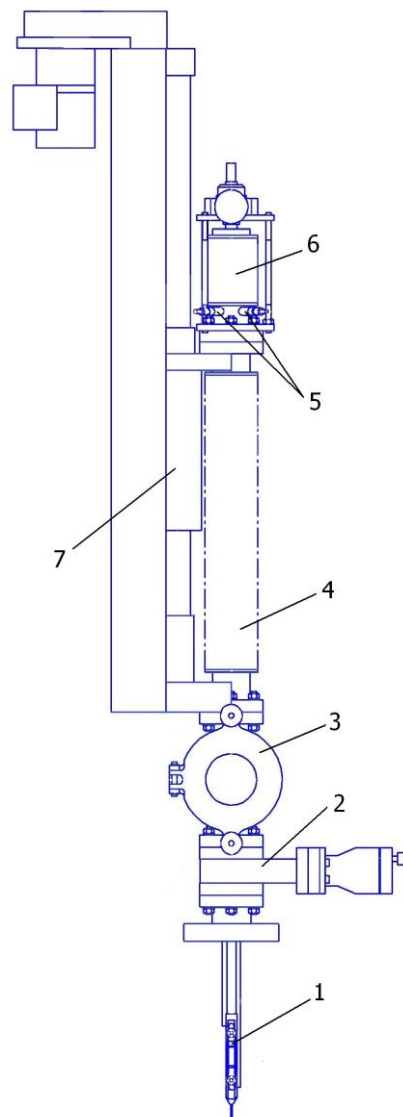


Figure 1. Schematic representation of the holder- positioner: 1) copper sample holder; 2) gate valve; 3) airlock; 4) bellows; 5) high vacuum current leads; 6) vacuum bellows inlet; 7) line translator

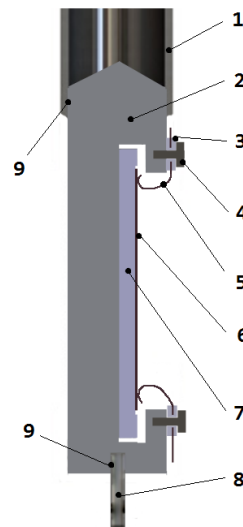


Figure 2. sample holder model profile with elements for annealing and cooling: 1 - stainless steel tube with refrigerant; 2 - solid copper sample holder; 3 – current leads electrical insulations; 4 - titanium fixing screws; 5 - tantalum leads in the form of hold-down clips; 6 - sample (silicon wafer); 7 - sapphire plate; 8 - steel latch; 9 - welded connections

The basis of holder-positioner is a vacuum-mechanical system (VMS). The VMS comprises the following elements: a vacuum shutter system, an airlock, the vacuum transport system, a nitrogen supply system, a sample heating system. The sample holder (Figure 2) is a coaxial design rod with a copper insert on which the specimen is mounted. The upper end of the rod is connected with the vacuum bellows inlet by means of tungsten-arc inert-gas (TIG). Catches mount with screws through the insulating sleeve on a copper insert. Catches provide a tight fixation of the specimen and serve as electrodes when the sample is heated by direct current passage.

Single-crystalline silicon substrate coated with a layer of SiO_2 was used for the holder-positioner temperature testing. Upon the sample holder nitrogen stream cooling temperature was being measured by a Pt100 resistance temperature detector (RTD). Figure 3 shows time dependences of the holder temperature and nitrogen pressure in the Dewar.

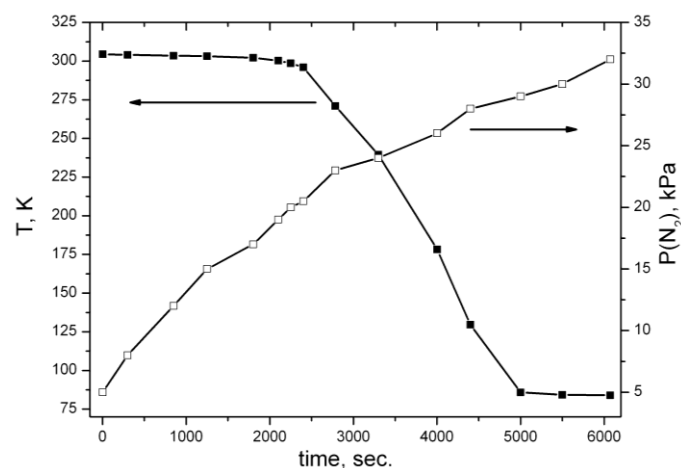


Figure 3. Time dependences of the holder temperature and nitrogen pressure in the Dewar

It can be clearly seen that the maximum sample cooling rate is approximately 0.1 K / s. After 2 hours of the cooling procedure the sample temperature has reached 84 K. To set an intermediate temperature in the range from 84 to 570 K a nitrogen flow heater is used. The flow heater power is smoothly given by a proportional-integral controller in the range of 0 to 1000 watts, which allows us to heat the nitrogen gas supplied to the sample holder from 76 to 800 K within a few minutes.

For the holder-positioner testing in the heating mode, voltage in current limiting mode was applied to the Si/SiO₂ sample. The sample surface temperature was measured by the infrared pyrometer IMPAC IGA 5 Advanced (LumaSense Technologies). The current increase through the sample was discretely carried out from 0.05 A up to 4 A in increments of 0.5 A. The maximum temperature of the sample was 1171 K. Figure 4 shows the electric current and sample temperature time dependences.

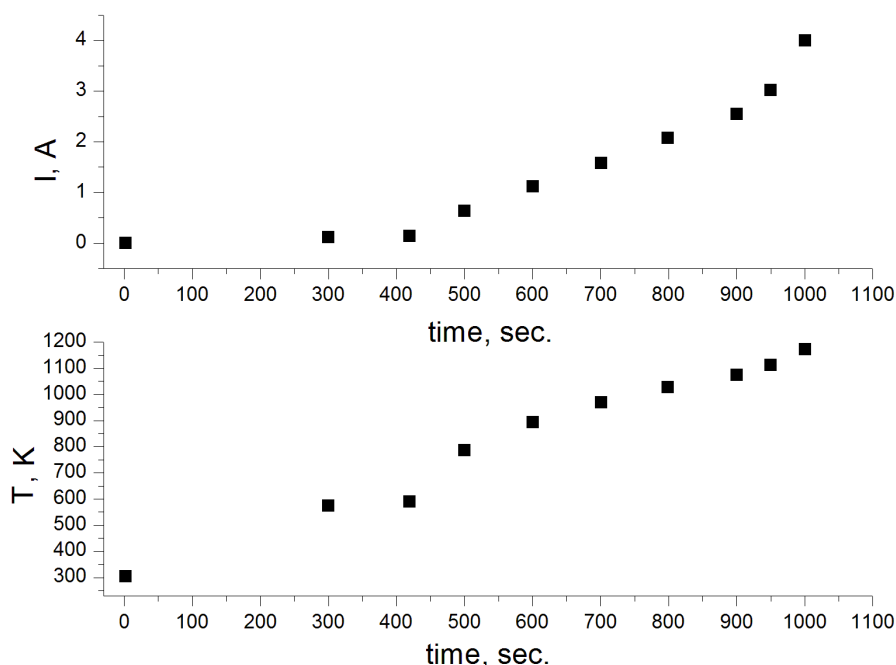


Figure 4. Time dependences of the electric current and sample temperature

As an example Figure 5 shows the results of the measurements of the ellipsometric parameter Ψ fulfilled on a silicon single crystal substrate with SiO₂ film under temperature changing in the range of 85-800 K. In situ ellipsometric measurements were made at a wavelength of 470 nm (2.64 eV) and with the polarizer and analyzer positions 45 °, 0 ° relative to the incidence plane, respectively [17]. To subtract the background illumination during ellipsometry measurements we were using a light beam mechanical chopper at a frequency of interruptions of 10-20 s⁻¹ [3].

The advantages of the holder-positioner construction are the ability to embed it in any vacuum system and two ways of the sample temperature setting: using the thermal contact with the heat-transfer and by the direct current passing.

The holder-positioner design is compact, the width of the copper holder with concomitant parts does not exceed 15 mm, which allows one to utilize standard ultra-high vacuum gate

valves and flanges. The sample loading system is integrated with a goniometer to ensure the required degree of freedom when setting up the optical part. Thus, it is possible to fine-tune the sample holder space position for carrying out in situ spectral ellipsometry measurements directly in the coating chamber. The manipulator is integrated with a cryostat-heater to achieve the desired temperature range.

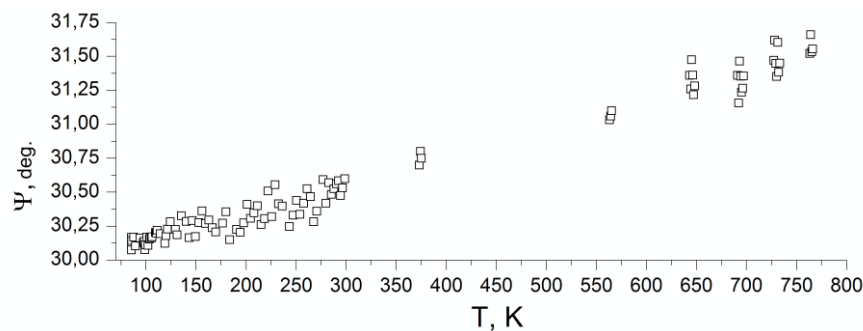


Figure 5. Temperature dependence of ellipsometric parameter Ψ for SiO₂/ Si sample

This feature distinguishes the system from the proposed analogues in which such functionally different parts are implemented as various units.

3. Conclusion

Taking into account peculiar properties of the holder-positioner design developed, one can conclude it is versatile. Independence from the other chamber components, the compactness and the large number of freedom degrees permit the use of the holder-positioner in experimental devices equipped with both optical and diffraction, electron spectroscopic surface analysis methods.

4. Acknowledgments

The work was supported by the Ministry of Education and Science of the Russian Federation (State task no.16.663.2014K), RFBR (Grant no.16-42-243058) and Program of the President of the Russian Federation for the support of leading scientific schools (Scientific School 7559.2016.2).

References

- [1] Fabian J, Matos-Abiague A, Ertler C, Stano P and Zutic I 2007 *Acta Phys. Slov.* **57** 565-907
- [2] Aspnes D E 1993 *Thin. Sol. Films* **233** 1-8
- [3] Lyaschenko S A, Varnakov S N, Tarasov I A, Shevtsov D V and Ovchinnikov S G 2012 *Vestnik SibGAU* (Krasnoyarsk) **4** (44) pp 162-167
- [4] Vonsovskij S V *Magnetizm* 1971 (Moscow: Nauka) p 1032
- [5] Rauer R, Neuber G, Kunze J, Bäckström J and Rübhausen M 2005 *Rev. Sci. Instrum.* **76** (2) 023910–023913
- [6] Norrman S H and Andersson T G 1977 *Vacuum* **27** 97
- [7] Belyaeva A I and Grebennik T G 1997 *Proc. SPIE, Optical Diagnostics of Materials and Devices for Opto-, Micro-, and Quantum Electronics* **3359** 401
- [8] Oxford Instruments. Nitrogen Cryostats 77 K. URL: <https://www.oxford->

- instruments.com/products/cryogenic-environments/optical-cryostats-for-spectroscopy/nitrogen-cryostats-77k (date of the application: 20.09.2016)
- [9] NIST Center for Neutron Research. Liquid Helium Cryostats. URL: <http://www.ncnr.nist.gov/equipment/Cryostat.html> (date of the application: 20.09.2016)
- [10] LTLab, Inc. The Cryogen-free measurement system: optCRYO105. URL: <http://www.ltlab.com/optcryo105.html> (date of the application: 20.09.2016)
- [11] Janis Research. Cryogenic Research Equipment. SuperTran Continuous Flow Cryostat Systems – Sample in Vacuum. URL: <http://www.janis.com/Products/productoverview/SuperTranContinuousFlowCryostatSystems.aspx> (date of the application: 20.09.2016)
- [12] Feng X H and Garfunkel E L 1989 *Vacuum* **39** 6
- [13] Hernandez L M and Goldman A M 2002 *Rev. Sci. Instrum.* **73** 162
- [14] Ekinici K L, Valles J M, Jr. 1997 *Rev. Sci. Instrum.* **68** 4152
- [15] Berman R 1951 *Proc. R. Soc. Lond. A.* **208** 90-108
- [16] Touloukian Y S and Ho C Y 1971 *Thermophysical Properties of Matter Volume 2: Thermal Conductivity: Nonmetallic* (NY: Macmillan) p 1172
- [17] Shvets V A, Spesivtsev E V and Rykhlytskii S V 2004 *Optics and Spectroscopy* **97** 483