Sensors and Actuators A 241 (2016) 87–95

Contents lists available at ScienceDirect

Sensors and Actuators A: Physical

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

Real-time SAXS study of a strain gauge based on a self-assembled gold nanoparticle monolayer

Karol Vegso[a,*], Matej Jergel[a], Peter Siffalovic[a], Mario Kotlar[a,b], Yuriy Halahovets[a], Martin Hodas[a], Marco Pelletta[a], Eva Majkova[a]

[a] Institute of Physics, Slovak Academy of Sciences, Dubravska cesta 9, 845 11 Bratislava, Slovakia
[b] STU Centre for Nanodiagnostics, Vazovova 5, 812 43 Bratislava, Slovakia

ARTICLE INFO

Article history:
Received 1 June 2015
Received in revised form 14 October 2015
Accepted 7 February 2016
Available online 9 February 2016

Keywords:
Flexible strain gauge
Gold nanoparticles
Nanoparticle monolayer
Langmuir film
Small-angle X-ray scattering

ABSTRACT

A strain gauge based on a monolayer of colloidal gold nanoparticles deposited on a flexible Mylar foil by a modified Langmuir-Schaefer method was tested *in situ* under external uniaxial stress by the small-angle X-ray scattering (SAXS) technique. Simultaneously, the stress-strain curve of the foil was measured. A high-flux laboratory X-ray source allowed fast data collection with 10 s temporal resolution. The monolayer exhibits a linear response in terms of the interparticle distances up to the 13% substrate strain while keeping its full integrity. This result indicates the dominant role of the pair potential function between the nanoparticle surfactant molecules and molecules of the substrate and compares well with previous quasi-static measurements of a similar strain gauge, suggesting none or negligible effect of the transient strain phenomena longer than the sampling interval on the gauge response. Keeping a constant strain on finishing the stretching, fast transient effects with characteristic times down to the limit imposed by the X-ray detector time resolution were not observed either during the SAXS pattern collection. A different stress behavior of the interparticle distance in the direction perpendicular to stretching comparing with a monolayer of colloidal iron-oxide nanoparticles studied previously reveals the surfactant effect on the gauge response controlled by the interparticle pair potential function. The results obtained suggest that the colloidal gold-nanoparticle monolayer on a flexible substrate is a prospective strain gauge with a very fast linear response in a broad strain range.

© 2016 Elsevier B.V. All rights reserved.

1. Introduction

Colloidal metallic and metal-oxide nanoparticles are characterized by the self-assembly effects that result in diverse two- and three-dimensional nanoparticle structures. In the last decade, great progress in the nanoparticle fabrication methods in terms of a low nanoparticle size dispersion has been achieved with positive impact on structural perfection of the nanoparticle assemblies and precise tuning of the interparticle distances [1–3]. Electrical resistivity of the assemblies depends on the nanoparticle size distribution, nanoparticle shape and interparticle distance. The electron transport is realized via electron tunneling through organic surfactant shells of the adjacent nanoparticles [4–8]. Therefore, the electron tunneling is strongly affected by the nature of organic molecules capping the nanoparticles [5]. The electrical resistivity controlled by tunneling is exponentially dependent on the interparticle distance, hence, it is extremely sensitive to any external stress as demonstrated by Mueggenburg et al. [9]. Indeed, colloidal nanoparticles provide promising alternative to conventional resistive strain gauges based on metallic thin films and semiconductor piezoelectric resistive strain gauges [10,11]. It has been shown that a gauge factor (the ratio between the relative resistivity change and the applied stress) of the nanoparticle strain gauges can outperform that of the conventional ones by up to two orders of magnitude [4,6,12–14]. An additional alternative to the strain gauge made of colloidal nanoparticles represents a lithographically patterned metal grating or mesh on the flexible polymer foil [15]. The advantage of lithography is its high accuracy and reproducibility. The narrow distribution of the widths of metal strips in the lithographically fabricated grating makes lithography extremely attractive for wide-scale commercial production of highly-sensitive resistivity gauges based on quantum tunneling.

Majority of papers on the nanoparticle resistive strain gauges have been devoted to electrical response while the underlying changes in the nanoparticle short-range order and interparticle distances have been rarely addressed. We have shown in our previous study at a synchrotron beamline that the small-angle X-ray scattering (SAXS) is a relevant technique for probing the stress-induced

* Corresponding author.
E-mail address: karol.vegso@savba.sk (K. Vegso).

http://dx.doi.org/10.1016/j.sna.2016.02.021
0924-4247/© 2016 Elsevier B.V. All rights reserved.

sub-nanometer structural changes in the nanoparticle assembly [16]. A comprehensive SAXS synchrotron study on the electromechanical behavior of the nanoparticle resistive strain gauges has been published recently [17]. In both studies, a linear shift of the nanoparticles beyond a 10% strain of the substrate has been observed. This demonstrates another advantage of the nanoparticle strain gauges as it is known that 10% strain produces cracks and discontinuities in the metallic thin film strain gauges [18].

The previous SAXS studies of the nanoparticle strain gauges were performed in a quasi-static regime, *i.e.* under equilibrium conditions. Namely, the SAXS patterns were collected at particular substrate strains for a rather long time to collect maximum intensity that was limited by radiation damage of the sample due to the intense synchrotron beam (tens or hundreds of seconds for each pattern). Here, we report on an *in-situ* laboratory SAXS study of a self-assembled monolayer of gold nanoparticles deposited on a flexible Mylar foil that was exposed to uniaxial stress. The major advantage of the *in-situ* approach is the exclusion of relaxation effects occurring during the quasi-static regime that could slightly affect the interparticle distance measurement. Application of a unique high-flux X-ray source enabled us a continuous tracking of the immediate SAXS response during the foil stretching with the temporal resolution given by the SAXS sampling interval. The SAXS results obtained in such a dynamic regime are directly comparable to the simultaneously measured stress-strain curve or electrical resistivity, hence, the microscopic and macroscopic properties may be related in a straightforward way. This has direct implications *e.g.* for development of fast strain gauges designed for monitoring dynamic loads. Moreover, correlation of the electrical resistivity measurement with the *in-situ* SAXS monitoring of the nanoparticle displacement can provide the electron tunneling constant among the adjacent nanoparticles. As a bonus, the gold nanoparticles under study exhibit the surface plasmon resonance effect (not shown here) which qualifies the gold nanoparticle monolayer also for other types of sensors such as those based on the surface-enhanced Raman scattering. Here, the studies of the stress-induced response in terms of the nanoparticle shifts are highly relevant to control and tune the plasmon coupling between the nanoparticles.

2. Experimental details

The colloidal gold nanoparticles capped with a mixture of mercaptoalkanes with dodecanethiol being the main component were purchased from PlasmaChem. The size distribution of gold nanoparticles dispersed in *n*-hexane (Sigma-Aldrich) was measured by SAXS. The SAXS pattern (Fig. 1a) was azimuthally averaged and fitted with Rayleigh scattering model with polynomial background (Fig. 1b) that provided diameter of spherically shaped nanoparticles of 5.9 ± 0.6 nm.

The nanoparticle monolayer was prepared by a modified Langmuir-Schaefer method [2]. First, the surface pressure for formation of a homogeneous compact monolayer was found by a measurement of the surface pressure-area isotherm (Fig. 2a). The gold nanoparticles dispersed in chlorophorm of spectroscopic purity (Sigma-Aldrich) at the concentration of 0.2 mg/ml were applied onto the water surface and left to spread at the air/water interface in a Langmuir-Blodgett trough (KSV-Nima Technology). After evaporation of chlorophorm residua during 30 min, barriers in the trough started to move increasing the surface pressure. Derivative of the surface pressure Π according to the surface area A

$$E = -A\frac{\mathrm{d}\Pi}{\mathrm{d}A} \quad (1)$$

provided the surface elastic modulus E of the gold nanoparticle Langmuir film with a sharp maximum at the surface pressure of 13.5 mN/m (Fig. 2b) where a homogeneous and compact nanoparticle monolayer was formed. Here, the barrier movement was stopped when preparing the sample for testing and the gold nanoparticle monolayer was transferred onto a substrate by a controlled removal of the water subphase. The substrate was a 23 μm thick Mylar foil (Lebow) of $7.5 \times 2.5\ \mathrm{cm}^2$ area that was cleaned ultrasonically in isopropanol (Sigma-Aldrich) and glued in the corners to a glass slide by epoxy to provide mechanical support for the nanoparticle deposition in the Langmuir-Blodgett trough. In addition to Mylar foil, a silicon substrate was used in the same deposition run for the microscopic characterization. The surface of silicon wafer (Si-Mat) was cleaned mechanically by a polymer coating and subsequently was exposed to UV radiation producing highly reactive ozone for 10 min.

The quality of the deposited nanoparticle monolayer was investigated *ex-situ* by several techniques. The scanning electron microscopy (SEM) performed on the field emission gun JFM-7500F apparatus (JEOL) operating at 10 kV reveals homogeneous large-area coverage of silicon by well-ordered nanoparticle domains with hexagonal symmetry separated by narrow boundaries (Fig. 3). The atomic force microscopy (AFM) image acquired in peak force mode (MultiMode 8, Bruker Nano) shows the hexagonal close-packed (hcp) structure inside the domains (Fig. 4a). The cross-section along the white line in Fig. 4a exhibits modulations corresponding to individual nanoparticles (Fig. 4b) which suggest the 8.7 nm interparticle distance. As both SEM and AFM are local probes, the grazing-incidence SAXS (GISAXS) technique was employed to get statistically relevant information of the nanoparticle assembly over macroscopic area. The measurement was performed on the same setup as the SAXS measurements (see below) at the angle of incidence of 0.7° that provided a footprint of the X-ray beam long enough to encompass the whole sample length. The sample was placed on a 6-axis miniature vacuum-compatible hexapod H-811 (PI). The GISAXS pattern collected for 10 min (Fig. 5a) shows distinct side maxima due to the nanoparticle ordering. The q_x and q_y axes refer to the components of the scattering vector parallel and perpendicular to the sample surface, respectively. The cut along the q_x axis at $q_y = 0.7\ \mathrm{nm}^{-1}$ exhibits the *10* diffraction peak of the hcp structure located at $q_x = 0.91\ \mathrm{nm}^{-1}$ (Fig. 5b) that corresponds to the interparticle distance $d = (4\pi)/(\sqrt{3}q_x) = 8.0$ nm. Comparing with the nanoparticle diameter of 5.9 nm as determined from SAXS, it suggests the thickness of the nanoparticle surfactant shell of 1.0 nm provided the nanoparticles are in contact and taking into account that SAXS probes the size of the metallic nanoparticle cores.

The SAXS and GISAXS measurements were performed in vacuum on a Nanostar device (Bruker AXS) equipped with a Ga liquid metal-jet X-ray microfocus source (Excillum). The source delivers the beam of GaKα radiation (0.134 nm) with the flux of 10^9 photons/s and the divergence of 0.3 mrad at the output of the collimating Montel optics (Incoatec). The SAXS setup was equipped with a 46 cm long double-pinhole collimator made of scaterless pinholes Scatex® (Incoatec) of 550 μm diameter. A two-dimensional Xe-based X-ray detector Vàntec-2000 (Bruker AXS) with the pixel size of $68 \times 68\ \mu\mathrm{m}^2$ was used.

For the tensile stress measurements, a sample with dimensions of $3 \times 0.5\ \mathrm{cm}^2$ was cut by a 355 nm pulsed nanosecond laser (TruMark station 5000, Trumpf) from the central part of the glass-supported Mylar foil covered with nanoparticles. The stretching of Mylar foil was performed continuously on a vacuum-compatible tensile stage TS 600 with a load cell operating in the range 1–600 N (Anton Paar). The sample was fixed between two grips of the load cell that were 23.5 mm away, defining thus the starting sample length. The starting sample cross-section was $5 \times 0.023\ \mathrm{mm}^2$. The whole stretching device was placed in the evacuated chamber of the SAXS setup. Schematic view of the *in-situ* SAXS measurement during the sample stretching is shown in Fig. 6a. The sample was

Download English Version:

https://daneshyari.com/en/article/736596

Download Persian Version:

https://daneshyari.com/article/736596

Daneshyari.com