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Heat-cured silicone rubber incorporating pyrite powders for actuation elements

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Two pyrite powder sorts were physically incorporated in a silicone rubber matrix consisting of high molecular mass (Mv=450000) polydimethylsiloxane, fumed silica and Fe_2O_3. The obtained composites processed as crosslinked thick (0.60-0.72 mm) films were tested as sensing elements in a nanoactuation device. An interferometric method was used for measurement of the linear displacement of polymeric films by applying a continuum electric field (dc). Displacements of 1600 and 3300 nm were registered under a voltage 475 V.

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1. Introduction

Materials consisting in polymeric matrix with incorporated metallic compounds are of high interest for micro-electro-mechanical systems (MEMS). Such materials combine the properties induced by the metal presence with those specific for polymers, among those being the easy processability. Taking into account the numerous metal derivatives and available polymers, there are possibilities to develop a large range of composite materials with desired properties. Silicones, the intermediates between organic and inorganic (silicates) compounds, bringing together the properties of the two material types, are considered as proper polymeric matrixes for various inorganic powders. The Si-O bond particularity is translated to macroscopic scale resulting in unique properties combination of the silicones: high flexibility, low glass transition temperature (Tg), low surface tension and surface energy, low dielectric constant, transparence to visible and UV light, resistance to ozone, and stability against atomic oxygen and oxygen plasmas, high refractive index, high gas permeability, high hydrophobicity, good thermal stability, etc. [1,2].

The conventional actuation principles applying to membrane actuators include piezoelectric, electrostatic, electrostrictive, thermopneumatic, electrochemical, bimetallic, shape memory alloy, and electromagnetic [2]. Among polymers, dielectric elastomers having an electrostrictive behavior can transform electrical energy directly into mechanical work and are able to generate in plane deformations of up 30% [3,4]. Therefore, the dielectric elastomer actuators have been proposed as large displacement actuators for use in mechatronic devices being simple, inexpensive, lightweight, and fracture resistance [3,5-7]. In addition, the elastomers also offer ease of molding, biocompatibility, robustness, and elasticity, all these features making them suitable for actuation applications [8].

The soft silicone elastomers based on polydimethylsiloxane (PDMS), having low Young's modulus are preferred for the actuation membranes even though traditionally, being insulators, they have played mainly structural role in MEMS, serving as protective layers, encapsulants, valve diaphragms, fluidic channel structures, etc. A number of "active" devices have been made using modified elastomers [2,9-12]. These devices use elastomers mixed with solid fillers, such as carbon black, multi-walled nanotubes (MWNT), or metallic powders that can confer the desired properties to the resulting composite material [10,13]. It has also been reported the *in situ* grown MWNT in PDMS to create strain gauges and field emission devices [14].

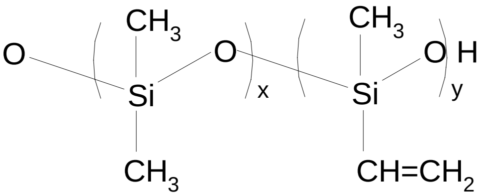
In this paper we attempted to obtain polymeric films for a micro-magnetovalve, microswitch or microscanner actuator. These films are based on a high molecular weight polysiloxane reinforced with fumed silica and crosslinked by radical mechanism. Two different sorts of pyrite powders were physically incorporated in such a composite before crosslinking. Taking into account that an electric impulse creates a magnetic field it is expected as the magnetic particles presence to induce actuation effects.

2. Experimental

2.1 Materials

The poly[dimethyl(methylvinyl)siloxane]-α,ω-diol (Scheme 1) having viscometric molecular mass Mv = 450000 was prepared by copolymerization of octamethylcyclotetrasiloxane, $[(CH_3)_2SiO]_4$, D_4, with a small portion of heptamethylvinylcyclotetrasiloxane,

$[(CH_3)_2SiO]_3[(CH_3)(C_2H_3)SiO]$, D_3V, at room temperature in presence of H_2SO_4 as a catalyst, according to the procedure described in ref. [15,16]. The average molecular mass was estimated on bases of the viscometer measurements in solution, using toluene as a solvent.



Scheme 1. Structure of poly[dimethyl(methylvinyl)siloxane]-α,ω-diol.

Fumed silica, Aerosil 380 (Degussa), 100% purity, specific surface 380 m^2/g, particle diameter 0,003 - 0,015µ was hydrophobized before it's using [16,17].

2,4-Dichlorobenzoil peroxide, having a critical temperature: 60-70^{0}C and set-cure temperature in the range 115 - 150^{0}C, was used as a paste in silicone oil.

Iron(III) oxide, Fe_2O_3, (Fluka): 98% purity, specific volume - 1,17cm^3/g was dried first in air oven 3h at 110 ^oC and then in vacuum, 1h at 110 ^oC.

Pyrites (Valea Calugareasca, Romania) had the following characteristics:

-ground reduced pyrite (Py1) with average diameter of the particles of about 73µm;

-unground pyrite with average particle diameter of 79 µm (Py4).

2.2 Procedure

2.2.1 Preparation of the composites

PDMS was mixed with about 20% hydrophobized silica and 2% Fe_2O_3 by mechanical blending. In the end, the vulcanizing catalyst (2,4-dichlorobenzoil peroxide) and pyrite (20%) were incorporated in pre-established amounts.

The resulted composites were pressed between two metallic plates into a rectangular frame. This ensemble was maintained in this manner at 150^{o}C for about 2h when the curing of the composite occurs in the presence of the radical initiator. The formed films having the thickness of 0.60 and 0.72 mm, respectively were easily detached from the plates surface.

3. Results and discussion

Hydrophobized silica was incorporated by the mechanical mixing into PDMS having a viscometric molecular mass of about 450000 as the continuous phase. The silica addition is necessary to improve the mechanical characteristics (abrasion resistance, tensile strength, tear strength, and modulus) of the polydiorganosiloxanes. The presence of the vinyl groups along the chain permits readily crosslinking by peroxide. 2,4-Dichlorobenzoyl peroxide was used in our case. Such a composite was used as matrix to mechanical incorporate two pyrite sorts in proportion of 20 %wt (Table 1). The mixtures were processed as films and heated to the critical temperature of the peroxide catalyst followed by post-curing to remove the volatile compounds formed by the peroxide decomposition. In fact, the matrix is a high temperature vulcanizing silicone rubber (HTV) [16,17].

Table 1. The prepared composites .

Sample	% wt Pyrite addition (type)	Film thickness, mm
HTV1	20 (Py4)	0.60
HTV2	20 (Py1)	0.72

The macroscopic aspect of the resulted films can be seen in Fig. 1.

HTV1 HTV2

Fig. 1. The photo images taken on the prepared films: a- HTV1; b- HTV2.

The resulted films were resistant and very flexible due to their composition and can be easily manipulated. The linear micro and nanodisplacements of these polymeric films were determined using an experimental setup for linear measurements, based on a Michelson type interferometer by an AGILENT 5529A system [18]. The device is able to determine minimum nanodisplacements above 2 nm. The experiments were performed using plane electrodes in contact with films under a force of 22 cN and a manual switch to control the electric field. The registered signals, which are visualized on the diagrams (Figs. 2 and 3), represent the polymer film actuation response to an applied continuum electric (dc) field with a voltage of 475V.

In Fig. 2 (a) is presented the microactuation (microdisplacement) obtained by using the sample HTV1 to a dc commutation regime with three different time durations (a); I – 6.6 s, II- 8.3 s, III -3.3 s. The speed characteristic is drawn in red. By inverse polarization of HTV1 film to 475 V, the diagram presented in Fig. 2 (b) was obtained. It can be seen that the maximum level of the obtained actuation is ~ 1600 nm, this being a good behavior from this point of view.

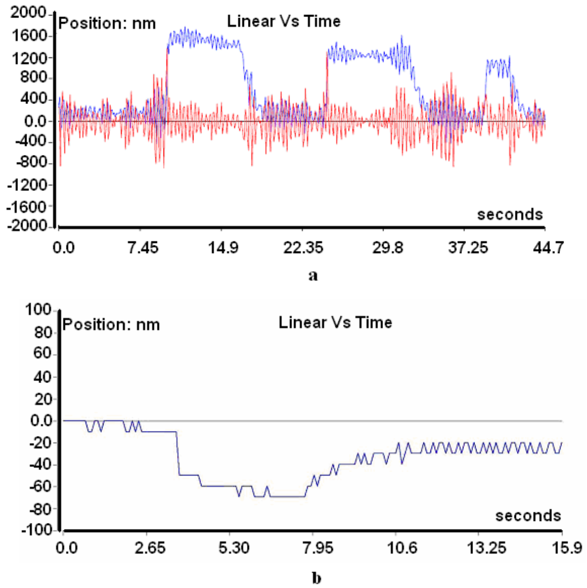


Fig. 2. The displacement obtained with silicone film HTV1 at 475 V in different conditions: a) – dc commutation regime with durations: I – 6.6 s, II- 8.3 s, III -3.3 s (the speed characteristic is showed in red); b- inverse polarization to 475 V.

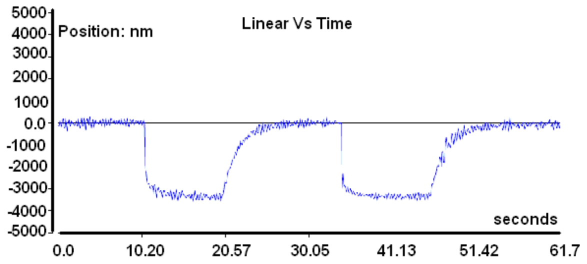


Fig. 3. The displacement obtained with silicone film HTV2 at 475 V dc.

However, the best proved to be the HTV2 for which a displacement of about 3000 nm was obtained. Even though the actuation results are good enough for the both prepared materials, it can be noticed that the presence of the reduced ground pyrite, Py1, is more effective as compared with unprocessed Py4. The smaller dimension of the Py1 particles as well as it's previously treatment (reduction) could explain this behaviour.

The presented results lead to the conclusion that silicone elastomers having incorporated pyrites can be used as active elements in different linear actuators.

4. Conclusions

Two composites were prepared by the incorporation of two pyrite sorts (which differ by the processing and particle diameters) in a classical silicone rubber composite consisting in mixture PDMS/silica/ Fe_2O_3 and processed as films with 0.60-0.72 mm thickness. The electromechanical tests of these films revealed a very good behavior for applications in nano and microactuation.

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