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TENSORESISTIVE PROPERTIES OF HETEROGENEOUS FILM MATERIALS BASED ON REFRACTORY METALS

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We present the experimental investigation results of the tensoresistive properties of heterogeneous film systems based on Re or Fe oxide phases and the metal component of hcp-Re (hcp is the hexagonal close-packed lattice) or a-Mo (a is the amorphous phase).

Keywords: HETEROGENEOUS FILM MATERIALS, REFRACTORY METALS, PHASE COMPOSITION, GAUGE FACTOR.

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1. INTRODUCTION

Heterogeneous metal film materials are of a great interest at the modern stage of magnetoelectronics and sensor technology development. Investigations [1] showed, that in many cases the single-layer heterogeneous films can replace the multilayer film materials since they have a number of features, namely, decrease in the electrical resistance with the increase in the magnetic field strength and its maximal value at zero magnetization (heterogeneous films based on ferromagnets); stability of the thermo- and tensoresistive operating characteristics of sensing elements in elevated temperature conditions (heterogeneous film materials based on refractory metals) [2-4].

Analysis of current publications [5-9] showed, that due to the high temperature stability the heterogeneous film materials based on refractory metals (such as nitrides, carbides, oxides, etc) can be used for production of passive components of the integrated circuits by the multilayer metallization method with prediction of the influence of diffusion processes and layer structure on the value of contact resistance and operating characteristics.

Many-component oxides, carbides and nitrides have high thermal stability and are the basis of functional nanocrystalline materials. In series of publications of Hrovat et al. [10, 11] there is the data, which allows to generalize information about applications of heterogeneous materials in the film resistive-strain sensor design; the strain-gauge parameters of thick-film resistors based on ruthenates (R) of Pb, Al, Zr, Cu or compounds RuO_2 and $RuO_2 + R$ and the thermal treatment influence on their values are presented.

Depending on the production conditions (pressure, temperature, condensation rate) the refractory metal films (Re, Mo) can have amorphous, bcc- or fcc-structures. According to [12-16] based on the production conditions the oxide (Mo₃O) or carbonitride (Mo(C,N)_x) phases are stabilized in Mo films. When two-phase bcc-Mo + Mo₃O films are heated the further oxidation to Mo_2O_3 composition occurs.

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Authors of [17] have established that under thermal treatment of twolayer Cu/Fe films the heterogeneous composition is formed on the basis of the following phases: fcc-Cu, bcc-Fe, fcc- Cu₂O, fcc-FeO and the traces Fe_3O_4 .

In [18] the phase composition of Re films obtained and annealed in the high-vacuum conditions is studied. Since Re almost does not interact with carbon, but reacts with oxygen and nitrogen, the oxides and oxynitrides are formed in Re films. According to these authors the phases with hexagonal (Re₃O), simple cubic (ReO₃), or rhombohedral (Re₂O₇) lattices can be formed in Re films. When Re films are obtained by the cathode sputtering method [19], the anomalous fcc phase with the lattice constant $a \approx 0,404$ nm is formed, the chemical composition of which, most likely, corresponds to the oxynitride Re(N,O)_x. This is confirmed by the fact that in bulk samples nitride Re_xN (x = 2-3) has the decreased lattice constant a = 0,393 nm [20].

Depending on the aforesaid, the aim of the present work can be stated as follows: to investigate the tensoresistive properties of heterogeneous film systems based on Re or Fe oxide phases and metal component of hcp-Re (hcp is the hexagonal close-packed lattice) or a-Mo (a is the amorphous phase).

2. EXPERIMENTAL TECHNIQUE

To obtain the film samples of two types: based on pure metals (two- and three-layer films based on a-Mo and bcc-Fe) and of heterogeneous composition $(\text{Re}(N,O)_x + \text{hcp-Re}, \text{fcc-FeO}_x + \text{a-Mo})$, the vacuum plant VUP-5M (vacuum $\sim 10^{-4}$ Pa) was used. Heterogeneous structures were formed by the variation of the condensation rate (ω) and the substrate (T_s) and the annealing (T_{ann}) temperatures. Evaporation was performed by the electron-ray method, and the thickness of separate layers (d) was controlled by the quartz resonator method (accuracy is 10%) and varied from 20 nm to 50 nm. The substrate temperature was changed in the range from 300 K to 500 K, the annealing temperature was 300-800 K, and $\omega \cong 1,0$ nm/s. The annealing duration at the maximal temperature was 15 minutes. Investigation of the phase composition was realized by the electron-diffraction method and the transmission electron microscopy (device PEM-125K). Measurements of the gauge factor (GF) were done by the standard technique described in [21].

3. EXPERIMENTAL INVESTIGATION RESULTS

In Fig. 1 we present the typical micrographs and the diffraction patterns (interpretation in Table 1) from heterogeneous fcc-Re(N,O)_x + hcp-Re film with the total thickness of $d \approx 20$ nm. The lattice constant of oxynitride Re varies from a = 0,407 nm (the unannealed sample obtained at $T_s \approx 450$ K) to 0,404 nm (annealed at $T_{ann} \approx 800$ K).

In the case of film systems based on Mo and Fe we have formed two types of the samples: bcc-Fe/a-Mo and bcc-Fe/a-Mo/bcc-Fe, and fcc-FeO + a-Mo. Results of microscopic and diffraction investigations of two-layer bcc-Fe/ a-Mo system and heterogeneous film are represented in Fig. 2 and Table 2.



Fig. 1 – Microstructure and the corresponding diffraction patterns from the unannealed (a) and the annealed (b) heterogeneous fcc-Re $(N,O)_x$ + hcp-Re film. $T_s \cong 450 \text{ K}$

Table 1 – Interpretation of the electron diffraction patterns from $Re(NO)_x$ fcc-phase

No	hkl	I, rel. un	d_{hkl} , nm	a, nm	I, rel. un	d_{hkl} , nm	a, nm		
1	100	S	0,236	0,409	V.S	0,234	0,405		
2	200	m	0,204	0,408	m	0,202	0,404		
3	220	m	0,144	0,407	m	0,142	0,402		
4	311	m	0,123	0,408	m	0,122	0,405		
5	222	w	0,116	0,405	V.w	0,116	0,402		
unanneal ā (Re(N	ed sample D) _x = 0,40	0.002	2 nm)	annealed to $T_{ann} = 800$ K sample \overline{a} (Re(NO) _x = 0,404 ± 0,002 nm)					
V.S – very strong, S – strong, m – medium, w – weak, V.w – very weak									



Fig. 2 – Microstructure and the corresponding diffraction patterns from the unannealed Fe(30)/a-Mo(20) film (a) and the annealed to 800 K Fe(40)/a-Mo(30) film (b). Thickness (in nm) is given in the brackets

During the investigations of the tensoresistive properties we have plotted the standard dependences $\Delta R/R$ versus the longitudinal deformation ε_l , where $\Delta R = R(\varepsilon_l) - R$ (*R* is the initial resistance) for the first five deformation cycles "load-reload". In Fig. 3 we present the typical deformation dependences for heterogeneous fcc-Re(N,O)_x + hcp-Re and two-layer Fe(30)/a-Mo(20) films. Calculation of the GF was performed using the V-th deformation cycle by the slope ratio.

No	hkl	I, rel. un	d_{hkl} , nm	a, nm	Phase	\overline{a} , nm			
Fe(30)/Mo(20) (unannealed sample)									
1	-	m	0,225	-	a-Mo				
2	2 110 S 3 200 m		0,202	0,286	bcc-Fe	\overline{a} (bcc-Fe) =			
3			0,144	0,288	bcc-Fe				
4	-	V.w	0,129	-	a-Mo	$= 0.287 \pm 0.001 \text{ nm}$			
5	211	m	0,117	0,287	bcc-Fe				
Fe(40)/Mo(30) (annealed to 800 K sample)									
1	111	V.S	0,252	0,436	FeO				
1'	110	diffuse	0,224	0,317	bcc-Mo	\overline{a} (bcc-Mo) = = 0,315 ± 0,001 nm			
2	200	m	0,216	0,432	FeO				
3	220	w	$0,\!154$	$0,\!436$	${\rm FeO}$				
4	311	w	0,132	0,438	FeO	a (reO) =			
5	211	diffuse	0,128	0,314	bcc-Mo	$= 0,435 \pm 0,003 \text{ nm}$			
-									

Table 2 – Interpretation of diffraction from Fe/Mo/Sub film systems



Fig. 3 –Deformation dependences for heterogeneous $fcc\operatorname{-Re}(N,O)_x + hcp\operatorname{-Re}$ and two-layer bcc-Fe(20)/a-Mo(20) films. The number of deformation cycle: $\circ - I$, $\circ - II$, $\Box - III$, $\diamond - IV$, $\bigtriangleup - V$

4. CONCLUSIONS

We have obtained the following results. In heterogeneous films based on Re as well as in fcc-FeO + a-Mo films the value of the GF is close to 8. In the case of two- and three-layer bcc-Fe/a-Mo and bcc-Fe/a-Mo/bcc-Fe film systems at the total thickness of the sample from 40 nm to 60 nm the value of the GF is about 10 (two-layer systems) or 12,4 (three-layer systems).

Based on the GF value one can state, that observed heterogeneous twoand three-layer film materials can be used as the sensing elements of resistivestrain sensors, although, depending on the data of [22], the vanadium singleoxide films, where the GF value changes from 285 (d = 40 nm) to 80 (d = 205 nm), can be the more effective sensing elements.

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