

ION-PLASMA DEPOSITION OF THIN QUASICRYSTALLINE Al-Cu-Fe AND Al-Cu-Co FILMS

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Al-Cu-Fe and Al-Co-Cu thin films were firstly deposited on sodium chloride or glass-ceramic substrates by modernized method of three-electrode ion-plasma sputtering. The nominal compositions of the films were chosen in the regions of quasicrystalline phases formation. The as-sputtered films were typically 85 to 260 nm thick. The films were annealed at temperatures ranging from 873 to 923 K for 10 min...3 h. The structure of films was studied by scanning and transmission electron microscopy and X-ray analysis. Electrical properties were determined by a four-probe method. The as-deposited Al-Cu-Fe film was found to consist of isolated quasicrystalline nanoparticles of icosahedral *i*-phase. With substitution of Fe for Co in Al-Co-Cu film, X-ray amorphous phase and only traces of quasicrystalline decagonal *D*-phase were revealed. After annealing, the films were predominately quasicrystalline due to transformation of metallic phases into quasicrystalline. At the same time, the size of coherent scattering regions for quasicrystals increased by two times from ~ 3 to 6 nm. Measurements of electrical resistivity showed that no phase transformations occurred in Al-Cu-Fe film up to 723 K and in Al-Co-Cu film up to 640 K. With following increase in temperature, electrical resistivity of Al-Cu-Fe film increased by six orders of magnitude (up to $6 \cdot 10^7 \Omega/\text{sq}$). In contrast, electrical resistivity of Al-Co-Cu film decreased by ~ 2 times. After cooling to room temperature, resistivity of Al-Cu-Fe film equaled to $\sim 3 \cdot 10^5 \Omega/\text{sq}$ and that of Al-Co-Cu film – to $8.7 \Omega/\text{sq}$. We concluded that Al-Cu-Fe thin film is more suitable candidate for application as precise high-ohmic materials.

PACS: 61.44.Br, 81.15.Fg, 61.43.Dq, 64.60.My, 81.07.Bc, 75.70.-i

INTRODUCTION

Al-Cu-Fe or Al-Cu-Co alloy systems have been identified as systems in which three-dimensional icosahedral (*i*) or two-dimensional decagonal (*D*) quasicrystals are stable at high temperatures [1, 2]. Both systems are known to yield quasicrystalline phases after slow solidification. Discoveries of the stable quasicrystals have inspired a great progress in the studies of quasicrystalline structure and properties. It has been found that these materials have unique mechanical and tribological properties, including low friction, high hardness, high wear, corrosion and oxidation resistance [3–5]. Other properties such as brittleness have hindered the use of quasicrystals in bulk form, although they have shown significant potential for use as thick composite coatings [6, 7] and thin films [8, 9].

To produce a large variety of thin quasicrystalline Al-Cu-Fe films on Al_2O_3 , MgO, NaCl etc. substrate materials, the methods of vapor deposition are now used [10]. The films were deposited at various substrate temperatures in either the amorphous or the crystalline state or as a stack of elemental layers [11, 12]. The icosahedral quasicrystalline phase (*i*-phase) was obtained by subsequent annealing. Thin quasicrystalline Al-Cu-Fe films were also formed directly by such methods as sputtering or evaporation, without need for post-deposition treatments [13]. Due to rapid cooling, quasicrystalline coatings based on Al-Cu-Fe quasicrystals have shown high resistance to wear, corrosion in aqueous solutions, low thermal conductivity [14–18]. They are ideal candidates for applications requiring sliding contact [19–21].

Thin films of Al-Cu-Co alloys were grown by thermal vapour deposition technique by varying the composition of the target material [22]. It was observed

that quasicrystalline decagonal phase (*D*-phase) did not form in thin films when the composition of the target material was close to that of quasicrystalline phase, i.e., $\text{Al}_{65}\text{Cu}_{15}\text{Co}_{20}$. The deposited films of lower thickness (< 200 nm) were always found to contain amorphous phase which transformed mainly to crystalline phases. By several trials of changing the composition of the target material it was found that target alloy composition close to $\text{Al}_{50}\text{Cu}_{20}\text{Co}_{30}$ yielded maximum amount of $\text{Al}_{65}\text{Cu}_{15}\text{Co}_{20}$ quasicrystalline phase in the films of 1000...2000 nm in thickness. The composition variation between the target alloy and the deposited film was explained by significant differences in partial vapour pressure among the constituent elements. The phase evolution under rapid solidification condition of the melt spun Al-Cu-Co-Ni ribbons may also open an interesting field of applications [23]. The thickness and width of the ribbons were found to be $\sim 50 \mu\text{m}$ and $\sim 0.75 \dots 1.0$ mm, respectively.

Thus, the structure of quasicrystalline coatings has been found to be sensitive to the cooling rate achieved during deposition [24–30]. Therefore, it is essential to monitor the rates of sputtered atoms which have a considerable dispersion not only in magnitude but also in direction. An acceleration of ions impinging on the target and, correspondingly, an increase in their kinetic energy by factors of 5 to 6 can be achieved by using a modernized method of ion-plasma sputtering [31, 32]. This method ensures a more uniform mixing of components upon their deposition on the substrates than traditional techniques [33]. The cooling rates for deposited films, theoretically estimated considering the time of atoms relaxation, are within the range of 10^{13} to 10^{14} K/s. The structure of the films is formed in extra non-equilibrium conditions.

It may be assumed that quasicrystalline materials appear to be a potential candidate for application as an efficient coating material on various substrates. However, it must be pointed out that in order to obtain a good quality quasicrystalline thin film, there is a need to pursue the study of their formation. Understanding the stages of quasicrystalline film growth is critical to reproducible film fabrication.

The aim of the present work is to deposit stable quasicrystalline thin films of Al-Cu-Fe and Al-Cu-Co alloys grown by the modernized ion-plasma sputtering technique. Coatings prepared of these alloys have been investigated as well for their electrical properties.

1. MATERIALS AND METHODS

Thin films were deposited on sodium chloride or glass-ceramic substrates by modernized method of three-electrode ion-plasma sputtering [31]. As targets, $20 \times 20 \times 5$ mm parallelepipeds were used made of pure components (99.99%). The parallelepipeds were separated by barrier cells performing like electrostatic lens. Details concerning schematic drawing of sputtering system can be found in [32].

The sputtering parameters to deposit quasicrystalline coatings by modernized method of three-electrode ion-plasma sputtering are listed in Table, where P_{start} is starting vacuum in the working chamber; P_{Ar} is a pressure of orifice gas (Ar); U_M is a target voltage; I_A is anode current; j is target current density; t_{dep} is a time of deposition on substrates; D is a thickness of films deposited on glass-ceramic substrates; d_{TEM} is a thickness of films deposited on sodium chloride substrates for TEM investigation; η is a rate of deposition.

Sputtering conditions for thin films

Parameter	Al ₆₀ Cu ₂₈ Fe ₁₂	Al ₆₆ Cu ₁₈ Co ₁₆
P_{start} , mPa	1.2	2.0
P_{Ar} , mPa	53	53
U_M , kV	-2	-2
I_A , A	2	2
j , A/m ²	4.8	6.8
t_{dep} , s	1660/120	1440/180
D , nm	260/19	105/14
d_{TEM} , nm	19	14
η , pm/s	160	73

The initial compositions of the targets used for sputtering were Al₆₀Cu₂₈Fe₁₂ and Al₆₆Cu₁₈Co₁₆ (at.%). The nominal compositions were similar to those of the quasicrystalline alloys reported in [1, 2]. The films thickness was varied from 85 to 260 nm. In addition, thin films were annealed in a tube furnace in vacuum at different temperatures (873...923 K) and times (10 min...3 h).

The films were characterized by scanning (SEM) and transmission (TEM) electron microscopy (EM) together with energy dispersive (EDX) and X-ray diffraction (XRD) techniques. Before examinations, the sodium chloride substrates were removed by dissolving in water to acquire free standing thin films. Electrical resistivity (R_s) of quasicrystalline films deposited on

glass-ceramic substrate was measured with the conventional four-probe method in the temperature range 293...923 K at heating rate of 18 K/min in vacuum (~ 1.3 mPa).

2. RESULTS AND DISCUSSION

Thin films of Al-Cu-Fe and Al-Cu-Co alloys adhere very well to the substrates and can withstand small plastic strains without failing. EDX measurements show that Al-Cu-Fe films growth reduces the Al present in the films, but Cu and Fe content is close to expected [17]. The average oxygen content is less than 13 at.%. In the deposited Al-Cu-Co film the Al content is higher, but the Co content is lower compared to calculated one demonstrating that the rate of Co deposition is lower than that of Al and Cu [22]. Composition of the films varies only slightly over the rather large substrate area.

The Al-Cu-Fe film consists of isolated quasicrystalline nanoparticles at the given composition and deposition conditions. The dispersed structure of Al-Cu-Fe film is deduced from EM patterns (Fig. 1). In electron microdiffraction pattern (see Fig. 1,b), the diffraction lines are diffused. No separate reflections are observed here in microdiffraction mode. This confirms the formation of nanosized quasicrystalline particles which takes place by a direct nucleation process.

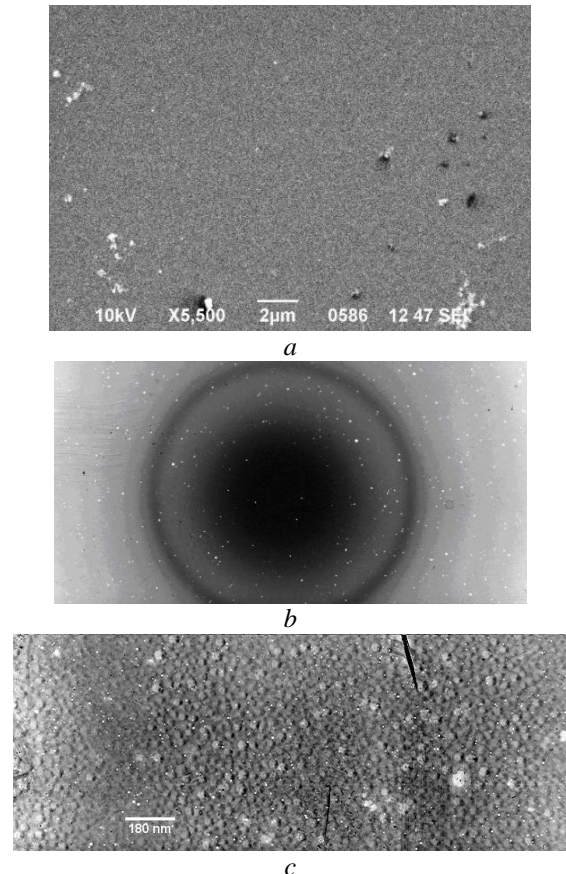


Fig. 1. EM patterns of as-sputtered Al-Cu-Fe film:

- a – SEM ($D \sim 260$ nm, $\times 5.5 \cdot 10^3$);
- b – electron microdiffraction pattern;
- c – TEM ($d_{TEM} \sim 19$ nm, $\times 4 \cdot 10^4$)

XRD pattern also reveals that Al-Cu-Fe film is not continuous but consisted of isolated nanoparticles (Fig. 2,a). The peaks angles are close to those indicated

for the icosahedral quasicrystalline phase [14]. Nanosize of *i*-phase is verified by a width of diffraction peaks. The size of coherent scattering regions for as-sputtered Al-Cu-Fe film is ~ 3 nm. The regions surrounding *i*-phases are dispersed crystalline and very thin compared to the quasicrystalline grains.

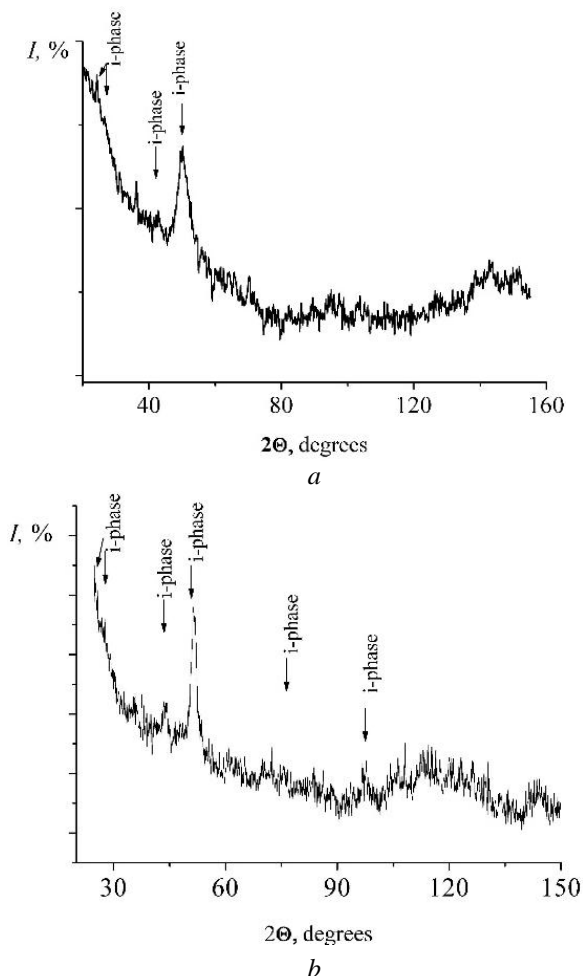


Fig. 2. X-ray patterns of Al-Cu-Fe films: a – before annealing, b – after annealing

After annealing for 3 h at 873 K, the Al-Cu-Fe film exhibits coarser structure. Post-annealing treatment reveals that coherent scattering regions of quasicrystalline icosahedral phase become ~ 6 nm in size. So, a twofold increase in size is observed as compared to that of as-sputtered film. In X-ray pattern additional diffraction peaks appear after annealing (see Fig. 2,b). All the peaks can be identified as icosahedral single phase. Diffraction peaks become narrow; their intensity increases. In addition, most of the peak positions are shifted towards lower angles because of the effect of thermal relaxation and volume diffusion of components in the films.

In the heating procedure with a rate of 18 K/min, measurements of electrical resistivity (R_S) show that the Al-Cu-Fe film is stable up to 723 K. Over a temperature range of 293...723 K, the slight reversible decrease in a resistivity from 20 to 19.9 Ω/sq is observed which indicates that no phase transformations take place (Fig. 3). The temperature coefficient of the resistivity (TCR) is of negative value $-2 \cdot 10^{-4} \text{ K}^{-1}$. As the temperature is raised from 723 to 803 K, irreversible

increase in electrical resistivity by six orders of magnitude (up to $6 \cdot 10^7 \Omega/\text{sq}$) evidences that the structure of the Al-Cu-Fe film starts changing. Activation energy of phase transformations calculated using Kissinger method equals to $\sim 103 \text{ kJ/mol}$. With following increase in temperature up to 873 K, R_S slightly decreases with the negative temperature coefficient of the resistivity. In cooling up to 753 K electrical resistivity decreases up to $\sim 3 \cdot 10^5 \Omega/\text{sq}$. Then, in cooling up to room temperature, it slightly lowers with the negative TCR amounting to $\sim -5 \cdot 10^{-6} \text{ K}^{-1}$. The observed changes in electrical resistivity of the deposits during heating are consistent with the above described changes of their nanoquasicrystalline structure. The presence of the three-dimensional quasi-periodicity contributes to the sharp increase in the electrical resistivity which can be explained by the specific properties of icosahedral quasicrystals [34].

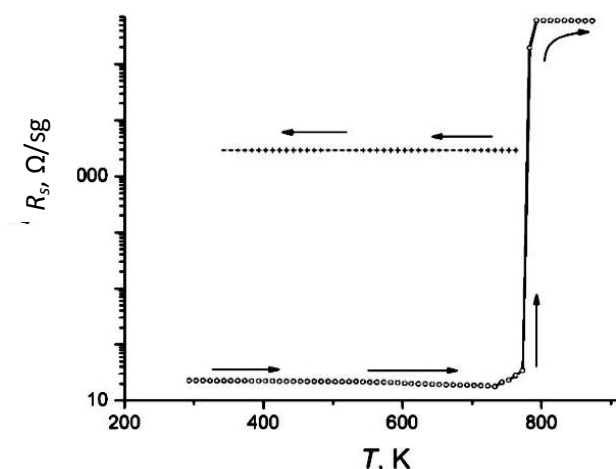


Fig. 3. Temperature dependence of electrical resistivity of Al-Cu-Fe film at heating rate of 18 K/min

As-sputtered Al-Cu-Co film is found to consist of X-ray amorphous phase. Some traces of quasicrystalline *D*-phase are revealed as well. The presence of these phases is verified by XRD (Fig. 4,a). The size of coherent scattering regions for *D*-phase is ~ 2.8 nm.

After annealing for 10 min at 923 K, the Al-Cu-Co film consists of a decagonal phase and an Al-based crystalline phase. This can be explained as follows. Enhanced diffusion leads to the rapid growth of quasicrystalline phases resulting in the expulsion of mainly aluminum to the surrounding matrix. Diffraction pattern from the decagonal phase and Al-based phase of post-annealed Al-Cu-Co film is shown in Fig. 4,b. Stresses relaxation and disappearance of amorphous phase may also be responsible for narrowing of some reflections in XRD pattern. In addition, due to solid state diffusion the significant shift in positions of diffraction peaks is revealed.

Annealing experiments show coarsening behavior. The coherent scattering regions for *D*-phase increase in size up to 6 nm after annealing. Thus, as-sputtered Al-Cu-Co thin film has mainly an amorphous structure. This amorphous phase is metastable and transforms to a mixture of quasicrystalline and crystalline phases upon short annealing. At temperature 923 K, diffusion is highly enhanced and results in a twofold enlargement of coherent scattering regions for *D*-phase.

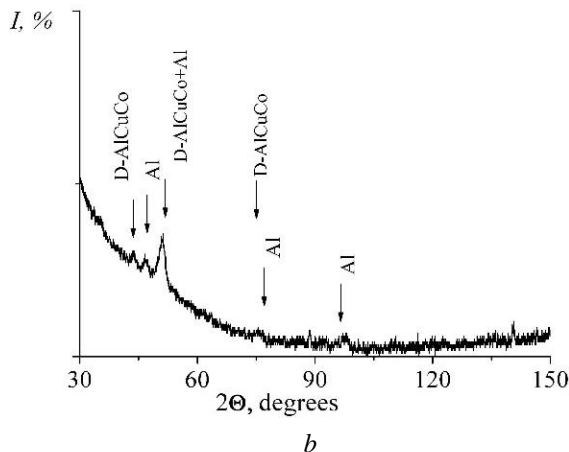
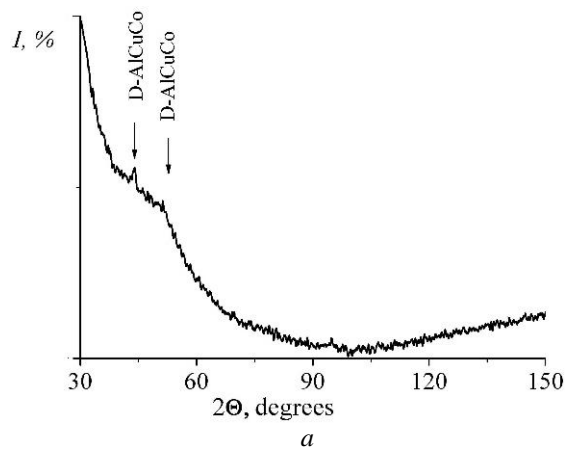


Fig. 4. X-ray patterns of Al-Cu-Co films:
a – before annealing, b – after annealing

The as-sputtered Al-Cu-Co thin film has relatively small values of electrical resistivity amounting to $\sim 22\text{--}25 \text{ } \Omega/\text{sq}$ (Fig. 5). Such values may be explained by the presence of amorphous phase with the positive temperature coefficient of the resistivity in the film. During heating, TCR for quasicrystalline and crystalline phases may mutually compensate each other. Investigations of electrical properties over a temperature range of $290\text{--}640 \text{ K}$ reveal that electrical resistivity shows reversible change from ~ 22 to $\sim 21.8 \text{ } \Omega/\text{sq}$. This means that structure of the film remains unchanged. The temperature coefficient of the resistivity for Al-Cu-Co film is negative and equals to $-6 \cdot 10^{-4} \text{ K}^{-1}$.

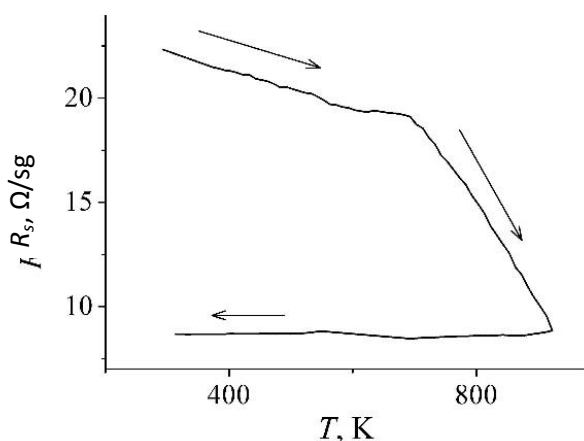


Fig. 5. Temperature dependence of electrical resistivity of Al-Cu-Co film at heating rate of 18 K/min

In a temperature range of $640\text{--}920 \text{ K}$, irreversible change of R_s from 19.3 to $8.8 \text{ } \Omega/\text{sq}$ is observed which indicates a start of phase transformations inducing structural changes. As annealing experiments show, this may be related to transformation from amorphous phase to a mixture of quasicrystalline and crystalline phases. Activation energy of structural changes reaches $\sim 51 \text{ kJ/mol}$. In the cooling from 920 K to room temperature, resistivity for Al-Cu-Co film decreases up to $8.7 \text{ } \Omega/\text{sq}$ and TCR amounts to $-1.2 \cdot 10^{-5} \text{ K}^{-1}$. The revealed difference in the electrical resistivity of Al-Cu-Co and Al-Cu-Fe thin films originates from differences both in their atomic arrangements and the accompanying electronic structure [34].

CONCLUSIONS

The quasicrystalline Al-Cu-Fe and Al-Cu-Co thin films have been first deposited by modernized method of three-electrode ion-plasma sputtering on NaCl or glass-ceramic substrates at cooling rates of $10^{13}\text{--}10^{14} \text{ K/s}$.

In the case of Al-Cu-Fe film, an almost pure icosahedral quasicrystalline *i*-phase can be obtained. Determination of a size of coherent scattering regions indicates that the *i*-phase of the Al-Cu-Fe film is in nanostructural state. Post-annealing treatment for 3 h at 873 K reveals an increase in the amount of *i*-phase and a twofold increase of the size of its coherent scattering regions due to solid state diffusion. The measurements of an electrical resistivity of the depositions show that the Al-Cu-Fe film is stable up to the temperature of 723 K . In a temperature range of 723 to 773 K , its electrical resistivity increases by six orders of magnitude. The Al-Cu-Fe film exhibits a negative temperature dependence of resistivity over a wide temperature range.

In the deposited Al-Cu-Co film, X-ray amorphous phase and traces of decagonal quasicrystalline *D*-phase are revealed. At annealing temperature of 923 K , transformation from amorphous phase to a mixture of decagonal quasicrystalline and Al-based crystalline phases is observed. Annealing results in almost twice larger coherent scattering regions of *D*-phase. As electrical resistivity measurements evidence, Al-Cu-Co film is stable up to 640 K . During cooling from 920 K to room temperature, the negative temperature coefficient of the resistivity changes as compared with that of the as-sputtered film.

Thus, by controlling the process parameters such as the deposition rate, thickness, substrate temperature, the composition as well as annealing procedure, it may be possible to obtain quasicrystalline Al-Cu-Fe and Al-Cu-Co films. The three-dimensional icosahedral quasicrystals possess much higher electrical resistivity than the two-dimensional decagonal quasicrystals. This leads us to conclude that the quasicrystalline Al-Cu-Fe thin film is more suitable candidate for fabrication of precise high ohmic resistors.

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Article received 26.12.2019

ОСАЖДЕНИЕ ТОНКИХ КВАЗИКРИСТАЛЛИЧЕСКИХ ПЛЕНОК Al-Cu-Fe И Al-Cu-Co ИОННО-ПЛАЗМЕННЫМ МЕТОДОМ

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Тонкие пленки Al-Cu-Fe и Al-Co-Cu были впервые нанесены на подложки из натрия хлорида и ситала модернизированным методом трехэлектродного ионно-плазменного осаждения. Номинальные составы пленок выбирали в области существования квазикристаллических фаз. Толщина напыленных пленок составляла от 85 до 260 нм. Пленки отжигали в интервале температур 873...923 К в течение 10 мин...3 ч. Структуру пленок изучали методами сканирующей и трансмиссионной электронной микроскопии и рентгеноструктурного анализа. Электрические свойства определяли четырехзондовым методом. Установлено, что в свеженапыленном состоянии пленка Al-Cu-Fe состоит из изолированных квазикристаллических наночастиц икосаэдрической *i*-фазы. При замене Fe на Co в пленке Al-Co-Cu обнаружены рентгеноаморфная фаза и следы квазикристаллической декагональной *D*-фазы. После отжига пленки имеют преимущественно квазикристаллическую структуру вследствие превращения металлических фаз в квазикристаллические. При этом размер областей когерентного рассеяния квазикристаллов увеличивается в два раза с ~ 3 до 6 нм. Измерения электрического сопротивления показали, что в пленке Al-Cu-Fe отсутствуют фазовые превращения вплоть до температуры 723 К, а в пленке Al-Co-Cu – до 640 К. При последующем нагреве электрическое сопротивление пленки Al-Cu-Fe возрастает на шесть порядков (до $6 \cdot 10^7$ Ом/кв). Напротив, электрическое сопротивление пленки Al-Co-Cu уменьшается в ~ 2 раза. После охлаждения до комнатной температуры сопротивление пленки Al-Cu-Fe составляет $\sim 3 \cdot 10^5$ Ом/кв, а пленки Al-Co-Cu – 8,7 Ом/кв. Сделан вывод о том, что пленка Al-Cu-Fe является более перспективным материалом для изготовления прецизионных высокоомных резисторов.

ОСАЖДЕННЯ ТОНКИХ КВАЗИКРИСТАЛІЧНИХ ПЛІВОК Al-Cu-Fe ТА Al-Cu-Co ІОННО-ПЛАЗМОВИМ МЕТОДОМ

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Тонкі плівки Al-Cu-Fe та Al-Co-Cu були вперше нанесені на підкладки з натрій хлориду та ситалу модернізованим методом триелектродного іонно-плазмового осадження. Номінальний склад плівок обирали в області існування квазікристалічних фаз. Товщина напилених плівок складала від 85 до 260 нм. Плівки відпалювали в інтервалі температур 873...923 К на протязі 10 хв...3 год. Структуру плівок вивчали методами скануючої і трансмісійної електронної мікроскопії та рентгеноструктурного аналізу. Електричні властивості визначали чотиризондовим методом. Встановлено, що в свіжонапиленому стані плівка Al-Cu-Fe складається з ізольованих квазікристалічних наночастинок ікосаедрічної *i*-фазы. У разі заміни Fe на Co в плівці Al-Co-Cu виявлені рентгеноаморфна фаза і сліди квазікристалічної декагональної *D*-фазы. Після відпалу плівки мають переважно квазікристалічну структуру внаслідок перетворення металевих фаз на квазікристалічні. При цьому розмір областей когерентного розсіювання квазікристалів збільшується в два рази з ~ 3 до 6 нм. Виміри електричного опору показали, що в плівці Al-Cu-Fe відсутні фазові перетворення аж до температури 723 К, а в плівці Al-Co-Cu – до 640 К. Під час наступного нагріву електричний опір плівки Al-Cu-Fe збільшується на шість порядків (до $6 \cdot 10^7$ Ом/кв). Навпаки, електричний опір плівки Al-Co-Cu зменшується в ~ 2 рази. Після охолодження до кімнатної температури опір плівки Al-Cu-Fe складає $\sim 3 \cdot 10^5$ Ом/кв, а плівки Al-Co-Cu – 8,7 Ом/кв. Зроблено висновок, що плівка Al-Cu-Fe є більш перспективним матеріалом для виготовлення прецизійних високоомних резисторів.