

A Study on the Possibilities of Obtaining Intermetallic Coating of Al-Cu and Cu-Zn Systems Deposited on Metal Carrier

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Abstract

The research aimed at investigating the possibility of obtaining thin intermetallic films, which can be used as thermal control coatings for spacecraft. The coating films build the passive part of the overall thermal control system of a spacecraft, thus they must provide strong adhesion with the carrier material and have high functional characteristics, optical ones in particular. The study concerned issues of synthesis of stable intermetallic phase of Al_4Cu_9 , Al_2Cu , Cu_5Zn_8 on aluminum and copper carriers by magnetron layer-wise sputtering of reagents. They have looked into patterns of emergence of intermetallic coating applying modes of "rapid" and "slow" sputtering of reagents, applying various thicknesses of sputtered layers of reagents, temperature of the carrier, heat treatment of sprayed coatings. Incomplete and complete modes of synthesis of intermetallic coverage have been discovered. Obtained cross-sectional images of coatings, outcomes of microanalyzer scanning of reagent distribution through the thickness of sputtered coatings, microhardness values, optical absorption and emission ratios, unit of electric resistances, adhesion to the carrier. Obtained prototypes of intermetallic thermostatic coatings could be used in "solar reflectors" and "solar absorbers" classes. The results of measurements of optical and strength characteristics revealed that the intermetallic films can be used not only as thermostatic coatings for space technology, but also in general mechanical engineering, due to high-end mechanical properties.

1. Introduction

When in Earth orbit, a spacecraft (SC) is exposed to strong thermal radiation of the Sun leading to some individual surfaces heating up to +150 °C. When at the shadow side of the orbit, SC surfaces cool down to -150 °C. Proper operation of SC electronics require comfortable temperatures close to the Earth ones. Required temperature is provided by thermal regulation system of SC, which protects electronics from overheating by solar radiation and heats colder parts of the working area. An important part of any thermal regulation system consists of thermostatic coatings (TSC) of a SC external surfaces [1]. TSCs are characterized by the following optical parameters: coefficient of absorption of solar radiation – A_s , degree of blackness or emissivity – ε [2].

The most important role belongs to the TSCs "Solar reflectors" (also called "white") and "Solar absorbers" ("black") classes [2]. "Solar reflectors" are designed to combat overheating of an SC by solar radiation.

Body temperature under the influence of direct solar radiation can be calculated by using the following connection [3]:

$$T = (J_s A_s / \sigma \varepsilon)^{1/4} \quad (1)$$

where T – equilibrium heating temperature, J_s – solar radiation heat flux, σ – Stefan-Boltzmann constant.

Connection (1) demonstrates that minimizing temperature of SC heating by solar radiation is achieved by minimization of coefficient ratios of A_s/ε TSC, meaning the less A_s and the more ε is,

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the less the body temperature becomes. TSC criterion for "Solar reflectors" class is the ratio of [4]:

$$A_s/\varepsilon \leq 1 \quad (2)$$

TSC class "Solar absorbers" is designed for maximum absorption of solar energy, which further is transported inside the SC to provide thermal energy to individual units. Such TSC require large values of A_s/ε .

TSC requirements are as follows: required optical characteristics of A_s and ε , resistance to oxidation by oxygen ions and space plasma, resistance to ionizing radiation of the Sun, electrical conductivity for removal of static electricity, strength (hardness and adhesion to the carrier) to counteract space particles [3].

"White" paints for TSC are produced from oxide powders and resins; they are sputtered upon the surface of the SC parts. «White» TSCs come also in the form of organic films or glass sputtered from the backside of the metal. "Black" paints for TSCs are made from black pigments and resins.

It is known that in the space outer space factors (OSF) have a strong degrading impact on TSC leading to significant changes in the A_s coefficient, in this case, as it turned out, the ε coefficient remains virtually constant.

Table 1 shows examples of "white" industrial TSC, as well as their A_s values in the conditions of outer space. As the data shows (experiments over 5 years, whilst SC fly for 15 years), TSC degradation especially actively happens under geostationary orbit conditions, it leads to gradual heating of SC [1].

Experts believe that new TSC classes, particularly "Solar reflectors" class with stable thermal radioactive properties at long-term service when applied to SC in space, are one of the most important priorities in the space industry of the 21 century [4]. Creation of such TSC will increase lifetime of an SC by more than 15 years.

Because of that there have been being conducted continuous search of innovative TSC types. Intermetallic compounds are of outlook interest in regards with TSC, they are distinguished by the variety of colors, durability, and corrosion resistance to oxidation by oxygen, and conductivity [5, 6]. Literature and patent study showed a lack of research in the area of applying intermetallic compounds as TSC.

This paper topic was defined for this same reason; it is devoted to obtaining intermetallic coatings and study of their characteristics as TSC test pieces. The coatings were created by magnetron

Table 1
Experimental data on degrading of "white" TSC at SC under outers pace conditions
(L – low orbits, GSO – geostationary orbit)

	TSC type	Composition, thickness, μm	Temperature range, $^{\circ}\text{C}$	Initial coefficients		As condition during the operation of an SC in orbits			
				A_s	ε	L < 1 000 km		GSO ~ 36 000 km	
						3 years	5 years	3 years	5 years
LCP	AK-512	White enamel, 100 μm	-150 +150	0.3	0.85	0.32	0.36	0.62	0.67
	AK-573	Same	-150 +200	0.24	0.85	0.21	0.21	0.45	0.5
Ceramic	TS-SO-1	Aqueous solution of potassium silicate, 240 μm	-100 +150	0.19	0.92	0.16	0.17	0.45	0.5
	TP-SO-10		-100 +150	0.18	0.9	0.09	0.09	0.57	0.6
Thin film	OSO-A	Glass plate K-208 with metallic 200 μm	-100 +100	0.13	0.9	0.17	0.18	0.26	0.28
	OSO-S		-100 +100	0.13	0.85	0.17	0.18	0.31	0.32
	SOT 1-A-100	Film F-4MB	-100 +100	0.17	0.8	0.13	0.13	0.37	0.42
	SOT-1-100		-100 +100	0.11	0.85	0.13	0.13	0.29	0.35

sputtering on a copper or aluminum carrier of alternating thin (nanoscale) layers of pairs of metal reagents Al-Cu and Cu-Zn. The goal is to obtain coatings made of sustainable intermetallic Al_4Cu_9 , Al_2Cu , Cu_5Zn_8 per the diffusion reaction mechanism. This technique is known as the method of coating flexible surfaces with strengthening intermetallic coatings [7], however, the possibility of using such coatings as TSC has not been investigated.

2. Experimental

Intermetallic coatings were generated using magnetron unit owned by the "Center for Earth Sciences, metallurgy and refining" JSC (CESMR) the unit schema is shown in Fig. 1. Aluminum with 99.995% purity, copper with 99.997% purity and zinc with 99.995% purity were selected as targets for sputtering. Target diameter was 110 mm, its thickness was 5 mm.

There have been implemented two regimes of metal spraying: rapid and slow with thinner layers of reagents. Current-voltage characteristics of magnetron for these modes are presented in Table 2. To calibrate the magnetron one common admission about the similarity of thicknesses of magnetron metal sputtering on metal and glass carrier have been considered. Glass carrier gives the opportunity to measure the thickness of sprayed layer using atomic force microscope JSPM-5200, sputtering time was measured with a chronometer. Thickness calibration results for Al and Cu, Zn for rapid and slow coating modes are show in Figs. 2 and 3.

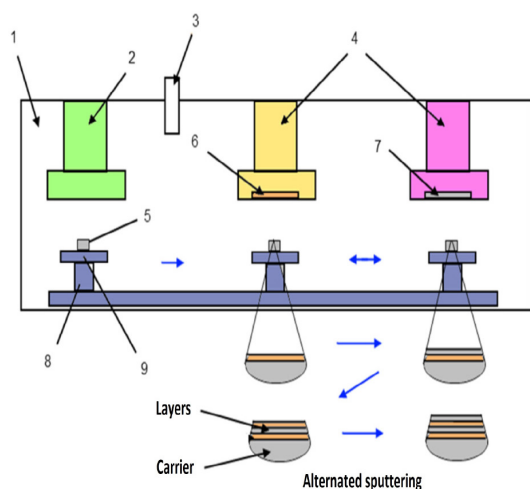


Fig. 1. Diagram of magnetron unit owned by CESMR: 1 – vacuum chamber; 2 – ion cleaning system; 3 – argon system; 4 – magnetrons; 5 – carrier; 6 and 7 – targets for sputtering, pumping system; 8 – sliding system; 9 – carrier holder.

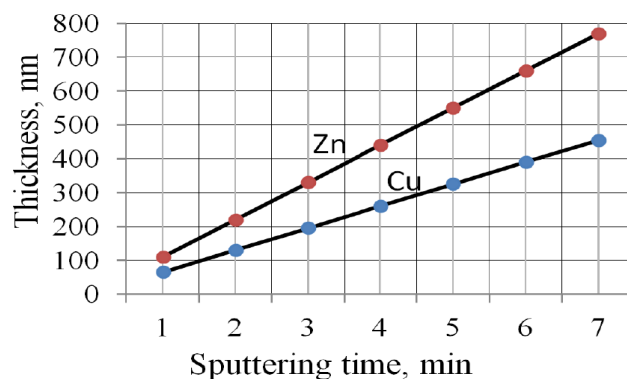
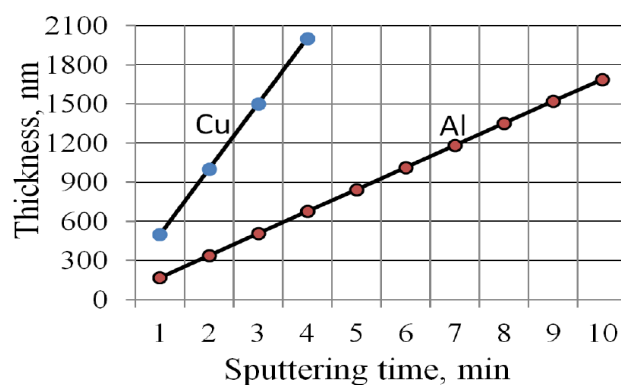


Fig. 2. Thickness calibration depending on sputtering time for one layer at rapid coating mode.

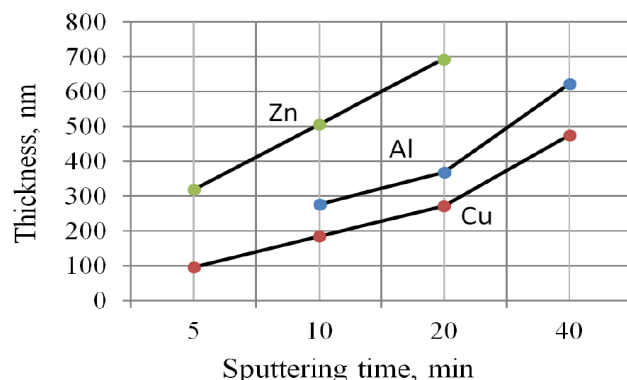


Fig. 3. Thickness calibration depending on sputtering time for one layer at slow coating mode.

Sputtering preparation process was carried out by vacuuming the operating chamber, cleaning the surface of the target and the carrier by ionic treatment, and setting voltage and current characteristics. Metal carrier coating of required thickness was performed according to the calibration data in Fig. 3, according to the sputtering time. It was expected to achieve the necessary phase by adjusting the thicknesses (mass) of reagent layers. The objective was to obtain sustainable intermetallic phases Al_4Cu_9 and Al_2Cu in Al-Cu coatings system, phase Cu_5Zn_8 in the Cu-Zn system. The objective should be achieved by sputtering stoichiometric ratio of the reagent masses, obtainable by choosing the

thickness ratio of coating layers. Reagent thickness ratio was calculated per the following connections: for phase Al_4Cu_9 – $h_{\text{Cu}} = 1.58h_{\text{Al}}$; for Al_2Cu – $h_{\text{Cu}} = 0.353h_{\text{Al}}$; for Cu_5Zn_8 – $h_{\text{Cu}} = 0.482h_{\text{Zn}}$. Experimenting plan is given in Table 2.

The resulting samples were tested to identify the following characteristics of the coatings: cross-sectional images and distribution of concentrations of reagents coated in terms of thickness (using micro analyzer of an electron-scanning probe JXA-8230), phase composition by frontal surface X-ray diffractometer D8 ADVANCE [9], values of microhardness using microhardness-meter PMT-3 (by indentation of a diamond pyramid indenter under a load of 5 g into the coating surface), light absorption ratios AS for wavelength range of 250–2400 nm using spectrophotometer Shimadzu UV-3600, emissivity ε using infrared pyrometer UNIT UT-302B for wavelength range of 800–1400 nm. Coating adhesion to the carrier was evaluated by scratching (GOST 9.302-88 "Metallic and non-metallic mineral coatings. Methods of control"), electric tester was used to evaluate conductivity.

The coating obtained by rapid sputtering were studied at room temperature, as well as after treating them with heat and subsequent cooling: for the Al-Cu system at 450 °C for one hour, for the Cu-Zn system at 350 °C for one hour. The coating obtained by slow sputtering were tested at room temperature, and some extra heat treatment was applied in some cases, at a temperature of 150 °C for one hour.

3. Results and discussion

3.1. Coatings obtained by rapid sputtering

3.1.1. Al-Cu System

Experiments were conducted using an aluminum carrier. Figure 4 shows a snapshot of the cross-section of coating # 1 (Al-Cu System), as well as the distribution pattern of concentration of Al and Cu reagents across the coating thickness. Snapshot reveals strips of reagents, as well as the blurred boundary between the carrier and the coating (the result of spreading reagents during

Table 2
Experimenting plan

Coating №	Magnetron setting mode	Current-voltage characteristics of the magnetron	Synthesized phases	Carrier temperature, °C	Coating layer thickness, nm	Number of layer pairs
1	Rapid Sputtering	Al - 360 V, 1A Cu - 400 V, 2A	Al_4Cu_9	25	Al – 1200 Cu – 1900	4
2			Al_4Cu_9	25	Al – 600 Cu – 950	6
3		Zn-480V, 0.27A CU-371V, 1A	Cu_5Zn_8	25	Cu – 260 Zn – 550	4
4			Cu_5Zn_8	25	Cu – 260 Zn – 550	6
5	Slow Sputtering	Cu – 330 V, 0.2A Al – 300 V, 0.12A	Al_2Cu	200	Al – 276 Cu – 96	2
6			Al_2Cu	200	Al – 368 Cu – 185	2
7			Al_2Cu	200	Al – 610 Cu – 220	2
8			Al_2Cu	200	Al – 610 Cu – 220	4
9		Zn – 300V, 0.1A Cu – 330 V, 0.2A	Cu_5Zn_8	200	Cu – 185 Zn – 320	2
10			Cu_5Zn_8	200	Cu – 217 Zn – 507	2
11			Cu_5Zn_8	200	Cu – 475 Zn – 694	2
12			Cu_5Zn_8	200	Cu – 217 Zn – 507	4

polishing the sample, as well as mutual diffusion of the coatings and the carrier), average thickness is estimated at 12.2 μm that is close enough to the calculated thickness of 12.4 μm . Data of Al and Cu concentrations across the thickness indicate that in reality the coating consists of Al and Cu layers, however concentrations of these elements in the layers is slightly lower than the one achieved in sputtering mode (comparison of the Al concentration in the carrier and the coating), which means partial reaction of intermetallic formation. The results indicate that sputtering reagent layers onto the carrier keeps their individuality, the reaction of intermetallic formation was not completed. To complete the synthesis of intermetallides in the coating, it requires to be treated with heat.

Coating # 2 displayed results similar to the results obtained for coating # 1. To trigger the reaction of intermetallic formation the coating # 2 was subjected to heat treatment in vacuum at a temperature of 450 $^{\circ}\text{C}$ for one hour. Snapshot of cross-section of coating # 2 after heat treatment is shown in Fig. 5.

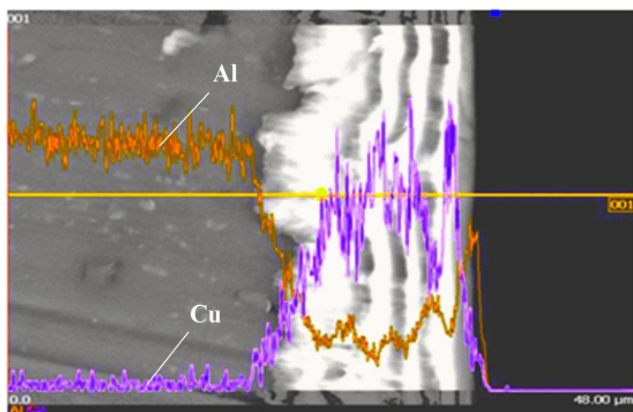


Fig. 4. Cross-sectional snapshot of coating # 1. Light section – the coating, section to the left – the carrier. Coating thickness is 12.2 μm .

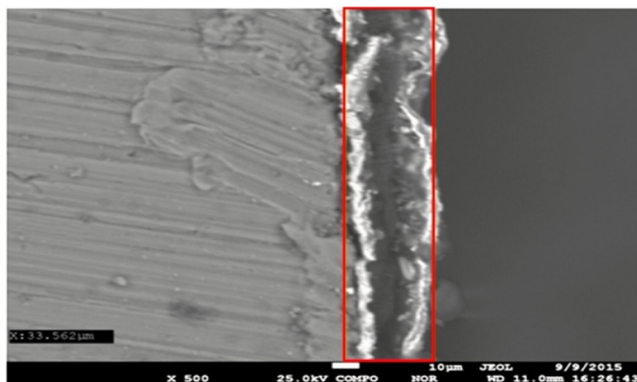


Fig. 5. Snapshot of cross-section of coating # 2 after heat treatment. Coating thickness is 33.5 μm .

It can be seen that the sandwich structure of the coating is gone, the coating surface and the border with the carrier have light stripes, presumably the stripes indicate presence of unreacted Cu. After thermal treatment the coating thickness has tripled, the reason for this phenomenon is not clear.

Table 3 shows the results of XRD of coating # 2.

Table 3

XRD Data for coating #2 with aluminum carrier

Phase	Formula	Content, %	
		prior to heat treatment	after heat treatment
Aluminum	Al	69.2	81.8
Aluminum in copper (solid solution)	Al-5.6%, Cu-94.3	22.4	5.4
Copper in aluminum (solid solution)	Al-99%, Cu-1%	8.4	7.2
Intermetallides	Al_4Cu_9	-	5.7

For interpretation of the data given in the Table 3 it must be noted that the frontal radiography of thin coating penetrates the aluminum carrier. Thereby, the "Aluminum" phase refers to the carrier, the other phases can be attributed to the coating.

It can be seen that before the heat treatment, of the coating, the Al_4Cu_9 phase is practically not visible, it appears after the heat treatment. Incompleteness of the intermetallic formation reactions in the coating is also obvious due to remnants of Al and Cu in the form of solid solutions. The most likely cause of incomplete synthesis is the excessive thickness of the reagent layers. Coating hardness after heat treatment is low – about 40 MPa.

3.1.2. Cu-Zn System

The results of XRD of coating #3 are shown in Table 4.

"Aluminum" phase in Table 4 should be attributed to the carrier, the remaining phases are related to the coating. It is obvious that a significant number of intermetallic Cu_5Zn_8 is formed during sputtering the coating; however, the process remains incomplete. Heat treatment of the coating at 350 $^{\circ}\text{C}$ for one hour led to formation of a number of unintended and fragile intermetallides, including aluminum from the carrier. During the heat treatment, the coating increased considerably in thickness.

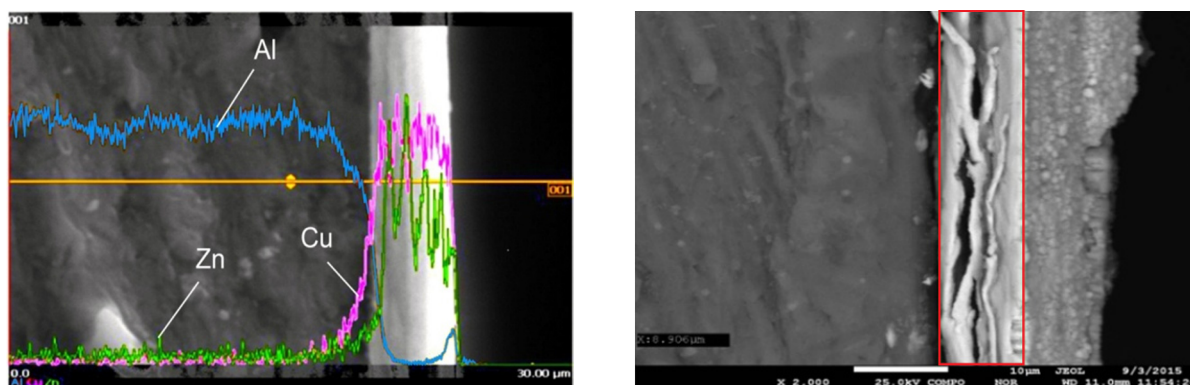


Fig. 6. Cross-sectional images of the coating # 4.

Table 4
The results of XRD of coating #3

Phase	Formula	Content, %	
		prior to heat treatment	after heat treatment
Aluminum	Al	71.6	64.8
Intermetallides	Cu_5Zn_8	11.4	-
Aluminum in copper (solid solution)	Al-5.6%, Cu-94.3%	9.9	-
Copper in aluminum (solid solution)	Al-99%,- Cu-1%	7.1	-
Intermetallides	$\text{Al}_{10.0565}\text{Cu}_{0.9434}$	-	16
Intermetallides	$\text{Cu}_{1.05}\text{Zn}_{0.95}$	-	10.6
Intermetallides	$(\text{Al}_{0.99}\text{Cu}_{0.01})$	-	5.7
Intermetallides	$\text{Cu}_{0.70}\text{Zn}_2$	-	1.6
Intermetallides	$\text{Cu}_{0.7}\text{Zn}_{0.3}$	-	1.3

Figure 6 shows cross-section snapshot of coating # 4. Here as well there are the results of the micro analyzer scan of presence of Zn, Cu reagents as well as Al from the carrier. There are clearly visible layers of reagents, as well as the mutual diffusion penetration of carrier and coating elements. After thermal treatment of the sample in vacuum at 350 °C for one hour (Fig. 6b), the coating thickened from the original value of 4.8 μm to 8.9 μm, along with formation of cracks and voids. Thicker coatings during thermal processing was caused by a complex process of chemical and physical interaction of intermetallic Cu_5Zn_8 , as well as residual Cu and Zn, with aluminum carrier.

Thus, during sputtering coatings # 3 and 4 the synthesis of intermetallic phase Cu_5Zn_8 happens only partially, considerable quantities of reagents remain in the form of solid solutions. Completion

of intermetallic formation is hindered by thick layers of reagents. When heat-treated, under the influence of active aluminum from the carrier, primary sputtered products transform into a number of unstable intermetallides, the coating thickens and cracks and voids occur. Hardness of the coatings is low – 41 MPa.

3.2. Coatings obtained by slow sputtering

Experiments were conducted on a copper carrier according to experimental plan shown in Table 2. Sample coatings # 5–12 were obtained. Coatings from this series had smaller thickness than the ones in rapid sputtering mode, which complicated research of the distribution of reagents across the coating thickness, as well as getting the XRD. The real method of characterizing the coating is assessing microhardness. Table 5 shows test results.

Experimental results have shown:

- coatings # 5–7, 9–11, which were obtained by combining 2 pairs of layers and were less than 2.9 μm thick, have the same microhardness after sputtering as the original metal microhardness, and after thermostating at 150 °C for one hour, their microhardness increases by an order. These data indicate that intermetallides do not form during sputtering; they are synthesized during heat treatment;
- coatings # 8 and 12, obtained from 4 pairs of reagents and having thickness equal to 2.9 μm or more, have a very high microhardness of 6000 MPa immediately after sputtering, heat treatment does not change this value. The resulting value of microhardness is normal for intermetallides [6], hence, it can be concluded that intermetallic compounds form during sputtering.

These data suggest that there is a certain critical thickness of coating sputtered to carrier. If thickness value is less than this critical thickness, then

Table 5
Strength characteristics of coatings obtained in slow sputtering mode

Coating #	Anticipated phase composition	Number of sputtered layer pairs	Coating thickness, μm	Microhardness, MPa	
				before heat treatment	after heat treatment
5	Al_2Cu	2	0.74	204	2430
6	Al_2Cu	2	1.1	220	2570
7	Al_2Cu	2	1.66	264	2600
8	Al_2Cu	4	3.32	6000	6000
9	Cu_5Zn_8	2	1.01	85	970
10	Cu_5Zn_8	2	1.45	170	1210
11	Cu_5Zn_8	2	2.34	185	1340
12	Cu_5Zn_8	4	2.90	2600	2400

intermetallic compounds form poorly. The most likely cause of this phenomenon is the effect of carrier, which diffuses with the sputtered layer and forms a solid solution with the latter, thereby passivates chemical activity of the reagent. Reaction triggering requires heat treatment. Increase in coating thickness leads to carrier influence weakening and acceleration of the intermetallide formation reaction.

To generalize the results, the following conditions of synthesizing intermetalline coatings during magnetron sputtering of reagents can be derived:

- reagent sputtering should be slow with the magnetron setting according to the order quoted in Table 2 and Fig. 3;

- carrier must be heated to the temperature of approximately $200\text{ }^\circ\text{C}$,

- thickness of sputtered reagent layer should be fairly thin – it should not exceed 610 nm ,

- the number of pairs of sputtered reagents must be at least 4, with a total thickness of coating more than $2.9\text{ }\mu\text{m}$.

Strictly speaking, these conditions only apply to the reviewed coatings systems; nonetheless they can be used as benchmarks for other systems.

3.3. Optical properties of coatings

There have been defined optical coefficients A_s and ε for coatings # 2 and 4, which were obtained in the rapid sputtering mode, as well as for coatings # 8 and 12 from the slow mode. Figure 7 shows examples of experimental data of reflection coefficient R (%) depending on the lengths of optical waves for coatings # 8 and 12.

Average integral A_s ratio is defined in the wavelength range of $240\text{--}2400\text{ nm}$. As Calculation was based on the ratio $A_s = 1 - R/100$. Experimental re-

sults are summarized in Table 6. Resistivity unit of individual phases was taken from cited sources [8, 9], followed by validation using electric resistance coating tester, which confirmed the order of these data. All coatings have sufficient electrical conductivity to dissipate any static electricity. The coatings are diffused and spliced with the carrier, which is well displayed by Figs. 5 and 6. Scratching the coating does not lead to its scaling from the carrier.

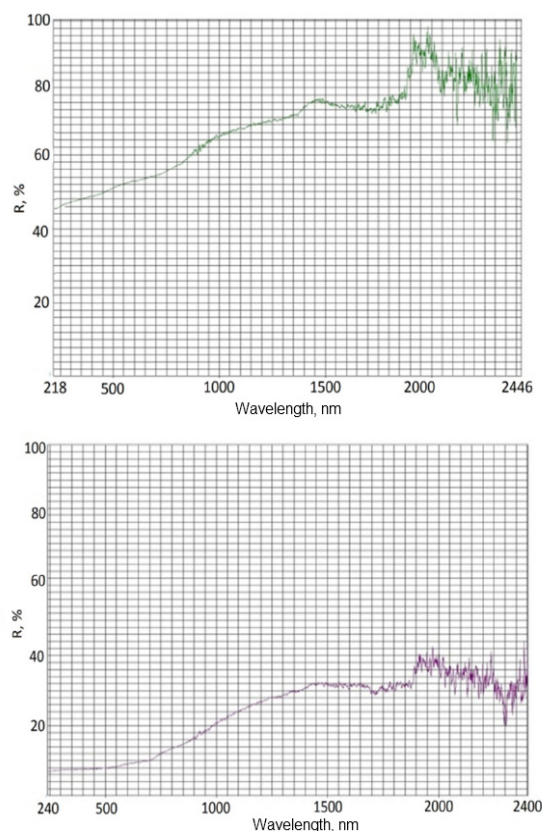


Fig. 7. Reflection coefficient of the coatings by wavelength.

Table 6
Optical, electrical and adhesive characteristics of coatings

Coating #	Sputtering mode	Phase composition	Microhardness, MPa	A_s	ε	A_s/ε	Resistivity, micro Ohm/cm	Adhesion to carrier
2	Rapid	Al_4Cu_9	43	0.82	0.44	1.96	14.2 [8]	Excellent
4		Cu_5Zn_8	42	0.79	0.38	2.1	11.1 [9]	Excellent
8	Slow	Al_2Cu	6000	0.3	0.49	0.61	7.0 [8]	Excellent
12		Cu_5Zn_8	2600	0.79	0.38	2.1	11.1 [9]	Excellent
		Al	263				2.4 [8]	
		Cu	818				2.0 [8]	
		Zn	570				5.9	

The values in Table 6 indicated that the following coatings can be taken as TSC prototypes:

- coating # 8 in the "Solar reflectors" category ("white"),
- coating # 12 in the "Solar absorbers" category ("black"),

Indeed, coating # 8 meets the requirement (2), has very high hardness, sufficient electrical conductivity. Intermetallide phases are corrosion resistant [10–11]. Coating # 12 absorbs energy from the Sun well, radiates it moderately, is durable and has sufficient electrical conductivity. Further research could focus on improving characteristics of the coatings.

Findings of this research shall be of particular interest to separate task of creating strengthening intermetalline coatings in mechanical engineering.

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4. Conclusions

1. The aim of present work is the synthesis of thermostatic coating prototypes for SC, which are to be based on metal carrier and consist of sustainable intermetalline phases Al_4Cu_9 , Al_2Cu , Cu_5Zn_8 . In order to synthesize it was decided to use the method of reaction diffusion among alternating layers of two reagents, which are to be sputtered upon a metal carrier by a magnetron and have to have thickness values compliant to stoichiometry of the phase being synthesized.

2. There have been used 2 reagent sputtering modes: "rapid" with the carrier temperature being equal to room temperature, and "slow" with a carrier heated to 200 °C. Configuring the magnetron and production of experimental samples of coat-

ings was conducted according to experimental plan set out in Table 2.

3. Modes of incomplete and complete synthesis of intermetallic coating were identified. Authors formulated conditions for intermetallic synthesis during reagent sputtering.

4. Acceptable results in terms of coating quality were obtained during the mode of slow sputtering. At the same time:

- coating # 8, which is 3.3 μm thick, with phase compound of Al_2Cu , has high microhardness of 6000 MPa, optical absorption coefficient $A_s = 0.3$, emissivity $\varepsilon = 0.49$, color criterion $A_s/\varepsilon = 0.61$, electrical resistivity of 7 micro Ohm/cm, excellent adhesion to the carrier, according to the sources – high chemical resistance. This coating can be used as the prototype of the "Solar reflectors" TSC class;
- coating # 12, which is 2.9 μm thick, with phase compound of Cu_5Zn_8 , has high microhardness of 2600 MPa, optical absorption coefficient $A_s = 0.79$, emissivity $\varepsilon = 0.38$, color criterion $A_s/\varepsilon = 2.1$, electrical resistivity of 2.1 micro Ohm/cm, excellent adhesion to the carrier, according to the sources – high chemical resistance. This coating can be used as the prototype of the "Solar absorbers" TSC class.

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