



ORIGINAL ARTICLE

Kinetic Features of the Oxidation of Zinc Alloy TsAMSv4-1-2.5 with Indium Additions

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ABSTRACT

This paper presents the results of a study on the kinetic features of the oxidation of the zinc alloy TsAMSv4-1-2.5 with indium additions in the solid state. The high-temperature oxidation process was investigated by the thermogravimetric method, which yields reliable quantitative data on the change in sample mass during heating in a controlled oxidizing atmosphere. It was established that the introduction of indium exerts a substantial effect on the oxidation kinetics of the alloy under study and alters the character of oxide-layer formation on the surface of the material. It is shown that alloying with small additions of indium changes the oxidation rate and affects the energy parameters of the process. The kinetic characteristics of oxidation, including the values of the apparent activation energy, were determined, making it possible to assess the thermal stability of the materials studied at elevated temperatures. It was found that the oxidation mechanism of the alloys is satisfactorily described by a hyperbolic law, indicating a gradual decrease in the process rate as the oxide-film thickness increases. The results obtained are of practical interest for the development and use of zinc alloys with enhanced heat resistance and stable properties under high-temperature conditions. The paper presents experimental dependences, mass-change curves, kinetic curves, and calculated data confirming the reliability of the study performed.

Keywords: zinc alloy TsAMSv4-1-2.5; indium; oxidation kinetics; thermogravimetric method; activation energy; high-temperature oxidation.

1 | INTRODUCTION

The oxidation of metals and metal alloys is among the most important physicochemical phenomena that directly affect the operational reliability and service life of structural materials. When metals interact with an oxygen-containing medium, an oxide layer forms on their surface, the characteristics of which — structure, density,

continuity, porosity, and degree of adhesion to the substrate — determine the protective properties of the material under high-temperature exposure. Zinc exhibits high chemical reactivity toward oxygen and is capable of oxidizing even at relatively low temperatures, forming predominantly ZnO oxide [1]. The resulting oxide film has a comparatively loose and porous structure and therefore does not provide complete isolation of the metal surface from the external environment. This promotes further diffusion of oxygen and continued growth of the oxide layer [8, 9].

One of the effective methods for increasing the heat resistance and corrosion resistance of zinc-based materials is alloying with various elements. The introduction of aluminum, magnesium, copper, tin, indium, and other components can substantially change the oxidation rate, the phase composition of the resulting oxides, and the morphology of the surface layers [10–15]. The effect of alloying additions depends on their chemical activity and the specific features of their interaction with oxygen, which can lead either to acceleration or to inhibition of the oxidation processes.

Of particular interest is the use of indium as an alloying element for zinc alloys. Indium can alter diffusion processes in the solid state, influence the structure of the surface oxide films, and modify the kinetics of high-temperature oxidation. According to published data, the introduction of indium promotes a change in the structure of the oxide layer, an increase in its density, and an improvement in its adhesive properties, which can have a positive effect on the protective capacity of the coating [2].

Studies of zinc-based systems show that the presence of alloying components changes the phase composition of the surface and promotes the formation of more stable oxide compounds. Such phases can perform a barrier function, reducing the intensity of oxygen and metal-ion diffusion through the oxide film. As a result, not only the oxidation rate but also the mechanism of the process itself changes, in some cases acquiring a diffusion-controlled character [4, 7].

It has also been noted in the literature that the effect of indium on thermo-oxidative processes depends substantially on its content in the alloy and on the temperature conditions of the experiment. At low concentrations, the effect of alloying may be only slight, whereas an increase in the indium content leads to the formation of more stable and denser oxide structures that enhance the protective properties of the material [8–17].

Thus, an analysis of the published studies indicates a significant effect of indium on the kinetic parameters of the oxidation of zinc alloys. At the same time, the regularities governing the formation of oxide films in the TsAMSv4-1-2.5 alloy at various temperature regimes and indium concentrations have not been studied in sufficient detail, which determines the relevance of the present work.

The aim of this study is to establish the temperature regularities of the oxidation kinetics of the zinc alloy TsAMSv4-1-2.5 with indium additions, and to assess the possibility of creating materials with enhanced thermal stability and improved protective properties.

2 | MATERIALS AND METHODS

The oxidation kinetics of the alloys were studied using the method of continuous thermogravimetric weighing of samples, which is widely used in studies of the high-temperature corrosion of metals and alloys. This method makes it possible to determine the principal kinetic characteristics of oxidation and to assess the resistance of materials to an oxidizing environment. Its advantages include high measurement accuracy, the possibility of conducting experiments at elevated temperatures, and the relative simplicity of the experimental setup [3, 5].

The experimental setup included a resistance furnace (1) fitted with a thermal-insulation lining made of aluminum oxide (2). The upper part of the system was sealed with water-cooled covers (7), which provided a controlled gas

atmosphere inside the working chamber. The covers were fitted with openings for gas removal (3), installation of the thermocouple (5), and placement of the crucible (4) containing the sample under study.

The crucible was suspended by means of a calibrated molybdenum spring (12) connected to a platinum suspension (6). The spring system was located in a glass vessel (11) with a ground-glass lid (14), mounted on a separate support (13), which made it possible to minimize the effect of mechanical vibrations on the measurement results. To prevent thermal effects on the measuring part of the apparatus, special protective screens and a cooler (15), located in the lower part of the glass housing, were used. The change in sample mass was recorded using a KM-8 cathetometer from the deformation of the calibrated spring. Crucibles made of aluminum oxide, 25–26 mm in height and 18–20 mm in diameter, were used as containers for the alloys. Prior to the experiments, the crucibles were preliminarily calcined at 1000–1200°C in an oxidizing atmosphere until a constant mass was reached, which made it possible to eliminate the effect of adsorbed moisture and extraneous impurities.

Temperature control was carried out using a platinum–platinum–rhodium thermocouple, whose hot junction was located in the immediate vicinity of the surface of the alloy under study. A ceramic sheath made of aluminum oxide was used to protect the thermocouple. The specified temperature regime was maintained by a thyristor controller with an accuracy of $\pm 2^\circ\text{C}$, and the temperature was recorded with a PP-63 potentiometer. After the tests were completed, the apparatus was cooled to room temperature, after which a control weighing of the crucible with the sample was performed. The oxide layer was then removed from the metal surface and examined by X-ray phase analysis to determine the phase composition and chemical nature of the oxidation products [6, 18].

Fig. 1. Diagram of the apparatus for studying the kinetics of oxidation of metals and alloys

The experimental error was calculated from the initial formula for the oxidation rate constant (K):

$$K = (dg/dt) \cdot (1/s) \quad (1)$$

where t is the time, s is the surface area of the metal, and g is the mass of the metal.

From the sum of the relative errors, the relative error was calculated, which amounted to 9.63%.

$$\Delta K/K = \Sigma(\Delta g_i/g_i) \quad (2)$$

3 | EXPERIMENTAL RESULTS AND DISCUSSION

To study the effect of indium on the kinetics of solid-phase oxidation of the TsAMSv4-1-2.5 alloy, a series of samples with different indium contents in the range of 0.05–1.0 wt.% was prepared. Oxidation tests were carried out in an air atmosphere at temperatures of 523, 573, and 623 K. The experimental data obtained are presented in the form of kinetic curves and summarized in tables (Figs. 1–6; Tables 1, 2).

Analysis of the thermogravimetric dependences showed that the oxidation process of the alloys studied proceeds most intensively at the initial stage. During the first minutes of heating, an accelerated increase in the mass of the samples is observed, due to the active formation of the surface oxide layer. It was established that an increase in temperature promotes an increase in the oxidation rate. At the same time, an increase in the holding time leads to a gradual decrease in the intensity of the mass gain. After 15–20 minutes of the process, the oxidation rate noticeably decreases, and the change in mass acquires a practically stationary character, which indicates the formation of a protective oxide film and the transition of the process to a quasi-stationary region.

The kinetic parameters for the initial alloy without indium additions were determined from the initial portion of the experimental curves by drawing tangents from the origin of the coordinates. It was established that at 523 K the true oxidation rate is $3.48 \cdot 10^{-4} \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$, and with an increase in temperature to 623 K it increases to $3.60 \cdot 10^{-4} \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$ (Fig. 2a). The thermokinetic analysis performed showed that the value of the apparent activation energy of the oxidation process, calculated from the linear dependence in the $-\lg K - (1/T)$ coordinates,

is 136.8 kJ/mol (Table 1). The activation energy value obtained indicates the predominance of a diffusion-kinetic mechanism of oxide-layer growth on the alloy surface.

For samples containing 0.05 wt.% indium, it was established that within the temperature range studied the oxidation rate changes from $3.62 \cdot 10^{-4}$ to $3.74 \cdot 10^{-4}$ $\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$ (Fig. 2b). Calculation of the kinetic parameters showed that the apparent activation energy of the process for this composition is 119.9 kJ/mol (Table 1).

A comparison of the results obtained indicates that the introduction of small amounts of indium does not change the fundamental mechanism of high-temperature oxidation of the TsAMSv4-1-2.5 alloy, but leads to a certain decrease in the activation energy of the process. This is probably associated with a change in the structural state of the surface layer, an increase in the concentration of defects, and an increase in the chemical activity of the surface, which facilitates the interaction of the material with oxygen.

Table 1. Kinetic and energy parameters of the oxidation process of the zinc alloy TsAMSv4-1-2.5 with indium in the solid state

Indium content in alloy, wt.%	Oxidation temperature, K	True oxidation rate $K \cdot 10^4$, $\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$	Effective activation energy of oxidation, kJ/mol
0.0	523	3.48	136.8
	573	3.53	
	623	3.60	
0.05	523	3.62	119.9
	573	3.68	
	623	3.74	
0.1	523	3.66	115.5
	573	3.72	
	623	3.80	
0.5	523	3.72	111.1
	573	3.77	
	623	3.85	
1.0	523	3.77	108.3
	573	3.82	
	623	3.89	

The kinetic curves for the oxidation of the TsAMSv4-1-2.5 alloy containing 1.0 wt.% indium are shown in Fig. 2d. The studies were carried out in an air medium at temperatures of 523, 573, and 623 K. Analysis of the experimental data showed that at 523 and 623 K the true oxidation rate values are $3.77 \cdot 10^{-4}$ and $3.89 \cdot 10^{-4}$ $\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$, respectively. According to the results of the thermokinetic calculations, the apparent activation energy of the oxidation process for this alloy is 108.3 kJ/mol.

The thermogravimetric analysis data indicate that the introduction of indium into the zinc alloy promotes an increase in the intensity of the oxidation processes. As the indium content increases, a decrease in the apparent activation energy of oxidation from 136.8 to 108.3 kJ/mol is observed, which indicates that the interaction of the alloy with oxygen is facilitated. It was established that the oxidation rate of the alloys studied is of the order of 10^{-4} $\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$.

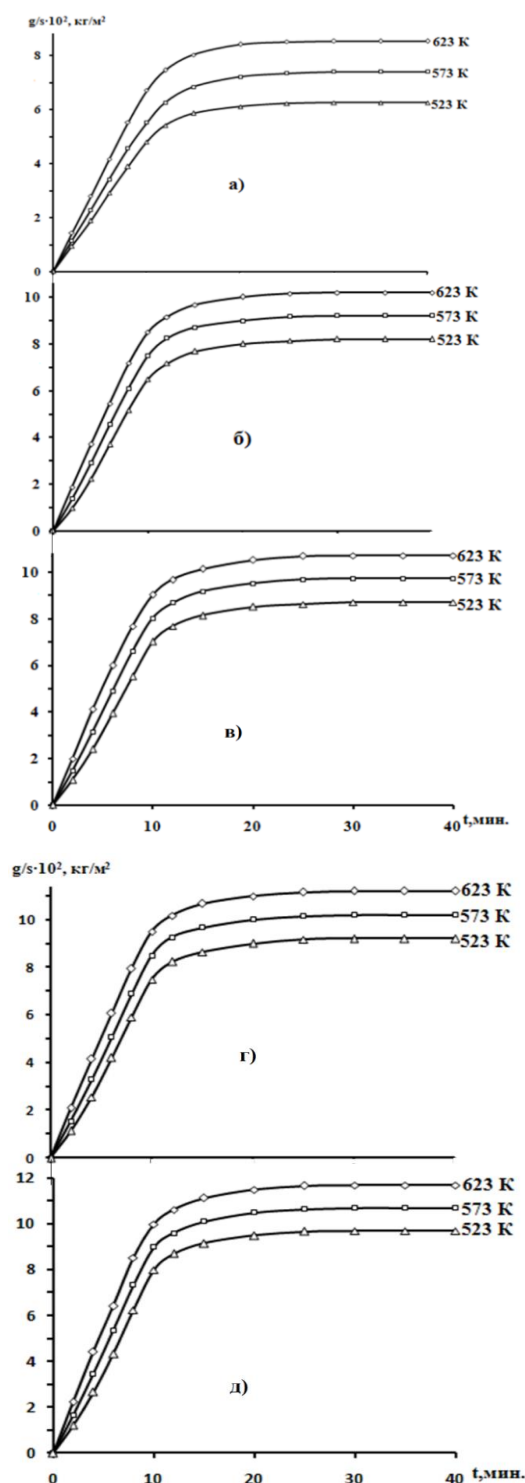


Figure 2. Kinetic curves of the oxidation process of the zinc alloy TsAMSv4-1-2.5 (a) with indium, wt.%: 0.05 (b); 0.1 (c); 0.5 (d); 1.0 (e)

The quadratic dependences of the oxidation kinetics of the TsAMSv4-1-2.5 alloys with an indium content of 0.05–1.0 wt.% in the $(g/s)^2$ – t coordinates are shown in Fig. 3. An examination of the curves obtained shows that they exhibit a pronounced nonlinear character, which indicates that the process proceeds according to a hyperbolic mechanism. This conclusion is confirmed by the values of the exponent n in the equation $y = kx^n$, which ranges from 1 to 3 (Table 2).

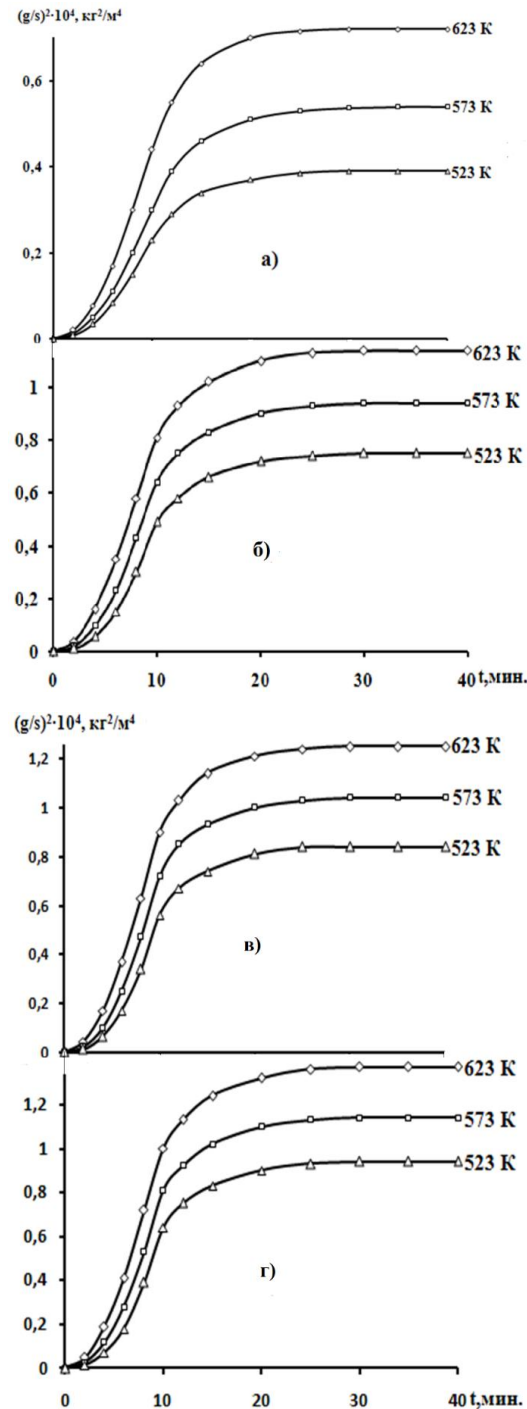


Fig. 3. Quadratic kinetic curves of the oxidation of the zinc alloy TsAMSV4-1-2.5 (a) with indium, wt.%, 0.1 (b); 0.5 (c); 1.0 (d)

Figure 4 shows the temperature dependences plotted in the $-\lg K - (1/T)$ coordinates for zinc alloys with an indium content of 0.05, 0.1, 0.5, and 1.0 wt.%. Analysis of the experimental data showed that these dependences are linear in character, which indicates that the oxidation process obeys the Arrhenius law. In this case, the lines corresponding to the indium-alloyed alloys lie above the curve for the unalloyed TsAMSV4-1-2.5 alloy, which indicates an increase in the oxidation rate upon the introduction of indium. The values of the apparent activation energy of the oxidation process were calculated from the slope of the straight lines in the $-\lg K - 1/T$ coordinates. The results of the calculations are given in Table 1.

Table 2. Results of the mathematical processing of the quadratic kinetic curves of the oxidation process of the zinc alloy TsAMSV4-1-2.5 alloyed with indium in the solid state

Indium content in alloy, wt. %	Oxidation temperature, K	Polynomials of the quadratic oxidation kinetic curves of the alloys	Correlation coefficient, R
0.0	523	$y^* = -0.05x^3 - 1.2 \cdot 10^{-2}x^2 + 0.591x^{**}$	0.992
	573	$y = -0.5 \cdot 10^{-3}x^3 - 1.5 \cdot 10^{-2}x^2 + 0.694x$	0.995
	623	$y = -0.5 \cdot 10^{-5}x^3 - 2.4 \cdot 10^{-2}x^2 + 0.874x$	0.994
0.05	523	$y = -0.5 \cdot 10^{-3}x^3 - 1.1 \cdot 10^{-2}x^2 + 0.743x$	0.986
	573	$y = -0.5 \cdot 10^{-5}x^3 - 2.6 \cdot 10^{-2}x^2 + 0.985x$	0.989
	623	$y = -0.5 \cdot 10^{-7}x^3 - 4.7 \cdot 10^{-2}x^2 + 1.211x$	0.993
0.1	523	$y = -0.05x^3 - 1.3 \cdot 10^{-2}x^2 + 0.804x$	0.985
	573	$y = -0.5 \cdot 10^{-7}x^3 - 3.2 \cdot 10^{-2}x^2 + 1.046x$	0.989
	623	$y = -0.5 \cdot 10^{-6}x^3 - 5.6 \cdot 10^{-2}x^2 + 1.331x$	0.994
0.5	523	$y = -0.05x^3 - 1.5 \cdot 10^{-2}x^2 + 0.867x$	0.984
	573	$y = -0.5 \cdot 10^{-4}x^3 - 3.2 \cdot 10^{-2}x^2 + 1.093x$	0.988
	623	$y = -0.5 \cdot 10^{-5}x^3 - 5.4 \cdot 10^{-2}x^2 + 1.363x$	0.993
1.0	523	$y = -0.05x^3 - 1.4 \cdot 10^{-2}x^2 + 0.904x$	0.982
	573	$y = -0.5 \cdot 10^{-4}x^3 - 3.5 \cdot 10^{-2}x^2 + 1.164x$	0.988
	623	$y = -0.5 \cdot 10^{-2}x^3 - 6.1 \cdot 10^{-2}x^2 + 1.460x$	0.993

y^* — specific weight gain of the samples (g/s);

x^{**} — oxidation duration of the samples (t).

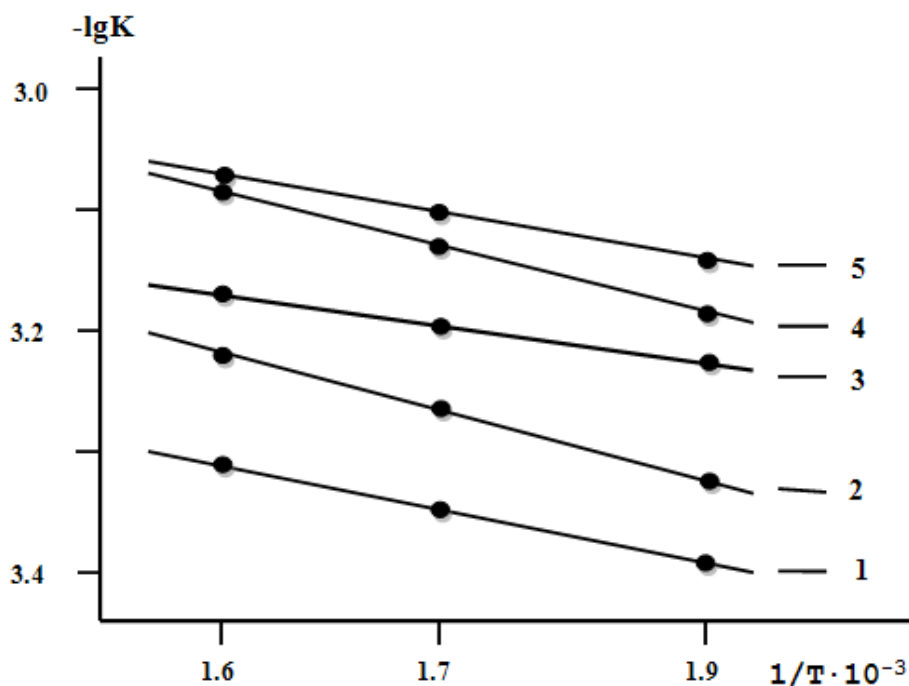


Figure 4. Dependence of $-\lg K$ on $1/T$ for the zinc alloy TsAMSv4-1-2.5 (1) containing indium, wt. %: 0.05 (2); 0.1 (3); 0.5 (4); 1.0 (5)

Based on the experimental data, isochrones of oxidation were constructed for zinc alloys with different indium contents (Fig. 5). Analysis of the resulting dependences showed that, both at an exposure time of 10 min and at a holding time of 20 min in an oxidizing atmosphere, a regular and smooth increase in the oxidation rate with increasing temperature is observed.

The established tendency is most pronounced within the temperature range studied and is confirmed by the decrease in the apparent activation energy of the oxidation process as the indium content in the alloy increases. This effect is presumably due to a change in the structure and phase composition of the oxide layers formed, which leads to an increase in their ionic conductivity and, consequently, to an acceleration of the oxidation processes.

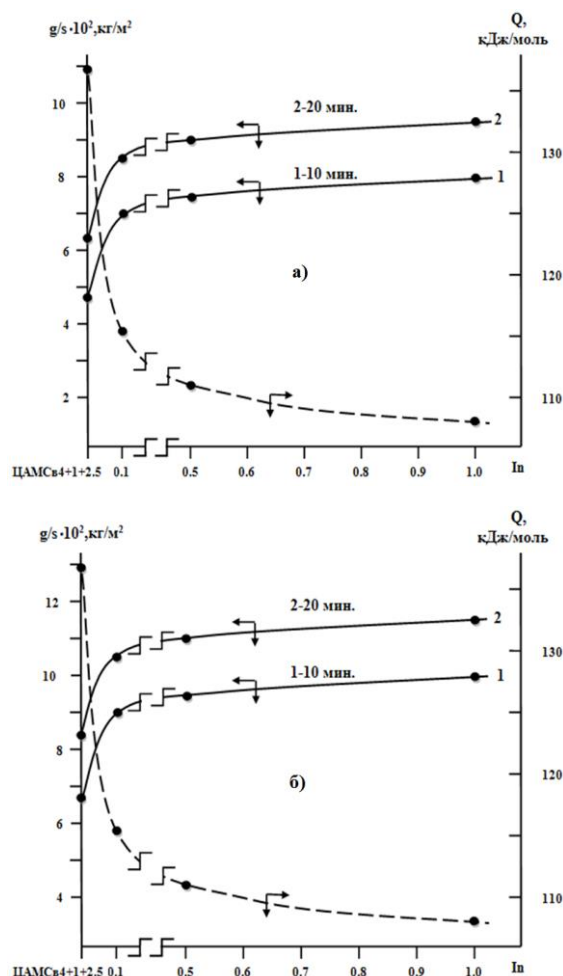


Figure 5. Isochrones of oxidation of the zinc alloy TsAMSV4-1-2.5 with indium at 523 K (a) and 623 K (b)

A dense oxide film acts as a protective barrier separating the metal substrate from gaseous oxygen. With sufficient access of oxygen to the surface of the oxide layer, the rate of high-temperature oxidation is determined by diffusion processes through the solid oxide layer formed. Such a film is characterized by high protective properties and, from a practical standpoint, represents an important factor in increasing the resistance of metals to oxidation.

The oxidation products of the zinc alloy TsAMSV4-1-2.5 with indium were investigated by X-ray phase analysis. As an example, Fig. 6 shows the X-ray diffraction patterns of the oxidation products of the TsAMSV4-1-2.5 zinc alloy alloyed with 1.0 wt.% indium. Investigation of the oxidation products of these alloys showed that the main oxidation products are Al_2O_3 ; $\text{Al}(\text{OH})_3$; Ga_2O_3 , $\alpha\text{-Ga}_2\text{O}_3$, $\text{CuO} \cdot \text{Al}_2\text{O}_3$; and ZnIn_2O_4 .

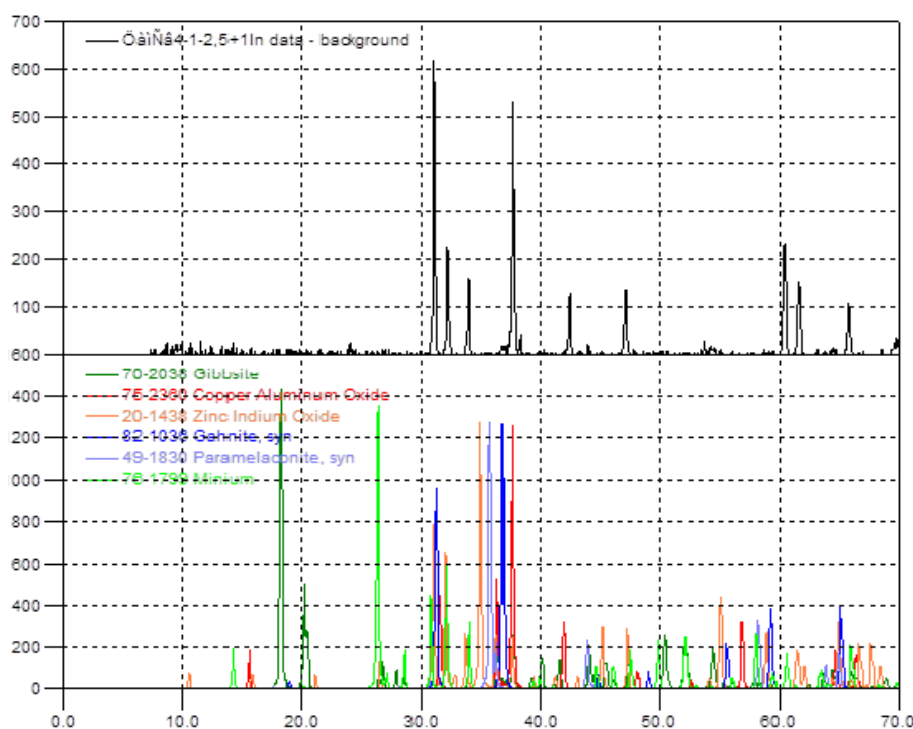


Figure 6. X-ray diffraction patterns of the oxidation products of the zinc alloy TsAMSv4-1-2.5 alloyed with 1.0 wt.% indium

4 | CONCLUSION

The thermogravimetric study of the oxidation kinetics of the zinc alloy TsAMSv4-1-2.5 with indium performed in this work showed that the addition of indium to the alloy substantially affects its oxidation processes in the solid state. It was established that even at a low indium content the structure and morphology of the oxide layer change: a less dense and more heterogeneous coating is formed, which promotes an increase in the rate of oxygen diffusion into the depth of the alloy. In addition, the presence of indium leads to a change in the phase composition of the surface oxide layer, as a result of which the stability of the protective oxide film decreases and the interaction of the material with the surrounding environment is enhanced.

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AUTHOR CONTRIBUTION

Conceptualization, I.N.G.; Methodology, P.N.A. and I.N.G.; Software, P.N.A.; Validation, P.N.A., I.N.G., A.E.B.; Formal Analysis, P.N.A., S.J.A.; Investigation, P.N.A., A.E.B.; Resources, I.N.G., A.E.B.; Data Curation, P.N.A.; Writing – Original Draft Preparation, P.N.A.; Writing – Review & Editing, I.N.G., S.J.A.; Visualization, P.N.A.; Supervision, I.N.G.; Project Administration, I.N.G. All authors have read and agreed to the published version of the manuscript.

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DATA AVAILABILITY

The datasets generated and analyzed during the current study are available from the corresponding author on reasonable request.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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