MECHANICAL PROPERTIES OF EXTRUDED MIXED COMPONENTS BASED ON ALUMINUM POWDERS AND TERNARY ALUMINUM-CONTAINING INTERMETALLIC COMPOUNDS SYNTHESIZED BY VARIOUS METHODS

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Abstract

This work is devoted to the study of the synthesis of various ternary intermetallic compounds based on aluminum in two alternative ways - self-propagating high-temperature synthesis (SHS) and mechanosynthesis (MS) and subsequent hot extrusion (HE) of pure aluminum powder mixtures as a matrix and the above-mentioned TIM as a hardening additive. Mechanical tensile tests showed an increase in tensile strength by 3.3 - 9.7% rel. (Except for one specimen, which stands out from the others) and an increase in microhardness by 9.3 - 49.1%. The microstructure analysis shows a uniform distribution of hardening particles over the matrix volume, which indicates a high uniformity of the distribution of TIM based on aluminum in the aluminum matrix.

Keywords: Hot extrusion of aluminum, intermetallic compounds, dispersed hardening, mechanosynthesis, powder metallurgy

1. INTRODUCTION

Powder metallurgy opens up wide possibilities for creating artificial ("synthetic") alloys, strengthened by double or triple IM, which can be realized by introducing pre-synthesized IM powders of a given chemical composition into pure aluminum (as well as other metals) powders or its low-alloyed alloys. As a result of such an introduction, the system will significantly differ from the equilibrium one and will contain significantly more than in accordance with classical phase diagrams, the number of strengthening phases. This content is determined only by the interests of the researcher, aimed at obtaining the best set of strength and service characteristics of the material after thermoplastic processing of the mixtures [1-3]. Triple intermetallic (TIM) compounds are found in many industrial alloys based on aluminum Aluminium and have a significant influence on their properties. They can be present in a structurally free state, enter the eutectic structure, or be in the form of scattered particles released during the dispersive decay of quenched alloys. One of the methods for producing aluminum-based intermetallic compounds, in addition to mechanosynthesis, is self-propagating high-temperature synthesis (SHS) of solid chemical compounds — a technological process for producing materials based on an exothermic chemical reaction of the interaction of the starting reagents in the form of combustion [4-6].

In this research, we study the patterns of the formation of ternary intermetallic compounds in various ways and their effect on aluminum alloys.

2. MATERIALS, METHODS AND DISCUSSION

2.1.1. Synthesis of Al – Ti – Zn systems

The synthesis was carried out according to a multistage scheme, including the mixing of titanium and aluminum powders, pressing the mixture, synthesis of the Al_3Ti intermetallic compound by SHS in vacuum. From the finished mixture, tablets Ø30 mm and a height of 10 mm were pressed at a pressure of 300 MPa. During SHS, a high-speed character of the Al_3Ti formation reaction was noted: after slow heating to 370 K for 160 minutes, the temperature of the space inside the retort rose to 1165 K within 10 minutes. The resulting material is an uneven structure consisting, according to the XRD data, of 70% Al_3Ti, the rest are oxides. From powders of 88.4Al-11.6Zn alloy (wt%), Zinc and synthesized Al_3Ti (all fractions 0 - 63 μm), batch mixtures corresponding to the stoichiometry of TIM $Al_{11}Ti_4Zn$ and $Al_{66}Ti_{25}Zn_9$ were prepared.

2.1.2. Synthesis of TIM systems of Al-Mg-X systems (where X– Mn, Cr)

The mixture corresponding to the stoichiometry of TIM $Al_{18}Mg_3Cr_2$ was prepared from powders of aluminum, PAM aluminum – magnesium alloy, and Al– β Cr alloy powder (all fractions are 0–63 μm). A mixture corresponding to the stoichiometry of TIM $Al_{18}Mg_3Mn_2$ was prepared from powders of aluminum, aluminum-magnesium alloy, and Al-27Mn alloy powder (all fractions 0–63 μm). Pressing tablets and conducting SHS repeated the procedure described in **2.1.1**. The system was heated uniformly to a temperature of 330 K (in the fore vacuum). A possible temperature jump, due to which it would be possible to judge the high energy release during the synthesis of Al – Mg – X TIM was not observed, in contrast to the Al – Ti – Zn synthesis. However, the obtained samples were very different from the original tablets: in addition to colour changes, the volume of tablets increased by 1.5 times. The XRD of the initial and obtained samples revealed the presence of various phases and their conversion from one to another during SHS (**Table 1** and **Table 2**).

2.1.3. Synthesis of TIM system Al-Mg-Cr

During SHS, a new TIM ($Al_{18}Mg_3Cr_2$) was synthesized, content of which amounted to 37% according to the XRD results. One can notice a sharp decrease in the aluminum content (almost three times) and complete dissolution of the phases $Al_{12}Mg17$, Al_8Cr_5 and $Al_{18.26}Cr_4$, which during the synthesis were transformed into $Al_{18}Mg_3Cr_2$ and $Al_{45}Cr_7$ (**Table 1**). SHS significantly changed (**Figure 1**) the diffraction patterns of the test sample.

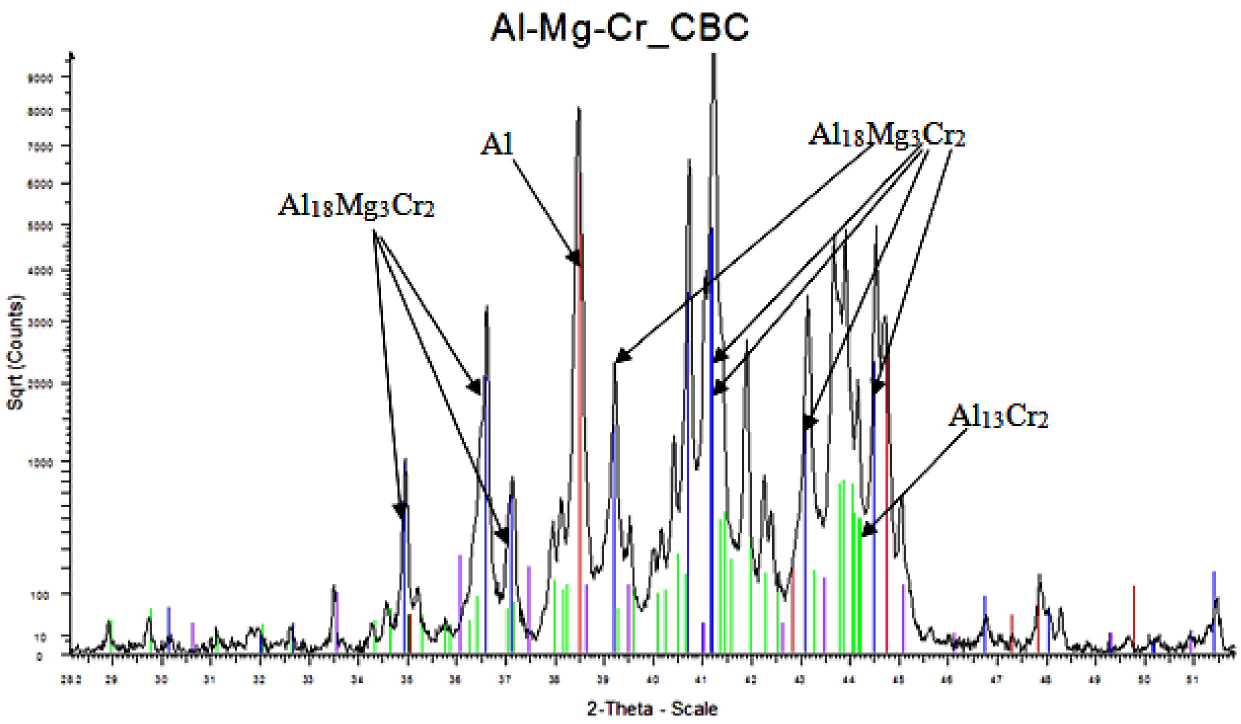

Figure 1 Diffraction patterns of the material of the Al-Mg-Cr system obtained as a result of SHS

Table 1 Comparison of the initial mixture of Al-Mg-Cr and the sample obtained by SHS

Powder sample		**SHS sample**	
Phase	Content (wt%)	Phase	Content (wt%)
Al	59.1	Al	20.3
$Al_{12}Mg_{17}$	25.7	$Al_{18}Mg_3Cr_2$	37.4
Al_8Cr_5	12.3	$Al_{13}Cr_2$	42.2
$Al_{18.26}Cr_4$	2.7	-	

Table 2 Comparison of the initial mixture of Al-Mg-Mn and the sample obtained by SHS

Powder sample		**SHS sample**	
Phase	Content (wt%)	Phase	Content (wt%)
Al	30.8	Al	23.4
$Al_{12}Mg_{17}$	37.6	$Al_{12}Mg_{17}$	10.8
Al_6Mn	19.8	Al_6Mn	32.8
$Al_{10}Mn_3$	2.3	$Al_{18}Mg_3Mn_2$	32.8
alpha-Mn	1.9	-	
$Al_{57}Mn_{12}$	7.3	-	

2.2. Obtaining TIM based on aluminum by mechanosynthesis (MS)

The batch was prepared in the same proportions as described in the materials of **Chapter 2.1.2**. The batch mechanosynthesis was carried out in the attritor according to the procedure with steel balls Ø 7-10 mm weighing 20 kg per 1150 g of powder mixture. Each powder mixture was processed for 8 ks. The MS mode was selected as follows: after 1 ks (16 min and 40 sec.), The machining took a break of 30 minutes to cool the system. Before the start of the MS and after every 2 ks of machining, a sample was taken and surfactant was added (0.6 wt% Stearic acid).

Diffraction patterns of the Al-Mg-Mn system are presented in **Figure 2**.

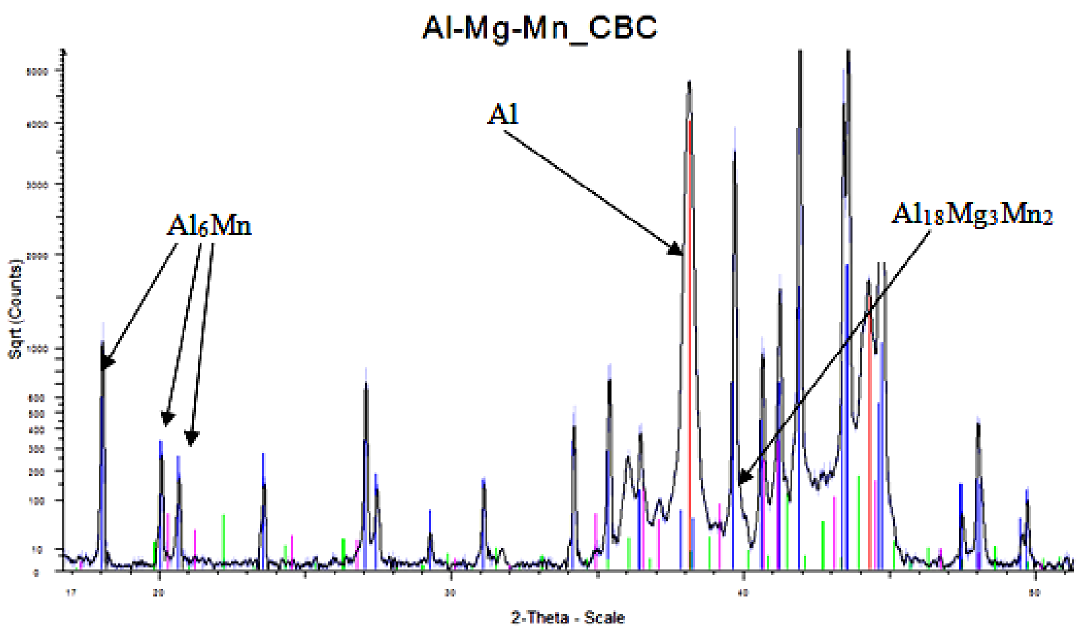

Figure 2 Diffraction patterns of the material of the Al-Mg-Mn system obtained as a result of SHS

2.2.1. Synthesis of Al-Ti-Zn Systems

The diffraction patterns of the TIM mixture powder corresponding to $Al_{66}Ti_{25}Zn_9$ and $Al_{11}Ti_4Zn$ at different activation times are shown in **Figure 3** and **Figure 4**. During the high-energy MS, the occurrence and constant, up to 74 wt. %, increase in the content of IMT $Al_{66}Ti_{25}Zn_9$. A similar increase in the content of IMT $Al_{11}Ti_4Zn$ was 54%.

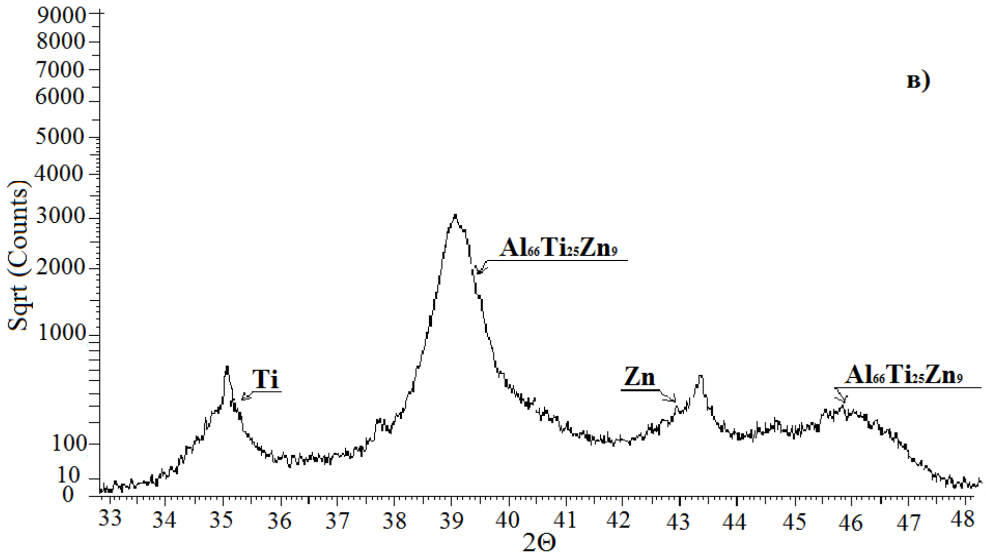

Figure 3 The diffractogram of the charge corresponding to the intermetallic $Al_{66}Ti_{25}Zn_9$ (the content of which is 74%)

Using SEM, the distribution of elements was definitely determined in the form of a finely dispersed structure, which is a homogeneous compound of aluminum, titanium, and zinc in concentrations corresponding to the composition of TIM $Ti_{25}Zn_9Al_{66}$ (**Figure 5**).

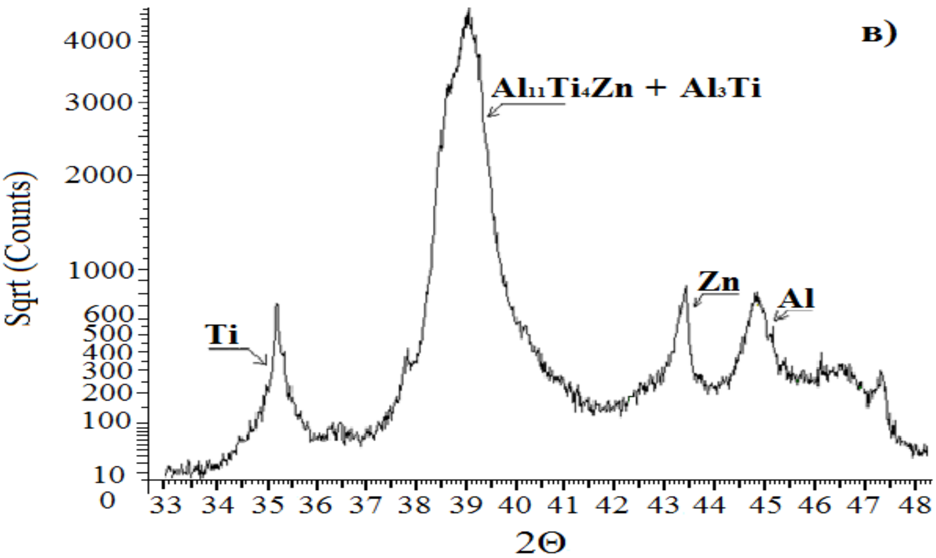

Figure 4 The diffraction pattern of the mixture corresponding to the intermetallic $Al_{11}Ti_4Zn$ (the content of which is 54%)

As a result of the work according to the results of **Chapter 2.1** and **Chapter 2.2**, new TIM-containing powders were synthesized, the phase composition of which is given in **Table 3**.

Table 3 The phase composition of the powders during the synthesis of TIM after the SHS and MS methods

Stage number	Synthesis method	Main phase	TIM content (%)	Other phases (%)					
				Al	$Al_{13}Cr_2$	$Al_{12}Mg_{17}$	Al_6Mn	Zn	Al_3Ti
1	SHS	$Al_{18}Mg_3Cr_2$	37	20	43	-	-	-	-
2	SHS	$Al_{18}Mg_3Mn_2$	34	23	-	11	32	-	-
3	MS	$Al_{11}Ti_4Zn$	53	24	-	-	-	9	14
4	MS	$Al_{66}Ti_{25}Zn_9$	75	14	-	-	-	4	7

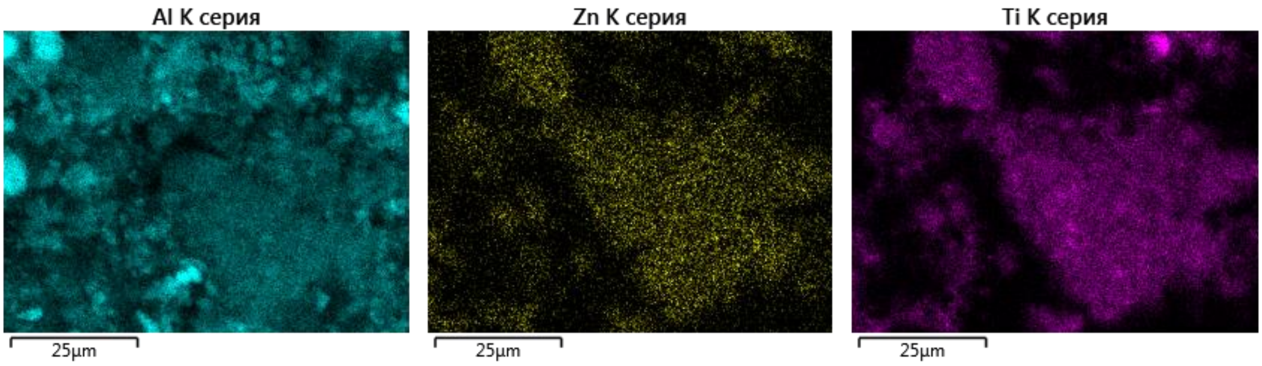

Figure 5 SEM image of the intermetallic compound $Ti_{25}Zn_9Al_{66}$

2.3. Obtaining compact semi-finished products by the method of GE powder mixtures

Hot extrusion of powders containing TIM phases obtained by performing steps 1 and 2 of this work was carried out in the following sequence **2.3.1** and **2.3.2**:

2.3.1. Preparation of a mixture of unalloyed aluminum powder and TIM - containing powder

The calculation of the required amount of TIM-containing phase from **Table 3** was made in such a way that the mass of "pure" TIM was 3 g/100 g of the charge (3 wt%) (**Table 4**).

Table 4 Preparation and creation of blends Al + TIM

TIM	The initial number of TIM in the samples (%)	The required amount in the mixture (%)	The total mass of the mixture (g)	% TIM of the total mass	Mass TIM (g)	Al powder mass (g)
$Al_{18}Mg_3Cr_2$	34	3	120	3.6	10.9	109.1
$Al_{18}Mg_3Mg_2$	37	3	120	3.6	9.7	110.3
$Al_{66}Ti_{25}Zn$	75	3	120	3.6	4.8	115.2
$Al_{11}Ti_4Zn$	53	3	120	3.6	6.7	113,3

2.3.2. Cold pressing of blends and auxiliary tablets of graphite

Briquetting the mixture into tablets took place under the same conditions for all powders: matrix diameter 30 mm, pressing pressure - 200 MPa. The process was as follows: a replaceable die, a graphite tablet, three briquettes of a compressed sample, a second graphite tablet, a press washer, and a punch were placed in the matrix. Then the matrix was heated for three hours and allowed to stand for 20 minutes.

2.4. Mechanical tensile tests

For tensile tests, two specimens from each bar were made of rods.

Table 5 Results of mechanical tensile testing of Al + TIM rods

Bar number	Chem. structure	**Tensile strength** (MPa)			Hardening (%)
		Sample 1	Sample 2	The average	
0	Al-powder	127	126	126	N/A
1	Al + (3%) $Al_{18}Mg_3Cr_2$	129	132	131	3.3
2	Al + (3%) $Al_{18}Mg_3Mn_2$	–	139	139	9.7
3	Al + (3%) $Al_{11}Ti_4Zn$	133	130	131	3.8
4	Al + (3%) $Al_{66}Ti_{25}Zn_9$	124	126	125	-1.1

From **Table 5** it can be concluded that the Al + (3 wt%) $Al_{66}Ti_{25}Zn_9$ composite does not exhibit the necessary strengthening effect. Aluminum rods containing 3% $Al_{18}Mg_3Cr_2$ and $Al_{11}Ti_4Zn$ slightly strengthen the matrix. A sample of Al + (3 wt%) $Al_{18}Mg_3Mn_2$ is released, which is almost 10% stronger than pure aluminum bar. It was at the rod of this chemical composition that one of the samples collapsed in the clamps along the thread. Both magnesium and manganese significantly increase the strength characteristics of aluminum, but both of them significantly embrittle the matrix (even when doped with a small amount). If we assume that the theory of the interaction of cast alloys is valid for composite materials "matrix + dispersed hardener", then such an increase in strength (and embrittlement) is theoretically justified.

2.5. Microhardness

Table 6 shows the results of microhardness measurements using a Hynistron TI 750 Ubi 1 Triboindenter scanning nanohardness tester. During the experiment, 7 measurements were carried out, two of which - the largest and smallest - were excluded from the calculation of the average value.

Table 6 The results of microhardness measurements of aluminum rods, dispersion hardened by intermetallic compounds

	Microhardness, HV							**The increase in hardness** (%)
Bar number	Mens. 1	Mens. 2	Mens. 3	Mens. 4	Mens. 5	Average	TIM	
0	39.0	38.5	38.4	38.9	38.9	38.7	–	–
1	43.2	43.6	44.2	42.8	43.8	43.5	463	12.3
2	48.5	64.5	43.0	45.9	49.6	50.3	406	29.8
3	61.0	59.5	56.0	53.2	59.1	57.8	568	49.1
4	40.8	42.2	43.9	43.9	40.9	42.3	393	9.3

After evaluating the results of **Table 5** and **Table 6**, one can observe both the absence of hardening and the smallest increase in microhardness of sample 4. A slight increase in the strength characteristics of sample 1 is also consistent. Sample 3 showed the greatest increase in microhardness, but a slight increase in tensile strength. In terms of the combination of strength properties, the second sample proves better than the others - a relatively good increase in microhardness and an increase in tensile strength by almost 10%.

3. CONCLUSION

The following conclusions can be drawn from the research work:

1) Five samples were obtained by hot extrusion: 1 pure aluminum sample and 4 composite Al rods + 3% MI at 400 °C and drawing coefficient 9. The microhardness index of all dispersively hardened samples increases. The best reinforcing composition (available) is $Al_{18}Mg_3Mn_2$. The introduction of 3% of which into the aluminum matrix increases the microhardness by 30%.
2) Pictures of the microstructure of the cross section, in which the intermetallic inclusions are distributed evenly, indicate the homogeneity of the matrix and hardener.
3) It is supposed that during further research it is possible to change the percentage of the MI phase in the Al matrix (both upward and downward), increase the dispersion, find the optimal exposure time and also increase the drawing coefficient.

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