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MICROSTRUCTURE AND THERMAL PROPERTIES OF AN Al–Mg ALLOY SOLIDIFIED AT HIGH TEMPERATURE IN THE ARGON ATMOSPHERE

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ABSTRACT

In this study, phase formation, the microstructure and the thermal properties of an Al-Mg alloy solidified at high temperature in the argon atmosphere were investigated. The maximum formation of a single-phase Al-Mg alloy was determined by the ratio of the primary aluminum and magnesium components Al – 50 at.% Mg and argon gas flow at a temperature of 750 °C. After solidification at pressures of 1 MPa and 2 MPa, the main phases are the β and γ phases of Al-Mg alloy, in equilibrium condition. The thermal properties of the Al-Mg alloy were studied using DTA-TG ($T_{melting}$ = 458.4 °C, $T_{oxidation}$ = 568.4 and 616.9 °C oxidation of pure Mg and pure Al, respectively).

Keywords: Al-Mg alloy, High-temperature diffusion bonding method, Microstructure, Rapid-solidification, Phase transformations.

1. Introduction

The initially high solidification rate inherent to liquid solidification, i.e rapid solidification from the liquid state, can a significant influence on the formation kinetics of aluminum-based alloys. Rapid solidification, compared to solid solidification, can result in an increased amount of excess vacancies and an enhanced degree of solute super saturation [1]. A solid solution becomes unstable through super saturation and decomposes partly but not completely into another crystalline phase as the equilibrium one is approached [2]. Structuring aluminum-based alloys with a rapid solidification process is considered to improve the thermodynamic properties of aluminum [3].

Al–Mg alloys have been widely used in wrought due to their corrosion resistance, low density, formability and release of high energy. For example, in structural materials [4], for hydrogen storage [5], in aerospace [6], in the manufacture of electronics [7], in the anti-corrosion coating [8], etc. Mg-Al solid solutions in comparison with pure Al have several advantages, namely, lower ignition temperature, shorter burning time, higher flame propagation rate, etc. In recent years, due to these advantages, the possibility of used as a fuel in explosives, propellant and pyrotechnic composition has been considered in many research areas [9-12].

These days, there are many methods for the preparation of Al-Mg alloy, such as, the mixing method [13], mechanical alloying method [14], electrical deposition method [15], high-temperature diffusion bonding method [16], molten salt electrolysis method [17], electron beam evaporation [18], etc. Al-Mg super-structured solid-phase alloys were obtained by the high-temperature diffusion bonding method. Compared to other extraction methods, the high-temperature diffusion bonding method is faster, cleaner, and less energy-intensive [19].

In this research work, the effects of the ratio of the initial components, the synthesis temperature and the environmental pressure on the formation of Al-Mg alloy were studied. The composition and structural anlysis of Al-Mg alloy synthesized at high temperatures were determined and the thermal properties were studied.

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2. Experimental part

2.1. Materials

Magnesium powder (Mg) – partial diameter around 200-250 μm and ≥ 99% purity. Melting point 659 °C. Aluminum powder (Al) – partial diameter around 65 μm and purity ≥ 99%. Melting point 669 °C.

2.2. Preparation of Mg-Al alloy

Pure Mg powder and pure Al powder are mixed and in placed in a high-pressure reactor, a solid solution of Mg-50% Al is synthesized by melting at a temperature of 750 °C. The scheme of the high-pressure reactor is shown in Fig. 1. The composition and structure of the Mg-Al alloy are determined by X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM).

2.3 Measurement of thermal decomposition behavior

Thermal analysis is a quick and effective way to study thermal ignition of Al-Mg alloy. Characteristics of thermal decomposition are determined and studied by using Thermogravimetry-Differential Thermal Analysis (TG/DTA) in the temperature range of 25–600 °C. The equipment operates in atmospheric pressure in a stream of nitrogen (300 cm^3/min). DSC is working with the heating rate of (β) 10 $Kmin^{-1}$. Aluminum pans (height 2.5 mm and diameter 5 mm) were used and 1 mg of the sample is placed in thermal connection with an aluminum pan. In TG/DTA equipment, the standard line of TG/DTA was measured four times for each sample.

3. Results and discussion

3.1. Influence of initial components on the formation of Al-Mg alloy

The degree of solubility of magnesium in aluminum is very important for the synthesis of Al-Mg alloy. Mg solution in Al for Al–40 at.% Mg composites are shown as 23%. In these experiments, the mixing temperature was stable at 45 °C. For Al–30 at.% Mg alloys the maximum solubility of magnesium is 14.1%. For Al–50 at.% Mg composites, around 45% solubility of Mg in the α-Al phase was shown [3].

In the synthesis of Al-rich Al-Mg alloy, the mechanical mixture from different concentrated magnesium is integrated with melting in the pressure reactor at 750 °C. The alloy is cooled by argon gas flow. The phase and crystal structure of all the samples were investigated by XRD and SEM. The results of XRD are shown in Table 1 and Fig. 2, respectively.

As shown in Table 1, Mg_2Al_3 and $Al_{12}Mg_{17}$ intermetallic components were synthesized during the synthesis of Al and Mg powders at high-temperature argon flow. As increasing the amount of aluminum, Al-rich Mg_2Al_3 intermetallic is decreased, but the Mg-rich $Al_{12}Mg_{17}$ amount is increased. Mg-rich $Al_{12}Mg_{17}$ alloy was formed for the Al–50 at.% Mg system. The Al–35 at.% Mg is an optimal ratio system for the synthesis of Al-rich Al-Mg alloy.

Structural analysis of synthesized alloys was carried out by scanning electron microscope. The microstructures for research results of the synthesized products are shown in Fig. 3.

As shown in Fig. 3, crystalline layers of aluminum and magnesium were formed at crystal

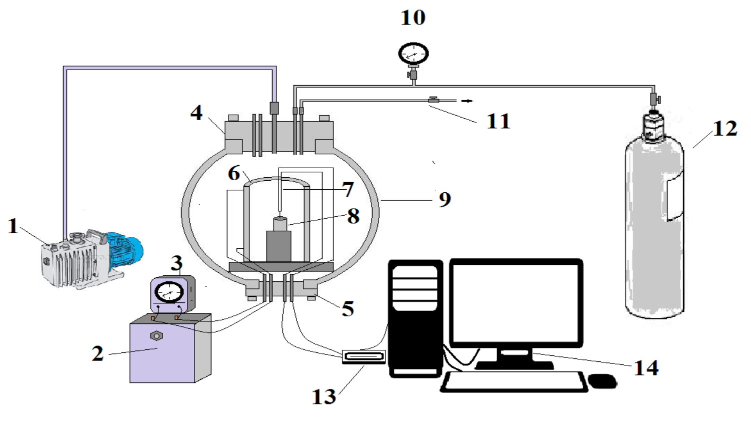

Fig. 1. Scheme of high-pressure reactor: 1 – vacuum pump; 2 – transformer; 3 – voltmeter; 4 – reactor top cap; 5 – bottom reactor cover; 6 – furnace; 7 – thermocouple; 8 – sample; 9 – reactor vessel; 10 – manometer; 11 – inlet and exhaust valves; 12 – argon gas; 13 – LTR-U-1 data acquisition systems; 14 – computer.

Table 1. Results of XRD analysis for synthesized products of Mg-Al systems

Ratio of primary components	Synthesis products, %				
	Mg_2Al_3	$Mg_{17}Al_{12}$	Al_2O_3	MgO	AlN
Al–50 at.% Mg	-	97.7	-	2.3	-
Al–35 at.% Mg	84.6	4.2	4.4	5.9	0.9
Al–30 at.% Mg	83.8	4.8	5.9	5.5	-
Al–20at.% Mg	82.1	5.5	6.2	5.1	1.1

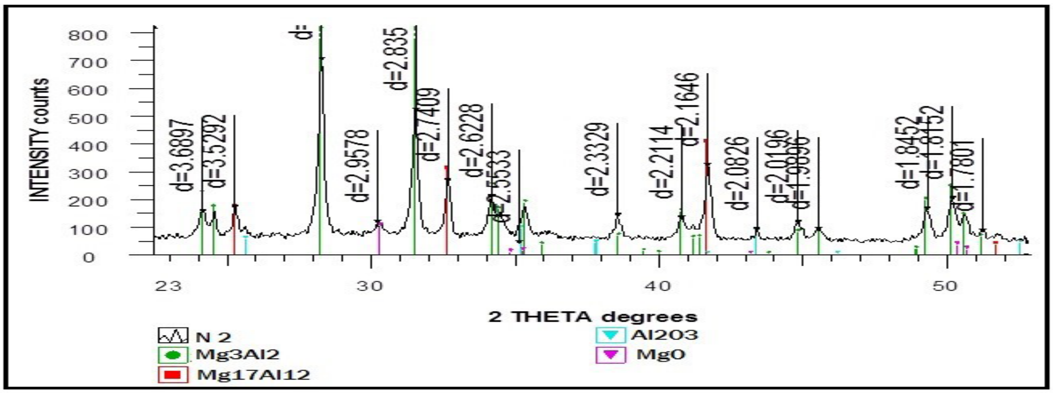

Fig. 2. X-ray pattern of Al 70 wt.%, Mg 30 wt.%.

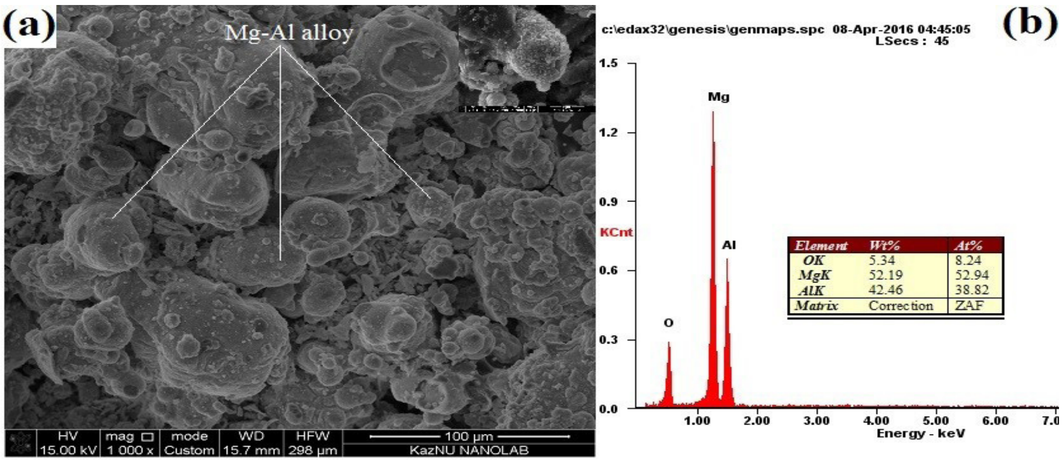

Fig. 3. Microstructure and elemental analysis of the Al–50 at.% Mg system: (a) – electron microscopic image; (b) – the energy dispersive spectrum and mass fraction of elements.

lattices of synthesis Al-Mg alloy production. In addition, the structure of alloy crystals is in submicron sizes. Single-phase alloys are formed when the magnesium content in aluminum is 43.9–62.5% [20]. Therefore, the degree of solubility of magnesium in aluminum is showed the maximum value. In addition, Al–50 at.% Mg alloy released maximum combustion heat.

3.2. Influence of synthesis parameters of the Al-Mg alloy structure

The formation of single-phase alloy in synthesis of Al–50 at.% Mg depends on the parameters of the formation temperature and synthesis pressure. XRD results of Al-Mg alloy synthesized at different temperatures and different pressures are shown in Table 2.

As shown in Table 2, the increase in pressure does not play an important role in our system. The change in the temperature of the synthesis determines the β and γ phases of Al-Mg alloy.

The results of quantitative and qualitative analysis of synthesized alloys was carried out using a SEM. Structural analysis of systems is shown in Fig. 4. The most effective point for the synthesis of a single-phase Al-Mg alloy is shown temperature 750 ^{o}C.

Table 2. Results of XRD for synthesized products at different temperatures and different pressures for Al-Mg systems

Ratio of primary components	Synthesis temperature, °C	Environmental pressure, MPa	Synthesis products, %			
			$Al_{12}Mg_{17}$	Al_3Mg_2	MgO	Al_2O_3
Al–50 at.% Mg	700	1	93.4	5.1	1.5	-
		2	65.1	31.2	3.7	-
	750	1	97.7	-	2.3	-
		2	97.6	-	2.4	-
	800	1	75.8	19.6	3.5	-
		2	50.8	45.7	3.6	1.0

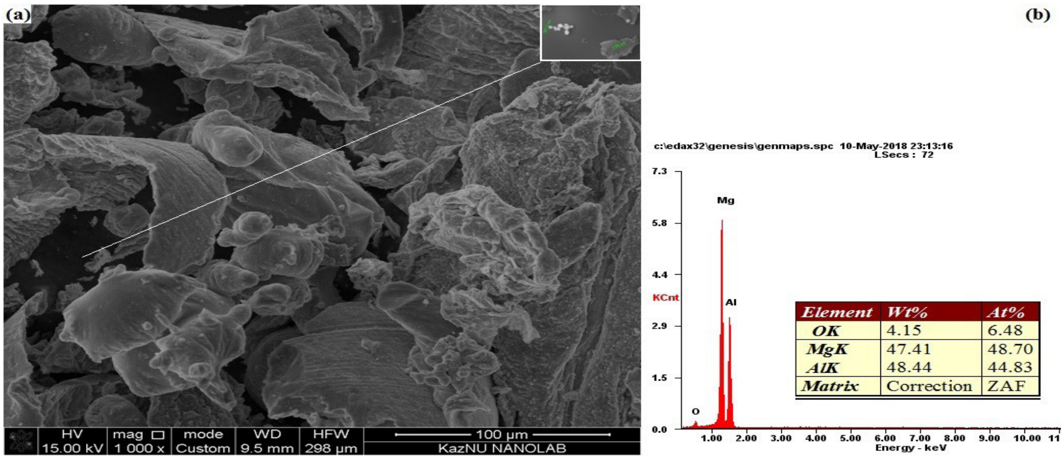

Fig. 4. Microstructure and elemental analysis of the Al–50 at.% Mg system (T = 750 °C, P = 2 MPa): (a) – electron microscopic image; (b) – the energy dispersive spectrum and mass fraction of elements.

The results of quantitative and qualitative analysis of synthesized alloys were carried out using a SEM. Structural analysis of systems is shown in Fig. 4.

According to the images of the electron microscope and elemental analysis of synthesized products, the formation of Al–50 at.% Mg alloy in the system can be predicted. Compared to Fig. 3, the increase in pressure does not have a significant effect on the structure of the alloy.

3.3. Thermal properties of Al-Mg alloy synthesized at high temperatures

The DTA-TG was used to investigate the thermal properties of the synthesized Al–50 at.% Mg alloy. In the nitrogen gas flow, at a heating rate of 10 °C/min and range of 30–650 °C the thermal characteristics of the system were measured. Thermal curves of Al–50 at.% Mg alloy is shown in Fig. 5.

The previous literature shows the DTA-TG values of single-phase Al–50 at.% Mg alloy [21]. According to the results, the oxidation process of double-step magnesium and aluminum occurs at temperatures of 542.6 and 599.2 °C along with the melting at 461.5 °C.

Figure 5 shows the thermogravimetric analysis of the Al–50 at.% Mg alloy at 458.4 °C, according to the previous literature endothermic peak is the melting point of the Al-Mg alloy. Also, the system has a two-stage exothermic peak alloy oxidation process.

Exothermic peaks at temperatures of 568.2 and 616.9 °C are oxidization processes of pure magnesium and pure aluminum, respectively. Together with the starting of two-phase exothermic peaks, the mass change in the system was shown. The temperature increases cause the reaction with oxygen in the air and forming high-density MgO and Al_2O_3.

The thermal properties of Al–50 at.% alloy in our system correspond to the thermal properties of the alloy in the previous literature [21]. Thus, single-phase Al–50 at.% alloy can be formed in the synthesized system.

4. Conclusion

Al-Mg alloys were synthesized at high temperatures in the argon gas flow and the effects of various parameters on the formation of the alloy and the thermal properties of the alloy were studied.

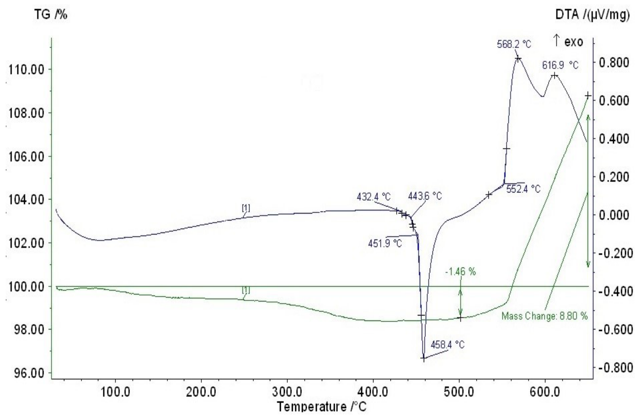

Fig. 5. Thermogravimetric analysis of Al–50 at.% Mg alloy.

The main conclusions of this study are:

1) Different ratios of the initial components to the maximum formation of single-phase Al-Mg alloys were studied. The ratio of the initial components of the Mg-Al alloy synthesized at high temperatures was determined Al–50 at.% Mg;

2) The effect of temperature on the formation of Al-Mg alloys was studied. The effective temperature for the formation of single-phase Mg-Al alloy was determined at 750 ^{o}C;

3) The effect of pressure on the formation of Al-Mg alloys was studied. The increase in pressure does not have a significant effect on the structuring of alloys;

4) The thermal properties of Al-Mg alloys synthesized at high temperatures were determined at different temperatures ($T_{melting}$ = 458.4 ^{o}C, $T_{oxidation}$ = 568.4 and 616.9 ^{o}C, oxidation of Mg and Al, respectively).

References

[1]. Mourik P.V., Maaswinkel N.M., de Keijser Th.H., E.J. Mittemeijer. Precipitation in liquid-quenched AI-Mg alloys; a study using X-ray diffraction line shift and line broadening // Journal of Materials Science. – 1989. – Vol.24. – P.3779–3786.

[2]. Jie J.C., Zou C.M., Brosh E., Wang H.W., Wei Z.J. , Li T.J. Microstructure and mechanical properties of an Al–Mg alloy solidified under high pressures // Journal of Alloys and Compounds. – 2013. – Vol.578. – P.394–404.

[3]. Schoenitz M., Dreizin EL. Structure and properties of Al–Mg mechanical alloys // Journal of Materials Research. – 2003. – Vol.18. – P.1827–2836.

[4]. Shih T.S., Wang J.H., Chong K.Z. Combustion of magnesium alloys in air // Materials Chemistry and Physics. – 2004. – Vol.85. – P.302–309.

[5]. Palma A.S., Iturbe-Garcia J.L., López-Muñoz B.E., Jiménez A.S. MgAl alloy synthesis, characterization and its use in hydrogen storage // International Journal of Hydrogen Energy. – 2010. – Vol.35. – P.12120–12124.

[6]. Rambabu P., Prasad E.N., Kutumbarao V.V., Wanhill R.J.H. Aluminium Alloys for Aerospace Applications // Aerospace Materials and Material Technologies. – 2016. – P.29–52.

[7]. Yang Z., Li J.P., Zhang J.X., Lorimer G.W., Robson J., Review on Research and Development of Magnesium Alloys // Acta Metallurgica Sinica. – 2008. – Vol. 21. – P.313–328.

[8]. Walsh F.C., Low C.T.J., Wood R.J.K., Stevens K.T., Archer J., Poeton A.R., Ryder A. Plasma electrolytic oxidation (PEO) for production of anodised coatings on lightweight metal (Al, Mg, Ti) alloys // The International Journal of Surface Engineering and Coatings. – 2009. – Vol.87. – P.1827–2836.

[9]. Kamunur K., Jandosov J.M., Abdulkarimova R.G., Hori K., Yelemessova Zh.K. Combustion Study of Different Transitional Metal Oxide based on AN/MgAl Composites Gas Generators // Eurasian Chemico-Technological Journal. – 2017. – Vol.19. – P.341–346.

[10]. Камунур К., Жандосов Ж.М., Абдулкаримова Р.Г., Хори К., Мансуров З.А. Влияние TiO_2 на характеристики горения газогенераторов на основе AN/MgAl // Химический журнал Казахстана. – 2017. – №4. – С.215–224.

[11]. Mohammed I., Santhosh K., Chakravarthy S.R., Jayaganthan R., Sarathi R., Srinivasan A. Study of burning rate characteristics of propellants containing Al-Mg alloy nanopowder // Nano Express. – 2020. – Vol.1. – P.1–14.

[12]. Yao M., Chen L., Yu J., Peng J. Thermoanalytical investigation on pyrotechnic mixtures containing Mg-Al alloy powder and barium nitrate // Procedia Engineering. – 2012. – Vol.45. – P.567–573.

[13]. Kim D.Y., Cheon S.S., Suh J.D. Solid-state processing of Al-Mg alloys // International Journal of Precision Engineering and Manufacturing. – 2018. – Vol.19. – P.1681–1688.

[14]. Gubicza J., Kassem M., Ungár T. Formation of nanocrystalline aluminum magnesium alloys by mechanical alloying // 3rd International Powder Metallurgy Conference. Ankara, 2002. – P.741–755.

[15]. Zheng Y., Zhang S., Lü X., Wang Q., Zuo Y., Liu L. Low-temperature Electrodeposition of Aluminium from Lewis Acidic 1-Allyl-3-methylimidazolium Chloroaluminate Ionic Liquids // Chinese Journal of Chemical Engineering. – 2012. – Vol.20. – P.130–139.

[16]. Chen M.C., Kuo C.W., Chang C.M., Hsieh C.C., Chang Y.Y., Wu W. Diffusion and Formation of Intermetallic Compounds during Accumulative

Roll-Bonding of Al/Mg Alloys // Materials Transactions. – 2007. – Vol.48. – P.2595–2598.
[17]. Yang S.H., Yang F.L., Liao C.F., Li M.Z., Wang X. Electrodeposition of magnesium-yttrium alloys by molten salt electrolysis // Journal of Rare Earths. – 2010. Vol.28. – P.385–388.
[18]. El Abedin S.Z., Giridhar P., Schwab P., Endres F. Electrodeposition of nanocrystalline aluminium from a chloroaluminate ionic liquid // Electrochemistry Communications. – 2012. – Vol.12. – P.1084–1086.
[19]. Shirzadi A.A., Wallach E.R. Temperature gradient transient liquid phase diffusion bonding: a new method for joining advanced materials // Science and Technology of Welding and Joining. – 1997. – Vol.2. – P.89–94.
[20]. Scudino S., Sakaliyska M., Surreddi K.B., Eckert J. Solid-state processing of Al-Mg alloys // Journal of Physics: Conference Series. – 2009. – Vol.144. – P.1–5.
[21]. Pourmortazavi S.M., Hajimirsadeghi S.S., Kohsari I., Fathollahi M., Hosseini S.G. Thermal decomposition of pyrotechnic mixtures containing either aluminum or magnesium powder as fuel // Fuel. – 2008. – Vol.87. – P.244–251.

References

[1]. Mourik PV, Maaswinkel NM, de Keijser THD, Mittemeijer EJ (1989) Ej. J Mater Sci 24:3779–3786. https://doi.org/10.1007/BF02385770
[2]. Jie JC, Zou CM, Brosh E, Wang HW, Wei ZJ, Li TJ (2013) J. Alloy. Compd 578:394–404. https://doi.org/10.1016/j.jallcom.2013.04.184
[3]. Schoenitz M, Dreizin EL (2013) J. Mater. Res. 18:1827–2836. https://doi.org/10.1557/JMR.2003.0255
[4]. Shih TS, Wang JH, Chong KZ (2004) Mater. Chem. Phys. 85:302–309. https://doi.org/10.1016/j.matchemphys.2004.01.036
[5]. Palma AS, Iturbe-Garcia JL, López-Muñoz BE, Jiménez AS (2010) Int. J. Hydrogen Energ. 35: 12120–12124. https://doi.org/10.1016/j.ijhydene.2009.09.073
[6]. Rambabu P, Eswara Prasad N, Kutumbarao VV, Wanhill RJH (2016) Aerosp. Mater. Mater. Technol. 1:29–52. https://doi.org/10.1007/978-981-10-2134-3_2
[7]. Yang Z, Li JP, Zhang JX, Lorimer GW, Robson J (2008) Acta Metallurgica Sinica. 21: 313–328. https://doi.org/10.1016/S1006-7191(08)60054-X
[8]. Walsh FC, Low CTJ, Wood RJK, Stevens KT, Archer J, Poeton AR., Ryder A (2009) Int. J. Surf. Eng. Coat. 87:122–135. https://doi.org/10.1179/174591908X372482
[9]. Kamunur K, Jandosov JM, Abdulkarimova RG, Hori K, Yelemessova ZhK (2017) Eurasian Chem.-Technol. J. 19:341–346. https://doi.org/10.18321/ectj682
[10]. Kamunur K, Jandosov JM, Abdulkarimova RG, Hori K, Mansurov Z (2017) Chemical Journal of Kazakhstan [Himicheskij Zhurnal Kazahstana] 4:215–224.
[11]. Mohammed I, Santhosh K., Chakravarthy SR, Jayaganthan R, Sarathi R, Srinivasan A (2020) Nano Express 1:020007. https://doi.org/10.1088/2632-959X/aba22f
[12]. Yao M, Chen L, Yu J, Peng J (2012) Pro. Eng. 45:567–573. https://doi.org/10.1016/j.proeng.2012.08.205
[13]. Kim DY, Cheon SS, Suh JD. Int. J. Precis. Eng. Man. 19:1681–1688. https://doi.org/10.1007/s12541-018-0195-8
[14]. Gubicza J, Kassem M, Ungár T (2002) Formation of nanocrystalline aluminum magnesium alloys by mechanical alloying. 3rd International Powder Metallurgy Conference Ankara, Turkey P.741–755.
[15]. Zheng Y, Zhang S, Lü X, Wang Q, Zuo Y, Liu L (2012) Chinese J. Chem. Eng. 20:130–139. https://doi.org/10.1016/S1004-9541(12)60372-3
[16]. Chen MC, Kuo CW, Chang CM, Hsieh CC, Chang YY, Wu W (2007) Materials Transactions 48:2595–2598. https://doi.org/10.2320/matertrans.MD200718
[17]. Yang SH, Yang FL, Liao CF, Li MZ, Wang X (2010) J. Rare Earths. 28:385–388. https://doi.org/10.1016/S1002-0721(10)60322-5
[18]. El Abedin SZ, Giridhar P, Schwab P, Endres F (2010) Electrochemistry Communications 12:1084–1086. https://doi.org/10.1016/j.elecom.2010.05.034
[19]. Shirzadi AA, Wallach ER (1997) Sci. Technol. Weld. Joi. 2:89–94. https://doi.org/10.1179/stw.1997.2.3.89
[20]. Scudino S, Sakaliyska M, Surreddi K, Eckert J (2009) Journal of Physics: Conference Series 144:1–5. https://doi.org/10.1088/1742-6596/144/1/012019
[21]. Pourmortazavi SM, Hajimirsadeghi SS, Kohsari I, Fathollahi M, Hosseini SG (2008) Fuel 87:244–251. https://doi.org/10.1016/j.fuel.2007.04.022

Микроструктура и термические свойства сплава Al–Mg затвердевшего при высокой температуре в атмосфере аргона

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Аннотация

В этой работе исследованы фазообразование, микроструктура и термические свойства Al-Mg сплава затвердевшего при высокой температуре в атмосфере аргона. Максимальное образование однофазного сплава Al-Mg соотношением компонентов первичного алюминия и магния Al–50%, потока Mg и газового аргона определялось при температуре 750 °C. После затвердевания при давлениях 1 МПа и 2 МПа основными фазами являются β- и γ-фазы сплава Al-Mg, находящиеся в равновесном состоянии. Теплофизические свойства алюминиево-магниевого сплава были исследованы методом DTA-TG ($T_{плавление}$ = 458.4 °C, $T_{окисление}$ = 568.4 и 616.9 °C окисление чистого Mg и чистого Al соответственно).

Ключевые слова: Al-Mg сплав, высокотемпературный диффузионный способ связывания, микроструктура, быстрое затвердевание, фазовые превращения

Аргон атмосферасындағы жоғары температурада қатайтылған Al-Mg қорытпасының микроқұрылымы және термиялық қасиеттері

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Аңдатпа

Бұл зерттеу жұмысында, аргон атмосферасында жоғары температурада қатайтылған Al-Mg қорытпасының фазалық құрылымдануы, микроқұрылымы және термиялық қаситтері зерттелінген. Бір фазалы Al-Mg қорытпасының максималды түзілуі бастапқы алюминий және магний компоненттерінің арақатынастары Al–50 at.% Mg және аргон газ ағынында 750 °C температурада анықталынды. Тепе-теңдік жағдайда, 1 МПа және 2 МПа қысымдарда қатайтылғаннан кейінгі Al-Mg қорытпасының басты фазалары β және γ фазалары болады. DTA-TG арқылы Al-Mg қорытпасының термиялық қасиеттері ($T_{балқу}$ = 458.4 °C, $T_{тотығу}$ = 568.4 және 616.9 °C сәйкесінше Mg және Al) зерттелінді.

Кілт сөздер: Al-Mg қорытпасы, жоғары температуралық диффузиялық байланысу әдісі, микроқұрылым, жылдам қатаю, фазалық ауысулар