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Arch. Metall. Mater. **65** (2020), 1, 201-206

DOI: 10.24425/amm.2019.131115

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PHYSICAL MODELLING OF THE PRODUCTION OF AN ALLOY VAPOUR SOURCE FOR THE SYNTHESIS OF DIELECTRIC MATERIAL

The paper reports the results of a physical modelling study of the production of a hypereutectic aluminium alloy to be used for making an alloy vapour source for operation in the magnetron. Within the study, targets from a hypereutectic aluminium-silicon alloy were made in laboratory conditions. Thus obtained material was subjected to heat treatment, porosity analysis, and the assessment of the microstructure and fitness for being used in the magnetron. The process of melting the hypereutectic Al-Si alloy was carried out at the Department of Foundry of the Czestochowa University of Technology. The investigation into the production of the alloy vapour source for the synthesis of the dielectric material from the hypereutectic aluminium alloy has confirmed.

Keywords: Aluminum-silicon alloy, magnetron sputtering processes, gravity casting, die casting

1. Introduction

Current requirements for the reduction of the mass of structures and the development of new materials of better functional properties force a continuous development in the field of materials engineering. Materials that perfectly fit into this area are light alloys, such as those based on aluminium or magnesium, multilayered or composite materials, as well as coated materials [1-4]. Classical surface engineering, whose main purpose is to produce a zone of strictly defined corrosion, mechanical or tribological properties in the top layer, has at the current state of technology a number of tools available. The basic means of top layer modification include mechanical, physical, chemical, electrochemical, thermal, thermochemical and thermomechanical methods [5-8]. Magnetron systems designed for the synthesis of layers under reduced pressure are currently used in many fields of industry and science. Magnetron sputtering is a variation of cold cathode ion-beam diode sputtering. Atoms or molecules ejected from the cathode referred to as the target are deposited onto the substrate. A stream of metal vapour is transferred to the substrate, where the growth of the layer follows. A negatively polarized solid target is sputtered with positively charged ions. The positively charged ions are produced during glow discharge with an electrode potential difference achieved in the presence of two mutually perpendicular magnetic and electric fields [1-6]. The synthesis of layers by the magnetron method using vapour sources chemically composed of aluminium and silicon has been the subject of many studies [7-10].

2. Methodology of research and results

The main objective of the study was to obtain a hypereutectic aluminium alloy that will enable the making of an alloy vapour source for operation in a WMK 100 circular magnetron.

It was assumed that using alloying aluminium as a source of vapour of a silicon content above 20% would enable the effective synthesis of nitride layers from a group of solid solutions in the Si – Al – O – N system. In view of the above assumption and a difficulty in purchasing the appropriate alloy in the market, we considered it advisable to prepare the alloy vapour source by ourselves based on available materials – see the composition of the material used in Tables 1 and 2 and their planned use in Table 3.

Based on the planned shares of alloying components from Table 3, melts were made in an induction furnace equipped with a silicon carbide crucible of a capacity of 6kg Cu. The inductor was current supplied with a frequency of up to 10 kHz and a maximum power of 40 kW. After melting, the alloy was

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TABLE 1

Chemical composition of silicon metal 2202

Chemical composition [% mass.]					Granulation [mm]
Si	Fe	Ca	Al	rest	
99,4578	0,1803	0,0137	0,0628	0,2854	10-40

TABLE 2

Chemical composition of aluminum alloy 6060

Chemical composition [% mass.]										Granulation [mm]
Al	Si	Mg	Fe	Cu	Cr	Mn	Zn	Ti	rest	
98,73	0.58	0,42	0,01	0,05	0,01	0,03	0,06	0,02	0,09	20-40

heated up to a temperature of 610°C and then was poured into a permanent mould – Fig. 1. The solidification process was designed so that any large irregular precipitates of the brittle primary silicon phase were taken out to the riser head. Figure 2 shows a view of the obtained cast. From the cast part that showed no casting defects, targets were made – Fig. 3. During machining, the selected material did not exhibit any of typical casting defects, such as porosity or contraction cavities, which would have excluded the target from being effectively used according to its purpose. Prior to being poured, a graphite mould of an inner diameter of 120 mm and a height of 120 mm was heated up to a temperature of about 180-190°C. The obtained casts were approx. 80 mm in height.

Fig. 1. Graphite mould

Fig. 3. Discs after cutting cast

TABLE 3

Weight comparison of substrates used for melting

Alloy	Weight [g]			Estimated silicon in the alloy
	aluminum 6060	silicon 2202	sum	
AlSi 23	1590	480	2070	≈ 23%
AlSi 25	1556	516	2072	≈ 25%
AlSi 30	1450	625	2075	≈ 30%
AlSi 50	1045	1030	2075	≈ 49,5%

After solidification, the cast was cut into approx. 8 mm-thick discs and, after visual assessment their parts with no surface defects were machined to the final shape – Fig. 4.

Next, the obtained material was subjected to a heat treatment operation – homogenization (at 550°C for 5 hours), examination for porosity and the assessment of the microstructure and suitability for using in the WMK 100 magnetron.

For the observation of the microstructure of the obtained Al-Si alloys and chemical analysis, a HITACHI S – 3500N scanning electron microscope equipped with an EDS. The measurement were made in selected four locations on each of the specimens

Fig. 2. Cast

Fig. 4. Si – Al – O target

TABLE 4

Analysis of chemical composition

Alloy	Sample 1 [% mass.]			Sample 2 [% mass.]			Sample 3 [% mass.]			Average [% mass.]		
	Al	Si	O	Al	Si	O	Al	Si	O	Al	Si	O
AlSi 23	73,82	23,42	2,76	71,93	25,26	2,81	75,67	22,66	1,67	73,81	23,78	2,41
AlSi 25	70,43	25,39	4,18	71,84	24,71	3,45	73,20	24,10	2,70	71,82	24,73	3,44
AlSi 30	65,76	29,95	4,29	67,53	28,06	4,41	67,76	28,62	3,62	67,02	28,88	4,11
AlSi 50	48,85	50,24	0,91	45,22	53,85	0,93	53,27	46,04	0,69	49,11	50,04	0,84

(centre, board and two point between). The obtained results, given in Table 4, confirm the correctness of the planning and making of the alloy vapour sources.

The examined chemical composition of the hypereutectic Al-Si alloys, shown in Table 4, confirmed that the share of silicon in the produced material was close to the share calculated based on supplied material attestations. The produced alloys contained from 0.7 to 4% oxygen. The affinity of aluminium to oxygen cause it to readily oxidize, especially in a liquid state. Another source of oxygen is here the aluminium charge, which is always covered with a film of Al_2O_3. Aluminium oxide, as being heavier, does not flow up by itself to the molten metal surface, where it could be removed with the slag. In Figures 5(a-d), the structures of the produced silumins are shown.

Isolated silicon lamellas observed in the microsection are a result of intersecting lamellar-skeleton silicon crystals in eutectic grains by the microsection plane. The microstructures are illustrated by the geometric forms of primary silicon crystals as a function of chemical composition variation. In repeatable liquid metal soaking and mould cooling conditions, a change in chemical composition might have influenced the number of condensation nuclei, hence the variation in the geometrical form and size of primary silicon crystals – Figs. 6a-d.

The assessment of the porosity of the obtained casts was made using a Nikon Eclipse MA 200 microscope and the NIS-Elements D program with mask function – Figs. 7 and 8. The assessment of microsection surfaces identified casting defects chiefly in the form of porosity and microshrinkage. The share of

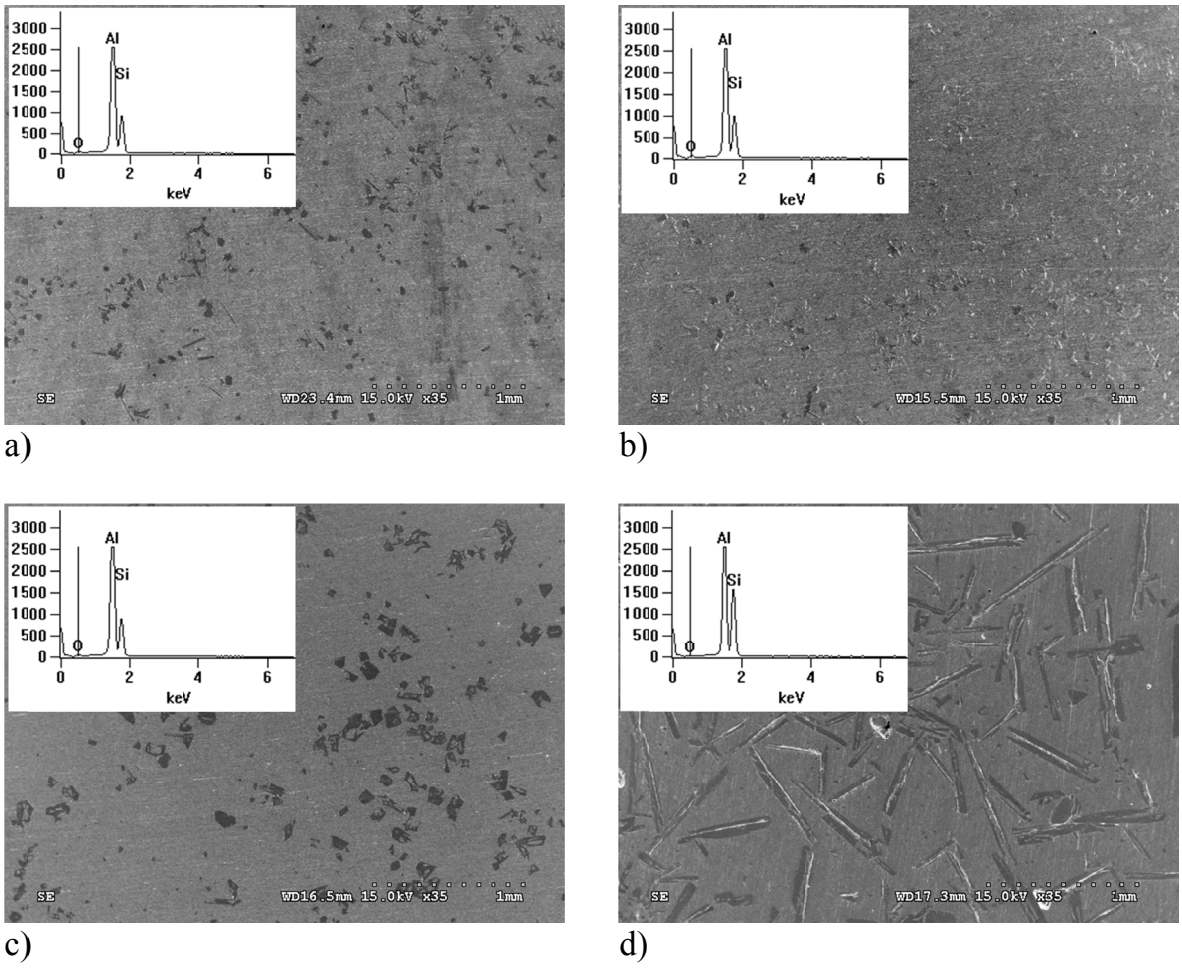

Fig. 5. SEM microstructure of produced silumins, EDS analysis of phase in sample – central parts of specimen: a) Si – 23%, b) Si – 25%, c) Si – 30%, d) Si – 50%

casting defects for the observed target surface was about 3%. The porosity value is the average of three measurements of the central part of the sample over the entire surface visible in the Fig. 7.

The participation of silicon in the produced targets was also identified, confirming the chemical analysis result. Figure 8 shows the microstructure of hypereutectic silumin of a silicon content of approx. 23%.

Using alloying aluminium as vapour sources of a silicon content above 20% enabled an effective synthesis of nitride layers from the group of solid solutions in the Si – Al – O – N system, which is confirmed by the macroscopic illustrative picture in Fig. 9 and the cross-section of the layer formed in Fig. 10 without identifying the phase components, obtained from a scanning microscope. It seems it is possible to effectively

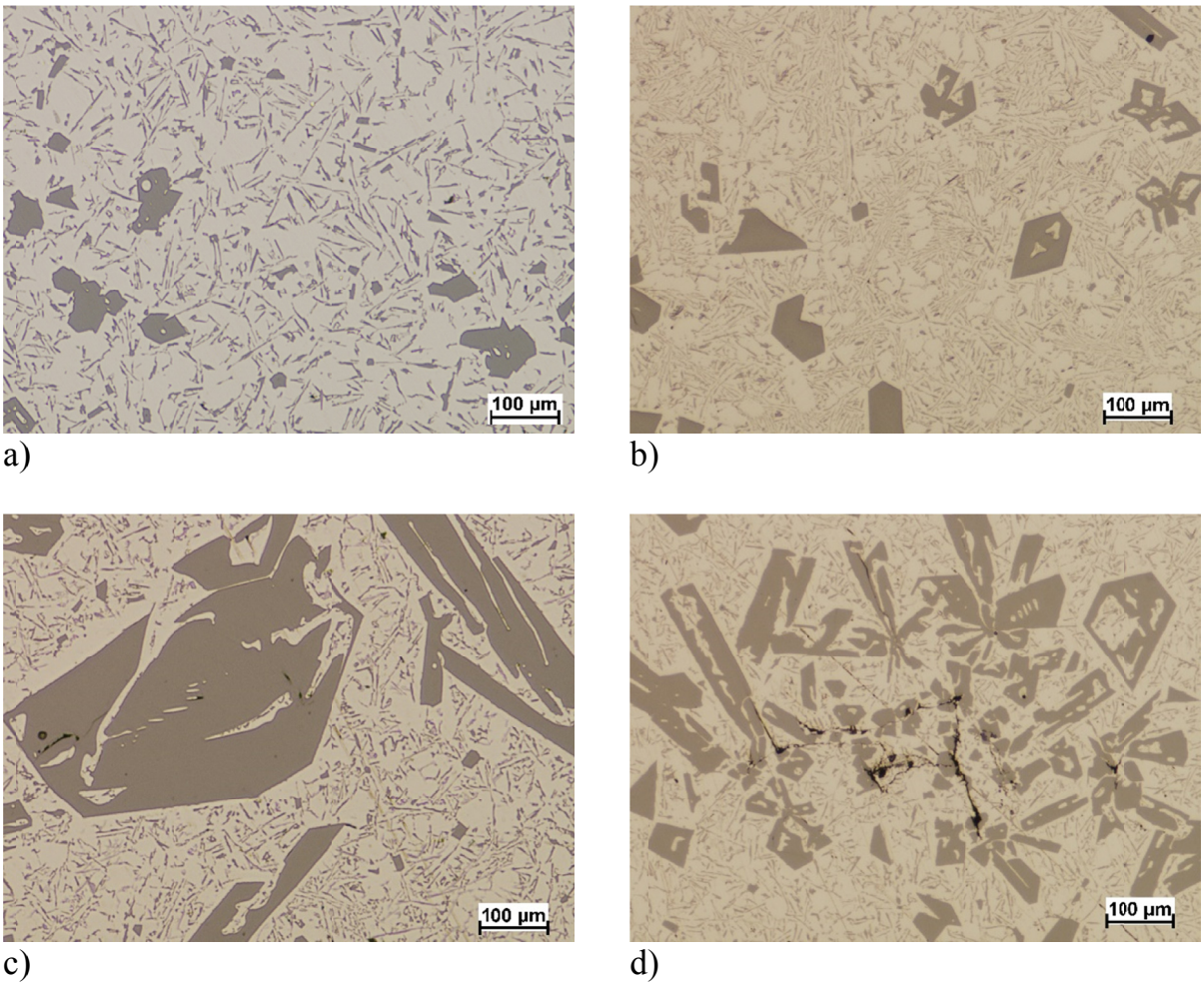

Fig. 6. Microstructure of produced silumins: a) Si – 23%, b) Si – 25%, c) Si – 30%, d) Si – 50%

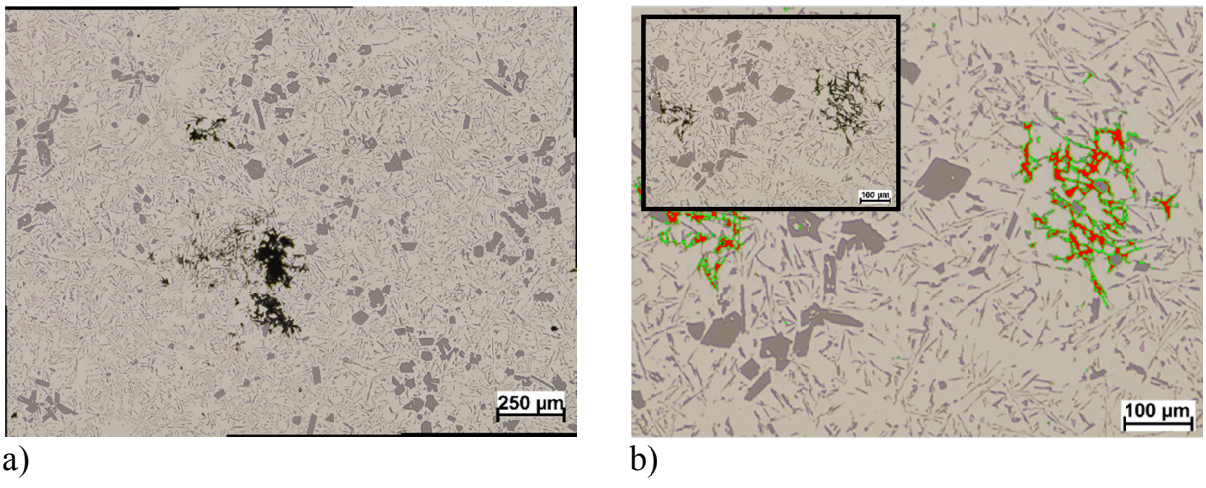

Fig. 7. Example of casting defects a) porosity, b) microshrinkage

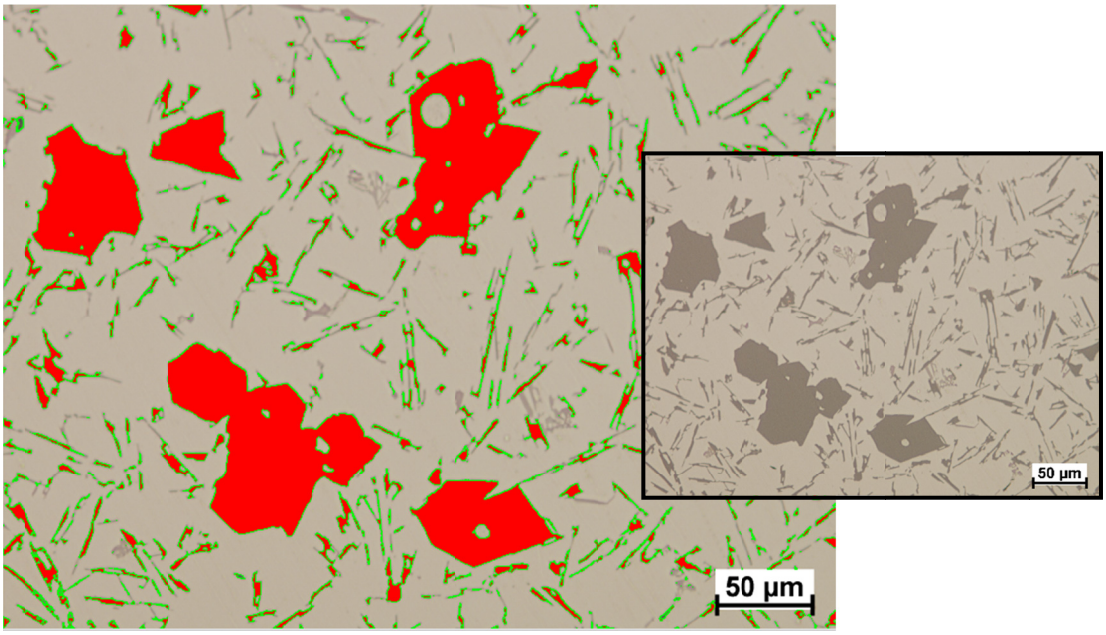

Fig. 8. Microstructure of Al-Si alloy; Si – 23%

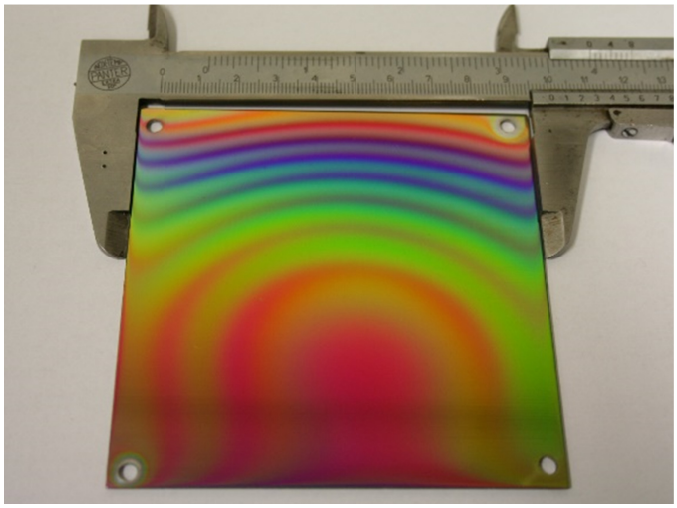

Fig. 9. Al – Si – O – N layer on aluminium base

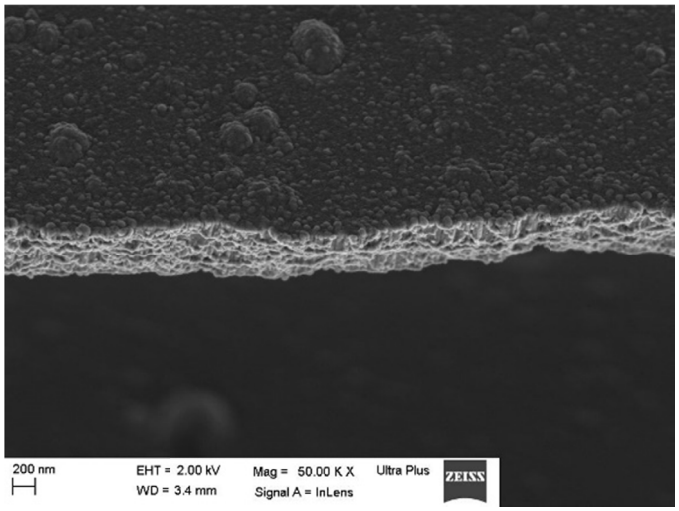

Fig. 10. Cross-section of nitride layer

produce good-quality vapour sources for magnetron sputtering processes by casting method. Studies of dielectric properties are beyond the scope of the proposed tests in this study.

3. Conclusion

The preparatory investigation into the production of the alloy vapour source for the synthesis of the dielectric material from the hypereutectic aluminium alloy has confirmed that it is possible to effectively produce good-quality vapour sources for magnetron sputtering processes. The advantage of preparing alloy targets independently is the possibility of freely designing the chemical composition. Melting processes can be conducted in vacuum conditions, minimizing thereby the number of undesirable elements, mainly oxygen and hydrogen, in the alloy.

- production of an alloy vapour source for the synthesis of dielectric material – a target of a hypereutectic AlSi alloy with a controlled share of silicon - is possible by a casting method;
- chemical composition of the produced hypereutectic Al–Si alloy conforms with the assumptions;
- a microstructure advantageous from the point of view of the synthesis and properties of the dielectric material has been obtained, and porosity and microshrinkage make only a technical problem, not affecting the vacuum sputtering process;
- the analysis of the chemical composition of the produced metal vapour sources has been confirmed by the analysis of the picture of their microstructure phases.

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