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Investigations of the Distribution of Elements in Phases Present in G-AlMg$_5$Si Cast Alloy with EDX/WDX Spectrometers and AES*

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Abstract. The mechanical properties of most aluminium alloys depend heavily on chemical composition, casting methods and heat treatment. Alloys of type G-AlMg$_5$Si are known for good corrosion resistance and mechanical properties at elevated temperatures. Under the designation Hydronalium (Hy 511) they are used for the production of heads for air cooled Diesel engines. To obtain better mechanical characteristics, titanium is added to the alloy. This paper deals with the results obtained during investigations of the distribution of elements in binary eutectic $\alpha \veebar Mg_2Si$ and ternary eutectic $\alpha \!-\!\!<^{Si}_{Mg_2Si}$ as well as the distribution of titanium in samples of Hy 511, obtained during casting of cylinder heads. Studies of the distribution of the elements were performed using EDX/WDX spectrometry, and the distribution of titanium was studied also with Auger electron spectroscopy.

Key words: cast aluminium alloy, EDX/WDX spectrometry, Auger electron spectroscopy.

Aluminium alloys used for castings differ from wrought alloys in many respects. In the first place they differ by having high fluidity thus permitting the casting of goods with thin walls and complicated cross sections. High purity aluminium does not possess this property and moreover has low strength. Therefore, pure aluminium is not often used for intricate castings. Rather it is used, for example, for parts of chemical apparatus, electrical devices, as are squirrel cages of electric motors, supports for brush holders, supporting rings of rotating converters and in the food industry for boilers, kettles and various containers.

The mechanical properties of most aluminium alloys depend heavily on chemical composition, casting methods and heat treatment. However, the basic properties such as normal and tangential moduli of elasticity and Poisson's ratio change very little.

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* Dedicated to Professor Günther Tölg on the occasion of his 60th birthday

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Some advantages of cast aluminium alloys are: (1) good castability, permitting the casting of parts of nearly any configuration, with a relatively small linear shrinkage and good machinability; (2) high specific strength, practically approaching that of straight carbon steel; (3) high corrosion resistance; (4) high thermal and electrical conductivity. All these advantages permit cast aluminium alloys to compete successfully with many industrial alloys based on other chemical systems.

During this study the phases present in an aluminium alloy with relatively high magnesium content, of type G-$AlMg_5Si$ (DIN 1725), known for good corrosion resistance and mechanical properties at elevated temperature [1,2], were investigated. Under the commercial name Hydronalium (Hy 511) this alloy is known to give satisfactory results in the production of cylinder heads for air-cooled Diesel engines [3].

Materials and Methods of Investigation

The materials which were investigated during this study were tensile test specimens, which were obtained during the production of cylinder heads for a 6-stroke, air-cooled Diesel engine. The alloy was melted in an induction heated coreless crucible electric furnace having a capacity of 600 kg. The melt was later degassed and the crystal grains were refined in another furnace. After this procedure, the melt stayed in the furnace for several hours. The cylinder heads were cast in metallic forms, where the pouring temperature was 1013 K and the temperature of the metallic form was 673 K. After having finished the casting of cylinder heads, they were subjected to a heat treatment procedure. For each melt of cylinder heads mechanical tests were performed in order to control the mechanical values of castings after the heat treatment procedure was finished.

According to DIN 1725 the elemental composition in wt.% of the investigated aluminium alloy should be within the following limits: Mg 4.5–5.5%; Mn 0.1–0.5%; Si 0.6–1.5%; Ti 0.2%; Fe max. 0.6%; Cu max. 0.2%; Zn max. 0.2%; Al balance. In order to improve the mechanical characteristics max. 0.5% Ni was added to this alloy.

After the heat treatment the following values of mechanical properties should be obtained: tensile strength 190–210 N/mm^2; hardness 75–85 BHN; elongation min. 3%. The broken tensile test specimens obtained during mechanical testings were further used as samples for the investigations of the distribution of elements in phases which were present in the cast Al-Mg-Si alloy.

The analysis of the distribution of elements was first performed with the X-ray microanalysis system TRACOR TN-2000 in connection with the scanning electron microscope JEOL JSM-35. In spite of the difficulties arising during the detection of titanium in the investigated samples, the EDXRF technique was also adopted, using X-rays obtained by excitation of samples with CuKα and CuKβ radiation having a diameter of 3 mm.

For further analysis wavelength dispersive analysis was applied, where an electron probe X-ray microanalyzer was used. The instrument in use during this stage, an X-ray microanalyzer 3 A was produced by JEOL Electron Optics Instruments, Tokyo.

In the final stage of investigation Auger electron spectroscopy was used to obtain images, chemical maps and spot analyses of the investigated aluminium samples.

During these investigations we used the Scanning Auger Microprobe SAM, PHI Model 545 A, manufactured by Physical Electronics Industries Inc., Eden Prairie, Minnesota. The energy of the electron beam was 3 keV, the current being 1 μA having a diameter of 40 μm.

Results

In Table 1, the results of chemical analysis, the results of tensile testings and the results of hardness measurements of some typical melts of investigated cast Al-Mg-Si alloy being selected after various values of elongation are presented.

The methods of determination of the elements are as follows: Silicon was determined by the modified gravimetric method after Regelsberger. Magnesium was determined by the sedimentary volumetric method and maganese by the volumetric determination method after Smith with ammonium-persulphate and silver nitrate. Iron, titanium and copper were determined by photometry, whereas the contents of nickel were determined gravimetrically. For tensile testings to be performed a Mohr-Federhaff tensile test machine was used.

As far as the results of chemical analysis of the melts investigated are concerned, the following conclusions can be made: The melt 160/30 showed the highest concentration of magnesium and also the highest content of nickel. The concentration of manganese was approximately the same in all the three melts investigated and the concentration of silicon was in good agreement with the values for silicon which were prescribed by standards.

The concentration of titanium was too high in melt no. 106/30, while the concentration of this element was within the prescribed limits in the two other melts investigated. The measured concentrations of nickel in all three melts were too high, nickel was added to the melts to improve the mechanical characteristics of the

Table 1. Results of chemical analysis, tensile testings and hardness measurements of the investigated aluminium alloy

	Chemical composition [wt %]							Results of mechanical tests		
Melt no.	Mg	Mn	Si	Cu	Ti	Ni	Fe	Tensile strength R_m [N/mm^2]	Elongation δ_5 [%]	Hardness BHN [N/mm^2]
84/36	4.51	0.38	1.47	0.31	0.19	0.16	0.24	268 298	1.2 1.6	103 104
106/30	5.58	0.38	1.24	0.32	0.40	0.26	0.18	285 298	4.8 3.2	102 102
109/10	5.36	0.39	1.28	0.33	0.20	0.19	0.20	277.5 270	2.2 2.2	102 102

castings. The iron concentration was in the prescribed limits. It is interesting to note that titanium and nickel showed the highest values in the melt no. 106/30, the only melt showing satisfactory results of elongation, being higher than the minimum value prescribed. The values of elongation of the other two melts investigated were not satisfying. The same problem of varying results of elongation measurements, while the other results of mechanical testings showed satisfactory results, arised already in the initial stage of this investigation of the cast Al-Mg-Si alloy. To solve the problem of varying values of elongation, we tried to apply various heat treatment procedures. Different heat treatment procedures did not result in satisfying results of mechanical values and the results of these investigations are already published [4].

In spite of these difficulties further studies of the chemical composition of the alloy and phases present in the investigated Al-Mg-Si cast alloy were recommended. Special attention was paid to the distribution of titanium, because titanium is added to the melt in order to obtain a smaller grain size of the cast alloy, thus influencing the mechanical values. Additions of nickel to the melt have a similar effect of increasing the strength at room temperature and also in the higher temperature range.

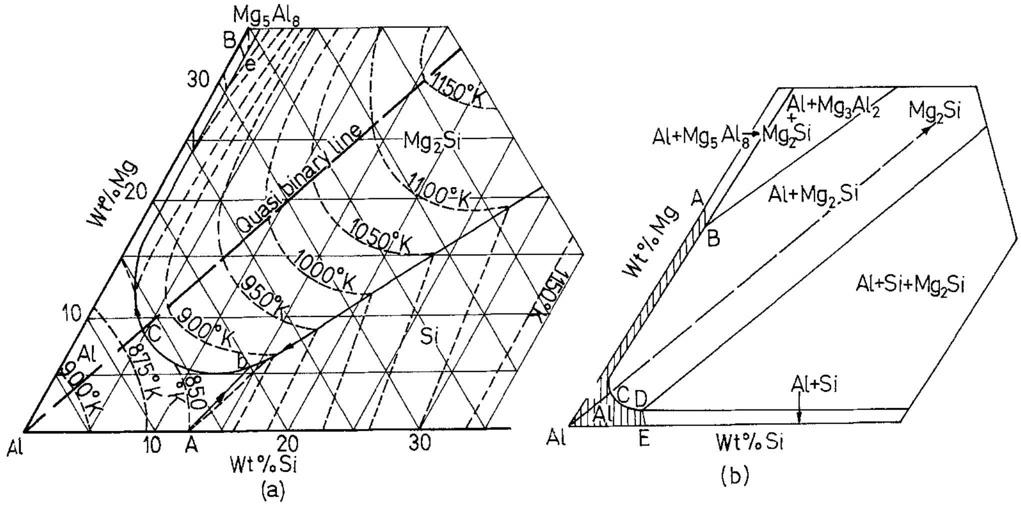

Fig. 1. Aluminium corner of the aluminium-magnesium-silicon diagram: (**a**) liquidus, (**b**) phase distribution in the solid. For location of points A, B, C, D, and E see the table below

	A	B		C		D		E
Temperature (K)	% Mg	% Mg	% Si	% Mg	% Si	% Mg	% Si	% Si
868	—	—	—	1.17	0.68	—	—	—
850	—	—	—	1.10	0.63	—	—	1.65
825	—	—	—	1.00	0.57	0.83	1.06	1.30
800	—	—	—	0.83	0.47	0.6	0.8	—
775	—	—	—	0.70	0.40	0.5	0.65	0.80
725	17.4	15.3	0.1	0.48	0.27	0.3	0.45	0.48
675	13.5	11	0.0X	0.33	0.19	0.22	0.3	0.29
575	6.7	5	0.0X	0.19	0.11	0.1	0.15	0.06

Table 2. Invariant reactions at the aluminum end of the aluminum-magnesium-silicon diagram

Reaction		Composition				Temperature (°K)
		Liquid		Al		
		% Mg	% Si	% Mg	% Si	
A liq.	Al + Si	—	12.5	—	1.65	850
B liq.	Al + Mg_5Al_8	34.0	—	17.4	—	723
C liq.	Al + Mg_2Si (quasibinary)	8.15	7.75	1.17	0.68	868
D liq.	Al + Mg_2Si + Si	4.96	12.95	0.85	1.10	828
E liq.	Al + Mg_2Si + Mg_5Al_8	32.2	0.37	15.3	0.05	722

The phases present in the investigated alloy are known from the aluminium corner of the Al-Mg-Si ternary system.

The equilibrium diagram is relatively simple and well established, good reviews of it can be found in [5]. The compound Mg_2Si is in equilibrium with aluminum and there is a quasibinary line Al-Mg_2Si at the Mg : Si ratio of 1.73 as can be seen from Fig. 1. The binary and ternary invariant reactions at the aluminum end are shown in Table 2.

The Si, Mg_5Al_8, and Mg_2Si phases in the reactions can be expected not to differ substantially from the stoichiometric ones. When in solution in aluminum magnesium and silicon tend to cluster together to form "molecules" of Mg_2Si. The solid solubility of Mg_2Si in aluminum is reduced slightly by the presence of silicon in excess over the Mg : Si = 1.73 ratio but an excess of magnesium greatly reduces the solubility. With a low magnesium content, the bulk of the binary eutectic, which is present in the aluminium corner of the ternary system, is found between the grains of the α-solid solution. The microstructure of this binary eutectic resembles Chinese script and presents a black phase in the metallographic structure of the investigated alloy.

The results of metallographic structure investigations of the cast Al-Mg-Si alloy are shown in Fig. 2 a–c, where the primary α-crystals can be seen together with the binary and the ternary eutectic, formed on the borders fo the α-crystals.

The metallographic structures of the investigated cast alloy, shown in Fig. 2 a–c, are obtained on tensile test specimens for mechanical testing. The metallographic structure in Fig. 2 a was obtained on a test specimen of the melt no. 84/36, the structure in Fig. 2 b on a specimen of the melt 106/30 and the metallographic structure in Fig. 2 c was obtained on the test specimen of the melt 109/10.

As far as the results of metallographic structure determinations shown in Fig. 2 a–c are concerned, no significant differences between these structures are observed, nevertheless these varying values of elongations are obtained during mechanical testings.

As mentioned above we investigated the causes of varying values of elongation and therefore further investigations of the elements present in phases in the investigated alloy were carried out.

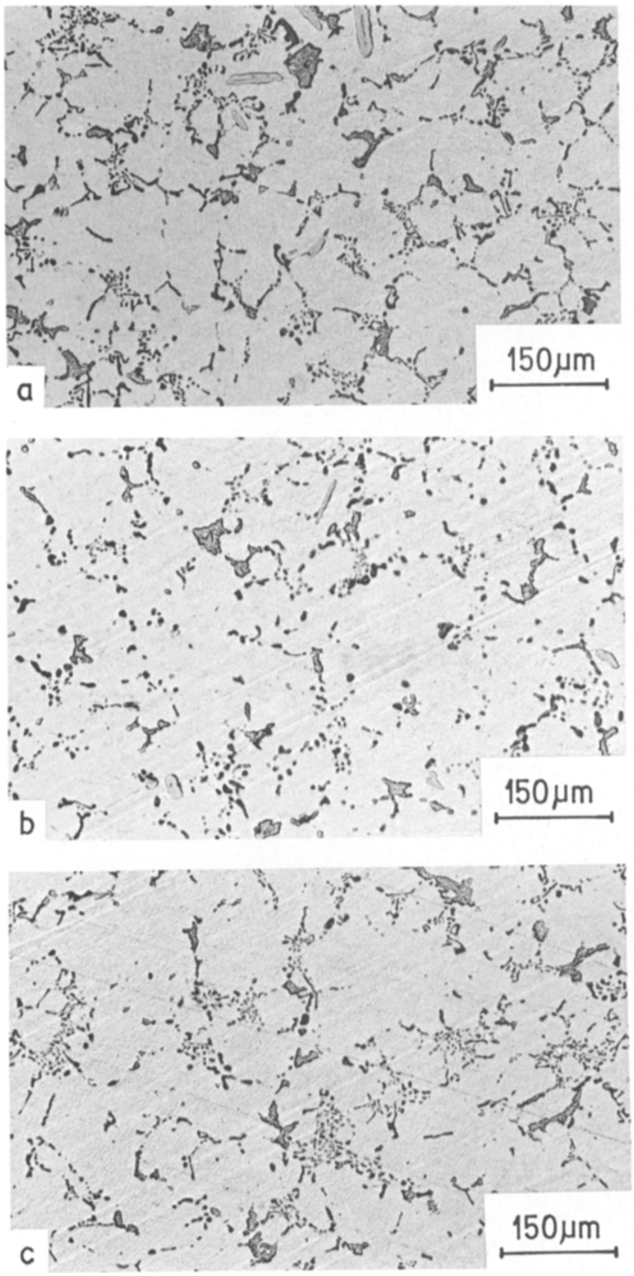

Fig. 2. The metallographic structure of Al-Mg-Si cast alloy. Magnification 100 X; **a**: melt no. 84/36, **b**: melt no. 106/30, **c**: melt no. 109/10

We investigated the distribution of elements in the α-solid solution and also the elements present at the interface of binary and ternary eutectic.

The measurements were performed with energy dispersive analysis and the EDXRF method of microanalysis was used.

The results of the investigations of the melt No. 109/10 are shown in Figs. 3 and 4. Fig. 3 presents the results obtained at the interface of the α-solid solution, and it can be seen that practically equal intensities of magnesium, aluminum and silicon peaks are obtained, with much lower intensity peaks for manganese and copper.

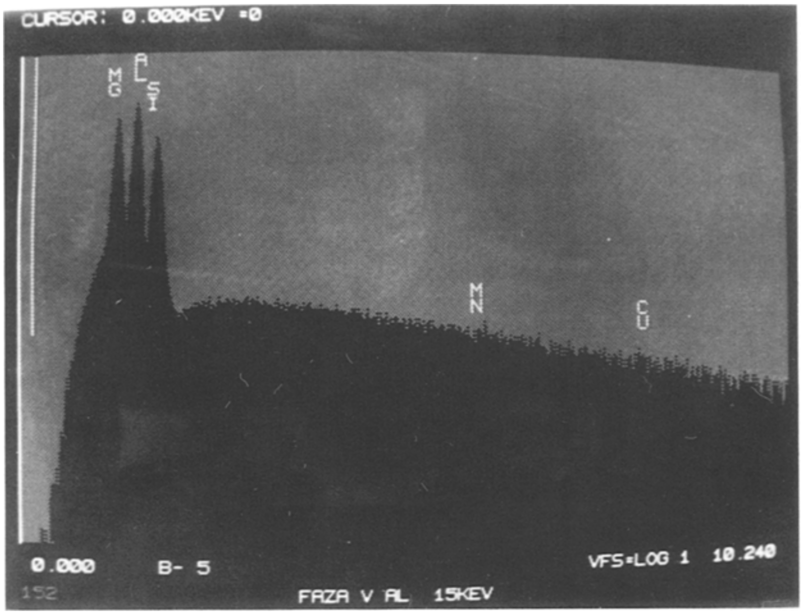

Fig. 3. Result of the X-ray microanalysis system TRACOR TN-2000 of grain boundary of the metallographic structure in Fig. 2

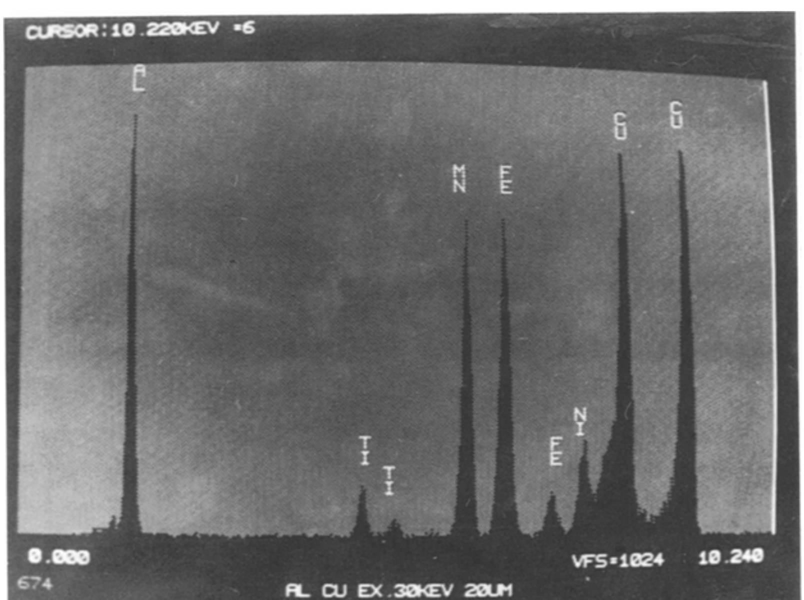

Fig. 4. Result of X-ray microanalysis of the solid solution in Fig. 2

Fig. 4 presents the result obtained in the α-solid solution and it may be observed that a very high aluminum intensity peak appears with a little lower intensity peak for copper and two practically identical but lower intensity peaks for manganese and iron. The lowest intensity peaks were obtained for nickel and titanium. The results of energy dispersive analysis show the distribution of elements in the α-solid solution and the binary and ternary eutectic on borders of the primary α-solid solution. Further investigations were devoted to the question of distribution of these elements in the interface between grains of the α-solid solution. For this purpose the wavelength dispersive analysis method was used and the results obtained on a sample of the melt No. 109/10 are shown in Fig. 5.

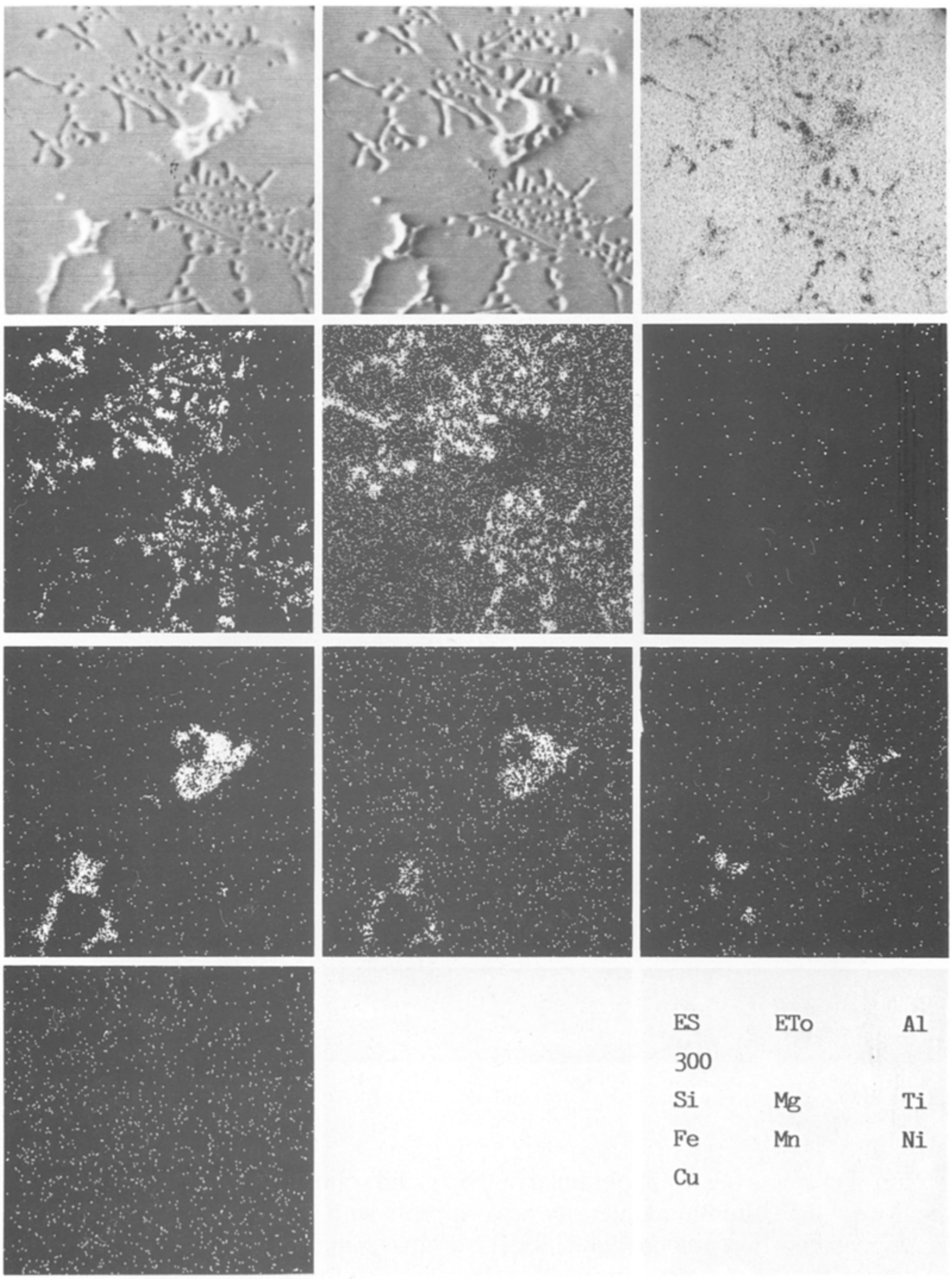

Fig. 5. Results of the wavelength dispersive analysis of the sample of the melt no. 109/10

The first two pictures in Fig. 5 show the electronic picture (ES) and the electronic topography (ETo) of some crystals of α-solid solution and the borders between these crystals with binary and ternary eutectic, presenting part of the metallographic structure shown in Fig. 2 c, at magnification of 300 x.

The results of the wavelength dispersive analysis in Fig. 5 show the distribution of aluminum, silicon and magnesium in the binary and ternary eutectic in the border of the α-solid solution. It is interesting to note that titanium was neither present in these phases nor in the α-solid solution. From the results shown in Fig. 5 it may be seen that iron, manganese and nickel are bound together in the investigated alloy.

The distribution of copper was identical to the distribution of titanium. As far as the distributions of titanium and copper are concerned, it must be noted that the measuring time for both elements was equal to 240 s.

During the casting procedure of aluminum alloys the process of desoxidation is important and therefore our attention was given also to the distribution of oxygen, eventually present in the alloy. For this purpose Auger electron spectroscopy was used. Analogous to the standard electron microprobe the Auger electron spectroscopy can be used to obtain images, chemical maps and spot analyses of surfaces. In addition, ion sputter etching enables to determine the in-depth profile of chemical elements present on the surface of the sample.

The Auger electron spectroscopy was used to detect titanium and eventually oxygen in the investigated melt no. 109/10. The result of the measurement on the surface is shown in Fig. 6.

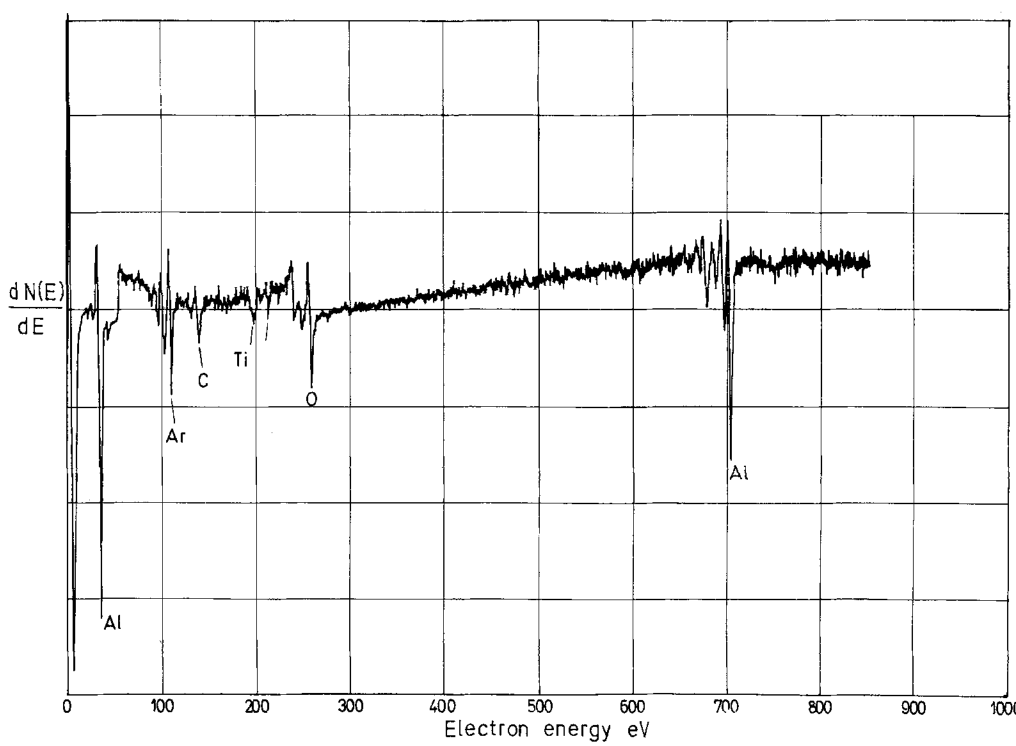

Fig. 6. Result of Auger electron spectroscopy of the phase in a sample of the melt no. 109/10

As far as the result shown in Fig. 6 is concerned it must be noted that small quantities of titanium and oxygen were detected on the surface. Also the in-depth profiling showed similar results, but with varying intensities of titanium and oxygen. These investigations will be continued and the results will be published later on.

Conclusions

Separately cast tensile test bars for quality control obtained during the production of the cylinder heads of air-cooled 6-stroke Diesel engines were investigated with EDX/WDX spectrometers and AES. The cylinder heads were cast from an Al-Mg-Si alloy of type G-AlMg$_5$Si known under the commercial name Hydronalium. After the casting procedure of the cylinder heads, they were subjected to a heat treatment procedure in order to obtain the prescribed values of mechanical properties. The principal aim of the investigation was to obtain, from the detailed analysis of phases present in the alloy, an explanation for the different values of elongation of the heat treated alloy. Special attention was paid to titanium, being added to the alloy in order to obtain a fine grained structure.

It was found that the highest values of nickel and titanium were followed by the highest values of elongation. Titanium was detected only in the primary α-crystals but only with the EDX-spectrometer. The results obtained with the WDX-spectrometer showed gradually distributed titanium in α-crystals and crystal borders. The AES method showed titanium bound to oxygen.

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