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HIGH-ACCURACY STRUCTURE IDENTIFICATION OF THE ALUMINIUM EUTECTIC ALLOYS USING THE COLOUR METALLOGRAPHY*

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Abstract. This paper presented the possibility to obtain the structural information using the colour contrast method for an eutectic aluminium alloy $Al_{12}SiCuMgNi$. The colour metallography of aluminium alloys represents a novelty in the area of aluminium alloys and improves the results obtained by optical metallographic microscopy concerning the internal structure of metals and alloys. This technique provides information on the structure and makes it possible to clarify the information on the structure in comparison with the black and white images. Also it allows to evaluation the individual types of phases in aluminium alloys, the identification of undissolved particles of metals (Zn, Si, Pb, etc), and evaluation of the quality of homogenizing, colour differentiation of the individual types of inclusions in aluminium alloys. The colour metallography results are correlated with the information obtained by means of TEM and XDR techniques. The photographs produced in a transmission microscope show the foreign particles and XDR analysis confirms that these foreign particles are based on silicon, Al-Ni-Fe-Mn and Al-Fe-Mn-Si. In this case, the colour contrast method allows the identification the aluminium alloy individual phases, identification nondissolved metal or master alloys particles like Si, identification the Fe particles penetrate during forming processes and colour distinguishing individual particles presented in aluminium alloy.

Key words: colour contrast method, microstructure, optical metallographic microscopy, XDR, TEM.

1. INTRODUCTION

Owing to their specific strength, low cost and superior corrosion resistance, aluminum alloys have been widely used in aerospace, automobile and construction industries. Also, by virtue of good thermal conductivity, they are often employed in heat exchanger manufacturing. Primary silicon is beneficial to enhance the wear/resistance ability and to reduce thermal expandability of Al-Si alloys, but it is detrimental to their strength and plasticity. Both the size and shape of the primary

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silicon phase determine the mechanical properties of the alloy [1-3]. The aim of the present work is to study the nature of the phases in an $Al_{12}SiCuMgNi$ eutectic alloy using the colour contrast method. X-ray diffraction (XDR) has been combined with transmission electron microscopy (TEM) to investigate the grain size and phase structure in eutectic alloy. It was confirmed that many phases are formed during the solidification process. The colour metallography technique is a set of procedures of optical metallographic microscopy utilizing the color contrast as a source of new information on the structure in comparison with classical approaches. The information and identification capacity concerning the structure are improved by the colour contrast, which can be obtained by the surface treatment of metallographic specimens (colour etching, vapour deposition) or by treatment of the surface of specimens (anodic oxidation) and using the additional devices of optical microscope (polarized light, Normanski prism) [4]. This method can be use to build up a database on phases forming during solidification process. Despite of these advantages, the extent of application of colour contrast in metallography of aluminium alloys is limited. TEM and XDR have proved the structural information obtained using the colour contrast method for an eutectic aluminium alloy analyses.

1.1. THE COLOUR CONTRAST METHOD APPLIED IN THE METALLOGRAPHY OF ALUMINIUM ALLOYS

Optical metallographic microscopy is the main method of examination of the structure of the materials. It is used the examination of the reflected light from planar sections of metallic samples by means of an optical light microscope. This method allows examining and evaluating the structure of metallic samples of the order 1 to 10^3 μm.

The colour contrast can be natural or induced and there are several methods for obtaining the colour contrast in the metallography of aluminium alloys [4]:

- The method of illumination of the specimen-polarized light;
- The method of surface preparation of the specimen-colour etching;
- The method of surface treatment of the specimen-vapour deposition of the interference layer.

During the casting process, a large amount of defects can be appearing. These defects affect both the macrostructure and the microstructure of aluminium alloys. The inclusions are regarded as foreign particles undissolved in aluminium. These inclusions (like oxide films, oxides of metals, nitrides, borides, carbides, remnants of moulding mixtures, etc) are undesirable in the melts because they decrease the quality of materials (the forming capacity of materials, impair the mechanical properties and corrosion resistance, etc). Colour metallography of aluminium alloys provides information on the structure and makes it possible to clarify the information on the structure in comparison with the black and white images. This method allows to evaluation the individual types of phases in aluminium alloys, the identification of undissolved particles of metals (Zn, Si, Pb, etc), and evaluation of the quality of homogenizing through colour differentiation of the individual types of inclusions in aluminium alloys.

The colour metallography results are correlated with the information obtained by means of TEM and XDR techniques. The TEM technique is used in the analysis of thin foils of aluminium alloys and allows obtaining important information about the substructure.

2. EXPERIMENTAL PROCEDURE

2.1. EXPERIMENTAL SETUP

Measurements have been made on samples of Al12SiCuMgNi with the composition, Si (12-12.2)%, Cu (1.1-1.3)%, Mg 1.3%, Mn 0.4%, Ni 1%, Ti 0.1% and impurities, Fe < 60 PPM and Zn < 20 PPM. The crucible is contained within a concentric stainless steel thermal shield. In addition, it is know that rapid solidification can better reflect the relationship between the structure of the melt and the microstructure after solidification, so the casting method used include normal permanent crucible casting and solidification under cooling rates. The crystallization process takes place during a controlled cooling by varying the air debit sent all around the crucible. The melt overcooling degree ΔT is detected by a chromel-alumel thermocouple encased in a quartz sheath of 1.7mm (outer diameter) placed at the center of the melt and a digital millivoltmeter type TR6656. The temperature measurement precision is 0.01°C. After solidification, transverse sections were cut from ingot ends and in central portion. The remaining portions were sectioned longitudinally for microstructure examination. The microstructure was observed by means of optical microscopy and transmission microscopy after metallographic preparation and etching. The phases as observed in the microstructure were identification using diffraction analysis (XDR).

2.2. COLOUR ETCHING IN ALUMINIUM ALLOYS

Both the reaction on the surface of metallographic section and the colour etching agent act in the formation of a transparent film important for interference coating. The thickness of this coating depends mainly on the chemical composition of the materials of the section and the etching conditions. The most important etching conditions are etching time, etching agent type, etching temperature and conditions of the prepared metallographic section. The different colours are produced as a result of the interference light and are depended on the thickness of the film and the individual microlocations of the structure [4].

For aluminium alloys there are a large number of etching agents that can be used in metallographic analysis. In case of eutectic AlSi12CuMgNi alloy, colour etching was carried out by conventional procedure: the composition of etching agent was 8 g $KMnO_4$ + 2 g NaOH + 200 ml distilled water. The preparation and application of etching agent: solution will be use maximum 2–3 hours after preparation and the etching time 20 s to 1.5 min. The section prepared in this way was examined in an optical microscope in light field.

3. RESULTS AND DISCUSSION

The photographs show the structure of the cast condition of AlSi12CuMgNi alloy. Microstructural observations present in Figs. 1 and 2 (a and b) displaying the matrix of an Al-Si eutectic phase, an AlFeMnSi phase, silicon particles, AlSiMn and AlNiFeMn phases. The XDR peaks confirmed the phases as observed in the microstructure of this AlSi12CuMgNi alloy. It is a non-modified eutectic silumin whose structure contains an eutectic in the form of needles of Si + α solid solution and large particles of undissolved silicon in the form of plates (Fig. 1). During the alloying procedure, violations of certain technological conditions may result in insufficient dissolution of the alloying elements and in formation of large independent particles. This effect appears when alloying elements present higher melting points than aluminium, so the solubility in aluminium of these elements is limited. Silicon is typical case.

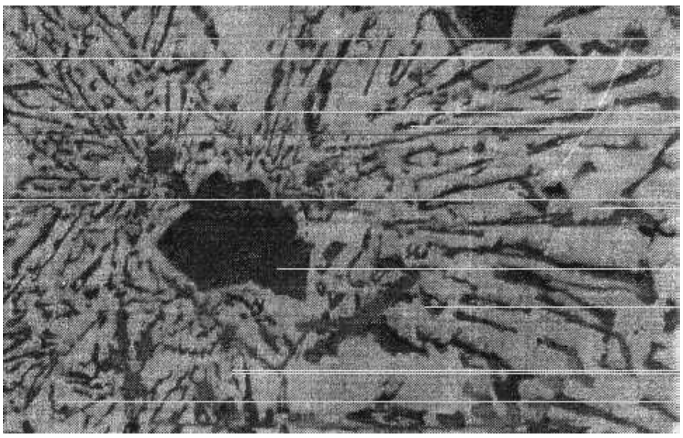
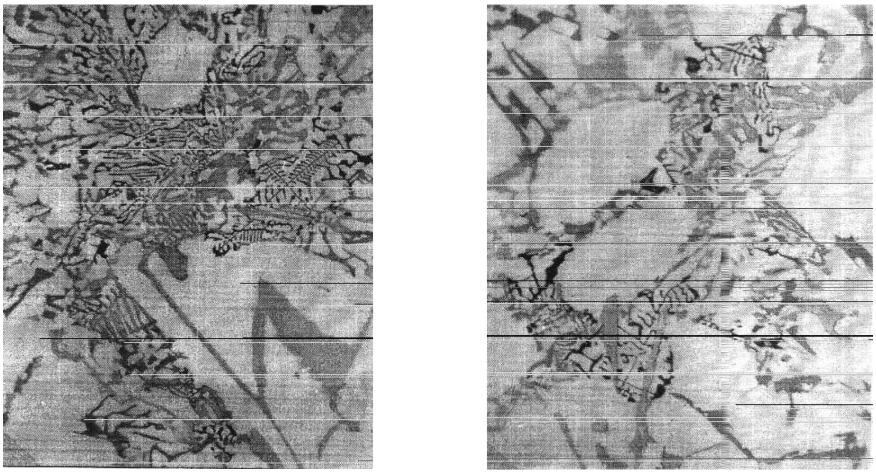


Fig.1 – An optical micrograph of the cast structure of the AlSi12CuMgNi alloy indicating the presence of large particles of silicon and a branched intermetallic phase based on AlFeMnSi (×200).

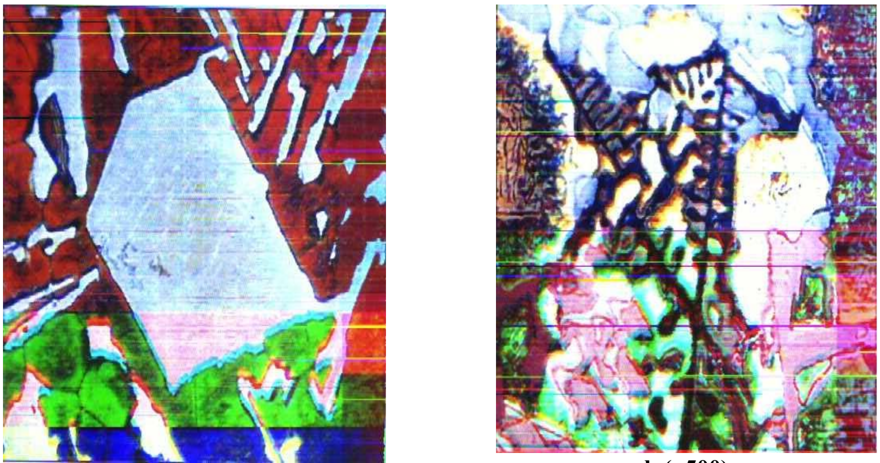


a (×150) b (×150)

Fig. 2 – (a and b). Optical micrographs of the cast structure of AlSi12CuMgNi alloy indicating branched light and black intermetallic phases based on AlSiMg (bright) and AlNiFeMn (black).

These undissolved particles are unacceptable inclusions due to their effects: they cause local inhomogeneity of the materials, may initiate cracking, they deplete the main material in the alloying elements, and these large particles decrease the mechanical and fatigue properties of the materials.

In Figs. 3 (a and b) are presented the cast structure of AlSi12CuMgNi alloy after colour etching. Fig. 3a shows a structure with a large blue silicon particle, light blue phase of AlFeMnSi and a brow solid solution. Fig. 3b shows a structure with large branched dark blue AlSiMg phase.



a (×500) b (×500)

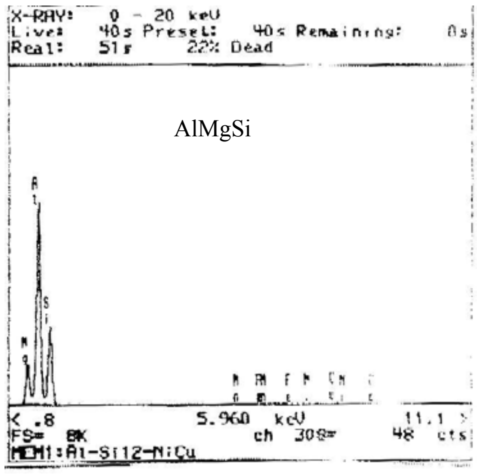
Fig. 3 (a and b) – The cast structure of AlSi12CuMgNi alloy after colour etching.

To determine the origin of the foreign particles and the different phases existing in structure of eutectic silumin alloy, the material was subjected to XDR analysis in a transmission electron microscope. Figures 4 and 5 show the photographs of a foreign particle produced in a transmission microscope and XDR analysis and confirm that the foreign particle is based on Al-Ni-Fe-Mn and Al-Fe-Mn-Si. In this case, the colour contrast method allows the identification the aluminium alloy individual phases, identification nondissolved metal or master alloys particles like Si, identification the Fe particles penetrate during forming processes and colour distinguishing individual particles presented in aluminium alloy.

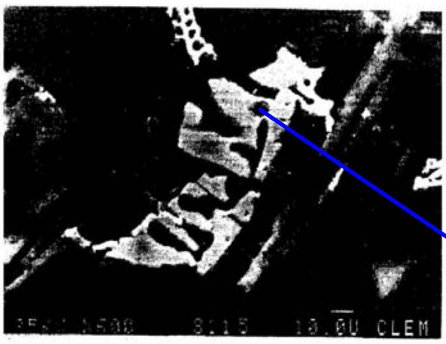
The temperature effect on the structure is one of the factors to modify the size and shape of the primary silicon. The onset temperature of nucleation exclusively determines the primary solidifying phase that depends on the cooling rate, the number of nucleation sites and the catalytic factors. These microstructures suggesting that the segregation of Si atoms is not very heavy and this process is strengthened at higher temperatures. Shingo *et al.* [5] found an interesting phenomenon of a "separated eutectic" occurring during unidirectional solidification of an Al12pctSi eutectic alloy with strong electric-magnetic stirring and some primary silicon appeared. Momono [6] also found that continuous convection flow intensified the gravity segregation of the primary silicon in an Al12pctSi eutectic alloy. The segregation can be attributing to convection transport, although in some cases, the segregation was due to sedimentation occurring during melting. It is not clear whether the segregation results from convection during solidification or from sedimentation during melting.

These results also show the possibility of segregation of Si atoms under additional forces like stirring and convection flow, somewhat similar to the controlled cooling of the molten metal. The possibility that fluid flow could disrupt the crystal bonding is negligible. The shear forces resulting from natural convection flow of melted metal are too weak to disrupt the bonding process during solidification, so these forces are not sufficient to break small dendrite arms (Figs. 2, 3 and 5). The structure exhibited a dendritic morphology in some location of sample (especially for outerlayer of casting sample). It is concluded that hydrodynamic forces are insufficient to cause breakage of dendritic arms under the present solidification conditions.

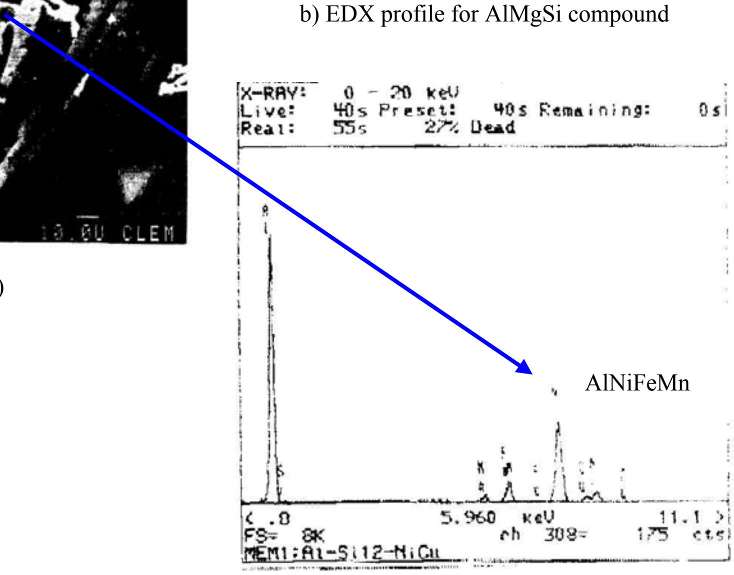
The atomic masses ($M_{\mathrm{Al}} = 27$ and $M_{\mathrm{Si}} = 28$) and atomic volumes ($V_{\mathrm{Al}} = 16.6\text{Å}^3$ and $V_{\mathrm{Si}} = 20.02\ \text{Å}^3$) of aluminium and silicon, respectively are similar [7], so it is expected that the segregation of Si atoms will not evolve into macrosegregation of Si phase. In these conditions, the presence of large independent Si particle (Fig. 1) shows that there are an insufficient dissolution of the alloying elements or, maybe, violation of certain technological conditions.



b) EDX profile for AlMgSi compound

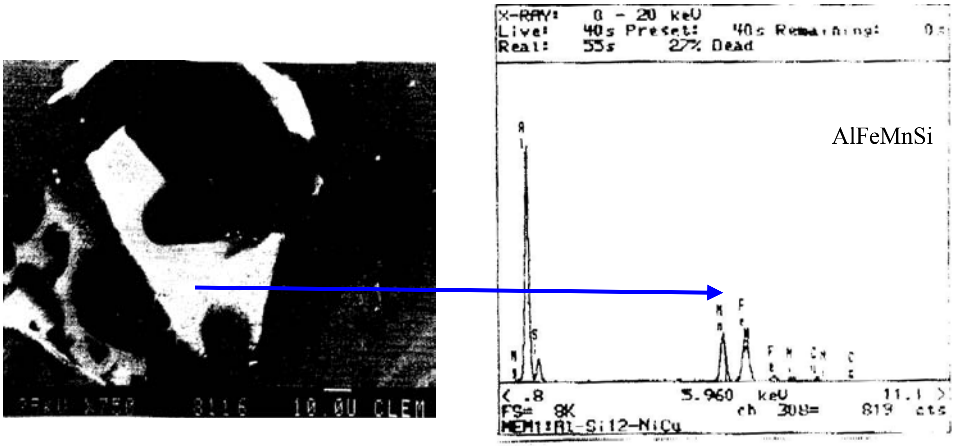


a) TEM image. (x600)



c) EDX profile for AlNiFeMn compound

Fig. 4 – Scanning electron micrographs (Fig. 4a) of the cast structure of AlSi12CuMgNi alloy and the results of EDX analysis (Figs. 4b and 4c) confirm that the gray phases are based on AlNiFeMn and the black phases on AlSiMg.



a) TEM image (×750) b) EDX profile for AlFeMnSi compound

Fig. 5 – Gray zone shows a particle composes basic to AlFeMnSi phase.

Despite of their proximity in the periodic table and similar crystallographic system (both-cubic), the structural component of the cast Al-Si alloy represent two completely different materials from the point of view of mechanical properties. α-Al solid solution has a ductile matrix and silicon particles are brittle precipitates. In Al crystals the atoms are connected whit metallic bonds. They change their relative position, in some range of displacement, without the loss of material compactness. In silicon crystals, the atomic bonds link the atoms. When internal stress surpasses the value of the cohesive forces in crystallographic planes, the bonds are broken without any atoms displacement and visible deformation. Also, the hard particles of the intermetallic phases can impinge on the mechanical properties of the materials, especially when containing iron (Figs. 2 and 3).

The morphology of the microstructural phase components influences very strongly their mechanical properties. The properties of the main structural components, such α-Al solid solution, primary and eutectic silicon particles and the particles of the intermetallic phases containing impurities or alloying elements, play an important part in the process of the microcrackes initiation and propagation.

4. CONCLUSION

The aim of this paper is to show how the colour metallography technique is proper to examine the structure of cast aluminium alloys. In this case, utilizing the colour metallography we can obtain more information about structure of aluminium alloys to comparing the classical method, it is enable to give better information about chemical structure, heterogeneity etc. Coupling with TEM and XDR techniques, the colour metallography give us detailed information about substructure of samples.

The results presented above suggest that the primary silicon phase is easy to coarsen and segregate under additional forces like stirring and convection flow induced by controlled cooling. The microstructure of this alloy includes an Al-Si solid solution, silicon particles and AlFeMnSi, AlSiMn and AlNiFeMn phases.

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