Journal of Achievements in Materials and Manufacturing Engineering

VOLUME 37
ISSUE 2
December
2009

Microstructure and mechanical properties of the Al-Ti alloy with cerium addition

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Received 20.09.2009; published in revised form 01.12.2009

Manufacturing and processing

ABSTRACT

Purpose: In this work there are presented the investigation results of mechanical properties and microstructure concerning mainly intermetallic phases of the aluminium – titanium alloy with a defined content of 2 and 4 % of cerium addition. The purpose of this work was also to determine the heat treatment conditions for solution heat treatment of the investigation alloys.

Design/methodology/approach: The reason of this work was to determine the heat treatment influence, particularly solution heat treatment time to the changes of the microstructure, as well to determine which intermetallic phases occur after the heat treatment performed, and how is the morphology of these particles.

Findings: After solution heat treatment for 4 hours the structure changes. The grains are larger and no more uniform as showed before. The most stable intermetallic in the Al-Ti system is the Al_3Ti phase. The solution heat treatment time should be greater than 4 hours to ensure a proper solution of titanium and cerium in the Al-α solid solution.

Research limitations/implications: The investigated aluminium samples were examined metallographically using optical microscope with different image techniques, scanning electron microscope and also analyzed using a Vickers micro-hardness tester, also EDS microanalysis was made.

Practical implications: As an implication for the practice a new alloy can be developed, some other investigation should be performed in the future, but the knowledge found in this research shows an interesting investigation direction.

Originality/value: The combination of light weight and high strength Ti-based alloys is very attractive for aerospace and automotive industries. Furthermore, the presence of calcium cerium into existence new unknown phases as well can enhance the thermal stability of ternary Al-Ti-Ce alloy because of its higher melting point then Al-Ti.

Keywords: Casting; Heat treatment; Aluminium alloys; Cerium

Reference to this paper should be given in the following way:

L.A. Dobrzański, K. Labisz, R. Maniara, A. Olsen, Microstructure and mechanical properties of the Al-Ti alloy with cerium addition, Journal of Achievements in Materials and Manufacturing Engineering 37/2 (2009) 622-629.

1. Introduction

New developed aluminium based alloys, especially with titanium, are getting more popularity due to their excellent properties. The combination of light weight and high strength makes Ti-based alloys very attractive for aerospace and automotive industries. There exists also a more and more increasing need for next sophisticated materials for various high – temperature applications. A number of studies on the phase diagram of the Al-Ti alloys are found in the literature. The methods used to calculate the phase diagram differ and some discrepancies still remain. Murray calculated the phase diagram by optimization of Gibbs energies with respect to phase diagram and thermochemical data. Kattner developed a phase diagram from calculations based on a last-square technique to optimize the thermodynamic quantities of the analytical description using experimental data available in the literature. Most commonly used of these methods is the casting in water as a cooling medium. Very high cooling rates can be achieved in the range of 104 – 108 Ks-1 during solidification from the molten state [1-2].

The mechanical behaviour of a material strongly depends on its microstructure, which can be controlled to a certain extent by the processing conditions. With the introduction of novel production methods like high-energy milling, inert gas condensation or electrodeposition, it became possible to synthesize materials with ultra fine grain sizes in the submicron or nanometer range [1, 2 and 3]. The very high density of two-dimensional defects like grain or phase boundaries was thought to impose a significant influence on the physical and mechanical properties of these materials. However, up to now, there is no clear understanding of the mechanisms that govern the mechanical behaviour of materials with ultra fine grain sizes. Generally, rules and equations describing the correlation between mechanical data and microstructural parameters of conventionally grained materials may either continue or break down, if the grain size falls markedly below 1 m [4,5].

On the basis of theoretical considerations, deviations may arise from the fact that size parameters gradually reach the dimensions of some characteristic lengths related to the original deformation mechanism, e.g. the width of a dislocation source in the case of glide deformation. Under these circumstances, additional constraints may complicate or impede single steps of the formerly prevailing deformation mode, thus contributing to changes in the mechanical behaviour. The extension of solid solubility limits afforded by rapid solidification offers greater flexibility in selection of alloying additions. In addition, the possibility of establishing thermally stable fine-scale dispersion of secondary phases in the as-quenched structure or during subsequent heat treatment offers potential improvement in mechanical properties of alloys via this route. Al-based alloys are particularly suitable to be developed by solution heat treatment and ageing, but only nine elements show appreciable solid solubility (greater than 1 at. %) in Al and only five of these have been exploited commercially [3-6].

Cerium was used as the alloying additive, because it:

- is mainly used in the form of rare earths elements with 50 to 60 % Ce content has been added experimentally to casting alloys. In the used alloy there are also other ere earth elements like Gd, Nd, La (Fig. 1)
- Increases fluidity
- Reduces die sticking
- Transforms acicular $FeAl_3$ into a noncicular compound

According to experimental investigations, nano- and submicrocrystalline alloys are usually characterized by high values of strength and hardness (e.g. [6]). Many studies have addressed the question of to what extent these observations may be explained by dislocation-controlled Hall–Petch behaviour, entailing a correlation between yield strength (y) and grain size (d) according to this dependence.

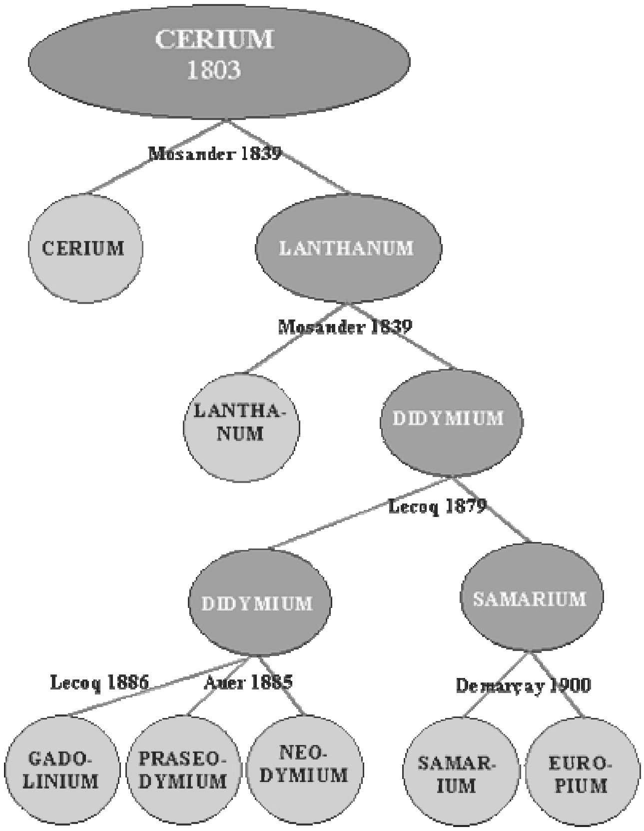


Fig. 1. Elements discovered in ceria [M.E. Weeks, P. van de Krogt]

Like Al-Fe also Al-Ti alloys with ternary and often quaternary additions typically have microstructures comprising a large volume fraction of thermally stable, dispersed intermetallic phases distributed a large volume fraction of thermally stable, dispersed intermetallic phases distributed uniformly in an Al matrix. Al-Ti alloy system is one of a group of Al-based peritectic systems with potential for development. The $TiAl_3$ intermetallic phase is intrinsically stable with a melting point of 1623 K [7-15].

Due to the low equilibrium solid solubility and diffusivity of Ti in Al, the potential exist for generating a refined microstructure comprising stable, fine-scale dispersion of intermetallic phases by additions or by controlled post-solidifications heat treatment. Furthermore, the presence of Ce can bring into existence of new unknown phases as well as can enhance the thermal stability of ternary Al-Ti-Ce because of its higher melting point then Al-Ti.

An additional interest in Al-Ti alloys arises from their common use in grain-refining in the casting of Al alloys [16-25].

2. Experimental procedure

The experimental aluminium-titanium alloys with cerium addition were investigated in this work. The exact chemical composition is showed in Table 1. Using an electro-resistance furnace (Fig. 2) all elements with the calculated and measured amount of the additives were melted in a ceramic crucible by induction heating and then melt into a carbon form, witch was cooled in air in a water-cooled aluminium block. in the furnace a controlled protective argon atmosphere was used to avoid contamination and oxidation of molten aluminium and additives.

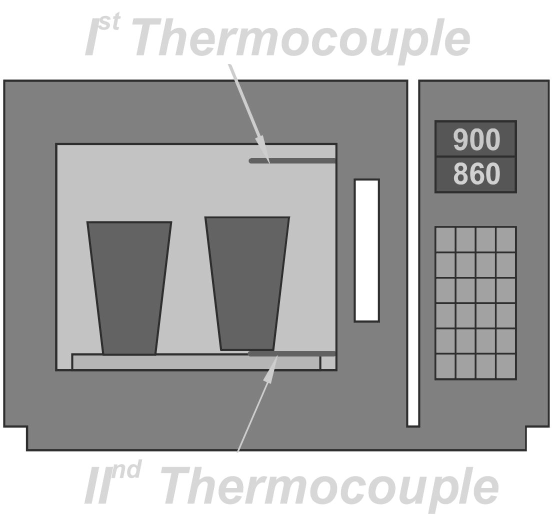


Fig. 2. Schematic thermocouple placement in the electro-resistance furnace

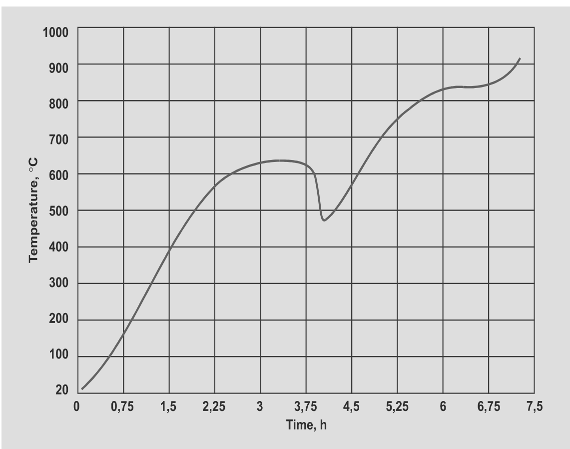


Fig. 3. Temperature curve registration for proper adjusting of the measured temperature value

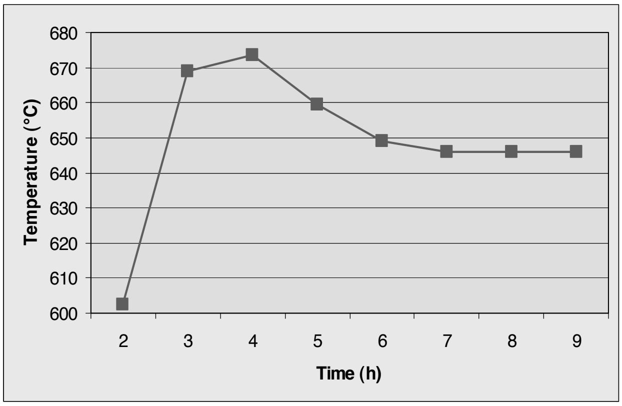


Fig. 4. Furnace temperature adjustment

The mould obtained, was 40 mm in diameter and about 30 mm in high. Samples were used of dimension of 10x10x2 mm for optical microscope and scanning electron microscope. The temperature was measured by a computer software connected to the thermocouples and registered as presented in Fig. 3. The furnace temperature was adjusted before heating (Fig. 4) to deliver probably measurements; a drying period for the whole furnace was also applied to avoid moisture.

An important point to emphasize is the fact that high purity raw materials of 99.99% Al and 99.99% Ti these were the highest purity industrially available. Special care has also been taken in the melting and casting process: an alumina and a graphite crucible made from a high purity material was used, together with argon of 99.9999% purity to avoid contamination by gas elements, especially hydrogen. After annealing the sample were polished and prepared for EDX analysis as well SEM investigations. This step is taken to ensure that the desired alloy was obtained without any undesirable phases such as oxides.

Table 1.
Chemical composition of the investigated alloys

alloy	Al	Ti	Ce
Al2Ti2Ce2	96	2	2
Al2Ti2Ce4	94	2	4

For casting a commercial grade Al-Ce alloy was used and Al-Ti alloy in form of sheets of approximately 4 mm in thickness. The main difficulty during casting process was to ensure a homogeny structure in the whole sample, therefore: the melt has to be mixed and a uniform cooling rate has to be applied. After casting a solution hest treatment was applied. EDS microanalysis on the SEM microscope was used to identify the chemical composition of the phases present in the alloy.

The phase identification was also performed using transmission electron microscopy together with energy dispersive spectroscopy (EDS). The hardness was measured with Vickers microhardness tester with a load of 0.05 kg and a measurement time of 10 s. A minimum of 8 indentations was made on each of the as-cast and solution heat treated samples. For temperature measurement a chromel-alumel thermocouple was applied.

3. Results and discussion

As a result of SEM investigation micrographs of the Al_3Ti and Al-Ce contained phase is presented on Figures 6 and 7 were also some other phases containing iron are present as well La, Gd, Nd and of course Ce. The same phases, especially the Al_3Ti phase could be observed also in the optical micrographs presented on Figures 8 to 15. The solid solubility of cerium is higher than that of titanium, so this cerium containing phases showed on Figures 14 and 15 occur mostly on the grain borders of the aluminium grains present on Figures 8, 9, 12 and 13 and build very small dispersive phases, this particles could be detected using TEM with very high magnification, about 200 000 x.

Titanium phase Al_3Ti showed on Figure 3b is present in form of bulk particles over the whole structure. To establish the highest possible solution heat treatment temperature, the samples were heat treated also in higher temperature until small areas with partial melting places were detected. So in finally a temperature of 550° C was determined as the highest possible. Furthermore structural changes were find in samples with different time of solution heat treatment compared to the structure of as cast alloy.

Generally a grain growth can be state according to the selected time. Some further more exact investigation belong this point will follow. Hardness measurement are presented in Fig 5, the highest value occurs for 4 hours solution heat treatment and is shorter than the values found in literature.

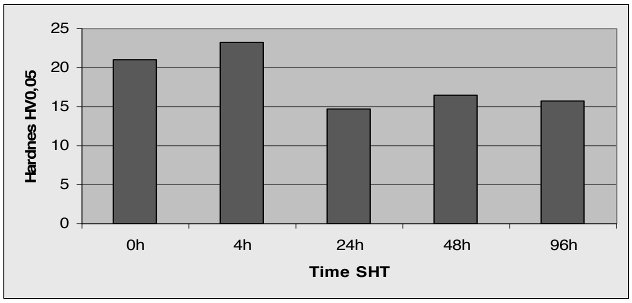
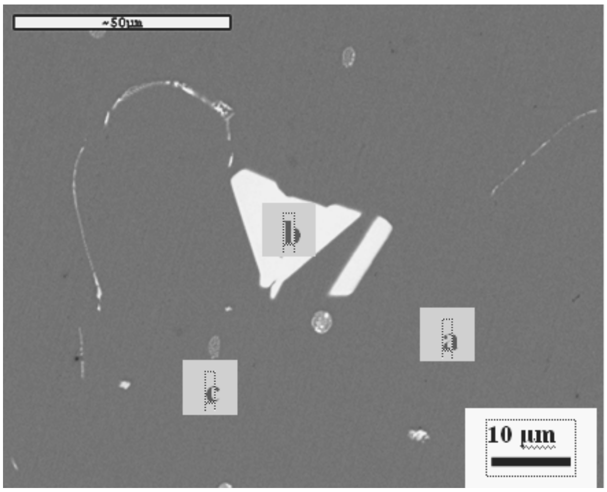
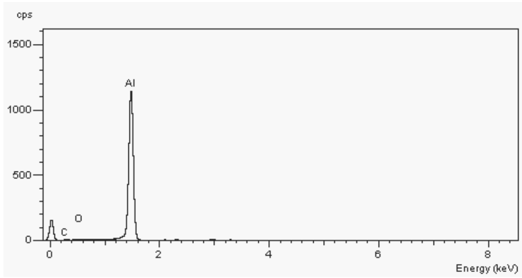


Fig. 5 Hardness measurement results after different times of solution heat treatment



a)



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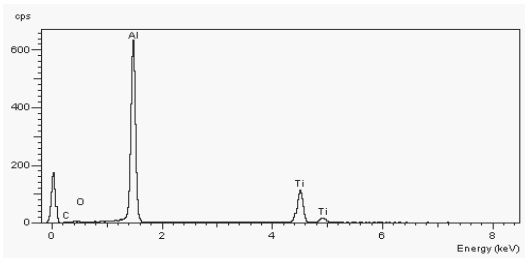

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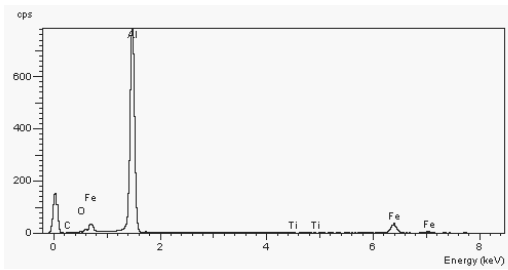

Fig. 6. EDS microanalysis performed on the marked places (a, b, c)

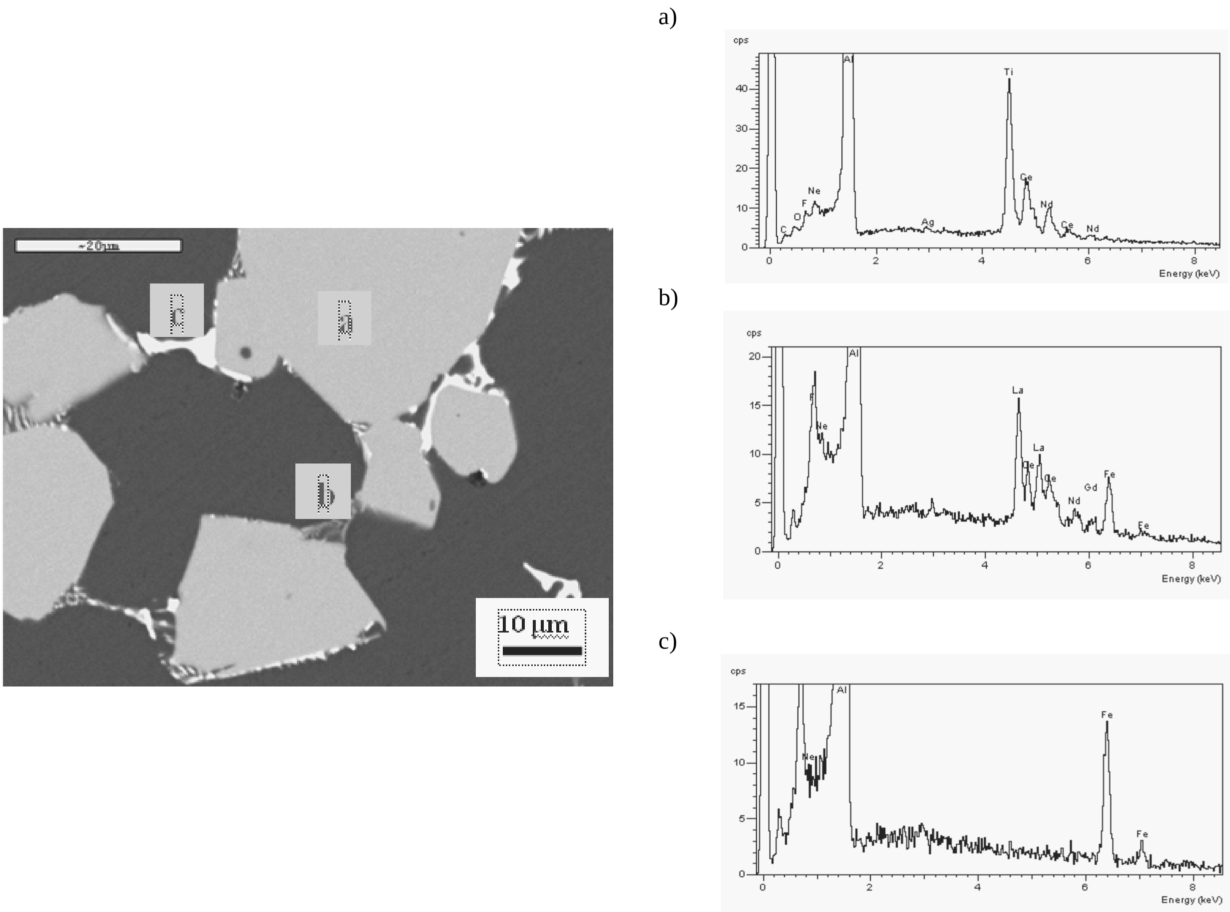

Fig. 7. EDS microanalysis performed on the marked places (a, b, c)

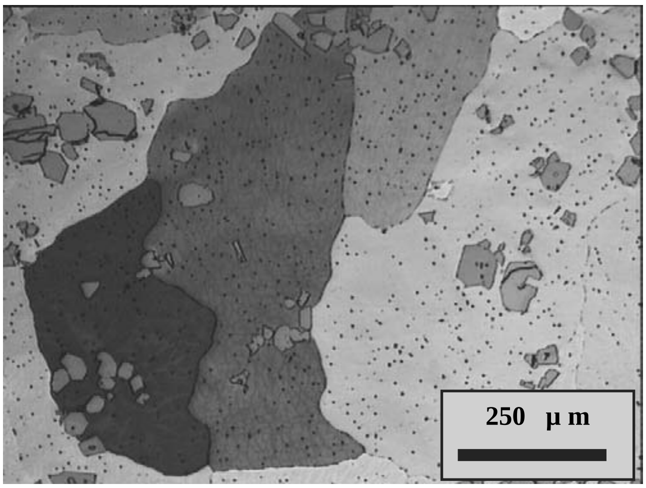

Fig. 8. Microstructure of the Al2Ti2Ce2 alloy in the as-cast state

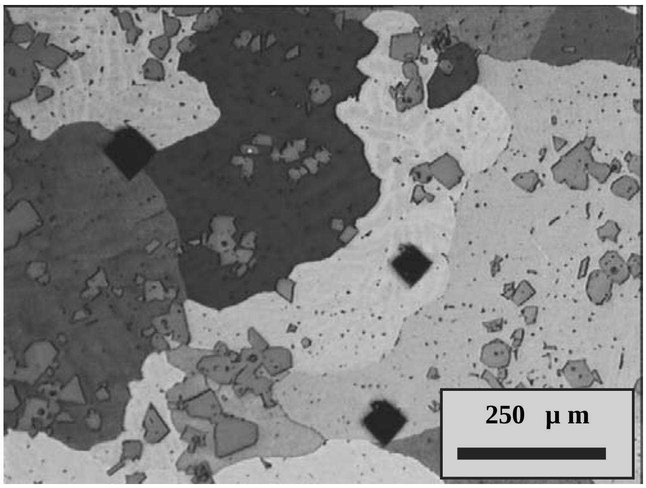

Fig. 9. Microstructure of the Al2Ti2Ce2 alloy after SHT in 595 º C for 4 h

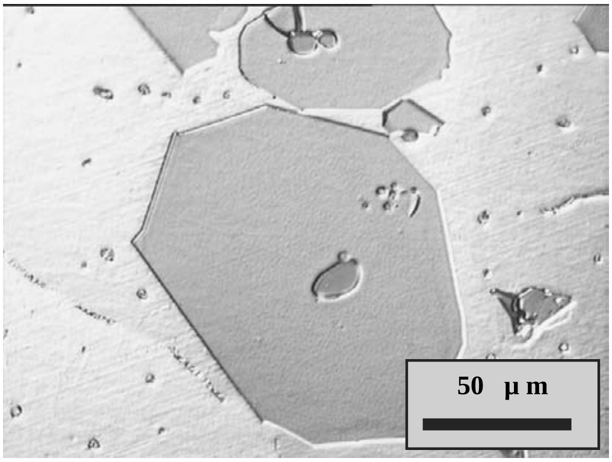

Fig. 10. Microstructure of the Al2Ti2Ce2 alloy in as-cast state

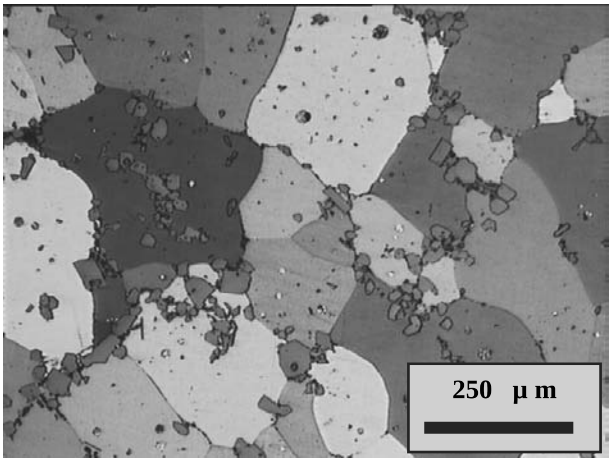

Fig. 13. Microstructure of the Al2Ti2Ce4 SHT in 600ºC for 4 h

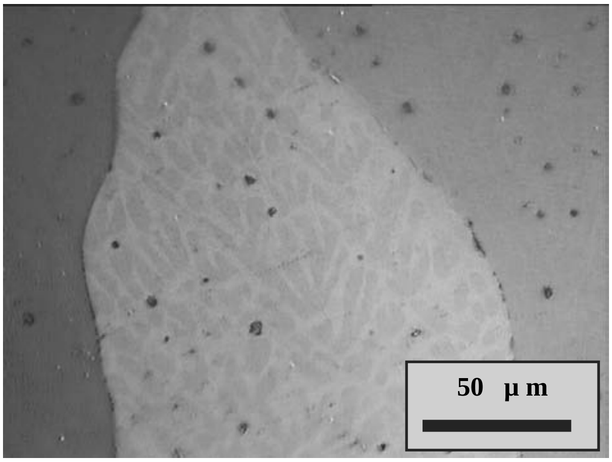

Fig. 11. Microstructure of the Al2Ti2Ce2 alloy in as-cast state

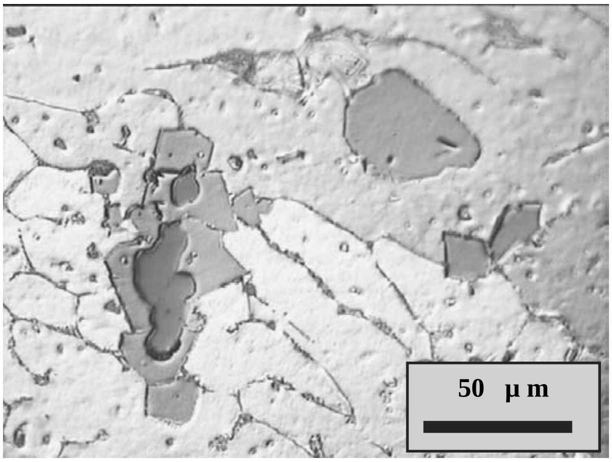

Fig. 14. Microstructure of the Al2Ti2Ce4 in as-cast state

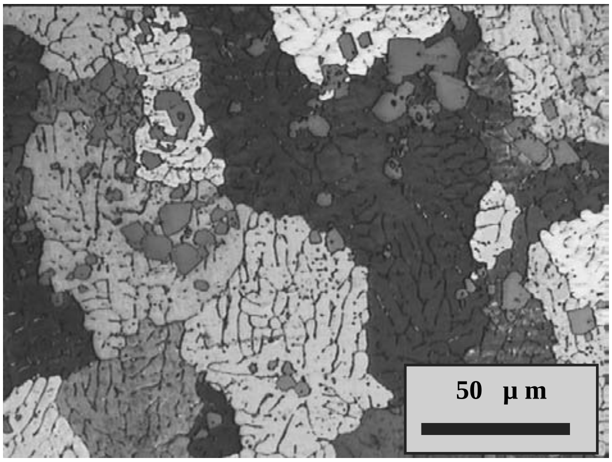

Fig. 12. Microstructure of the Al2Ti2Ce4 in as-cast state

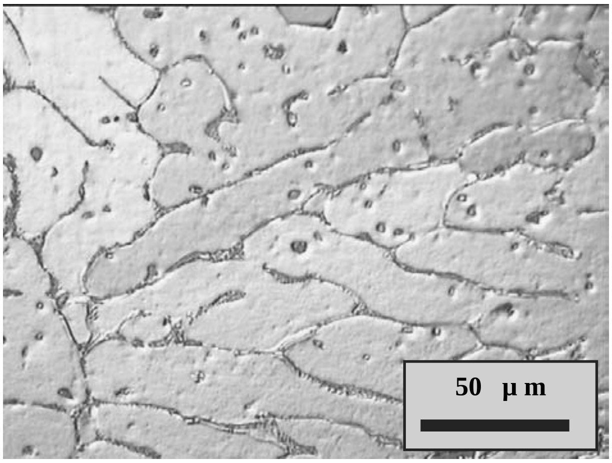

Fig. 15. Microstructure of the Al2Ti2Ce4 as-cast

4. Conclusions

It could be state on the basis of the structure micrographs that directly after casting the as-cast alloy has very small grains and a uniform structure. After solution heat treatment for 4 hours the structure changes in a way, that the grains are larger and no more uniform as showed before. This process is continuing during a longer solution heat treatment time of 24, 48 and 96 hours. We can observe that with a prolongation of the solution heat treatment time the phase

Al_3Ti is growing up and breaking up in smaller parts. This process is continuing with increasing of the solution hat treatment time. The titanium of the particles is going into the matrix.

This process is confirmed with the hardness measurements where after 4 h solution heat treatment 23 HV 0.05 is detected. After 24 hours SHT and more the hardness decreases below the value of as-cast alloy - 15 HV 0.05 (Fig. 16). The reason for that can be, that the particles are growing together so that the embedded phase in the supersaturated Al-α solid solution is no more uniform spread; this lead to decreasing of the hardness, because the strengthening influence of small dispersive particles is loosed.

We can conclude that:

- The most stable intermetallic phase in the investigated Al-Ti system is the Al_3Ti phase.
- The solution heat treatment time should be greater than 4 hours to ensure a proper solution of titanium and cerium in the Al-α solid solution.

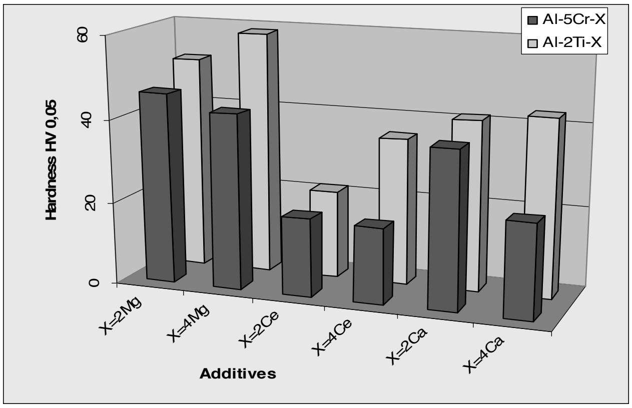

Fig. 16. Hardness measurements results of the alloys with magnesium cerium and calcium addition

Acknowledgements

The author will express his great thanks to the Center of Materials Science, especially to the Structural Physics group for the leading and scientistifically support in elaboration of this project. The authors are grateful to Mr. Arne Olsen, Mr. Przemysław Zagierski and Mr Eric Sørbreden for his valuable and very usefully comments and assistance in preparing of the investigation.

The investigations carried out were partially financed within the framework of the Norwegian Government Scholarship and of the Structural Physics, Centre for Materials Science at the University of Oslo.

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