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

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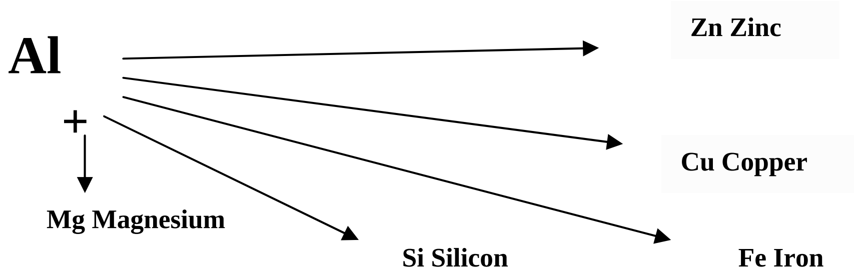
ABSTRACT

Purpose: In this paper there are presented the investigation results of mechanical properties and microstructure with intermetallic phases of the aluminium – titanium alloy with a defined content of Ca 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 in a way, that 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 calcium in the Al-α solid solution.
Research limitations/implications: The investigated aluminium samples were examined metallographically using optical microscope with different image techniques, SEM, TEM and 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 can bring into existence new unknown phases as well can enhance the thermal stability of ternary Al-Ti-Ca alloy because of its higher melting point then Al-Ti.
Keywords: Casting; Heat treatment; Aluminium alloys; Calcium

1. Introduction

Aluminium based intermetallics, 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

Table 1.
The most important alloying additives and their effect [15]



Additive	Effect
Zn Zinc	Increased strength and hardness. Possibility for stress corrosion. Gives heat-treatable alloys, when combined with Mg.
Cu Copper	Gives heat-treatable alloys. Increased strength and hardness. Reduced corrosion resistance.
Mg Magnesium	Increased strength. Increased hardness. Good corrosion resistance. Increased weld ability.
Si Silicon	Gives heat-treatable alloys, when combined with Mg. Good corrosion resistance.
Fe Iron	Most common impurity found in aluminium. Reduces the grain size.

Table 2.
The most important alloying additives and their effect [6]

alloy	Al	Ti	Ca
Al2Ti2Ca2	94	2	2

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 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 (Table 1) of these have been exploited commercially [3-6].

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 [7-10].

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 Ca can bring into existence of new unknown phases as well as can enhance the thermal stability of ternary Al-Ti-Ca 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 [11-16].

2. Experimental procedure

The experimental aluminium-titanium alloys with Ca addition were investigated in this work. The exact chemical composition is showed in Table 2. Using an electro-resistance furnace 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. The mould obtained, was 40 mm in diameter and about 30 mm in high. Two types of sample were used: 10x10x2 mm for optical microscope, SEM and thin foils for TEM investigations.

The furnace temperature was adjusted before heating (Figure 1) - Especially the thermocouple - 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 as TEM and SEM investigations. This step is taken to ensure that the desired alloy was obtained without any undesirable phases such as oxides.

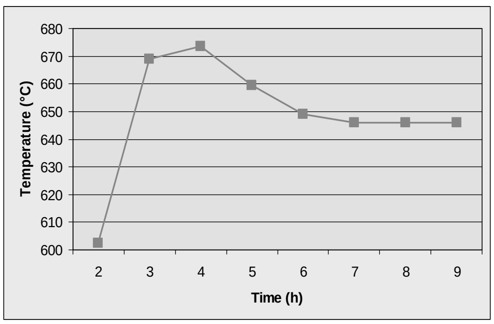


Fig. 1. Furnace temperature adjustment

For casting a commercial grade Al-Ca 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 (TEM) 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 a micrograph of the Al_3Ti and Al-Ca contained phase is presented on Figure 2. The same phases, especially the Al_3Ti phase could be observed also in the optical micrographs presented on Figure 4. The solid solubility of calcium is lower than that of titanium, so this calcium containing phases showed on Figure 3b occur mostly on the grain borders of the aluminium grains present on Figure 3c 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 3a 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.

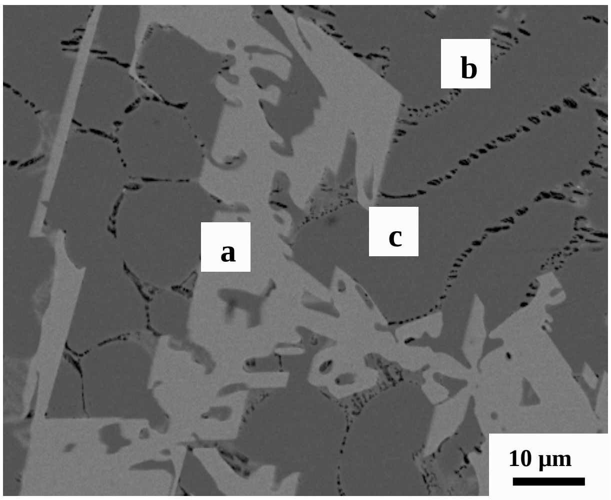


Fig. 2. Al_3Ti particle (a) and calcium containing phase (b) occu-rred in the as cast alloy, (c) aluminium matrix, SEM – image

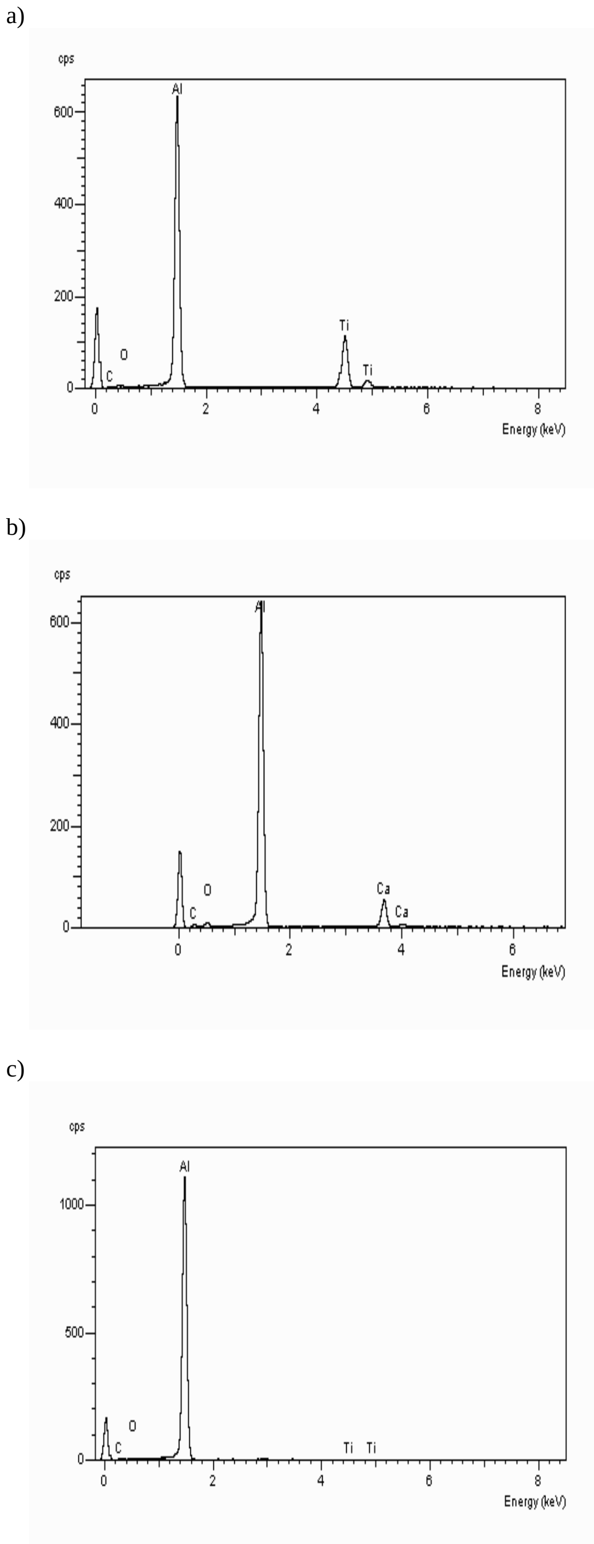


Fig. 3. EDS microanalysis performed on the marked places (a-c) on Figure 2

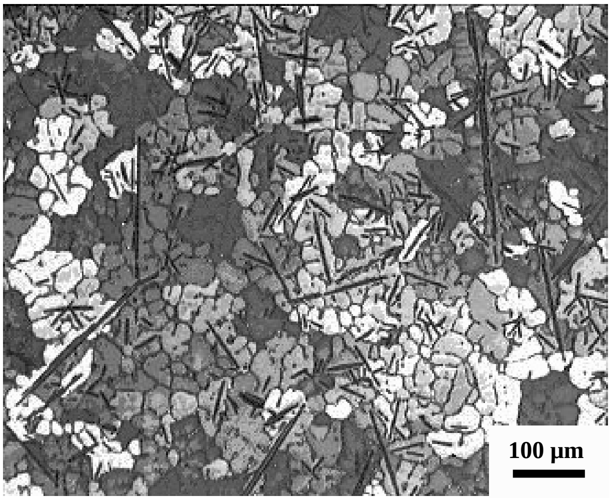


Fig. 4. Microstructure of the as-cast alloy

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 29 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. 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:

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

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