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Microstructure investigations and hardness measurement in Al-Ti alloy with additions of Ce after heat treatment

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Abstract: Mechanical properties and intermetallic phases of the aluminum – titanium alloy have been investigated over a defined content of Ce additives. Experimental results of the hardness measurements showed a dependence on the kind and conditions of heat treatment.

Keywords: Cerium, Aluminium, Phases, Al_3Ti

1. INTRODUCTION

Aluminum 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 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 [1].

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 10^4 – 10^8 Ks^{-1} during solidification from the molten state [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 [3].

Al-based alloys are particularly suitable to be developed by solution hest treatment and ageing, but only nine elements show appreciable solid solubility (greater than 1 at. %) in Al and only fife of these have been exploited commercially [4]. 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 [5, 6, 7]. 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 [8].

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 [9, 10, 11].

A number of studies have been undertaken to investigate the microstructure of rapidly cooled Al-Ti alloys [12, 13, 14, 15], but not very few was done about Al-Ti alloys with additions of especially cerium. The purpose of this work is to investigate phase- and microstructural evolution and microhardness of the as-cast Al-Ti-Ce alloy as well as after solution hest treatment.

2. EXPERIMENTAL PROCEDURE

The experimental aluminum-titanium alloys with Ce addition were investigated in this work. The exact chemical compositions are showed in table [1]. 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 aluminum block. In the furnance a controlled protective argon atmosphere was used to avoid contamination and oxidation of molted aluminum and additives. The molt (Fig. 1) obtained was 40 mm in diameter and about 30 mm in high. Two types of sample were used 10x 10 x 2 mm for TEM investigations.

Table 1.
Chemical composition of the aluminum alloys investigated (wt. %).

alloy	Al	Ti	Ce
Al2 Ti2 Ce4	94	2	4

The furnance temperature was adjusted before heating (Fig. 2.) - Especially the thermocouple - to deliver probably measurements; a drying period for the whole furnance 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.

For the casting was used a commercial grade Al-Ce in form of rod of approximately 10 mm in diameter and Al-Ti alloy in form of sheets of approximately 4 mm in thickness (supplied by).

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Figure 1. The molt obtained was 40 mm in diameter and about 30 mm in high

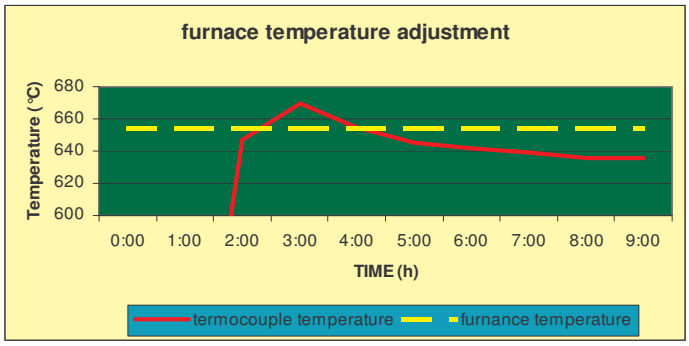

Figure 2. Furnace temperature adjustment.

It was the main difficulty during casting process to ensure a homogeny structure in the whole sample, therefore: the melt has to be mixed and an uniform cooling rate has to be applied. After casting a solution hest treatment was applied.

X-ray diffraction (XRD) was used to identify the phases present in the as-cast alloy directly after casting. The phase identification of the as-cast alloy phases was also performed using XRD with Kα and (Kβ) radiations. The microstructure of the samples was characterized using transmission electron microscopy (TEM) together with energy dispersive spectroscopy (EDS). The foils for TEM observations were punched and prepared using electro polishing device. 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 o SEM investigation two micrographs of the Al_3Ti and AlCe phase are presented in figure 4 and 5. The same phases, especially the Al_3Ti phase could be observed also in the optical micrographs in figure 6-11. The solid solubility of cerium is higher than that of titanium; this is the reason for so huge amount of titanium phase present in the presented micrographs. Cerium figure 4 builds very small dispersive phases, which could be detected using TEM with very high magnification (200 000 x).

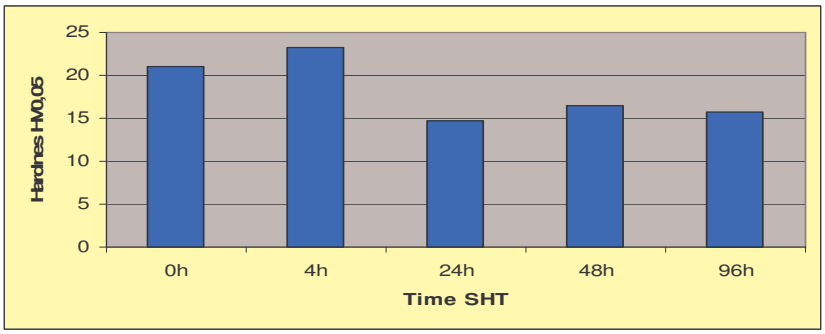

Figure 3. Results of microhardness measurements of the of the Al2 Ti2 Ce4

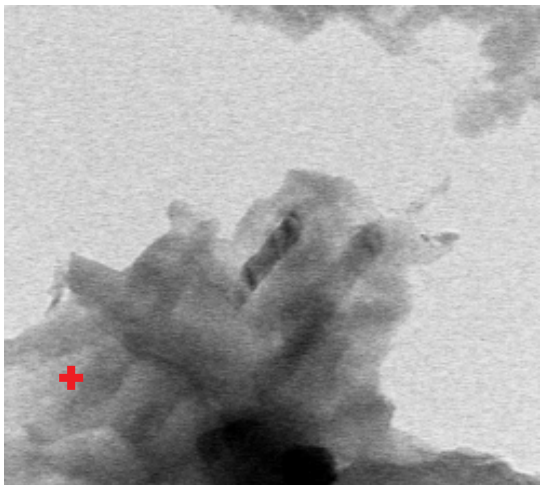

Figure 4. AlCe (1) particle in the aluminum matrix (2), TEM - image, mag. 200 000 x

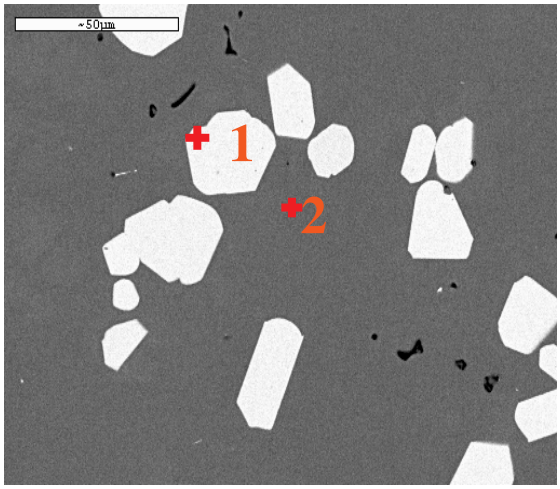

Figure 5. Al_3Ti particle (1) in the aluminum matrix (2), SEM - image, mag. 5000 x

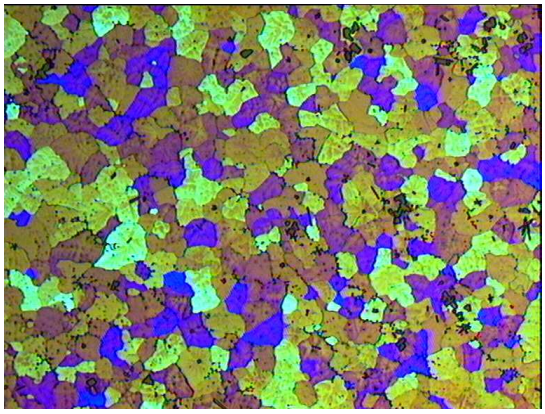

Figure 6. Optical micrograph of the Al2 Ti2 Ce4 – as cast alloy, mag. 200 x

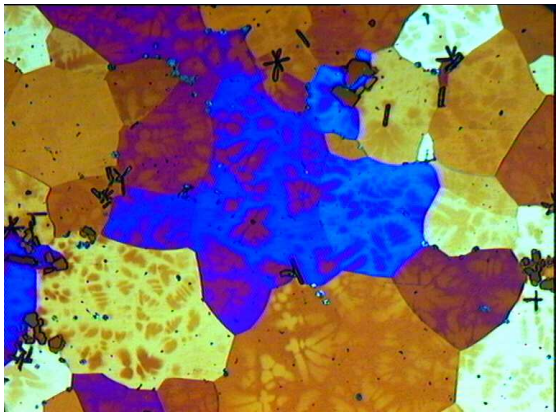

Figure 7. Optical micrograph of the Al2 Ti2 Ce4 – after SHT in 550° C for 4 hours, mag. 200 x

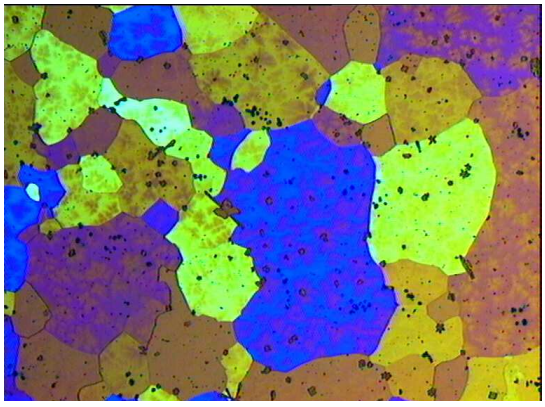

Figure 8. Optical micrograph of the Al2 Ti2 Mg4, after SHT in 550° C for 24 hours, mag. 200 x

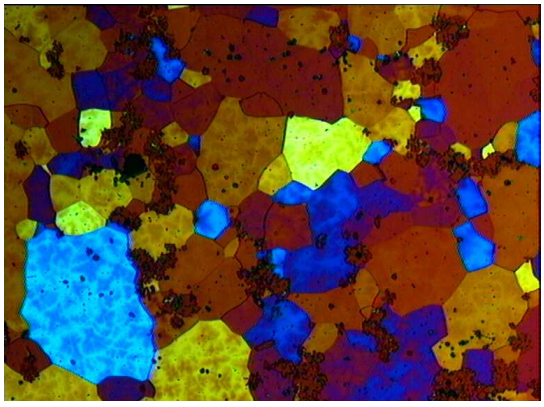

Figure 9. Optical micrograph of the Al2 Ti2 Mg4, afterSHT in 550° C for 48 hours, mag. 200 x

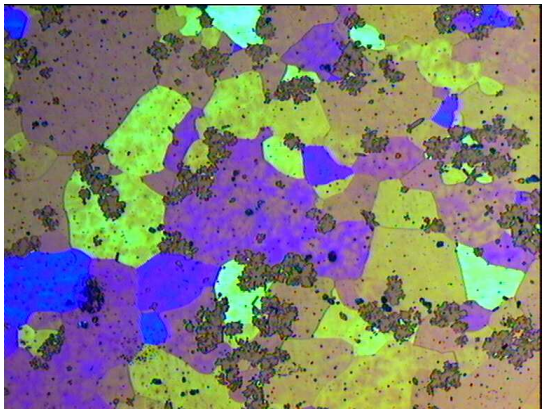

Figure 10. Optical micrograph of the Al2 Ti2 Ce4, after SHT in 550° C for 96 hours, mag. 200 x

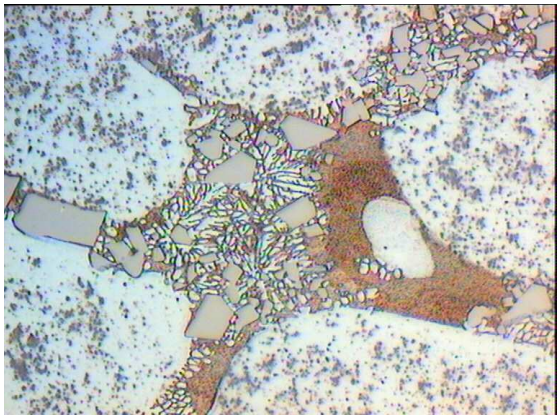

Figure 11. Optical micrograph of the Al2 Ti2 Ce4, after SHT in 560° C for 4 hours, mag. 400 x, partial melting areas

4. CONCLUSION

It could be seen on the basis of the structure micrographs showed in figure 6, 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 results showed in figure 3 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 - 21 HV 0,05. The reason for that is, 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 properties of Al.-Ti system were determined using an EDX method. The most stable intermetallic in the 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 cerium in the Al-α solid solution.

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