

Executive Summary of the Thesis Entitled

Characterization of 7075 Aluminium Alloy Using Modifier and Heat Treatment

Submitted to

THE MAHARAJA SAYAJIRAO UNIVERSITY OF BARODA

In the partial fulfilment of the requirements for the award of degree

DOCTOR OF PHILOSOPHY

in

METALLURGICAL AND MATERIALS ENGINEERING

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May 2023

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Background of the thesis

Aluminium alloys are lightweight materials with a versatile combination of properties that make them widely usable and popular among engineering applications. For sustainable development, a need for lightweight material in combination with high strength increases its demand because of excellent technological properties. For advanced applications in aviation industries, and automobile industries as structural applications, the high-strength and lightweight aluminium alloys are the most appropriate. The 7XXX aluminium alloys are a combination of the aforementioned properties as high strength-to-weight ratio. The 7XXX aluminium alloy having major alloying elements like zinc, magnesium, and copper, makes it precipitation hardenable. Amongst, the 7075 aluminium alloy is widely used as a high strength alloy in wrought conditions. The utilization of Al 7075 in wrought conditions is costlier because of its process and heat treatment. The casting route is cheaper and the most viable route for manufacturing alloy, but because of the poor castability of the alloy, alternative routes can efficiently improve the mechanical properties of this alloy. Mechanical, chemical, and thermal treatments or a combination of the above treatments can also help to enhance mechanical properties.

Research Problem Formulation

As discussed above, producing 7XXX aluminium alloys by casting route reduces the cost of the final product and increases the application. The casting of 7XXX aluminium alloys is associated with three major problems.

- Segregation of solute.
- Wide solidification range (freezing range).
- Hot tearing.

The above issues are coupled with the mechanical properties, tribology properties, grain size, fraction and size of the eutectic phases, intermediate phases, and macro and micro-segregation.^[11] The quaternary Al-Zn-Mg-Cu, 7075 aluminium alloy, has the problem of solute segregation due to high solute concentration, and the wide solidification range in the mushy zone creates the problem of solidification cracks during the solidification^[14–18]. Due to high solute concentration, the microstructure of as-cast Al7075 forms α -Al grains with the

formation of intermediate phases like $\eta(\text{MgZn}_2)$, $\text{S}(\text{CuMgAl}_2)$, $\text{T}(\text{Al}_2\text{Mg}_2\text{Zn}_3)$, $\theta(\text{CuAl}_2)$, Mg_2Si , and $\text{Al}_7\text{Cu}_2\text{Fe}$ [19, 20]. The distribution of the intermediate phases within the $\alpha\text{-Al}$ grains and at grain boundaries plays a significant role in the alloy's mechanical, tribological, and corrosion properties. For this reason, it is interesting to note that studying solute segregation in the microstructure during solidification, and its effects on the properties, is essential. The segregation pattern can be changed either chemically, mechanically, thermally or a combination. Post-treatments like heat treatment and thermomechanical processing can also alter as-cast microstructural changes to optimize the mechanical properties [21–26]. Adding grain refiners, modifiers, and micro-alloying elements can act as the heterogeneous nucleation site and help to control the microstructure [26–31]. Mold vibration by mechanical, electromagnetic, and ultrasonic action can also change the segregation pattern [32–34]. Thermally, as-cast can be treated by changing the casting methods or quenching in some media by controlled diffusion solidification [30, 35–41]. Synergistically, mentioned treatments change the segregation pattern's morphology and refine the grain structure from dendritic to non-dendritic.

In the present research, the 7075 aluminium alloy was characterized by the addition of modifiers like ZrO_2 , TiO_2 , and ZrTiO_4 into the cast Al 7075, made a cast Al 7075 by using different quenching media, studied post-heat treatment of modifiers added Al 7075, cast Al 7075 by different casting techniques, and developed economical Al 7075. The study was divided into the five phases.

A research gap was identified after a thorough literature review. The major challenge is the application of Al 7075 by the casting route because of the solute segregation problem. The production of Al7075 by casting route might replace the costlier processing of wrought 7075 aluminium alloy. As per the literature survey, the wrought 7075 aluminium alloy has high strength, nearly similar to steel. The forming processes and secondary operations to get the final product are significantly costlier. Many researchers studied cast Al 7075 by adding grain refiners, modifiers, and micro-alloying elements to achieve significant mechanical properties. After an extensive literature survey, the present study focuses on changing the eutectic phase segregation pattern by adding high-temperature oxides like ZrO_2 , TiO_2 , and ZrTiO_4 in the cast Al7075 by die casting route. The comparative effect of these three oxides on the cast Al 7075 and their natural ageing is a novel study. However, the study on the solute segregation pattern and the intermediate phase formation within the microstructure is

quite complex but essential to solving the problem of cast Al 7075.

A detailed discussion in the literature, the addition of grain refiners or modifiers (chemically), controlled diffusion solidification (thermally), mould vibration (mechanically), or a combination of either can alter solute segregation patterns. Another aspect of the study is focused on microstructural morphology and mechanical properties by quenching cast Al7075 in ice, hot water for 30 minutes, and hot water until cooled down. A similar work is hardly reported in the present literature. Further, a study on the different casting techniques is performed to produce the cast Al 7075 by permanent mould casting (gravity die casting), sand casting, and investment casting. The mould characteristics play a significant role in the solidification of cast Al 7075, which influences the solute segregation and phase morphology of the final microstructure.

The double step-ageing of oxide-added cast Al 7075 was studied to compare microstructure and mechanical properties before and after the heat treatment. SEM-EDS and XRD analysed the precipitate phase to understand the precipitation behaviour. The double-step ageing cycle is unique, as most literature studied conventional T6 treatment, retrogression and re-ageing or either homogenization treatment.

The cast Al 7075 was developed by alloying and compared the microstructure and properties with the wrought Al 7075. The motivation for development is the high cost of the wrought Al 7075. The chemistry was achieved after the four successive heats, but achieving mechanical properties is challenging. However, the developed as-cast results are promising compared to as-cast Al 7075 produced from Al 7075-T6.

Objective of the present work

The following objectives based on the research gap are discussed below.

1. To study the effect of the addition of high-temperature oxides in the cast Al 7075 to understand the segregation pattern.
2. To study the effect of quenching media on cast Al 7075 to understand the eutectic phase morphology change.
3. To study the effect of double-step ageing treatment on oxide-added Al 7075 to understand the effect of modified heat treatment on microstructure, mechanical, and tribology properties.

4. To study the microstructure and mechanical properties of cast Al 7075 by different casting techniques.
5. To develop cost-effective cast Al 7075 by alloying and compare its properties with wrought Al 7075.

Structure of the thesis

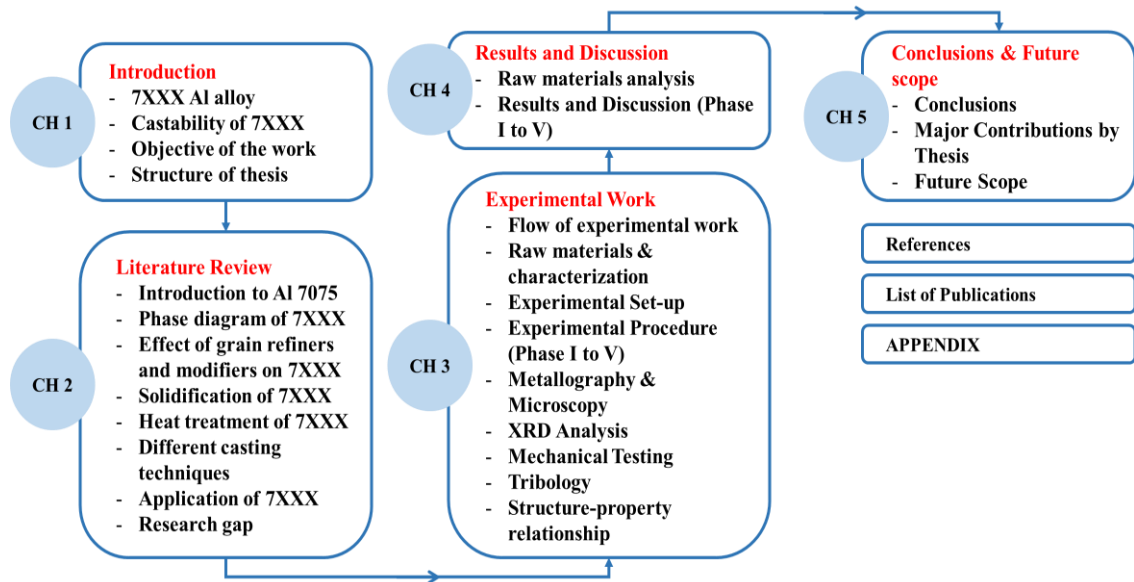


Figure 1 Structure of the thesis

Flow of the Experimental Work

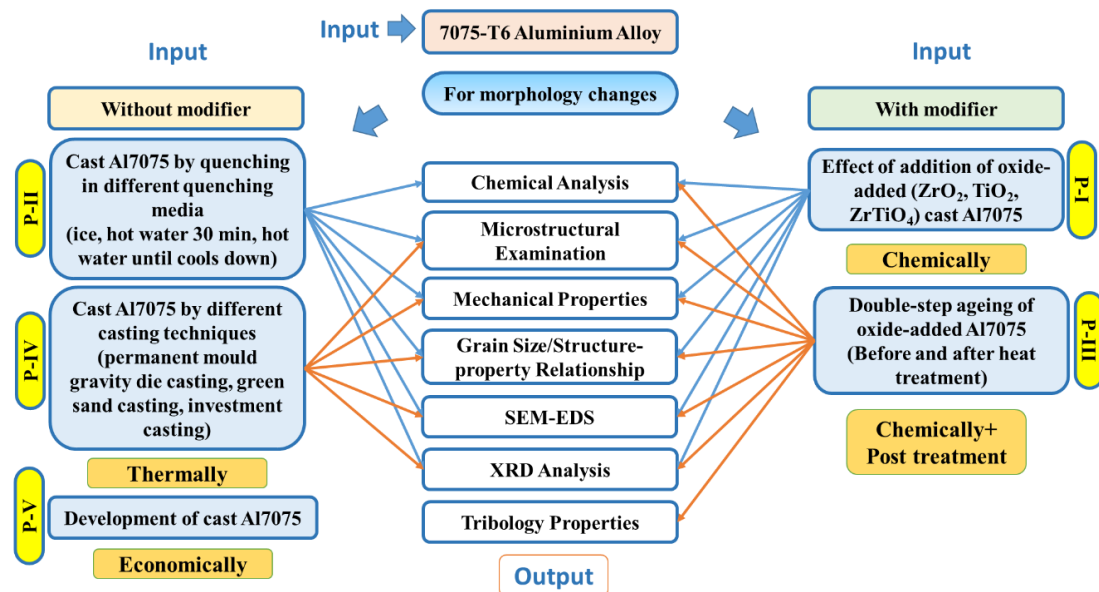


Figure 2 Plan of experimental work: Phase I & II with modifiers addition (right side), and Phase III, IV, and V without modifiers (left side).

Experimental Set-up

The experimental set-up for melting of Al 7075 is shown in Figure. A crucible-type furnace with kanthal wire winding, RTD sensor, and controller is used for resistance heating. Metallic die is used to pour the liquid melt, and the dimensions are also shown in Figure 3.



Figure 3 Experimental set-up for melting Al7075 (left), and metallic die with dimensions to pour liquid melt.

The resistance heating furnace temperature is set at 730 °C. The schematic of the metallic die is shown in 4.

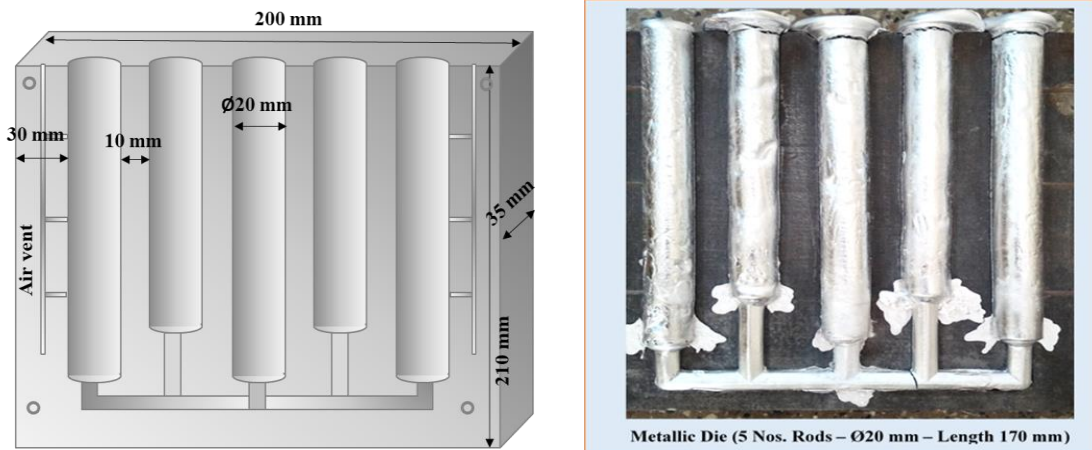


Figure 4 Schematic of metallic die (right), and solidified casting in the die (left).

Research Methodology

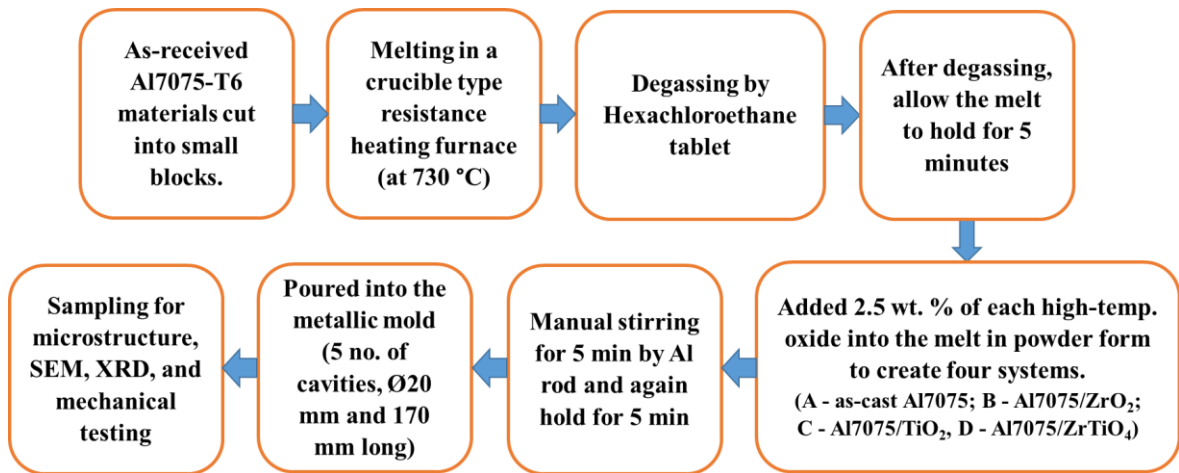
The experiments are divided into five phases to characterize the Al 7075 by using modifiers and heat treatment. Five phases are planned as below, out of that Phase I and Phase II with

modifiers addition, and Phase III, and Phase IV without modifier addition.

- Phase I** Effect of the oxide addition into cast Al 7075.
- Phase II** Effect of quenching medium on cast Al 7075.
- Phase III** Effect of double-step ageing on the oxide-added cast Al 7075.
- Phase IV** Effect of different casting techniques of cast Al 7075.
- Phase V** Development of cast Al 7075 by alloy addition.

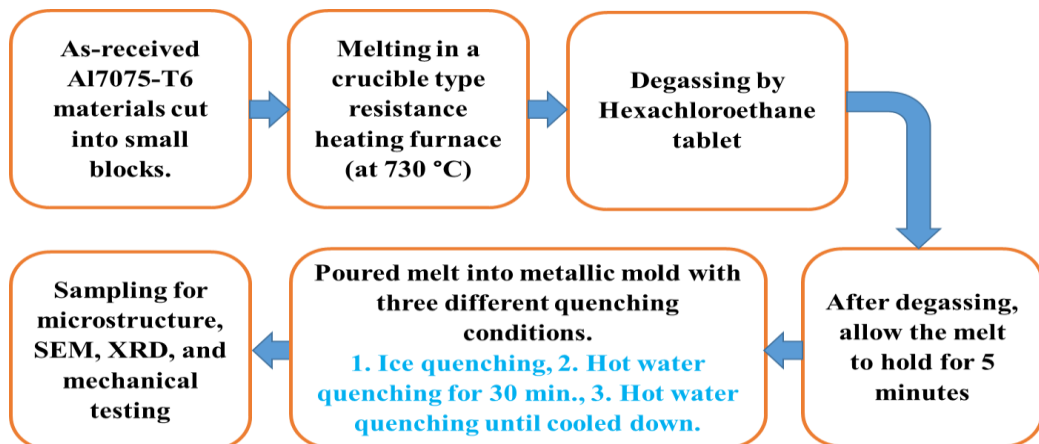
The process flow of the above phases is outlined below.

Phase I Effect of the oxide addition into cast Al7075.



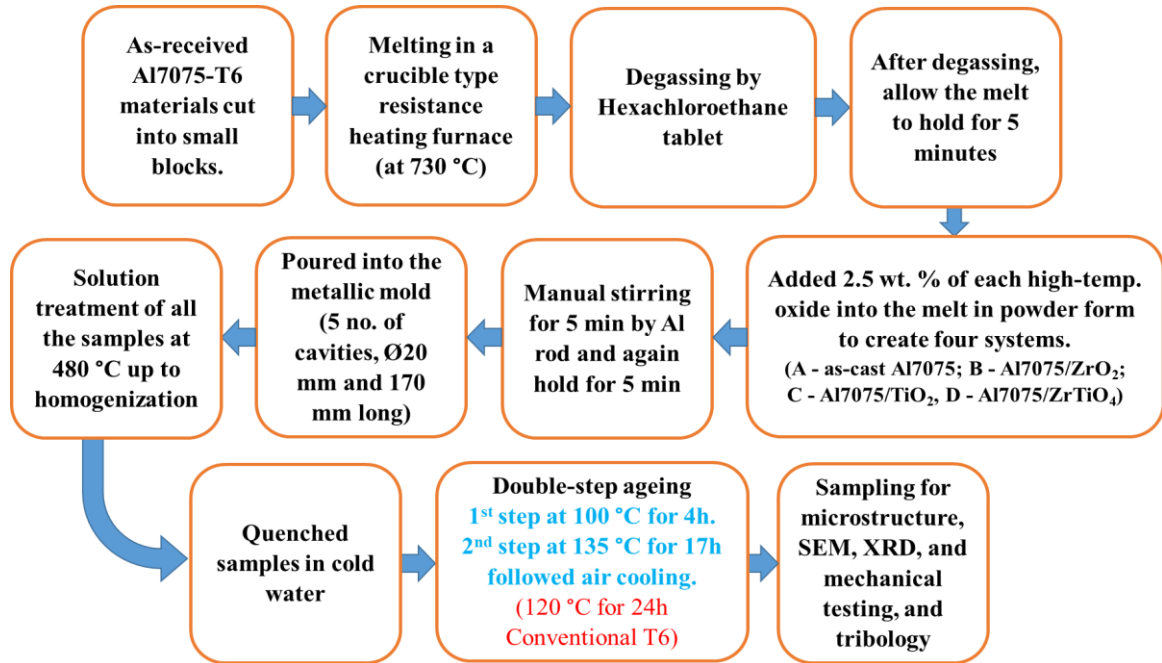
Process flow diagram for Phase I.

Phase II Effect of quenching medium on cast Al 7075.



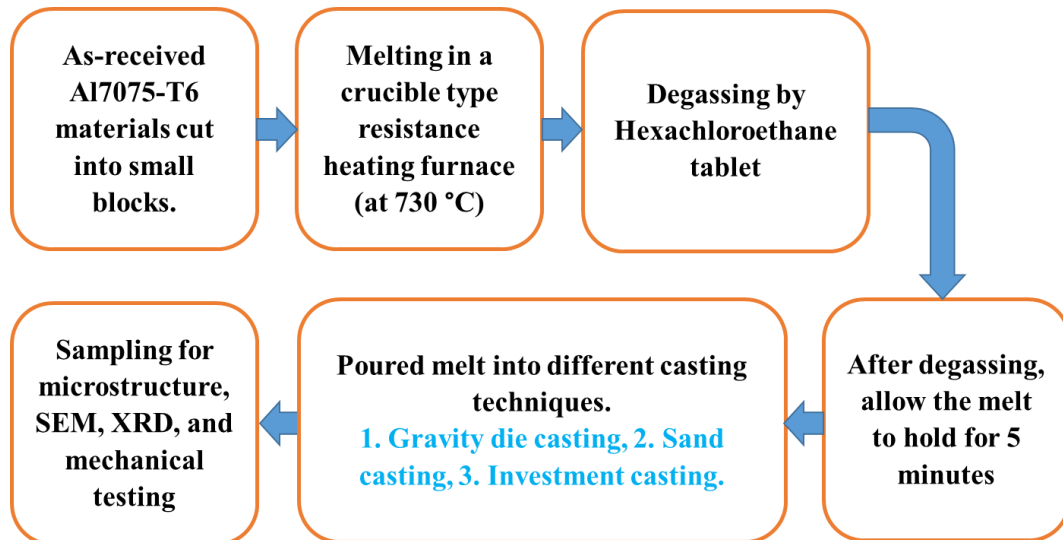
Process flow for Phase II.

Phase III Effect of double-step ageing on the oxide-added cast Al 7075.



Process flow for Phase III.

Phase IV Effect of different casting techniques of Al 7075

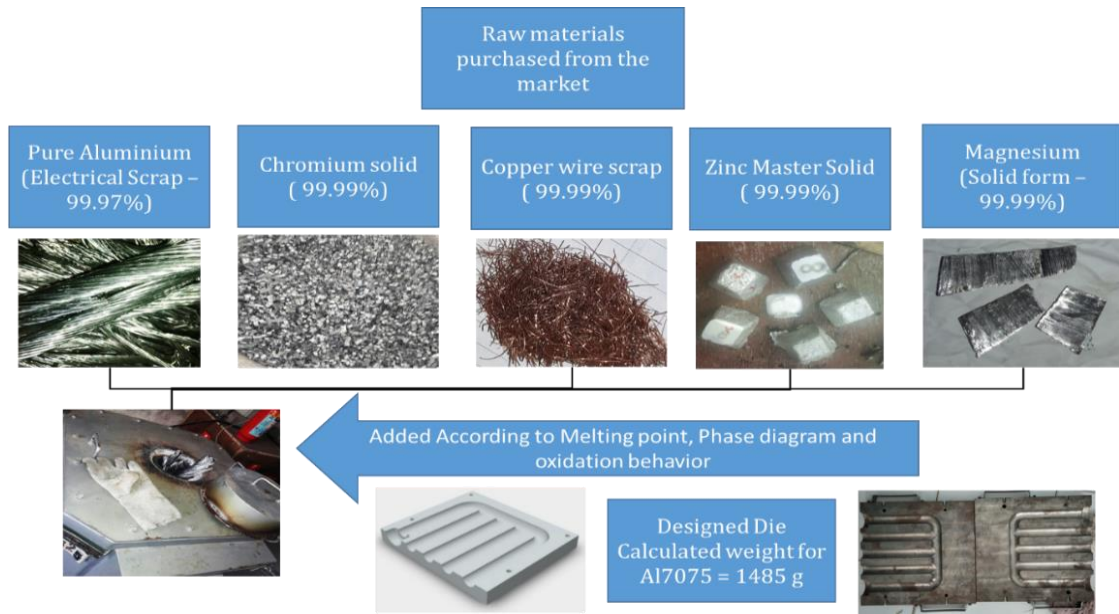


Process flow of Phase IV.

Phase V Development of cast Al 7075 by alloy addition.

For the economic development of cast Al 7075, the different scraps are used to make an alloy of Al 7075. The recycling of the materials definitely reduces the cost of the alloy. The

process adopted to develop it is described below.



Process adopted for Phase V to develop cast Al 7075.

Research Findings

Phase I: The segregation pattern was altered in the ZrO_2 added Al 7075 with improved mechanical properties. After natural ageing, the precipitation of phases at grain boundaries was observed in oxides of Zr and Ti individually, while the combined addition of Zr and Ti oxides added Al 7075 dissolves intermediate phases within the grain matrix.

Phase II: The cast Al 7075 was quenched in different quenching media like ice, in hot water for 30 minutes, and hot water till cooled down. The hot water for 30 minutes has good mechanical properties compared to others. Also, for naturally aged conditions, it shows higher hardness values.

Phase III: The morphological changes of the oxide added Al 7075 were quite interesting before and after heat treatment. The ZrO_2 added Al 7075 has shown excellent properties after the heat treatment but at the cost of losing elongation.

Phase IV: The study of the effect of different casting techniques on the grain morphology observed and found that permanent mould casting has good mechanical properties. The main objective is to study the segregation pattern of the solutes within the matrix.

Phase V: The cost of the wrought Al 7075 is very high and not economical to use it for domestic applications. The trials were made to develop the cast Al 7075 and compare it with the wrought and cast from the wrought. Though the chemical balance got perfect, the developed Al 7075 has considerable hardness and tensile strength values compared to wrought, but it is achieved higher than cast from wrought.

Summarized Conclusion from all the Phases

1. 2.5 wt. % ZrO₂ addition altered the eutectic segregation phase and converted α -Al grains into small equiaxed grains.
2. XRD analysis of ZrO₂-added cast 7075 alloy shows the presence of eutectic phases like η (MgZn₂), T(Al₂Mg₃Zn₃), (Al₃Zr), and S(Al₂CuMg).
3. The highest mechanical properties are 196 MPa tensile strength and 112 BHN hardness observed in ZrO₂ addition.
4. Using localised chemical analysis by EDS and Zn/Mg ratios, (η) MgZn₂ and (T) Al₂Mg₃Zn₃ were confirmed.
5. The hot water quenching for 30 mins offers the highest tensile strength of 197 MPa and 100 BHN hardness value in the cast condition.
6. Double-step ageing treatment after ZrO₂ oxide addition offers 366 MPa tensile strength and 212 MPa before heat treatment.
7. By changing casting techniques, there is no significant to alter the eutectic phase segregation pattern, and finally, no substantial improvement in the mechanical properties.
8. Attempts were made to develop a cost-effective 7075 alloy by using scrap and changes in the alloying sequences.

Major Contribution

This thesis focuses on the subjective research gap of cast Al 7075. The following are significant contributions:

1. Development of ZrO₂, TiO₂, and ZrTiO₄ oxide-added cast Al 7075.
2. Investigation of segregation pattern and phase morphology and their effect on mechanical properties.
3. Study on the effect of quenching media during casting of Al 7075.

4. Study on the effect of double-step ageing on oxide-added cast Al 7075.
5. Comparison of the microstructure, mechanical, and tribology properties of cast Al 7075 after double-step ageing (before and after).
6. Study the changes in the different casting techniques to alter the segregation pattern of the eutectic phase.
7. Development of cost-effective cast Al 7075 by alloying additions.

Future Scope

- ✓ The future scope of the work can be extended by studying the corrosion properties of the samples.
- ✓ Further, heat treatment like homogenization, and RRA (Retrogression and Re-ageing) of the cast Al 7075 alloys.
- ✓ Apply the thermomechanical process.

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