

1.1 IMPORTANCE OF Al-Si ALLOYS

With the rapid advancement of technology in industries such as automotive, aerospace, and electronic packaging, materials need to evolve to meet the demands of these applications (C.Robles-Hernandez and Jose Martin Herrera Ramirez, 2017). In the pursuit of lightweight metals, aluminium (Al) and its alloys have emerged as an alternative to steel. Aluminium and its alloys possess a unique combination of properties, making them suitable for high-strength engineering applications in the form of highly ductile foils. However, pure aluminium alone does not meet the required strength criteria. By incorporating alloying elements such as copper, manganese, silicon, magnesium, and others, the strength of aluminium can be enhanced to meet specific component demands.

Aluminium (8.23%) and silicon (28.2%) exist in abundance in the Earth's crust next to oxygen (46.1%). While Al and Si, on the one hand, possess some comparable properties like densities of 2.7g/cm³ and 2.33g/cm³, respectively, they have quite different properties on the other hand, like Mohs hardness of 2.75 and 6.5, respectively (Haynes, 1942). The combination of these two elements in the form of Al-Si alloys or *in situ* composites provides the ability to obtain lightweight material of high specific hardness. Other than this, Al-Si alloys exhibit excellent properties, including low density, lightweight, high strength, high thermal conductivity, low coefficient of thermal expansion, high corrosion resistance, and easy castability and recyclability (Hogg *et al.*, 2006; Velasco and Nino, 2011; Raghukiran and Kumar, 2015; Jia *et al.*, 2017; Haga, Imamura and Fuse, 2021). These properties can be influenced by the silicon content. Based on the silicon content (wt.%), Al-Si alloys are categorized into three groups: hypoeutectic (<12.6% Si) Al-Si alloys, eutectic (12.6% Si) Al-Si alloys, and hypereutectic (>12.6% Si) Al-Si alloys. Al-Si alloys with hypoeutectic (Al-6 to 8Si) (Javidani and Larouche, 2014), eutectic (Al-11 to 13Si) (Jung *et al.*, 2016), and hypereutectic (Al-20Si) (Orlowicz *et al.*, 2015) compositions, along with their variants or impurities, are widely employed in the automotive, aerospace, and electronic industries (C.Robles-Hernandez and Jose Martin Herrera Ramirez, 2017; Godbole *et al.*, 2024). Owing to their lightweight and satisfying physical and mechanical properties, including good formability, Al-Si alloys are treated as basic light materials, upon which evolved are the alloy modifications and process developments for identified applications.

As-cast Al-Si alloys consist of the α -Al matrix, eutectic phase, and primary silicon phase. The α -Al matrix is ductile and easily deformable, whereas the primary silicon phase is brittle. This incompatibility between the two phases results in brittleness during plastic deformation for the higher Si content alloys. The mechanical properties of the as-cast alloys are influenced by microstructural parameters such as primary dendrites and secondary dendrite arm spacing (SDAS), α -Al matrix size, and the morphology of Si particles (Osório *et al.*, 2006; Yajjala, Inampudi and Jinugu, 2020). Beyond the eutectic composition (> 12.6% Si), silicon is typically present in the form of primary silicon precipitates due to its very low solubility limit (1.65 wt.% Si) in aluminium at eutectic temperature, and it reduces to 0.05% at room temperature (Murray and McAlister, 1984). As the silicon content increases, the size, shape, and distribution of silicon particles become more heterogeneous, leading to a deterioration or limitation in mechanical properties, particularly ductility. Therefore, effective modification and refinement techniques

are required to alter the silicon morphology. Over the years, researchers have investigated various techniques for modifying and refining silicon particles (Hegde and Prabhu, 2008), including chemical modification (Mazahery and Shabani, 2014; Li *et al.*, 2015), thermal modification (Dang, Jian and Xu, 2020), and mechanical processing (Ma, Sharma and Mishra, 2006; Kucukomeroglu, 2010; Rajinikanth *et al.*, 2011; Immanuel and Panigrahi, 2015; S. M. Aktarer *et al.*, 2015; Guan and Tie, 2017; Choudhary *et al.*, 2024).

Chemical modification techniques often involve the addition of modifiers or refining agents such as Na, P, Sr, K, Sc, Ca, B, and rare earth elements to the Al-Si alloys, which restrict the nucleation and growth of silicon particles (Li *et al.*, 2015). Thermal modification methods alter the solidification/cooling rate during alloy casting. While these methods are useful for hypoeutectic to eutectic Al-Si alloy compositions, they are not applicable to hypereutectic Al-Si alloys due to the presence of large Si particles. Both approaches can refine the microstructure to some extent, but they may have adverse effects on other aspects such as compositional variation (ternary or quaternary elements), porosity, and shrinkage defects (Samuel *et al.*, 2023). Moreover, they can introduce microstructural inhomogeneities, further compromising the mechanical properties. Therefore, it is time to look into binary Al-Si alloy processing in view of several mechanical working methods (Meyers *et al.*, 2009; Dieter, 2011), including severe plastic deformation (Segal, 2018; Bagherpour *et al.*, 2019; Rakshith and Seenuvasaperumal, 2021), evolved in the recent past.

1.2 DEFORMATION METHODS AND MICROSTRUCTURE DEVELOPMENT

The refinement of microstructure and homogeneous distribution of particles can be achieved through various mechanical processing methods such as extrusion, rolling, and a number of severe plastic deformation (SPD) techniques like friction stir processing (FSP), high pressure torsion (HPT), equal-channel angular pressing (ECAP), accumulative roll bonding (ARB), etc. (Rakshith and Seenuvasaperumal, 2021). Although the cold rolling or extrusion process can produce a fine-grain material, the grain size is typically limited to approximately 10 μm in Al alloys (Cui *et al.*, 2010; Immanuel and Panigrahi, 2015). However, in order to achieve a more dramatic refinement or ultrafine-grain microstructure down to the nano-sized level, SPD techniques are employed. These techniques involve subjecting the bulk material to severe plastic deformation, resulting in the development of ultrafine grains (UFG) smaller than 1 μm (Ma, Saito, *et al.*, 2005) and reaching the nanocrystalline level (<100nm). Friction stir process (Mishra and Ma, 2005) and high pressure torsion techniques were used extensively to get ultrafine grains in different types of material (Edalati and Horita, 2016; Ito, Edalati and Horita, 2017), including metallic, composites, ceramics, polymers, and biomaterials.

The flow behaviour of two-phase materials depends on factors such as the matrix grain size and the size, shape, and distribution of second-phase (Si) particles existing outside as well as within the matrix. Additionally, the flow properties vary with changes in deformation conditions like strain, strain rate, and test temperature. Several authors have investigated the room temperature tensile, compressive, and fatigue properties of Al-Si alloys, which were found to be enhanced after undergoing mechanical processing (Dighe, Gokhale and Horstemeyer, 2002; Joseph, Kumar and Babu, 2015; Wang *et al.*, 2018). At high temperatures, the ductility (elongation) is observed to improve but at the expense of a loss in strength (flow stress) (Rajaram, Kumaran and Rao, 2010). The eutectic structure of Al-Si alloy upon HPT processing can lead to the development of fine inter-dispersion of two phases, which can provide higher strength at lower temperatures and higher elongation to the extent of superplasticity at elevated temperatures. The effects of test temperature and strain rate were also reported (Liao *et al.*, 2015) on the flow behaviour of Al-Si alloys. At low temperatures, the plastic deformation occurs by dislocation motion in the slip process, whereas at high temperatures, the deformation slip is facilitated by a combination of dislocation glide and climb mechanisms. Some researchers have even reported the attainment of superplasticity in Al-Si alloys after SPD processing (Mishra,

Bieler and Mukherjee, 1997; Ma, Takagi, *et al.*, 2005; Cao *et al.*, 2013), where the deformation mechanisms comprise grain boundary sliding, intragranular dislocation slip, diffusion, grain switching, grain rotation, and their combinations.

Al-Si alloys with different silicon contents are widely utilized in the automotive industry (Javidani and Larouche, 2014). For instance, pistons are typically composed of Al-12Si, cylinder liners are made of Al-20Si, and engine blocks contain Al-7Si. These components are able to sustain significant thermal stresses during the combustion process, with the engine block temperatures reaching as high as ~673K while the piston temperatures range between 523–603K (Wang *et al.*, 2019; Li *et al.*, 2021). Thus, it is very crucial to analyse the effects of temperature, strain rate (or stress), and composition on the deformation behaviour of Al-Si alloys for closely relating with the performance of such components. Hu *et al.* (Hu, Wang and Deng, 2013) produced a processing map for Al-Si eutectic from constant true strain rate compression tests and reported the range of optimum temperatures and strain rates for processing depending on progress in deformation (strain). Furthermore, the influence of Si content on the parameters of constitutive relationships for deformation in binary Al-Si alloys remains unreported in the existing literature. Owing to the absence of such data, the prediction of the deformation behaviour of Al-Si alloys across diverse processing conditions or applications cannot be made. Thus, it could otherwise provide guidelines for selecting high-temperature deformation processes such as extrusion, rolling, and forging, as commonly employed for manufacturing simple to intricate components needed in the automotive, aerospace, and construction sectors.

1.3 OBJECTIVES OF PRESENT WORK

Currently, the development of bimodal grain structures, UFG, or nano-sized grain microstructures in Al-Si alloys has become an intriguing area of research due to their unique structural patterns and exceptional mechanical properties. It appears from the literature that most of the previous studies on Al-Si alloys were focused on hypoeutectic and eutectic compositions, along with some studies on AlSi10Mg alloys (Saumyadeep Jana *et al.*, 2010; Wang *et al.*, 2015; S. M. Aktarer *et al.*, 2015; Bösch *et al.*, 2015; Meenia *et al.*, 2016; Qin *et al.*, 2018; Lu *et al.*, 2019; Bian *et al.*, 2020; Xi *et al.*, 2020; Du *et al.*, 2022; Soleymanpour, Aval and Jamaati, 2022a; Neuser, Schaper and Grydin, 2023). Only limited studies seem to have been reported on hypereutectic Al-Si alloys (Hong and Suryanarayana, 2005; Yoon *et al.*, 2007; He *et al.*, 2011; Jiang *et al.*, 2012; Rao *et al.*, 2013, 2016; Golafshani, Nourouzi and Jamshidi Aval, 2019a; Dang, Jian and Xu, 2020). These studies mainly focused on the refinement of the microstructure and its effect on enhancing mechanical properties. However, to date, no systematic study has been reported that investigates the effects of conventional processing and SPD processing methods on microstructure and flow behaviour at different deformation conditions like test temperature and strain rate across a range of compositions from hypo to hypereutectic Al-Si alloys.

Given the reported effects of mechanical processing on various materials, it would be interesting to investigate the compositional, microstructural, and mechanical property changes in hypoeutectic, hypereutectic, and eutectic binary Al-Si alloys together. While many researchers have studied the effects of deformation conditions like test temperature and strain rate on flow behaviour and microstructure evolution, a comprehensive understanding over a wide range of strain rates and temperatures in the Al-Si alloys has not been appreciable. This thesis, therefore, aims to systematically study the microstructure evolution and flow behaviour across a broad range of temperatures and strain rates using differential strain rate and differential temperature test techniques (B.P. Kashyap and K. Tangri, 1985; Bakshi and Kashyap, 1995; Thakur, Kashyap and Malik, 1996; Kashyap *et al.*, 1999). The present investigation focuses on Al-Si alloys of different compositions, spanning from hypoeutectic (2-8wt.% Si) to hypereutectic (20wt.%, 30wt.% Si), including the eutectic composition (12wt.% Si). The selected alloy compositions are planned to be processed from commercial purity aluminium and silicon,

beginning with the melting and casting stage to conventional mechanical processing and severe plastic deformation stages. The objectives of the current work are as follows

- To develop a range of microstructures for hypoeutectic to hypereutectic, including eutectic Al-Si alloys
- Tailoring of mechanical properties for ambient and elevated temperature conditions by application of mechanical working
- To express the deformation behaviour in the form of possible parameters of the constitutive relationship from as-cast to mechanically processed Al-Si alloys
- Development of superplasticity in hard-to-work hypereutectic Al-Si alloys
- To examine the possibility of *Composition-Process-Structure-Property* relationships

The following steps are undertaken to accomplish the objectives of this thesis work:

- Select the composition and perform melting and casting for hypoeutectic to hypereutectic Al-Si alloys, including the eutectic composition. Investigate the microstructure and mechanical properties of the as-cast alloys at room temperature and elevated temperatures.
- Modify the microstructure through extrusion mechanical processing and FSP, and HPT severe plastic deformation techniques for investigating the tensile, compressive, and hardness mechanical properties at room temperature and tensile properties at high temperatures for selected processing conditions.
- Conduct differential strain rate and differential temperature tests to establish the constitutive relationships for high-temperature deformation of differently processed Al-Si alloys.
- Conduct differential strain rate tests followed by constant strain rate tests at the highest test temperature to evaluate the parameters of the constitutive relationship for high-temperature deformation along with simultaneously exploring the elongation possible for the alloys made.
- Study the microstructure evolution after deformation under different processing and test conditions.
- Analysis to explore the interrelationship between ambient and high-temperature properties as emerging from different processing methods.

To achieve the aforementioned objectives, the planned experimental works are summarized in the flow chart outlined below:

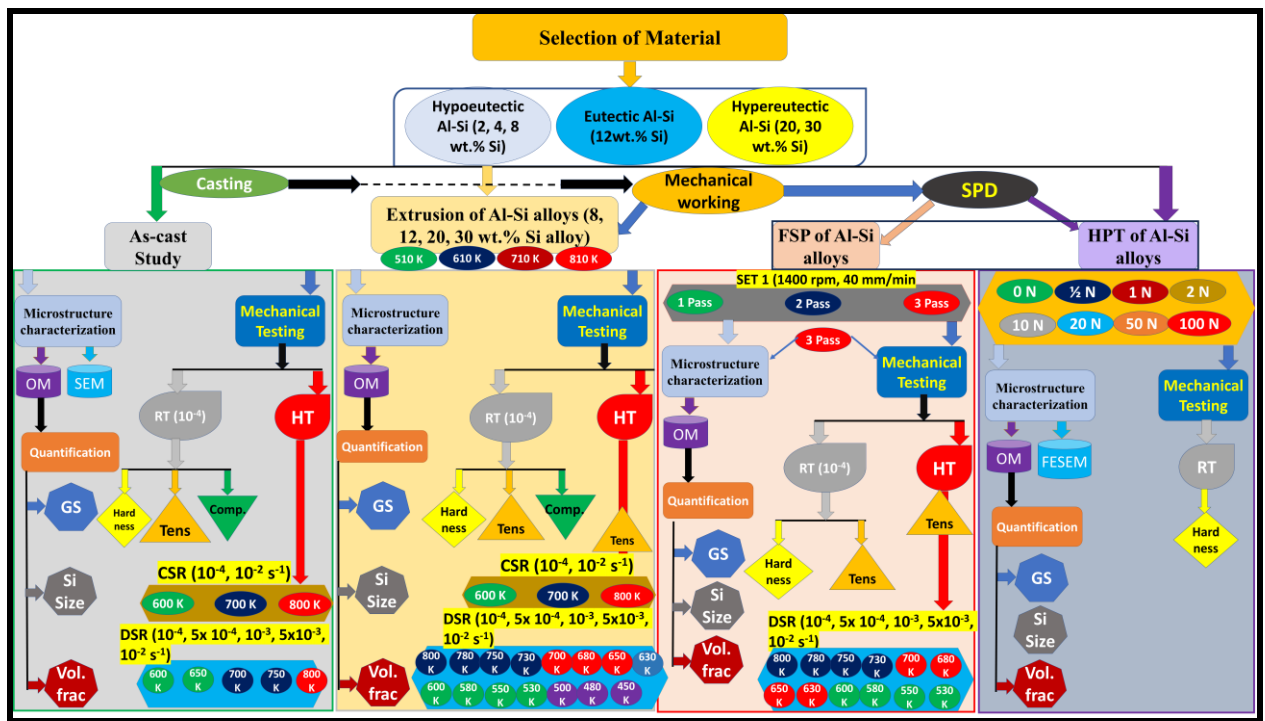


Figure. 1.1 : A schematic flow chart of the experimental work

1.4 OUTLINE OF THE THESIS PRESENTED

The present work is divided into six chapters, including the *chapter 1 Introduction*, which highlights the importance of Al-Si alloys and the need for a systematic study of their microstructure-flow property relationships through the variation in composition and processing method at different test conditions. *Chapter 2* gives *Literature Review* describing in brief the works reported on Al-Si alloys along with the broader aspects of light metals and modern mechanical processing methods. This helped in designing *chapter 3 Materials and Experimental Methods*, to reflect an attempt to utilize a range of variants from processing at the casting stage to severe plastic deformation and closely follow towards obtaining the data for gaining insights into the composition-process-microstructure-mechanical property mutual relationship. The outcomes of experimental works planned to meet the set objectives are presented in *Chapter 4* as *Results*. This chapter is divided into two major sections, *viz. 4.1 Microstructure Evolution*, which describes different microstructures evolved by changes in alloy compositions and processing methods, including from as-cast to conventional extrusion and severe plastic deformation mechanical working methods. Section *4.2 Deformation Behaviour and Mechanical Properties* presents the results from tensile and compression tests at room temperature and from tensile tests using constant initial strain rate and differential strain rate test techniques at elevated temperatures. The data thus obtained were analysed to relate with microstructure. The results presented in these two sections of Chapter 4 are taken up in *Chapter 5 Discussion* to emphasize the importance of varying microstructures and mechanical properties emerging from the variations in alloy composition, processing method, and test technique in a comparative manner. *Chapter 6* presents the *Summary and Conclusions*, as suggested by the results and discussion in the preceding chapters. Also, a few *suggestions for future work* and, finally, the *closing comments* are provided in this chapter.

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