Preface

The modern transportation industry, particularly the automotive sector, increasingly emphasizes weight reduction as a crucial strategy to enhance fuel efficiency. While low-density materials such as aluminum (2.7 g/cm^3) and magnesium (1.738 g/cm^3) alloys are being considered for automotive applications, their inherent limitations namely, high costs, low stiffness, and inadequate dent resistance restrict their use primarily to non-critical components. In contrast, steel remains the most economical and widely utilized material in automobile manufacturing, accounting for approximately 55% of the weight of a typical passenger vehicle.

Recent advancements in alloy design and processing have led to the development of advanced and ultra-high-strength steels (UHSS), which present a cost-effective and recyclable alternative to conventional steels. These materials facilitate significant weight reduction; however, they often exhibit reduced ductility and stiffness due to down-gauging processes. To address these challenges, lightweight high-strength steels, particularly iron-manganese-aluminum-carbon (Fe-Mn-Al-C) alloys, have emerged as a promising and sustainable solution for automotive structural applications. These alloys demonstrate remarkable potential for use in crash-resistant car body structures, enhancing both safety and performance while contributing to overall vehicle weight reduction. The current PhD Thesis consists of seven chapters.

Chapter-1 reviews the existing literature on lightweight steels and outlines the objectives of the present thesis. Fe-Mn-Al-C alloys are recognized for their low-density and exceptional mechanical properties, making them highly suitable for weight-saving applications. The incorporation of 7-10% aluminum reduces the density of these steels to approximately 7 g/cm^3, achieving a reduction of about 1.3% in density per 1 mass % of aluminum. Manganese and carbon play a crucial role in stabilizing the austenite phase at lower temperatures. The microstructure of these alloys can vary widely, encompassing ferritic, austenitic, and duplex phases, as well as combinations that include kappa-carbides and B2 precipitates, depending on the alloying elements and their concentrations. The ultimate tensile strengths of these steels range from 800 to 1500 MPa, depending on their composition and processing conditions, with total elongation varying between 30% and 80%. Austenitic steels typically contain higher amounts of manganese and aluminum. The high percentages of Mn, Al increase costs and cause challenges such as manganese evaporation during melting. Additionally, higher aluminum content creates issues by choking flow due to excessive oxidation during casting. Reducing aluminum content to below 7% and manganese content to below 20% may help mitigate the issues partially mentioned above. Developing a steel within this composition range while maintaining similar mechanical properties could offer advantages during melting and casting.

Literature has shown that severe plastic deformation strengthens materials by refining grain size to the nanoscale, albeit at the cost of ductility. Annealing severely deformed material above the secondary recrystallization temperature for short periods can create a bimodal grain size distribution, enhancing both ductility and strength. Thus, it can be anticipated that there is a possibility of getting bimodal grain size distribution by short annealing above the recrystallization temperature of selected austenitic low-density steel to achieve both high strength with high ductility.

As mentioned earlier, the addition of aluminum in Fe-Mn-Al-C alloys significantly reduces density but also results in a decreased elastic modulus. Alloying elements such as chromium (Cr) and silicon (Si) can increase the modulus of elasticity; however, both are ferrite stabilizers, which can adversely affect the ductility and formability essential for shaping automotive components. Thermodynamic calculations indicate that the incorporation of carbides like NbC and TiC in austenitic steels can substantially improve Young's Modulus. While the addition of NbC to low-density steel increases its density, the introduction of TiC may further reduce density. Bonnet et al. found that adding reinforcing particles, such as TiC, with a high Young's Modulus to iron enhances its modulus of elasticity. Nonetheless, there is currently insufficient experimental evidence regarding the extent of modulus recovery in austenitic low-density steel due to the addition of TiC. The incorporation of fine-sized, lighter particulate reinforcements into liquid steel poses challenges related to flotation and agglomeration due to their low-density and poor wetting properties. In-situ formation of TiC can be achieved by adding titanium (Ti) and carbon (C) in a designed composition.

Significant advancements in the strengthening through alloy design of Fe-Mn-Al-C steels have been made through the incorporation of 1-5% nickel, which enhances hardness and tensile strength. Although these steels with banded microstructures exhibit reduced formability, they demonstrate superior tensile strength compared to steels lacking a band-free structure. Previous research has predominantly focused on cold-rolled products, with relatively few studies investigating hot-rolled variants. While hot-rolled steels typically exhibit lower strength levels compared to their cold-rolled counterparts, they offer a cost advantage by eliminating additional processing stages. However, achieving uniform B2 precipitation and effectively controlling the microstructure in hot-rolled low-density steels presents a significant challenge. This challenge arises from the continuous dynamic recrystallization commonly observed in materials with high stacking fault energy at elevated temperatures, as well as the larger grain sizes characteristic of hot-rolled steels. Till now about maximum 5 mass pct Ni is added in high-carbon low-density steel. It is worth studying duplex low-density steel of high Ni content to get increased amount of B2 precipitates to achieve high strength and hardness with in the hot rolled products there by reducing the cost of additional cold rolling process.

Additionally, the presence of coarse banded B2 phases within the microstructure can induce anisotropy and diminish the strengthening effect when compared to finer platelets. Therefore, refining these phases is essential for attaining isotropic properties and improved formability. Although cold rolling followed by annealing can effectively distribute B2 platelets uniformly, pre-existing elongated bands and globular B2 precipitates formed during hot rolling remain largely unaffected. While most studies have concentrated on optimizing B2 platelet size and distribution, limited research exists on refining banded and globular B2 phases. Electropulsing techniques have demonstrated promise in refining high-temperature precipitates at low temperatures/much below solvus temperature in various alloys; however, their application to austenite-based duplex low-density steels, particularly refinement of B2 remains largely underexplored.

Based on the Literature review, the identified research gaps are summarised as: Most studies have focused on lower B2 content (8-20%) with the addition of 3-5% nickel; however, very few investigations address the incorporation of higher B2 contents along with higher nickel additions. Additionally, no literature exists on the refinement of microstructure through electropulsing in Fe-Mn-Al-C-Ni-based low-density steel. TiC has been utilized to enhance the modulus of low-carbon medium manganese steels and austenitic steels, yet no studies have reported on the effect of TiC on the mechanical properties of TiC added low-density steels. Furthermore, research on the effect of cyclic thermal treatment (repeated annealing) on microstructural modification and mechanical properties of low-density steel remains sparse.

Based on the above identified research gaps, the objectives of the present thesis are to design austenitic and duplex steels based on austenite for low-density applications, to standardize process parameters to achieve high strength in low-density steels. The other objectives are to investigate the impact of repeated annealing treatment for short time on the microstructure and tensile properties of austenitic low-density steel and to design an austenitic matrix composite with an improved modulus. The further aims of the present investigation are to explore the effect of electropulsing on the microstructure and tensile properties of nickel-added low-density steel.

Chapter 2 outlines the experimental procedures employed in the present study. Three types of low-density steels are produced in the present work. Fe-18Mn-6.5Al-0.75C alloy (P1) is designed to get austenitic low-density steel. Fe-18Mn-6.5Al-1.25C-2.5Ti alloy (P2) is formulated to get in-situ TiC reinforced austenitic matrix low-density composite steel. Fe-18Mn-10Al-1C-6Ni (all by mass %) alloy (P3) is designed to obtain duplex low-density steel. A mixture of raw materials except Mn or Fe-Mn is melted in a vacuum induction furnace at 1600°C under a pressure of 10^{-4} mbar. Following 10 min of stirring, the temperature is dropped to 1570°C, the required quantity of Mn/Fe-Mn are added to get approximate quantity of 2 kg melt. Immediately vacuum is broken and Ar is purged at 600 mbar and temperature is maintained at ~1560°C. After complete melting of Fe/Fe-Mn, melt is stirred for ~3 min and cast into a plate of 15 mm thickness in a copper mould.

The dissolution temperatures of potential precipitates are calculated using the Thermo-Calc database, establishing a homogenization temperature of 1200°C for duplex steel and 1080°C for both the austenitic steel and composite. Rectangular bars are cut from the cast plates using wire-cut electrical discharge machining (EDM), followed by homogenization and hot rolling with a 75% reduction in thickness. The austenitic and composite steels are air cooled and solution treated followed by quenching in water. The quenched austenitic steel is cold rolled and repeatedly annealed for 2 min at 900°C (PD1-SCR). Duplex steel samples are quenched in water at 950°C to maximise dislocation density and inhibit undesired precipitation (PD3). The rolled and quenched steels are annealed at 930°C for 30 min and quenched in water again (PD3-A). The quenched steels are aged at 550°C. Annealed and peak aged steel (PD3-AB_{30}) are electropulsed.

All the samples are studied under an optical microscope, and scanning electron microscope (SEM) to get the morphology and grain size of austenite, TiC and B2 precipitates. Additionally, the compositions of the austenite, TiC and different types of B2 are estimated using energy dispersive spectroscopy (EDS). The samples are further examined using X-ray diffraction (XRD) technique to identify phases, phase fractions, crystallite size, lattice microstrain, and dislocation density. The samples are further scanned using SEM equipped with electron backscatter diffraction (EBSD) to get phase fractions, grain size, residual strain, high angle and low angle grain boundaries, kernel average misorientation maps and microtexture. The finer and coarser phases are further conformed using SAED patterns obtained from Transmission Electron Microscope (TEM) and morphology, size of kappa carbide, ferrite (K-pearlite) are measured based on the TEM bright field imaging. ImageJ software is used to analyse the images.

Mechanical properties of all the samples are measured by Vickers microhardness measurement and tensile testing. Yield strength, tensile strength, plastic elongation and product of strength and plastic elongation (PSE) are calculated from tensile data. The work-hardening rate versus true plastic strain are plotted using engineering stress-strain data, and fitted with flow models such as Hollomon, Ludwik, Swift, Ludwigson, and Voce to analyse flow behaviour. Fracture surfaces of tensile tested samples are examined by SEM to determine the size and volume fraction of dimples and facets. The elastic modulus of the samples is measured using ultrasonic technique. The results of the present investigation are discussed from Chapter 3 to Chapter 6 and major summary of the work is reported in Chapter 7.

Chapter 3 investigates the microstructure and mechanical properties of austenitic low-density steel in the annealed condition(PD1-A), as well as the effects of cyclic thermal treatment (repeated annealing) on this steel. The austenitic steel sample exhibits a low-density of 6.99 g/cm^3 and a Young's Modulus of 169 GPa. Tensile testing of the steel reveals yield strength (YS) of 435 MPa, ultimate tensile strength (UTS) of 732 MPa, and a plastic elongation of 59%. The strengthening mechanisms in the austenitic low-density steel are primarily attributed to solid solution strengthening, followed by dislocation and grain boundary strengthening. Repeated annealing treatment refined the austenite grain size from 82.6 ± 8.9 μm to 5.24 ± 1.51 μm and increased the YS to 635 MPa and UTS to 958 MPa with decreasing ductility from 59% to 51%, while tensile toughness is improved from 43.3 GPa% to 48.1 GPa% at a low strain rate of 10^{-3}/s. Fractography analysis confirmed that the fracture behaviour is ductile within the strain rate range of 10^{-3}/s to 10^{0}/s, with a noticeable decrease in dimple size with increasing strain rate.

Chapter 4 reports the enhancement of elastic modulus, strength through the incorporation of Titanium carbide (TiC) reinforcement in low-density steel. Addition of Ti in Fe-18%Mn-6.5%Al-1.25%C produces in-situ TiC reinforced austenite matrix composite. Presence of TiC reduced grain size, density but increases Young’s Modulus, yield strength and ultimate tensile strength but reduces elongation. Strengthening mechanisms in the steel primarily arise from solid solution effects, with enhanced dislocation and grain boundary strengthening due to the presence of TiC; however, precipitation strengthening is limited by the coarse size of the TiC particles. Solutionized and quenched composite displays 3 stages of work hardening i.e., easy glide, steady linear work hardening rate and dynamic recovery. In both stage I and stage II, the work hardening rate decreases much faster than that of base alloy with increasing true strain. Work hardening rate in stage I is much faster than that in stage II.

Chapter 5 investigates the microstructure and strengthening mechanisms in duplex lightweight steel. The incorporation of 10.5 mass% aluminum in the selected low-density steel (PD1) reduces density to 6.67 g/cc. Homogenization and hot rolling of the selected low density duplex steel precipitates 13% B2. Annealing of the hot rolled steel at 930°C, increases the B2 content to 18%. Ageing of the annealed steel precipitates 8% kappa pearlite which consists of 3% kappa carbide and 5% ferrite. Hot rolled low density steel reports high yield strength due to high amount of solutes, dislocation density and low grain size. Strength decreases in annealed material as a result of reduction in dislocation density. Ageing recovers strength to highest level due to precipitation hardening. Strengthening of homogenized and hot rolled steel/ annealing of it is dominated by solid solution followed by dislocation and grain boundary strengthening. In case of aged low density steel major contribution to yield strength comes from solid solution followed by dislocation and precipitation. The ductility decreases with increasing precipitation of B2/Kappa carbides. Homogenized and hot rolled, annealed steel follows 3 stages of work hardening i.e., easy glide, steady linear work hardening rate and dynamic recovery whereas aged steel with reduced ductility displays first two stages. Coarse grain size and low volume fraction of B2 in hot rolled steel induces lower work hardening which results in low UTS. High volume fraction, wide range of B2 size distribution leads to high amount of work hardening in annealed steel. Aged steel displays highest UTS due to high yield strength and high amount of work hardening resulting from high fraction of B2 and appreciable amount of kappa pearlite.

Chapter-6 explores the influence of electropulsing treatment on the microstructure and tensile properties of duplex low-density steel. Electropulsing of annealed duplex low density steel raises the temperature to 346°C, partially dissolves banded, globular and platelets of B2 at this low temperature which is lower than the equilibrium solvus temperature by reducing activation energy. Electropulsing deforms B2 as well as recrystallizes austenite and reduced the intensity of deformation texture. At low current density of later pulse of electropulsing signal, the material is supersaturated due to rapid cooling but the energy is sufficient to accelerate precipitation of B2. Dissolution and precipitation refine the size of B2 and improves its distribution. Electropulsing also spheroidizes the existing B2 precipitates. The improvement in yield strength and work hardening of electropulsed steel is due to refinement, enhancement in uniformity in size distribution and additional B2 precipitation. Improvement in ductility and higher amount of work hardening leads to higher ultimate tensile strength. Annealed as well as electropulsed duplex steel follows the stages of work hardening of easy glide, steady linear work hardening rate and accelerated dynamic recovery, Ludwigson flow behavior. The work hardening rate of annealed steel decreases more rapidly due to more dislocation activities and higher interparticle spacing than that of electropulsed sample and material quickly goes to accelerated dynamic recovery. Steady linear hardening rate is prolonged for electropulsed duplex low-density steel due to higher ductility. Electropulsing of aged duplex steel breaks kappa carbide partially and spherodizes it due to reduction in barrier energy of dissolution and diffusion, respectively. Refinement of B2 and dissolution of kappa carbide decrease the YS and UTS but recover tensile toughness due to increased ductility. The work hardening rate of aged and electropulsed aged materials are similar to those of annealed and electropulsed annealed ones but with only two stages of easy glide and steady linear hardening stage.

Major conclusions drawn from the present study are

Three light weight steels with 10% reduction in density are successfully produced. Annealing of austenitic low-density steel (PD2-SC) at 900°C for brief 2-minute intervals results in 99.5% recrystallization which significantly improved yield strength (YS) by 200 MPa and ultimate tensile strength (UTS) by 228 MPa.

Incorporation of 4.5% TiC in austenitic steel (PD2-S) increases the elastic modulus by 7 GPa and decreases density by 2.1%, leading to a 6.6% improvement in specific stiffness to the composite. The grain size of austenite is greatly reduced due to the presence of TiC at grain boundaries.

Presence of 10.5% Al majorly reduces the density of PD3 to 6.67 g/cc. PD3-AB$_{30}$ sample has showed the highest yield strength, and ultimate tensile strengths with lower ductility than all samples. Solid solution strengthening mechanism is the major strength contributor to the yield strength of PD3, PD3-A and PD3-AB$_{30}$. Flow Behaviour of PD3 & PD3-A exhibit three stages (easy glide, steady-state work hardening, dynamic recovery), whereas PD3-AB$_{30}$ lacks stage III due to limited ductility.

Electropulsing treatment refines the B2 through dissolution, deformation & fragmentation, precipitation, spheroidization and promotes the recrystallization in austenite. Athermal electron wind effects are the major contributor to the refined microstructure by reducing activation energy of the processes. Reduction in dislocation density, refined precipitate size, improved uniformity, and partial recrystallization of austenite increases the plastic elongation in electropulsed sample. Electropulsing treatment fragments the lamellar kappa carbides through electron wind force.