

Preface

The demand for carbon reduction, production efficiency, and energy conservation in the steel industry necessitates a new design strategy for the development of advanced high-strength steel, employing simple and fast processing routes. The strength of steel can be enhanced through the production of ultrafine-grained (UFG) structures in bulk using severe plastic deformation (SPD) techniques such as equal-channel angular pressing, or the generation of nanostructured bainites through heat treatment. UFG materials created through SPD exhibit high strength; however, they often suffer from poor ductility due to reduced dislocation activity and the non-equilibrium nature of grain boundaries. The maximum ductility in low carbon steel is achieved when the grain size falls within the range of 1 to 10 μm . Therefore, there is a possibility of recovering ductility in SPD materials by producing a combination of UFG and fine grains, without significantly sacrificing strength.

Nanostructured bainitic steel exhibits an excellent combination of strength and ductility, attributed to the presence of fine bainite plates and a ductile retained austenite phase. These steels are typically produced by undergoing isothermal transformation at extremely low temperatures (below 300 °C). The growth of bainite plates is impeded by the higher yield strength of austenite at low temperatures, which increases the resistance to dislocation movement. However, this transformation process at low temperatures can be time-consuming, taking several days, due to the slow kinetics involved. This is because carbon diffusion is required during the nucleation stage of the process.

Electropulsing (EP) is a rapid processing technique that involves the application of high-density current pulses to modify the internal structure of a material. This technique has the capability to accelerate recrystallization and phase transformation kinetics, as well as improve plasticity. One potential benefit of EP is its ability to enhance the diffusivity of carbon, which

is necessary for the nucleation of bainite. This, in turn, can accelerate the kinetics of bainitic transformation in a less stable or metastable austenite phase. Therefore, the objective of the present investigation is to produce UFG and nanostructured bainitic steel and to see the possibility in plasticity and improvement in kinetics of bainitic transformation in these materials. The thesis is divided into EIGHT chapters, discussed below.

Chapter I provides a brief literature survey on UFG materials processed by equal-channel angular pressing and the austempering of alloys to produce nanostructured bainite. The chapter highlights the challenge of poor ductility in UFG materials, despite their excellent strength, and explores the potential of EP as a means to recover ductility without compromising strength. The characteristics of nanostructured bainite and the significant issue of slow kinetics are also discussed. Furthermore, the chapter explores the possibility of kinetic acceleration and the stability of different phases when subjected to high-density current pulses. Finally, the chapter concludes by outlining the objectives of the present investigation as given below.

- The effect of electropulsing on ultrafine-grained ferritic steel processed by Equal-channel angular pressing.
- Design of suitable steel composition to achieve nano-structured bainite with minimum carbide content.
- The influence of different austempering durations on the microstructural evolution of nanostructured bainite in the selected steel.
- Effect of austempering time on variant selection of nanostructured bainite in the steel.
- Strain hardening behavior of high carbon low alloy bainitic steel containing different amounts of retained austenite.
- The impact of electropulsing on the microstructural stability of retained austenite, bainite, and martensite in the high carbon low alloy steel.

Chapter II describes the design, development and characterization techniques used for microstructural and mechanical property analysis. To produce UFG steel, coarse grained low carbon steel of 0.07 mass % C is equal-channel angular pressed for 10 passes followed by EP for one pulse and five pulses. An alloy composed of Fe-1.06C-2.1Si-2.08Mn-1.3Cr-0.28Mo-1.5Co-1.2Al-0.44Ni is designed using JMatPro and ThermoCalc software to achieve an Ms temperature of -8°C . The aim is to produce nanostructured bainite by austempering at a temperature of 250°C , while maintaining reasonable kinetics. The alloy is prepared using an induction furnace. After hot rolling at 1000°C , the material undergoes austenitization, followed by austempering at 250°C for durations of 25 hours, 33 hours, and 48 hours. Another set of samples is directly air cooled after austenitization at 970°C . Additionally, the air-cooled and 48 hours austempered samples are subjected to further EP at various current densities. The resulting microstructural modifications are analysed using optical microscopy, scanning electron microscopy, electron backscattered diffraction, transmission electron microscopy and X-ray diffraction. Furthermore, mechanical properties are evaluated by tensile and hardness testing.

Chapter III illustrates the effect of EP on UFG low carbon steel processed by ECAP. It is observed that single-pulse EP of low carbon steel processed by ECAP results in a bimodal distribution of grain sizes and a reduction in defect density. On repeated EP, a few sub-grains coalesce resulting in the formation of a relatively defect-free region. Fine carbides that precipitate during ECAP is completely dissolved due to the accelerated movement of carbon atoms. Electropulsing of ultrafine-grained materials can enhance ductility by promoting the coarsening of some ultrafine grains into micron-sized grains through the migration of high-angle grain boundaries.

Chapter IV focuses on microstructural characterization aspects of nanostructured bainitic steel austempered for different timings. It is observed that in the early stages of bainitic

transformation, there is a continuous decrease in austenite grain size and an increase in dislocation density, which enhances the yield strength of austenite. However, the increased dislocation density hinders the growth of bainite plates, resulting in a slower rate of plate thickening. The omission of dynamic nature of austenite grain size and dislocation density overestimates in calculation of plate thickness in the existing empirical equations. However, longer austempering leads to the saturation of bainitic transformation and partial annihilation of dislocations due to slower kinetics. The reduction in dislocation density in austenite results in the lowering of hard impingement which increases the thickening of bainite plates at the edge of a sheaf. The orientation relationship determined from EBSD shows the bainite formed at 250 °C consists of both Kurdjumov–Sachs and Nishiyama–Wasserman types. Among the various crystallographic variant pairs, the V1-V6 pair, associated with the Kurdjumov–Sachs orientation relationship, dominates as the transformation progresses.

Chapter V describes the influence of carbon distribution in retained austenite and its impact on the mechanical properties of the material. In the short-time austempered sample, the presence of carbon inhomogeneity within the blocky retained austenite promotes early strain-induced martensitic transformation. This transformation negatively affects elongation due to the creation of discontinuities within the ductile austenite phase. On the other hand, in the longer austempering time sample, homogeneous distribution of high carbon in austenite leads to a delayed strain-induced martensitic transformation. This transformation contributes to an improvement in ductility, mainly attributed to the occurrence of the transformation-induced plasticity effect. Analysis of the tensile fractured sample reveals a larger volume fraction of tetragonal martensite ($c/a=1.01$) in the 48 hours sample, which can be attributed to the conversion of a higher fraction of retained austenite into martensite.

Chapter VI explores the stability of nanostructured bainite and retained austenite when subjected to electropulsing. Instrumented hardness testing reveals that the passage of electric

current leads to a decrease in the elastic modulus of the material. This decrease is attributed to the weakening of bonds within the material caused by the flow of current. The reduced elastic modulus facilitates easy shearing, promoting diffusionless transformation in the material. The rapid migration of carbon to generate a potential nucleation site and the lowering in elastic modulus in the presence of current pulse enable the blocky RA to transform into bainite. The initial electric pulse can induce carbide precipitation both in the retained austenite and the bainite phases. Estimating the growth kinetics of precipitates indicates that the observed sizes cannot be solely explained by thermal effects during heating and cooling processes. The kinetics of carbide precipitation are accelerated by the enhanced mobility resulting from the athermal effect of EP, rather than relying solely on thermal effects. Carbide precipitation in blocky retained austenite leads to a decrease in chemical stability. As a result, the carbon-depleted austenite undergoes partial transformation into martensite during cooling to room temperature. After five pulses carbide precipitates are completely dissolved in austenite of higher solubility, on the other hand it is decreased in bainite. Hardness of austempered steel increases at initial pass of EP due to additional bainite and carbide formation. On further pulsing the material is softened due to dissolution of carbides and partial recovery of defects.

Chapter VII focuses on the effect of EP on stability of martensite obtained through the air-cooling process of a high carbon low alloy steel sample. The boundary between the martensite and retained austenite phases in the D2-A material predominantly consists of low-angle boundaries. However, under the influence of EP, these boundaries undergo a transformation and convert into high-angle boundaries through migration. After pulsing the material for one pass at a high current density, a significant amount of carbide formation is observed in the martensite phase. The retained austenite phase undergoes a transformation into both bainite and martensite. Furthermore, when subjected to EP, the retained austenite phase is converted into fine carbides. Hardness of the electropulsed sample increases due to additional

bainite/martensite formation and it is more in case of low current density sample where temperature effect is less for softening.

Chapter VIII outlines the major conclusions drawn from the present research work as given below.

Electropulsing is very much effective in recovering ductility in the SPD materials by forming bimodal sized grains with less defect density in a fraction of second.

The bainite plate thickness is found to be dependent on the dynamic nature of dislocation density and austenite block size which was not included in the empirical equations developed in the earlier works. Nanostructured bainite produced by prolonged austempering, annihilates dislocation, as a result thickness of plates at the edge of sheaf is increased. The V1-V6 variant pairing is more dominating in the high carbon low alloy nanostructured bainitic steel.

On deformation of nano structured bainitic steel homogeneous distribution of carbon in austenite in longer austempering sample leads to gradual strain-induced martensitic transformation which adds to ductility because of transformation-induced plasticity, while, inhomogeneous distribution of carbon behaves opposite by forming martensite in the early stage of deformation.

Electropulsing of high carbon bainitic steel leads to precipitation of carbides with different morphologies along with bainite and martensite phase. Estimation of growth kinetics of precipitates confirms that observed sizes are not feasible by only thermal effect during heating as well as cooling. The kinetics of carbide precipitation are accelerated due to enhanced mobility by athermal effect of electropulsing along with the thermal one. Repeated pulsing can redissolve carbide precipitates completely in austenite on the other hand its content is decreased in bainite. Instrumented hardness testing shows a decrease in elastic modulus as a result of the weakening of the bonds caused by the passage of electric current. Easy shearing promotes

diffusionless transformation like bainite and martensite because of reduction in elastic modulus.

Electropulsing applied to steel containing martensite and RA undergo decomposition to cementite in martensite phase and induces bainite and martensite in RA phase. Instead of softening, application of EP effectively increases the hardness of the steel and it is more in case of low current density where temperature effect is less.