Hot deformation behaviour of Ni-30Fe and Ni-30Fe-Nb-C model alloys

Parvez Mannan

University of Wollongong
Hot Deformation Behaviour of Ni-30Fe and Ni-30Fe-Nb-C Model Alloys

Parvez Mannan, B.Sc.

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I, Parvez Mannan, declare that this thesis, submitted in partial fulfilment of the requirements for the award of Doctor of Philosophy, in the School of Mechanical, Materials and Mechatronic Engineering, Faculty of Engineering and Information Sciences, University of Wollongong, is wholly my work unless otherwise referenced or acknowledged. The document has not been submitted for qualifications at any other academic institution.

Parvez Mannan

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Abstract

The studies of the influence of processing parameters and the effect of Nb content (from 0 to 1.4 wt. %) on the dynamic recrystallisation behaviour in model Ni-30 wt. % Fe alloys were conducted using a Gleeble-3500 thermo-mechanical processing simulator. Different processing parameters, such as solution treatment and deformation temperatures, strain and strain rate, were used to elucidate their effects on softening processes. It was shown that the deformation activation energy has increased from 362 KJ/mol to 419 KJ/mol with an addition of 0.332 wt. % Nb to Ni-30 wt. % Fe alloy.

In order to clarify the evolution of dynamic recrystallisation, electron backscattering diffraction and transmission electron microscopy were used for systematic investigation of the interrupted strain specimens of Ni-30 wt.% Fe-0.331 wt.% Nb-0.04 wt.% C alloy subjected to plane strain compression at 1075 °C, 0.1 s\(^{-1}\) strain rate. Segmentation of partially recrystallised maps into dynamically recrystallised and unrecrystallised fractions has performed based on the grain orientation spread criterion.

It was shown that the discontinuous dynamic recrystallisation occurred through the nucleation of new strain-free grains at bulged pre-existing boundaries leading to the formation of the first necklace-like layer of dynamically recrystallised grains. At higher strains, the DRX evolves via both subsequent nucleation at DRX grains/deformed matrix interfaces leading to more layers formation and by independent nucleation events within the deformed grains interior. The growth of DRX grains occurs via strain-induced boundary migration.

The micro-texture of Ni-30 wt.% Fe-0.331 wt.% Nb-0.04 wt.% C in \(\varepsilon = 0\) condition comprises of cube-RD (\(\{013\}\{100\}\)), cube-ND (\(\{001\}\{310\}\)) and cube (\(\{001\}\{100\}\)) orientations. At strains \(\geq 0.68\), the dynamic recrystallisation texture showed the dominance of cube orientation, which intensifies and stabilises during the progress of dynamic recrystallisation. The texture of unrecrystallised grains also
showed the dominance of cube orientation. This could be explained by the low stored energy of cube grains and their overall orientation stability.

The influence of NbC particles on deformation substructure and softening were investigated using scanning transmission electron microscopy. It was shown that the role of NbC precipitates is twofold. Comparatively smaller semi-coherent particles (~10-150 nm) severely restrict the dislocations re-arrangement and subgrain boundary mobility, thus leading to sluggish or even suppressed dynamic and metadynamic recrystallisation. However, the evidence of particle stimulated nucleation at coarse particles of ≥ 400nm size was also described.

Due to the high deformation temperature and fast diffusion rates, the minimum size of the NbC particles in the alloys with 0.87-1.4 wt. % Nb content was found to be ~10 nm, despite a significant increase in their number density after ε = 1.2 indicating their formation and growth during deformation. The detailed analysis of particle size, shape and distribution was carried out. The shape of the particles changes from nearly elliptical to octahedral followed by hexagonal and tetra-kai-decahedral by truncation of {111} facets by {001} ones, since the boundary energy of the latter is lower. The detailed crystallographic analysis of different size NbC particle and austenite matrix interfaces reveals that 10-150 nm particles are semi-coherent with the austenite matrix and show additional planes in the matrix and misfit dislocations at interface every four to five atomic planes when the observed interfaces are edge on.
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# Table of Contents

Certification .......................................................................................................................... II
Abstract ................................................................................................................................. III
Acknowledgement ................................................................................................................ V
Table of Contents .................................................................................................................... VII
List of Figures ........................................................................................................................ XI
List of Tables .......................................................................................................................... XX
List of Publications ............................................................................................................... XXI

Chapter 1: Introduction ........................................................................................................... 1
  1.1 General background ........................................................................................................... 1
  1.2 Scope ............................................................................................................................... 2
  1.3 Outline of thesis ............................................................................................................... 4

Chapter 2: Literature Review .................................................................................................. 6
  2.1 Ni-30Fe model alloys ...................................................................................................... 7
  2.2 Ni-Fe phase diagram ....................................................................................................... 7
  2.3 Work hardening .............................................................................................................. 9
  2.4 Softening mechanisms ................................................................................................... 12
    2.4.1 Static recovery .......................................................................................................... 12
    2.4.2 Static recrystallisation ............................................................................................. 13
  2.5 Dynamic softening phenomena ..................................................................................... 14
    2.5.1 Dynamic recovery ................................................................................................... 15
    2.5.2 Dynamic recrystallisation ......................................................................................... 17
      2.5.2.1 Flow behaviour ................................................................................................ 17
      2.5.2.2 Nucleation ....................................................................................................... 19
      2.5.2.3 Microstructure evolution ................................................................................ 24
      2.5.2.4 Texture development ....................................................................................... 25
      2.5.2.5 Factors affecting dynamic recrystallisation ................................................... 28
2.5.2.6 Influence of solute elements and precipitates on dynamic recrystallisation ............................................. 30

2.6 Summary .......................................................................................................................................................... 38

Chapter 3: Experimental and Analytical Procedures ................................................................. 40

3.1 Chemical composition and production of alloys................................................................. 40
3.2 Solubility of NbC ....................................................................................................................... 41
3.3 Thermo-mechanical Processing in Gleeble-3500 Simulator ......................................... 44
   3.3.1 Determination of non-recrystallisation temperature .................................................. 46
   3.3.2 Thermo-mechanical processing schedules ................................................................. 48
3.4 Microstructure characterisation .......................................................................................... 50
   3.4.1 Sample preparation ............................................................................................................. 50
   3.4.2 Optical microscopy and scanning electron microscopy ............................................. 51
   3.4.3 Electron backscattering diffraction ............................................................................. 53
   3.4.4 TEM ............................................................................................................................... 54
3.5 Calculation of dynamic recrystallisation fraction ............................................................ 55

Chapter 4: Hot Deformation Behaviour of Ni-30Fe-C and Ni-30Fe-Nb-C Model Alloys ................................................................. 58

4.1 Results ......................................................................................................................................................... 58
   4.1.1 Flow curve analysis ............................................................................................................... 58
   4.1.2 Optical microscopy and scanning electron microscopy ............................................. 62
   4.1.3 Characterisation of work hardening and softening behaviour ................................. 66
   4.1.4 Interrupted strain observation ......................................................................................... 67
   4.1.5 Zener-Hollomon parameter (Z) and deformation activation energy (Q) ............ 68
   4.1.6 Fraction of dynamically recrystallised grains ............................................................... 72
4.2 Discussion ................................................................................................................................................. 74
   4.2.1 Flow behaviour .................................................................................................................... 74
   4.2.2 Grain structure ..................................................................................................................... 74
   4.2.3 Work hardening and softening behaviour ................................................................. 75
   4.2.4 Effect of alloying elements on deformation activation energy ............................. 76
4.2.5 Influence of processing parameters on dynamic recrystallisation evolution ................................................................. 78

4.3 Conclusions ........................................................................................................................................................................... 80

Chapter 5: Insight into Microstructure and Micro-texture Evolution During the Dynamic Recrystallisation of Ni-30Fe-Nb-C Model Alloy ........................................... 82

5.1 Results ..................................................................................................................................................................................... 82
  5.1.1 Flow curve analysis .............................................................................................................................................................. 82
  5.1.2 Microstructure characterisation .......................................................................................................................................... 83
  5.1.3 Intragranular local misorientation and substructure ........................................................................................................... 90
  5.1.4 Nucleation and progression of dynamic recrystallisation ................................................................................................. 96
  5.1.5 Development of dynamic recrystallisation texture ........................................................................................................... 99

5.2 Discussion ................................................................................................................................................................................. 105
  5.2.1 Microstructure evolution ...................................................................................................................................................... 105
  5.2.2 Development of micro-texture ........................................................................................................................................... 107

5.3 Summary .................................................................................................................................................................................. 108

Chapter 6: Effect of Nb on Dynamic and Post Dynamic Recrystallisation of Ni-30Fe-Nb-C Model Alloys .......................................................... 110

6.1. Results ..................................................................................................................................................................................... 110
  6.1.1 Flow behaviour .................................................................................................................................................................. 110
  6.1.2 Microstructure in optical microscopy ................................................................................................................................... 111
  6.1.3 Particle analysis in scanning electron microscopy ........................................................................................................... 115
  6.1.4 Substructure development and interaction of particles with dislocations and boundaries .................................................... 119
  6.1.5 Nucleation mechanisms during DRX .................................................................................................................................. 122
  6.1.6 Particle shape evolution with size ...................................................................................................................................... 125

6.2. Discussion ................................................................................................................................................................................ 132
  6.2.1 Effect of the alloy composition and processing on NbC precipitate behaviour ........................................................................ 132
  6.2.2 Precipitate and solute interaction with dislocations and boundaries ..... 134
6.2.3 Flow behaviour, softening mechanism and microstructure evolution ... 136

6.3 Conclusions ............................................................................................................ 138

Chapter 7: General Conclusions and Future work .................................................. 139

7.1 Conclusions ............................................................................................................ 139

7.2 Contributions to original knowledge .................................................................. 142

7.3 Future directions .................................................................................................. 144

Bibliography ............................................................................................................... 146
List of Figures

Figure 2.1 Ni-Fe phase diagram at elevated temperatures..................................................8
Figure 2.2 Lattice parameters as a function of mole fraction of Ni [47]. .........................8
Figure 2.3 Schematic representation of (a) stress-strain (b) work hardening plots showing work hardening stages [24, 49].................................................................10
Figure 2.4 The process of recovery of plastically deformed materials.........................13
Figure 2.5 schematic representation of (a) discontinuous static recrystallisation during annealing of work hardened materials [56] and (b) continuous static recrystallisation controlled by particle coarsening [61]..................................................14
Figure 2.6 Representative stress-strain curves for Al-1% Mg deformed at 400 °C [47] showing dynamic recovery and the effect of strain rate on dynamic recovery..15
Figure 2.7 Development of microstructure in cDRX of AA1421 subjected to hot equal channel angular extrusion of a strain of 12 at 673 k [48].................................16
Figure 2.8 Typical flow curve of dynamic recovery and dynamic recrystallisation..18
Figure 2.9 Schematic of determination of critical point in DRX flow curve..........19
Figure 2.10 Schematic representation of dislocation density at a dynamic recrystallisation front [75]. $\rho_m$ is the maximum dislocation density for DRX and $\rho_x$ is the dislocation density in a distance of x from DRX front. ........................................20
Figure 2.11 Nucleation of polycrystalline copper compressed at $10^{-3}$ s$^{-1}$ to (a) $\varepsilon = 1.3$ at 573 K and (b) $\varepsilon = 0.2$ at 723 K [64, 66]. (c) Schematic representation of DRX nucleation [25, 64, 66, 76]. Taken from [56]. HAGBs are delineated by thick black lines, LAGBs by thin black lines. Twin boundaries are marked in yellow. ..............21
Figure 2.12 Three types of dislocation density in DRX grains [56, 65]: (a) a DRX nucleus, (b) a growing DRX grain, and (c) a critically work-hardened DRX grain and corresponding TEM micrographs (d), (e) and (f). $\rho_c$ is the critical dislocation density for DRX nucleation and $\rho_0$ is the dislocation density at annealed stage...............23
Figure 2.13 Recrystallisation nuclei associated with silica particles in a Ni-0.03 wt. % Si alloy during rolling to $\varepsilon = 0.9$ and annealing at 600 °C for 15 min: (a)
secondary electron image and (b) planar recrystallised front is observed in transmission electron micrographs [83].

Figure 2.14 Schematic of the microstructure evolution during DRX by necklace formation as suggested by Sellars et. al [85]: (a) no DRX grains exist below the critical strain ($\varepsilon_c$), (b) first necklace layer formed at pre-existing grain boundaries, (c) the formation of the subsequent necklace layer, (d) consumption of the deformed grains by successive necklace layers, (e) full DRX microstructure and (f) typical DRX flow curve.

Figure 2.15 Schematic representation of cold rolling texture of fcc materials in plane strain compression [24].

Figure 2.16 Crystallographic texture development in Ni-30Fe during PSC as a function of strains: (a) overall micro-texture at different temperature and (b) DRX grains at deformation temperature of 900 °C [15].

Figure 2.17 The flow curves of (a) Ni-30Fe alloy and (b) 0.15C steel deformed in plane strain compression at 900 °C with varying strain rates [28].

Figure 2.18 Stress-strain curves of (a) Ni-30Fe alloy and (b) 0.15C steel deformed with 0.7 s$^{-1}$ strain rate at various deformation temperatures [28].

Figure 2.19 Strengthening by solid solution (a) substitutional atoms, (b) interstitials and (c) introduced new strain fields around substitutional and interstitial solute atoms.

Figure 2.20 The interaction of particles with boundary or dislocations [117].

Figure 2.21 The Zener dragging force on grain boundary by a particle [119].

Figure 2.22 The Orowan mechanism of dislocation interaction with particles (a) bowing of a dislocation around particles and (b) dislocation passing through particles leaving a loop [116].

Figure 2.23 Dislocation motion past hard particle (a) without or (b-d) with cross-slip showing Orowan and prismatic loop formation [118].

Figure 2.24 Transmission electron micrographs of prismatic loops formed by cross-slip on outer Orowan loop in Cu-30% Zn-0.22% Al$_2$O$_3$, taken from [117].
Figure 2.25 Bright-field TEM micrographs of strain induced precipitation of NbC on microbands during deformation of Ni-30Fe alloys at 925 °C to \( \varepsilon = 0.2 \) and holding for 30 s [17].

Figure 3.1 The schematic diagram of the original rolling schedule to produce 15 mm thick plates of model alloys (except Alloy L\(_0\)) after casting.

Figure 3.2 (a) The solubility curve used to calculate Nb in solution in Ni-30Fe alloys from DeArdo et. al [115]. (b) Amount of solute Nb and C in Ni-30Fe solid solution as function of temperature based on Wang et. al. [104] method.

Figure 3.3 The solubility curve of studied alloys calculated by Zurob et al. [129].

Figure 3.4 Gleeble-3500 thermo-mechanical processing simulator.

Figure 3.5 Schematic of sample orientation in Gleeble-3500 TMP simulator.

Figure 3.6 The schematic diagram for determination of non-recrystallisation temperature (Tnr).

Figure 3.7 The procedure for determination of Tnr: (a) an example of true stress vs true strain curve of multiple hit test in alloy H and calculated Tnr for (b) alloy H, (c) alloy M and (d) alloy L.

Figure 3.8 Schematic diagram to study the hot deformation behaviour of alloys L and L\(_0\) (presented in Chapter 4).

Figure 3.9 Schematic diagram to investigate the evolution of hot deformation microstructure and micro-texture in alloy L (Ni-30Fe-0.331Nb-C) (presented in Chapter 5).

Figure 3.10 Schematic diagram to study the effect of NbC precipitates on dynamic and metadynamic recrystallisation (presented in Chapter 6).

Figure 3.11 The cutting direction for optical and SEM microscopy of Gleeble samples and planes of specimen with respect to compression axis. The black coordinates represent PSC convention and the coordinates in red represent rolling convention.

Figure 3.12 Determination of maximum softening \( (\varepsilon_m) \) strain from work hardening rate vs strain curve [87].
Figure 4.1 Flow curves of model alloys (a, c, e) alloy L and (b, d, f) alloy L₀ after a plane strain compression with $\varepsilon = 1.2$ at different deformation temperatures and strain rate of (a, b) $1 \text{s}^{-1}$ (c, d) $0.1 \text{s}^{-1}$ and (e, f) $0.01 \text{s}^{-1}$.

Figure 4.2 Determination of the critical point (a) first derivatives and (b) second derivatives of stress strain curves obtained from plane strain compression of 1.2 at a deformation temperature of 1000 °C for L alloy.

Figure 4.3 Effect of (a, c) temperature on peak stress and strain for a deformation strain rate of 0.01s$^{-1}$ and (b, d) strain rate on peak stress and strain for a deformation temperature of 1075 °C for (a, b) alloy L and (c, d) alloy L₀.

Figure 4.4 Representative optical microstructures of alloys (b, c) L₀ and (d, e) L after roughing at 1175 °C with 0.3 strain and quenched before deformation temperature of (b, d) 1150 °C and (c, e) 1000 °C.

Figure 4.5 Microstructures of L alloy after $\varepsilon = 1.2$ at a strain rate of (a-d) 1 s$^{-1}$, (e-h) 0.1 s$^{-1}$, (i-l) 0.01 s$^{-1}$ and deformation temperature of (a, e, i) 1150 °C, (b, f, g) 1075 °C, (c, g, k) 1000 °C and (d, h, l) 925 °C.

Figure 4.6 Grain size distribution after final deformation; (a, c) for different temperature at 0.01 s$^{-1}$ strain rate and (b, d) for different strain rate at a deformation temperature of 1150 °C of alloys (a, b) L and (c, d) L₀.

Figure 4.7 Work hardening rate curves in alloys (a, b) L and (c, d) L₀ at deformation temperature of 925-1150 °C and strain rate of (a, c) 0.01 s$^{-1}$ and (b, d) 1 s$^{-1}$.

Figure 4.8 (a) Flow curve of alloy L at a deformation temperature of 1075 °C and 0.1 s$^{-1}$. Optical micrographs for intermediate strain of (b) 0.23 (arrows indicate serrated boundary), (c) 0.35, (d) 0.68 and (e) 0.85.

Figure 4.9 Determination of (a, c) n from sinh($\alpha \sigma_p$) vs strain rate plot and (b, d) the deformation activation energy (Q) from lnA vs -1/RT plot for alloys (a, b) L and (c, d) L₀.

Figure 4.10 Effect of Zener-Hollomon parameter (Z) on (a, c) critical and peak stress and (b, d) critical and peak strain for (a, b) L and (c, d) L₀ alloys.

Figure 4.11 Evolution of DRX fraction of L and L₀ model alloys at different deformation temperatures and strain rate of (a) 0.01 s$^{-1}$ (b) 0.1 s$^{-1}$ and (c) 1 s$^{-1}$.
Figure 5.1 (a) Flow curve and work hardening curve of alloy L after 1.2 strain at 1075 °C, the red circles on flow curve are the points of interrupted strain experiments and (b) recrystallised fraction with respect to strain calculated from EBSD and empirical equation.................................................................83

Figure 5.2 EBSD inverse pole figure maps at strains of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85, and (f) 1.2. LAGBs = grey, HAGBs = black, 60° 111, Σ3 TBs = red lines ..................................................................................................................84

Figure 5.3 EBSD band-contrast maps at strains of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85, and (f) 1.2. LAGBs = blue lines, HAGBs = thick black lines and 60° 111, Σ3 TBs = red lines........................................................................................................85

Figure 5.4 EBSD inverse pole figure maps of (a, c, e, i) recrystallised and (b, d, f, h, j) unrecrystallised fractions at (a, b) 0.23, (c, d) 0.35, a clear necklace layer delineates the pre-existing grain boundaries (e, f) 0.68, (g, h) 0.85 and (i, j) 1.2 deformation strain. ........................................................................................................88

Figure 5.5 (a) Misorientation distribution as a function of strain with typical misorientation axis distributions in crystal coordinate system for Σ3 and Σ9 angular range, (b) grain boundary area fraction as a function of deformation strain, (c) the initial grain size is measured before any deformation and average DRX grain size at different strain with and without TBs as grain boundaries, (d) the area fraction Σ3 annealing TBs of cube and non-cube DRX grains.................................................................89

Figure 5.6 Intragranular local misorientation maps at strain of (a) 0.23, (b) 0.35, (c) 0.68, (d) 0.85 and (e) 1.2. Black lines are HAGBs, high local misorientation is delineated by green area, reds are twin. ........................................................................................................91

Figure 5.7 Intragranular local misorientation of (a) DRX grains at different strains and (b) mean intragranular local misorientation of recrystallised, unrecrystallised subsets and full maps as a function of true strain. .............................................................................92
Figure 5.8 (a) TEM bright-field image using two beam with [111] excited condition at $\varepsilon = 0$. TEM bright-field images where beam direction close to [011] zone axis, showing dislocation cell walls and sub-structure development during DRX at (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85, and (f) 1.2. The inset in (f) shows the diffraction pattern with [011] zone axis. The white arrows in the micrographs (b-f) indicate the dislocation walls and subgrain boundaries while the opposite arrows in the top right inset show the deformation direction.

Figure 5.9 (a) Zoomed-in area of EBSD local misorientation maps of (a) 0.23, (b) 0.35 and (c) 0.68 marked by red rectangle in Figures 4.6 a, b and c respectively. TEM bright-field image, beam direction close to [011] zone and samples deformed to (d) 0.23, (e) 0.35 and (f) 0.68. The white arrows in EBSD maps showed high intragranular local misorientation areas while in TEM red arrows indicate the arrays of dislocations and dislocations cells.

Figure 5.10 (a) EBSD band contrast maps, thick black lines are HAGBs, blue lines are LAGBs and red lines are first order TBs and (b-d) STEM bright field images at $\varepsilon = 0.23$, the white arrows in EBSD and STEM micrographs depict the origination of new DRX nuclei at pre-existing boundaries (a, b, c) and twins (a, d) by subgrain formation. (e) A SADP along [011] zone showing both twin and matrix lattices and (f) DF image of the twin in (d) using [200] spot of twin lattice.

Figure 5.11 Selected area EBSD band contrast maps from samples at (a) 0.68 and (b) 0.85 strain showing nucleation inside deformed grains. TEM bright-field micrographs of the samples deformed at (e) 0.68 showing the incipient DRX nuclei, and (f) 0.85 revealing dislocation cells and subgrains inside deformed grains. The beam direction in TEM is close to [011] zone axis. The white arrows in all images indicate some DRX nuclei or subgrains.

Figure 5.12 Schematic representation of important texture components in fcc metals and alloys.

Figure 5.13 $\varphi_2 = 0^\circ$, 45° and 65° ODF sections of coarse map with 10 µm step size at (a) 0, (b) 0.23 and (c) 0.35, strain. Contour levels = $2\times$. 
Figure 5.14 $\varphi_2 = 0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of full maps at strain of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85 and (f) 1.2 with a step size of 1 µm. Contour levels = $2\times$.

Figure 5.15 $\varphi_2 = 0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of recrystallised subsets at strain of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85 and (f) 1.2 deformation strain. Contour levels = $2\times$.

Figure 5.16 $\varphi_2 = 0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of unrecrystallised subsets at strain of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85 and (f) 1.2 deformation strain. Contour levels = $2\times$.

Figure 6.1 Flow curve of model alloys (a) after final deformation of 1.2 at 1075 °C at 0.1 s$^{-1}$ strain rate. (b) First derivatives (work hardening rate), (c) second derivatives and (d) the relationship of critical strain and critical stress with wt. % (Nb + C).

Figure 6.2 Optical microstructure of (a, d, g, j, m) alloy L, (b, e, h, k, n) alloy M and (c, f, i, l, o) alloy H after (a-c) reheating at 1250 °C for 300 s, (d-f) roughing at 1250 °C, $\varepsilon = 0.3$ and quenched from 1075 °C and (g-o) final deformation of $\varepsilon = 1.2$ at 1075 °C and subsequent hold for (g-i) 0 s, (j-l) 30 s and (m-o) 300 s.

Figure 6.3 (a) Average recrystallised grain size and (b) recrystallised fraction in alloys L, M and H after $\varepsilon = 1.2$ at 1075 °C followed by 0 s, 30 s and 300 s holding.

Figure 6.4 Representative SEM images of (a) large and (b) small NbC particles with (c) EDS spectra for large and small size NbC particles and matrix.

Figure 6.5 SEM micrographs of (a, c, e, f, i) alloy M and (b, d, f, h, j) alloy H in (a, b) reheated to 1250 °C for 300 s, (c, d) non-deformed 1075 °C condition. The alloys deformed to $\varepsilon = 1.2$ at 1075 °C, then hold for (e, f) 0 s, (g, h) 30 s and (i, j) 300 s.

Figure 6.6 The particle size distribution in (a, c) alloy M and (b, d) alloy H. (a, b) after reheating and non-deformed 1075 °C condition and (c, d) after a final strain of 1.2 at 1075 °C and subsequent holding for 0 s, 30 s and 300 s.
Figure 6.7 TEM bright-field images of NbC particles and substructure development obtained with the beam direction close to [011] zone in (a, b) alloy L, (c, d) alloy M and (e, f) alloy H at 1075 °C with a strain of (a, c, e) $\varepsilon = 0$ and (b, d, f) $\varepsilon = 1.2$. The white circles locate the position of NbC particles.

Figure 6.8 Bright-field TEM micrographs of the specimen deformed to 1.2 and beam direction close to [011] zone axis showing the substructure and interaction of different size NbC particles with dislocations and (sub)grain boundaries in (a, c, e) alloy M and (b, d, f) alloy H. (e) The restriction of a dislocation array by a 40 nm and (f) Interaction with a subgrain boundary by 130 nm and 240 nm NbC particles. The white circles locate the position of NbC particles and the arrows in the top right corner indicate the compression direction.

Figure 6.9 (a, c, e) Optical micrographs and (b, d, f) bright-field TEM images, the beam direction close to the [011] zone axis, of the classical DRX nucleation mechanism in (a, b) alloy L, (c, d) alloy M and (e, f) alloy H after a final strain of 1.2 at 1075 °C.

Figure 6.10 STEM images, the beam direction close to the [011] zone axis, delineating particle stimulated nucleation in (a) alloy M and (b-d) H after $\varepsilon = 1.2$, 0 s at 1075 °C. The red numbers on the images are the misorientation angles of the boundaries and black arrows in the inset represent the compression direction. The misorientation angles in (c, d) were determined using CBED patterns.

Figure 6.11 Bright field TEM micrographs obtained with the beam direction close to the [011] zone axis in (a) alloy L, (b) alloy M and (c) alloy H after deformation of 1.2. The zoomed in view of dislocation network, arrays and subgrain boundaries in inset of each image shows the absence of strain induced precipitation.
Figure 6.12 STEM observation of different size NbC particles for the samples deformed to $\varepsilon = 0$ or $\varepsilon = 1.2$ in (a-d, f) alloy M and (g, h) in alloy H: (a-c) Detailed bright-field micrographs of (a) 17 nm, (b) 10 nm and 26 nm and (c) 46 nm faceted particles with the Fast Fourier transformed (FFT) pattern in the inset of each image at $\varepsilon = 1.2$. (d) A 130 nm NbC particle and the corresponding selected area diffraction pattern ($\varepsilon = 0$). (e) Pattern indexing revealing the [011]$_{\text{NbC}}$ zone axis and cube-on-cube orientation relationship between the $\gamma$ and NbC lattices, DD denotes the double diffraction. (f) A 155 nm particle without an orientation relationship to the matrix. (g) A 70 nm faceted particle at $\varepsilon = 1.2$. (h) Bright-field STEM image of a tetra-kai-decahedral particle enclosed by {$100$)$_{\text{NbC}}$ and {111}$_{\text{NbC}}$ planar facets and the corresponding SAD patterns with [011]$_{\text{NbC}}$ zone axis at $\varepsilon = 0$. (i) Schematic representation of the tetra-kai-decahedral particle shape.

Figure 6.13 STEM HAADF images of NbC/$\gamma$ interfaces of the samples deformed to 1.2: (a) from 10 nm and (b, c) from 26 nm particles observed in alloy M, bright-field image shown at Figure 12 a, b and (d) from 46 nm particle of alloy H. The lattice fringes are marked on a red box and the white lines in all images are the approximate location of interfaces.

Figure 6.14 STEM HAADF and corresponding lattice images of NbC/$\gamma$ interfaces in alloy H at $\varepsilon = 0$: (a) two types of interfaces (facets and oblique) on a 67 nm particles (b) The interfaces of {$100$}$_{\text{NbC}}$ and {$111$}$_{\text{NbC}}$ planar facets in a 150 nm particles. The locations of additional lattice planes are denoted by the red dotted lines in lattice images while the white dotted lines are the approximate location of the interface.
List of Tables

Table 2.1 Coordinates of main α- fibre and β- fibre components in Euler space. .....26

Table 3.1 Chemical compositions in wt. % of model alloys. Dissolution and solidus temperature for the model alloys. ........................................................................................................40
Table 3.2 Solubility temperature and nose temperature with wt. % of Nb and C in solution for alloy L, M and H at 1250 °C reheating. .................................................................42
Table 3.3 Determined recrystallisation stop temperature (Tnr) (°C) values for alloys L, M and H. ........................................................................................................................................47
Table 3.4 Sample preparation steps in Tegrapol-21.................................................52

Table 4.1 Measured critical stress, strain and peak stress, strain for L and L₀ model alloys for different strain rates (s⁻¹) and temperatures. .........................................................61
Table 4.2 Average recrystallised grain size (µm) after final deformation of L and L₀ alloys. ........................................................................................................................................65
Table 4.3 The power law relationships between Zener-Hollomon parameters (Z) and peak (σp) or critical (σc) stresses and peak (εp) or critical (εc) strains in two sets of model alloys..................................................................................................................................70
Table 4.4 Deformation activation energy for different C-Mn steels, stainless steels and Ni based alloys ......................................................................................................................................77

Table 5.1 Euler angles and Miller indices for common texture components in fcc metals and alloys [178]. ..................................................................................................................100

Table 6.1 Average grain sizes at reheating and non-deformed 1075 °C and average recrystallised grain sizes (µm) of alloys at ε = 1.2, 1075 °C and subsequent holding for 0 s, 30 s and 300 s........................................................................................................114
Table 6.2 Average particle size and particle number density in alloy M and H at different deformation conditions........................................................................................................117
List of Publications

- "Hot deformation behaviour of Ni-30Fe-C and Ni-30Fe-Nb-C model alloys"  
  **P. Mannan**, A.G. Kostryzhev, H.Zurob, E.V. Pereloma  

- "High temperature dislocation structure and NbC precipitation in three Ni-Fe-Nb-C model alloys"  
  A.G. Kostryzhev, **P. Mannan**, O.O. Marenchy  

- "Effect of thermo-mechanical processing on Nb precipitation and austenite recrystallisation in Ni-30Fe-Nb-C model alloys"  
  **P. Mannan**, A.G. Kostryzhev and E.V. Pereloma  

- "Effect of Strain on Microstructure and Texture Development in Model Ni-30Fe-Nb-C Alloy During High Temperature Plane Strain Compression."  
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- "Insight into microstructure and micro-texture evolution during the dynamic recrystallisation of Ni-30Fe-Nb-C model alloy."  
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Chapter 1: Introduction

1.1 General background

The plastic deformation of metallic materials has started as early as around 300 BC in Iron Age by blacksmith despite having very narrow knowledge about metallurgy and not knowing about phase transformations at all. Another common way of enhancing properties through alloying started back in 2000 BC during Bronze Age via addition of foreign elements Sn, As during smelting of copper. The plastic deformation processes in those days were limited to making tools and decoration purposes. The modern civilisation strongly relies on the proper knowledge of materials science achieved by research and development from thousands of years. Nowadays, the technology also advanced many folds and could precisely control the change of materials on macroscopic and microscopic scales during forming. Thousands of millions of superior and specific parts make it possible to build giant aircrafts Airbus A-380, Boeing-747 to miniature mobile phones in our pocket.

High strength low alloy steels are widely used as structural material, while advanced high strength steels are mostly used for weight reduction in cars. Modern advanced steels, possessing excellent mechanical and physical properties compared to conventional grade of steels, contain micro-alloying elements such as Nb, Ti, Mo, and V. These micro-alloying elements combine with C or N to give precipitation of carbides, carbonitrides, and sometimes more complex precipitated particles. In general, micro-alloying elements have an enormous effect on the strength of steel either by precipitation strengthening, grain refinement, solute dragging and solid solution strengthening or by all mechanisms operating concurrently, as for example, occurs in high strength low alloy steels [1].

It is believed that grain size, orientation of grains and the distribution of second phase determine the properties of materials [2]. Grain size refinement by hot deformation or by cold deformation and annealing is used to enhance mechanical properties. The microstructure and micro-texture of steels during hot deformation is controlled by the processing parameters and stacking fault energy (SFE) of the
materials. However, the study of high temperature phases at room temperature in commercial steels is impossible, as low temperature phases (martensite, ferrite, bainite and pearlite) obscure the high temperature austenite and second phase precipitate. The austenite microstructure at elevated temperature of steels can be replicated in some model alloys owing similar SFE to steels.

### 1.2 Scope

Ni-30 wt. % Fe as a model alloy offers the advantages of having only single phase austenite microstructure and the SFE similar to that of austenite in HSLA, C-Mn and low C steels [3, 4]. In addition, Ni-30 wt. % Fe alloy also has a similar to austenite in steels crystallographic texture and the same orientation dependence during formation of new grains [5]. Hereafter, Ni-30 wt. % Fe will be referred to as Ni-30Fe and that when numbers are given in compositions they are always in wt. %.

During hot deformation, new dislocations form leading to material work hardening. The work hardening rate increases up to a critical strain as dislocation density increases continuously, then after reaching the critical stored energy, recovery of material takes place by rearrangement of dislocations into lower energy configurations and by annihilation of dislocations of opposite sign via dislocation glide and climb [6]. Low to medium SFE face centred cubic materials soften by discontinuous dynamic recrystallisation (DRX) during hot deformation above recrystallisation stop temperature [7]. This is a two-stage process of nucleation and growth. Initially DRX nuclei originate at pre-existing grain boundaries by bulging and serration when dislocation density reaches the critical dislocation density. Then these nuclei transform and grow into new DRX grains. The evolution of DRX microstructure is quite interesting and greatly influenced by solute and precipitation. Electron backscattering diffraction and high-resolution transmission electron microscopy might be suitable to analyse the dislocation interactions and DRX phenomena in model Ni-30Fe alloys containing Nb. Although a significant research on model Ni-Fe and Fe-Ni alloys was carried out [3, 5, 8-15], a very limited work was done on these alloys containing Nb [16-18].

Nb is frequently used as an alloying element in modern steels to enhance the mechanical properties, as it is found to be the most effective grain refining element. Thus, it provides the largest strength increase. The effect of Nb on hot deformation
characteristics and the recrystallisation kinetics depends on whether it is present as a solute or as discrete second phase precipitates. Nb precipitates as NbC and/or NbCN during deformation and cooling restrict the grain boundary and dislocation movement. The large amount of strain during thermo-mechanical treatment leads to strain-induced precipitation (SIP). These SIP particles precipitate mostly on dislocations, microbands, tangles of dislocation and dense dislocation walls [17, 19-21].

Large particles may accelerate the process of DRX by providing additional sites for nucleation; hence, new DRX grains originate at the particle/matrix interface of coarse particles [22, 23]. Besides, Nb in solution is believed to play a role in the retardation of austenite recrystallisation through a process of solute drag [16]. The model alloys studied here have a wide range of Nb content and might be effective in characterising different size particles and their interaction with dislocations and grain boundaries. The aims of the research are concisely listed below:

- To investigate the effects of processing parameters and Nb content on flow behaviour and DRX during hot deformation of Ni-30Fe-Nb-C alloys in a wide temperature window.
- To elucidate the evolution of DRX microstructure and micro-texture as a function of strain using electron backscattering diffraction and scanning transmission electron microscopy.
- To investigate the relationship between recrystallisation behaviour and Nb states either as a precipitate or as a solute in the studied alloys.
- To study the Nb precipitation at different stages of thermo-mechanical processing schedule and the interactions of NbC particles with dislocations and grain boundaries.
- To evaluate the possibility of particle stimulated nucleation during dynamic recrystallisation in Ni-30Fe-Nb-C alloys.
- To study the crystallographic relationship of the different size NbC precipitates and austenite matrix interface using scanning transmission microscope.
1.3 Outline of thesis

The thesis consists of five main chapters. The literature review is presented in Chapter 2. It includes up to date knowledge and observations of Ni-30Fe alloys and other fcc metallic alloys behaviour at high temperature during plastic deformation. The influence of important hardening and softening mechanisms on microstructural evolution during hot deformation are highlighted. The mechanisms of recrystallisation and the role of Nb in these phenomena are discussed in Chapter 2. The present knowledge on the texture development during dynamic recrystallisation is also summarised.

The compositions of alloys, the detailed experimental techniques, sample preparation and analysing techniques are described in Chapter 3. The solubility temperatures of NbC in model alloys are calculated and compared with the previous solubility data in this chapter. All different thermo-mechanical processing schedules designed for this project are outlined. The electropolishing methods for both electron backscattering diffraction (EBSD) and transmission electron microscopy (TEM) are described.

Chapter 4 comprises of the effect of processing parameters and Nb addition on hot deformation behaviour of two model alloys: Ni-30Fe-C and Ni-30Fe-0.33Nb-0.04C. The flow curves of the alloys deformed in the temperature interval from 925 °C to 1150 °C at three strain rates of 0.01, 0.1 and 1 are described. The critical and peak strains and stresses for DRX are determined. The DRX fractions are calculated based on Avrami type model using the parameters obtained from flow curves. These DRX values are compared with the actual DRX fractions measured from the optical micrographs of specimens after interrupted strain tests along the flow curve. This chapter also addresses the effect of alloying elements on deformation activation energy.

Chapter 5 focuses on the evolution of dynamically recrystallised microstructure and micro-texture as a function of strain, which characterised by advanced microscopy techniques. Electron backscattering diffraction uncovered the mechanism of DRX grain formation that was further validated by the scanning transmission electron microscopy. The nucleation mechanisms at all strain levels up to 1.2 are discussed in this chapter. The micro-texture development in Ni-30Fe-0.331Nb-C model alloy is presented in Chapter 5.
The influence of the Nb content on the DRX behaviour in model alloys is outlined in Chapter 6. The effect of solute Nb and different size NbC particles on substructure formation and DRX grains nucleation and growth are elucidated. The shape change of NbC particles with respect to their size is characterised. The crystallographic relationship between austenite matrix and NbC precipitates is outlined and discussed in Chapter 6. The metadynamic recrystallisation behaviour was also investigated and discussed in this chapter. The particle stimulated nucleation mechanism on large NbC particles is delineated in Chapter 6.

Chapter 7 summarises the main results, lists the key contributions to the original knowledge, as well as suggests some future directions of research.
Chapter 2: Literature Review

There are two aspects of the metal forming processes: macroscopic scale to obtain the desired shape and microscopic scale related to the physical and mechanical properties [24]. Thermo-mechanical processing (TMP) refers to the combination of thermal and mechanical operation where the basic materials are transformed to a specific shape with superior properties. The elevated temperature phase in most of the commercial steels is austenite (γ) with face centred cubic (fcc) lattice. The characterisation of high temperature phases during deformation in these materials is extremely important. Some model alloys, 304 and 316 austenitic stainless steels have been examined to track the changes in elevated temperature austenite microstructures [9, 25-27], as low temperature transformation products, e.g. ferrite, pearlite bainite or martensite, in most commercial steels hinder direct observation of austenite. The microstructure development during hot deformation depends mainly on the SFE of materials. The SFE is related to the atomic bonding in the material and it defines the degree of dissociation of perfect dislocations into partial dislocations [6]. However, the alloying elements significantly alter the SFE of materials, hence the softening kinetics during deformation. The softening mechanism during and/ or after deformation has also seen to be a function of solute elements, strain, strain rate and deformation temperature [26, 28-30].

The alloying additions remain in solution at elevated temperature deformation, but these solute atoms could precipitate as carbide or carbonitrides during final deformation or during isothermal holding at lower temperature. The solutes, via the solute drag effect and the precipitates, control the microstructure and micro-texture evolution during dynamic recrystallisation [1, 6, 31]. The following review summarises the existing knowledge on the work hardening behaviour, deformation substructure formation, softening mechanisms and the effects of processing parameters on dynamic recrystallisation and microstructure and micro-texture development, as well as the influence of different sizes particles on dynamic and post dynamic softening.
2.1 Ni-30Fe model alloys

The two indirect methods to characterise high temperature austenite in different grades of steels are: (i) to reveal the prior autenite grain boundaries (GBs) by etching and/or analyse the response to hot deformation via the flow curve behaviour [32] and (ii) to use a model alloy in which the austenite is retained down to room temperature. 304 austenitic stainless steels (ASS) 316 ASS, Ni-30Fe, Fe-30Ni and Fe-Co model alloys have been studied by researchers in order to gain insight into the high temperature austenite characteristics [25-27, 33, 34].

The SFE of 304, 316 ASS (SFE = 21-23 mJ/m² [35, 36]) is lower than that of plain carbon steels [37], and twinning is the main deformation mechanism in this alloy at a lower deformation temperature [3]. Thus, the Shefield research group developed Ni-30Fe model alloys to examine the high temperature deformation behaviour of austenite [3, 5, 10, 14, 17, 18, 38]. The SFE of pure Ni is about 220 mJ/m² [39], whereas SFE of 70 mJ/m² [40] in Fe-40 Ni can be obtained. SFE of iron (75 mJ/m²) is not significantly altered when the concentration of alloying elements in plain carbon steel is very low [41]. The advantages of using Ni-30Fe alloy lies in: (i) it has a single phase austenite over the entire temperature range, (ii) the comparable stacking fault energy (~50-70 mJ/m²) [41] of Ni-30Fe model alloys to commercial steels signifies that analogous microstructure and texture to microalloyed, low C and C-Mn steels are expected to develop after hot deformation and (iii) acceptable solubility of NbC is maintained [38].

2.2 Ni-Fe phase diagram

Ni-based superalloys have exceptional combination of high temperature strength, toughness and resistance to degradation in corrosive or oxidising environments [42]. Figure 2.1 illustrates the phase diagram of Fe-Ni alloys above 600 °C, corresponding to the zone of reheating and deformation. The main features of the diagram are: the γ-(Fe, Ni) solid solution with an fcc structure extending over the complete composition range at high temperature and forming a very narrow two-phase field with liquid [43]. The liquidus temperature of model alloy is around 1440 °C – 1418 °C at a composition of approximately 67 to 70 % of Ni [44-46] which is far below than that for most commercial steels (~1517 °C). The lattice parameter of Fe-Ni binary alloys is a function of crystal structure (body centred cubic (bcc) or fcc)
and the percentage of alloying elements [44]. The lattice parameter of Ni-Fe alloys as a function of Ni content is depicted in Figure 2.2. The lattice parameter of Ni-30Fe (fcc) is ~0.356 nm and is very close to lattice parameters of austenite in carbon steels [47]. Owing to the similar lattice parameters, it is believed that the matrix accommodates the solute atoms and plastic deformation in steels and Ni-30Fe alloys in identical fashion.

Figure 2.1 Ni-Fe phase diagram at elevated temperature.

Figure 2.2 Lattice parameters as a function of mole fraction of Ni [47].
2.3 Work hardening

The concept of work hardening (WH) was introduced with the discovery of crystal dislocations and since then has developed continuously [48]. Work hardening also known as strain hardening is one of the typical properties of most metallic materials; it may be the most desirable one during plastic deformation. Dislocations are continuously generated during plastic deformation that regulates the plastic flow of crystalline materials. These dislocations impose obstacles to their movement; hence the shear stress of plastic flow increases with strain. Thus, further stress is required to overcome the barriers to dislocation movement. The critical shear stress \( \tau_c \), causes slip of dislocations in particular crystallographic directions on specific crystallographic planes of easy glide with a maximum inter-planar separation; e.g. the combination of close-packed direction in close-packed plane forms the slip system. The advent of every dislocation creates a displacement by a Burgers vector of dislocation (\( b \)) in slip direction. As prerequisite of the Von Mises criterion (five independent slip systems for plastic deformation), different combination of slip planes and directions operate and interact. The increase in flow stress with dislocation density can be written as:

\[
\tau_c = \tau_0 + \alpha G b \sqrt{\rho}
\]

(2.1)

where \( \tau_0 \) is the intrinsic glide stress, \( \alpha \) is a constant depends on the atomic structure and the type of substructure, usually 0.2 – 0.3 for fcc and 0.4 in the case of bcc materials, \( G \) is the shear modulus of the materials and \( \rho \) is the dislocation density (m\(^{-2}\)). The established square-root law is a result of the fact that the glide stress of dislocations is inversely related to the spacing of the obstacles, \( l \), i.e.

\[
\tau_c = \tau_0 + \alpha G b \frac{\rho}{l}
\]

(2.2)

Work hardening during plastic deformation of polycrystalline materials can be illustrated by several stages as shown in Figure 2.3 [49, 50]. Stage I is based on the orientation of crystals for single slip which is readily observed in single crystals. Typically, the dislocations are confined to slip planes without any interactions of dislocations; hence the WH rate is quite low. However, the reorientation of crystals towards double slip orientations during plastic deformation tends to favour significant dislocation interactions at stage II.
At the beginning of plastic deformation of polycrystalline materials, stage I is minimal as mobility of first few dislocations inhibited by GBs [24]. The restriction of GBs to the movement of dislocations introduces Hall-Petch grain size (D) hardening relation [51, 52]:

\[ \sigma (D) = \sigma_\infty + k_1 D^{-1/2} \]  \hspace{1cm} (2.3)

where, \( \sigma_\infty \) is flow stress of materials at extremely coarse grain size. Eq. (2.3) greatly affects the hardening of polycrystalline materials particularly when the grain size is
close to 20 μm or less. Generally, the $k_1$ value varies from 0.7 to 1 for C-steels, from 0.4 to 0.2 in hcp metals and from 0.1 to 0.07 for fcc Cu and Al.

Stage II is characterised by the high and constant work hardening rate due to the rapid multiplication of dislocations arising from the interactions of dislocations gliding on different slip planes. This stage of work hardening is linked to the orientations of the crystals and insensitive to deformation parameters (temperature and strain rate) [50] as seen in Figure 2.3. However, as a result of glide on primary planes, coplanar arrays of dislocations are formed and dislocation glide on different planes and their mutual interactions give rise to complex arrays of dislocations in a low SFE materials while tangles and cellular structure form in high SFE materials [24].

Stage III of work hardening (usually above a strain of 0.3) is identified by the drop in hardening rate and the flow curve becomes parabolic as multiplication of dislocations is concurrently counterbalanced by annihilation of opposite sign dislocations by dislocation glide and dislocation climb at elevated temperature and localised cross-slip [50]. Work hardening rate in this stage is a strong function of temperature, as shown in Figure 2.3. In stage III, the cell walls that comprise complex dislocation tangles have a tendency to collapse into thin, clean sub-boundaries. These boundaries form subgrains and usually have low angle misorientation of 1 – 4° during stage III and continue to develop sub-boundaries in stage IV. Stage IV could be reached at a very high strain (strains $\geq$ 1), deformation substructures progressively disintegrate to form microbands or shear bands in multiple orientations [24].

The discernible difference in flow behaviour and work hardening during hot deformation is delineated by the activation of climb and cross-slip mechanisms. The hardening rate is associated with recovery via glide, climb and cross-slip of dislocations and is determined by the SFE of materials, deformation temperature and strain rate. Nonetheless, the climb and cross-slip is hampered in low to medium SFE materials (austenitic stainless steels, Ni-Fe model alloys). Hence, the dislocation densities increase rapidly to a critical level during deformation leading to the initiation of DRX.
2.4 Softening mechanisms

Plastic deformation is used to shape the materials such as tubes, rods, bars, plates and sheets are produced by rolling, forging, extrusion etc. Work hardening during plastic deformation has a considerable effect on the mechanical energy consumed during shaping of the materials. Most of the deformation energy involved in plastic deformation of materials is lost by heat dissipation and the remnant energy is stored in the materials in the form of dislocations. Plastic deformation of materials increases the grain boundary length as pre-existing GBs are elongated during deformation. The internal substructure forms via the accumulation of dislocations [6]. Thus, the stored energy of plastic deformation can be represented by the sum of the energy from the increased area of interfaces and all of the dislocations with minor influence from any survived vacancies and interstitials.

The release of the stored energy is the driving force for softening via recovery and recrystallisation. During recovery, the stored energy decreases via dislocation annihilation and rearrangement of dislocations to lower energy configurations such as cells and subgrains. By contrast, recrystallisation is generally used to describe the replacement of deformed microstructure by new grains [6, 53, 54]. Static recovery dominates the softening phenomena during annealing at temperatures near the half of the melting point (i.e. 0.4-0.5 Tm), and the static recrystallisation (SRX) occurs at prolonged annealing, while the softening mechanisms prevail during elevated temperature (i.e. above 0.5 Tm) plastic deformation are referred to as dynamic recovery (DRV) and dynamic recrystallisation. Although the prime attention is paid to the softening phenomena at high temperature deformation: the DRV and dynamic recrystallisation, a brief description of static recovery, static recrystallisation is also outlined in the following sections.

2.4.1 Static recovery

The stored energy is reduced during recovery, by either of the two concurrent primary dislocation movement processes: annihilation of opposite sign dislocations and rearrangement of dislocations into lower energy configuration. Those two processes of dislocation motion are associated with the glide on same glide plane, climb and glide in different glide planes and cross-slip of screw component of the
dislocations. Static recovery (SRV) takes place during early stages of annealing that decreases the stored energy by creation of subgrains [6, 53-56]. There are several stages of SRV, as described in the Figure 2.4. The dislocations initially form tangles by dislocation interactions during deformation and then these tangles are rearranged into dislocation cells. Then, the interior of the cells are cleaned by annihilation of dislocations [57] followed by a subgrain formation having well-defined, narrow low angle boundaries (LAGBs). The mechanism of recovery depends on many parameters such as the SFE of the material, purity, strain, deformation and annealing temperature.

![Figure 2.4 The process of recovery of plastically deformed materials.](image)

2.4.2 Static recrystallisation

The deformed materials are softened during an annealing process where new recrystallised grains consume elongated grains through nucleation and growth [6]. From this definition, it is clear that SRX process consists of nucleation of a strain-free region whose boundary further moves into the strained matrix to form a strain-free material [58, 59]. At the initial stage of SRX, the nuclei are delineated by the LAGBs which gradually transform to high angle grain boundaries (HAGBs) [6, 54, 60]. SRX microstructure is characterised by a decrease in the number of work hardened grains and an increase in the fraction of recrystallised grains. This recrystallisation phenomenon refers to as discontinuous static recrystallisation, and is depicted in Figure 2.5a. On the other hand, recrystallised microstructure could develop homogeneously throughout entire deformed area in some high SFE materials, such as work hardened Al alloys comprising second phase particles [61]. In this case, new grains are formed via progressive increase of subgrain boundary misorientation in the whole microstructure accompanied by particle coarsening and the process is called in-situ or continuous static recrystallisation, which is shown in Figure 2.5b.
2.5 Dynamic softening phenomena

The softening processes during deformation at high temperature are termed DRV and dynamic recrystallisation. Depending on the SFE of materials, dynamic softening can be associated with two coexisting processes: DRV tends to decrease the stored energy through dislocation glide, climb and cross-slip while new grains originate from deformed matrix by discontinuous dynamic recrystallisation (dDRX) in medium to low SFE materials or by continuous dynamic recrystallisation (cDRX) in high SFE materials [25, 62-66]. The occurrence of dDRX or cDRX depends on the rate of recovery and the velocity of migrating GBs. The microstructure is evolved by nucleation and growth in dDRX process where the rate of recovery is slow, whereas geometrically necessary boundaries develop that gradually transformed into HAGBs during cDRX. The SFE of the Ni-30Fe model alloys is in the range of low to medium SFE, the primary softening phenomena are DRV and dDRX. Hereafter,
the discontinuous dynamic recrystallisation is abbreviated to DRX and used as such throughout the entire thesis unless otherwise stated.

2.5.1 Dynamic recovery

In high SFE materials, like aluminium, α-iron and ferritic steels, dislocations climb and cross-slip occurs readily [6]. Opposite sign dislocations on same glide plane annihilate each other and thermally activated climb occurs to annihilate dislocations from different glide planes. However, cross-slip of dislocations is observed to occur at relatively lower temperature in high SFE materials [6]. Therefore, rapid and extensive DRV is noticed in high SFE materials and presumably represents the only restoration mechanism. The flow curve of material undergoing DRV is typically characterised by an increase in flow stress to a plateau followed by a steady state flow stress as demonstrate in Figure 2.6.

![Figure 2.6 Representative stress-strain curves for Al-1% Mg deformed at 400 °C [47] showing dynamic recovery and the effect of strain rate on dynamic recovery.](image)

As dislocation density increases, the driving force for recovery also increases, hence the rate of DRV increased and rearrangement of dislocation leads to the microstructure consisting of LAGBs and subgrains similar to SRV shown in Figure 2.4.

Afterward, the rates of recovery and work hardening reach a dynamic equilibrium where the dislocation density remains constant and the steady state condition is reached. The development of substructure and growth of subgrains
during DRV was described by Sandström et al. [67]: (i) pair annihilation of dislocations in the subgrain interior, (ii) pair annihilation of dislocations in the cell boundaries, (iii) transfer of dislocations from the interior to the grain boundaries through the absorption at the boundaries, (iv) transfer of dislocations from the grain boundaries to the interior through the emission of dislocations. Pair annihilation of dislocation is more rapid than transfer processes at high temperature as long as the dislocation densities are reasonably high [67].

The phenomena of dislocation generation and recovery operate in a wide range of deformation conditions. Dislocation generation factor is dominant at low temperatures and higher strain rates while prominent DRV is favourable at higher temperatures and lower strain rates. A representative example of the effect of strain rate on DRV is shown in Figure 2.6 [68], where it is clearly seen that the flow stress is increased with an increase in strain rate. The increase in deformation temperature tends to increase the rate of DRV as dislocations and boundaries can move faster at higher temperature.

The microstructure development in an aluminium alloy during hot working from Musin et al. [69] is shown in Figure 2.7, where new grains appear as a result of continuous increase in misorientation of sub-boundaries and formation of HAGBs due to constant accumulation of dislocations [56].

Figure 2.7 Development of microstructure in cDRX of AA1421 subjected to hot equal channel angular extrusion of $\varepsilon = 12$ at 673 k [48].
2.5.2 Dynamic recrystallisation

Generally new DRX grains are nucleated at the pre-existing GBs and gradually consume the deformed microstructure. Since the material continues to deform, the dislocation density of new DRX grains increases, thus reducing the driving force for their further growth [70].

The typical features of DRX flow curve and microstructure development are: (i) stress peak on the flow curve followed by the steady state stress, (ii) a critical strain ($\varepsilon_c$) to initiate DRX [71], (iii) the size of dynamically recrystallised grains ($D_R$) remains constant when stress corresponds to steady state, (iv) nuclei of DRX primarily appear on pre-existing GBs and twin boundaries (TBs) though intragranular nuclei are observed at a low strain rate and at a large initial grain size [6]. Metadynamic recrystallisation (MDRX) occurs when the nuclei of DRX are growing during inter-pass holding [72]. The detailed characteristics of DRX flow curve, nucleation mechanism, microstructure and texture evolution and the factors influencing DRX are overviewed in the next sections.

2.5.2.1 Flow behaviour

A typical DRX flow curve comprises of initial WH followed by a stress peak and final attainment of the steady state condition, as shown in Figure 2.8. The flow curve can be divided into three regions; (i) WH plus DRV region where dislocation density increases rapidly by dislocation generation and multiplication of dislocations hence increases in flow stress, (ii) WH, DRV and DRX operate simultaneously after the critical strain ($\varepsilon_c$) of DRX is reached. After reaching the peak stress ($\sigma_p$), the softening mechanisms become dominant; and (iii) the steady state region where new DRX grains continuously replace the deformed grains [62, 63].
Figure 2.8 Typical flow curve of dynamic recovery and dynamic recrystallisation.

Although the presence of the peak on the flow curve is generally accepted as an indication of initiation of DRX, DRX starts before peak strain which is 0.6-0.8 times of the peak strain ($\varepsilon_p$) [29]. DRX usually begins at a critical strain prior to the peak and work hardening decreases up to a strain-independent steady state condition. At the steady state, there is a dynamic balance between work hardening and work softening.

Identification of the critical point of DRX is crucial to predict final microstructures and estimate load drops in rolling mill or during plastic deformation. It is difficult to accurately judge the critical point directly from stress-strain curve because of a smooth transition from the critical point to the peak in the flow curve. Moreover, some material groups, such as Nb microalloyed low carbon steel, 321 stainless steel and alloyed austenitic stainless steel have a broad stress peak on flow curves [6]. The critical point of DRX can be determined from the inflection point on the first derivatives (work hardening rate ($\theta$) vs true stress ($\sigma$) plot) of flow curve [71, 73], which is depicted in Figure 2.9.
The critical point is further elucidated by the distinct minimum in the second derivatives of flow curve (\(-d\theta/d\sigma\) vs. \(\sigma\) plot) as depicted in the top right inset of Figure 2.9. WH rate (\(\theta\)) is easily derived from the true stress and true strain values using \(\theta = d\sigma/d\varepsilon\) relationship.

2.5.2.2 Nucleation

There are two counter-acting forces during hot deformation; WH is liable for the accumulation of strain by increasing the dislocation density and DRV tends to reduce the stored energy of plastic deformation. These two opposing forces try to neutralise each other, but in materials with low to medium SFE, the dislocation density raises gradually by overcoming the recovery component. DRX initiates, if the rate of boundary migration of the potential nucleus is high in relation to the rate of re-accumulation of dislocations behind the boundary [74]. The distribution of dislocation density in DRX mechanism was proposed by Sandström et al. [75] and is depicted in Figure 2.10.
It is illustrated in Figure 2.10 that when the boundary at point A is moving from left to right with a velocity of $\dot{X}$, the dislocation density is going to decrease and it becomes nearly zero at the tip of migrating boundary where new DRX nucleus originates. The dislocation density at the front of boundary is the maximum ($\rho_m$). During continuous straining, dislocations accumulate at the back of DRX nucleus and their density increases to $\rho_x$ at a distance $x$ from the point A, finally reaching $\rho_m$ at a large distance. Generally, the growth of dynamically recrystallised grain depends on the distribution and density of dislocations [6].

A typical nucleation mechanism during DRX is illustrated in Figure 2.11[56], where polycrystalline copper is compressed at $\dot{\varepsilon} = 10^3 \text{s}^{-1}$ to (a) $\varepsilon = 1.3$ at 573 K and (b) $\varepsilon = 0.2$ at 723 K [64, 66]. It is obvious from Figure 2.11a that the dislocation pressure across the boundary serrates the original boundaries; which leads to the formation of sub-boundaries at a higher strains and/ or $\Sigma 3$ TBs (yellow lines) at a lower strains (Figure 2.11b). The $\Sigma 3$ boundary completion is readily observed at a higher deformation temperature as their mobility is higher at an elevated temperature.
Based on the experimental observations, a nucleation model was suggested and is depicted in Figure 2.11c [25, 64, 66, 76]. It comprises of the following steps; firstly, a dislocation density gradient is developed across grain boundaries accompanied by subgrain formation. Then serration of existing GBs followed by strain induced sub-boundary or twin boundary formation at the back of the bulged front. The former process is also accompanied by the rotation of the bulged region.

DRX nucleation is associated with operation of either of the two fundamental mechanisms [77]. (i) Strain-induced boundary migration (SIBM) was first introduced by Beck and Sperry [78] for a wide variety of metals. According to this mechanism, a dislocation-free region is created behind the migrating pre-existing...
bulged boundary and a new area enclosed by the bulged boundary maintains an analogous orientation relationship with the old grain. The recrystallisation by SIBM is restricted by the amount of deformation above which this mechanism is not in operation. In Al, this limiting deformation is approximately \( \varepsilon = 0.4 \) [78], and Bellier and Doherty [79] determined it to be \( \varepsilon = 0.2 \) during compression. Due to an orientation relationship between new grains and prior grains, the operation of this mechanism is expected to have a similar recrystallisation texture to the deformation texture. (ii) At higher deformation strain, a new nucleus is formed preferably and independently at grain boundaries, twin boundaries, and shear bands, at inclusions or at second phase particle by continuous evolution of sub-boundaries to new DRX grains.

The substructure development during DRX [65] is depicted in Figure 2.12 and can be described as: (a) DRX nucleus appears when the critical dislocation density \( \rho_c \) is achieved and the nucleus has a clean interior (dislocation density corresponds to that in annealed state \( \rho_o \)), (b) a growing DRX grain that continuously accumulates dislocations; it contains dislocation cells and tangles in its interior and (c) a critically work hardened grain after impingement having dense dislocation walls at a large distance from the recrystallisation front [56].

In the presence of large particles, some DRX nuclei originate from these particles, which is called particle stimulated nucleation (PSN) [22, 23]. The earlier prediction showed that PSN in DRX may occur at 0.5-1 \( \mu \)m size particles [22], but the mostly accepted particle size for PSN is greater than 1 \( \mu \)m and sometimes the particle exceeds 5 \( \mu \)m [6, 80-82]. In Figure 2.13 the recrystallised grains with a planar recrystallisation front are visible around large particles in a Ni alloy [83] after compression and annealing at 600 °C for 15 min. PSN also frequently generates a set of continuous recrystallised grains separated by twin boundaries [80].
Figure 2.12 Three types of dislocation density in DRX grains [56, 65]: (a) a DRX nucleus, (b) a growing DRX grain, and (c) a critically work-hardened DRX grain and corresponding TEM micrographs (d), (e) and (f). $\rho_c$ is the critical dislocation density for DRX nucleation and $\rho_0$ is the dislocation density at annealed stage.

Figure 2.13 Recrystallisation nuclei associated with silica particles in a Ni-0.03 wt. % Si alloy during rolling to $\varepsilon = 0.9$ and annealing at 600 °C for 15 min: (a) secondary electron image and (b) planar recrystallised front is observed in transmission electron micrographs [83].
2.5.2.3 Microstructure evolution

The DRX microstructure development by necklace layer formation initially suggested by Roberts et al. [84] and further developed by Sellars et al. [85] is shown in Figure 2.14. DRX microstructure evolves by the successive necklace layer formation above a critical strain where the initial layer of DRX grains is nucleated at parent boundaries. The necklace layers continuously form at the interface between deformed and recrystallised grains, when the nucleation sites at parent grain boundaries are saturated. Then, gradually the necklace layers consume the entire deformed volume.

Figure 2.14 Schematic of the microstructure evolution during DRX by necklace formation as suggested by Sellars et. al [85]: (a) no DRX grains exist below the critical strain ($\varepsilon_c$), (b) first necklace layer formed at pre-existing grain boundaries, (c) the formation of the subsequent necklace layer, (d) consumption of the deformed grains by successive necklace layers, (e) full DRX microstructure and (f) typical DRX flow curve.

Ponge and Gottstein [86] agreed with others [84, 85] on the creation of the initial necklace layer, but have a different view on the expansion of necklace layer at higher strain levels. They suggested that new DRX grains at higher strain could...
rather randomly nucleate from the continuously developed substructure within the deformed grains.

The volume fraction of dynamically recrystallised material \( X(t) \) can be derived from the Eq. (2.4).

\[
X(t) = 1 - \exp(-2S_p Gt)
\]  

(2.4)

Here, \( S_p \) is the active grain boundary area per unit volume, \( G \) is the radial growth velocity and \( t \) is the time. This expression is no longer valid when recrystallisation is well under way and the existing boundary is covered by newly formed nuclei. Roberts and Ahlblom [63, 74] found that the grain boundary area per unit volume increases with the time because continuous deformation leads to formation of a large number of new GBs. In this situation, the grain boundary area fraction is described as follows:

\[
S_p = S_0 (1 - \exp(\varphi t))
\]  

(2.5)

Where, \( \varphi \) is a constant, \( S_0 \) is the initial grain boundary area per unit volume which is inversely proportional to the initial grain size, \( S_p \) is the total available specific grain boundary area. In this way Eq. (2.4) could be replaced by the approximate relation:

\[
X = 1 - \exp\left\{-\left(\frac{K}{D_0}\right)t^n\right\}
\]  

(2.6)

Where, \( n \approx 1.3 \) and \( D_0 \) is the initial grain size which influences the rate of recrystallisation. Finally, the fraction of dynamically recrystallised grains could also be obtained by an Avrami [87] type model which is described in detail in Chapter 3.

2.5.2.4 Texture development

The alteration of orientation of grains during softening may lead to a significant change in mechanical and physical properties. The crystallographic texture development after cold rolling of fcc materials in plane strain compression can be represented by the ideal orientations in Figure 2.15 [24]. The main texture components in cold rolling consist of two groups of orientations, \( \alpha \)-fibre and the \( \beta \)-fibre respectively. The \( \alpha \)-fibre is characterised by the orientations having \( \{011\} \) axis parallel to the rolling plane normal and which extends from Goss \((G, \{110\} \{001\})\) orientations to Brass \((B, \{110\} \{112\})\). On the other hand, the \( \beta \)-fibre can be described as the orientations generated at Brass going through S \((\{123\} \{634\})\) and
Goss and ending at Copper (Cu, \{112\} \{111\}). The ideal coordinates of these two main fibre components in Euler space are listed in Table 2.1.

Table 2.1 Coordinates of main α-fibre and β-fibre components in Euler space.

<table>
<thead>
<tr>
<th>Texture component</th>
<th>Euler angles</th>
<th>Miller indices</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\phi_1$</td>
<td>$\Phi$</td>
</tr>
<tr>
<td>Cube (C)</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Goss (G)</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>G/B</td>
<td>74</td>
<td>90</td>
</tr>
<tr>
<td>Brass (B)</td>
<td>55</td>
<td>90</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>90</td>
<td>35</td>
</tr>
<tr>
<td>Copper Twin (CuT)</td>
<td>90</td>
<td>74</td>
</tr>
<tr>
<td>S</td>
<td>59</td>
<td>37</td>
</tr>
</tbody>
</table>

The texture development in fcc materials during cold rolling and SRX is well understood and it strongly depends on the SFE [88-93]: copper (Cu) type texture is developed in high SFE materials that is described by the β-fibre that spreads from the Cu (\{112\} \{111\}) to the Brass (B, \{110\} \{112\}) through the S (\{123\} \{634\}) orientations [91]. On the other hand, Brass (B) type textures are evolved in low SFE materials and characterised by α-fibre that extends from the Goss (G, \{110\} (001))
to Brass orientations [89]. Wusatowska-Sarnek et al. [64] concluded from the investigation of copper in compression that a deformation texture near (101) direction developed during low temperature deformation (before the initiation of DRX), which weakens and randomises by the progress of DRX at higher temperatures. The hot deformation texture in Ni-30Fe model alloy deformed to 700-900 °C at 1 s\(^{-1}\) strain rate is similar to rolling/plane strain compression (PSC) texture in typical medium to high SFE fcc materials and the orientations are mainly composed of B, Cu, G and S components, accompanied by limited presence of C [15] which is illustrated in Figure 2.16a.

Figure 2.16 Crystallographic texture development in Ni-30Fe during PSC as a function of strains: (a) overall micro-texture at different temperature and (b) DRX grains at deformation temperature of 900 °C [15].

27
Additionally, the hot deformed texture in medium to high SFE fcc materials is characterised by texture components located along α-fibre to β-fibre through crystallographic slip during PSC [15, 94, 95]. However, a weak DRX texture exhibiting a stronger than others cube orientation is observed in Ni-30Fe model alloy after PSC to $\varepsilon = 1$ at 900 °C with 1 s$^{-1}$ strain rate [15] (Figure 2.16b). The weak DRX texture comprising a cube component was also corroborated by the investigation of Fe-30Ni model alloy deformed to 0.6 strain at 950 °C with a strain rate of 10 s$^{-1}$ [26]. Moreover, the weakening of texture with the progress of DRX during compression of copper was ascribed by Hasegawa et al. [96] to randomisation of texture due to high frequency of twinning. Recently, the observation in 304 austenitic stainless steel in PSC at 950 °C to 1100 °C showed a strong cube DRX texture formation by multiple twinning and limited grain rotation during deformation [97]. Nevertheless, Sakai et al. [56] stated that there is a significant lack of experimental evidence on DRX texture evolution.

2.5.2.5 Factors affecting dynamic recrystallisation

Deformation temperature, strain rate, chemical composition and presence of particles are the main components determining the DRX kinetics. The combined effect of temperature and strain rate on DRX is represented by the Zener-Hollomon parameter [98]. The Zener-Hollomon parameter is represented by Eq. (2.7)

$$Z = \dot{\varepsilon}\exp\left(\frac{Q}{RT}\right) = A_0(\sinh\alpha\sigma_p)^n$$

(2.7)

Where, $\dot{\varepsilon}$ is the strain rate, $\sigma_p$ is peak stress, $T$ is absolute temperature in K, $R$ is the universal gas constant (8.314 JK$^{-1}$mol$^{-1}$), $Q$ is the deformation activation energy and $A_0, \alpha, n$ are the constants. Medina et al. [99] summarised the activation energy data for various steels and concluded that the higher content of alloying elements increases the activation energy, except for C, which has the opposite effect. It has also been reported that Ti, Nb, Mo and Si significantly increase the deformation activation energy, whereas Mn and V have a moderate effect on the deformation activation energy. The flow curves of Ni-30Fe alloy and 0.15C steel as a function of strain rates and temperatures are shown in Figures 2.17 and 2.18 respectively.
The flow stress of Ni-30Fe alloy is higher than the flow stress of 0.15C steel. The increase in flow stress is attributed to the solid solution strengthening from the addition of 30 wt.% Fe to Ni [3]. Generally, the flow stress is increased with a decrease in deformation temperature or an increase in strain rate (i.e. high Z), while a decrease in flow stress is observed at a higher deformation temperature or at a lower strain rate (i.e. low Z). Most importantly, the shape of the typical DRX flow curve remarkably deviates from the ideal condition at high Z where WH dominates over DRX and DRV. There is no evidence of occurrence of DRX at high strain rate because the flow rate is limited by the DRV [100]. The critical strain for initiation of DRX also raises with the increase in strain rate [101] or deformation at a lower temperature. At higher strain rate, the accumulation of dislocations is rapid and there
is a quicker increase in dislocation density at grain boundaries. Moreover, at a high strain rate the deformation time is very low and the deformed grains may not be recrystallised within this short period of time. Thus, softening by DRX would not likely take place for material that is deformed at a high strain rate.

The driving force for DRX decreases with increase in deformation temperature, because the dislocation density generated at any given level of strain is lower [101]. Besides, below a certain temperature recrystallisation will stop due to inadequate driving force for SIBM or nucleation. This recrystallisation stop temperature is known as Tnr. The critical and peak stresses are readily achieved when the materials are deformed at a considerably high temperature and slow strain rate; the steady state condition of DRX is easily obtainable (see flow curves in Figures 2.17 and 2.18 for 1100°C or 0.07 s\(^{-1}\) strain rate).

Chemical composition of materials considerably alters the SFE, i.e. pure Ni has a SFE of 128 mJm\(^{-2}\) while Ni-30Fe has a SFE of 70 mJm\(^{-2}\) [6]. SFE of material is a critical factor in determining the softening phenomena prevailing during hot deformation or during annealing. It is already mentioned and well known fact that the high SFE materials soften by cDRX, whereas DRX is the main softening mechanism for low to medium SFE materials [25, 62-66]. An initial grain size also plays an important role in determining the kinetics of DRX. Although the size of DRX grains is independent of the initial grain size, the rate of recrystallisation is greatly accelerated by a smaller starting grain size [102]. It is suggested [103] that the rate of recrystallisation for fine grained materials is nearly 20 times faster than that of coarse grained materials at the constant temperature.

2.5.2.6 Influence of solute elements and precipitates on dynamic recrystallisation

The small amount of micro-alloying elements added to steel, such as Ti, Nb, V, significantly enhances the mechanical properties of steels [104]. The solute elements could be present as solute, precipitated particles and dispersed second phase particles in austenite microstructure leading to a significant increase in strength [105-107]. Therefore, these alloying elements, present in most commercial grade steels, nickel and titanium alloys have a remarkable impact on softening kinetics [6, 54, 108-110].
Nb is a powerful alloying element in microalloyed steels and high strength steels. The addition of Nb as an alloying element in steels acts in threefold ways [4, 108, 111-113]: firstly, Nb restricts the austenite grain growth during reheating, secondly, it suppresses the recrystallisation by solute drag or strain induced precipitation of NbC and finally, it provides precipitation hardening from NbC in the low temperature transformation product.

The solute Nb exerts a significant retarding force on DRX by solute drag effect [114]. The Nb and C atoms are present in the matrix as substitutional and interstitial elements, respectively. The variation in the atomic radius of alloying and matrix atoms creates a strain field around the solute atom in crystal structure (Figure 2.19).

![Figure 2.19 Strengthening by solid solution (a) substitutional atoms, (b) interstitials and (c) introduced new strain fields around substitutional and interstitial solute atoms.](image)

This lattice strain imposes inhomogenities in the microstructure during plastic deformation by restricting dislocation movement. The first quantitative model of solute drag was proposed by Lücke et al. [105] and stated that the interaction between solute elements and grain boundaries increases with the concentration of solute atoms. The model also suggested that the moving boundaries are held back by solute atoms at the boundary and the diffusivity of solute atoms behind the moving boundary control the speed of boundary migration. At high grain boundary velocities, the faster diffusing elements have the greater drag effect whereas at low velocity the opposite is true [106]. However, the solute dragging effect on the grain boundary mobility is greatly affected by the temperature: at lower temperature the solute drag controls the boundary mobility while above a transition temperature the drag effect becomes negligible [107]. The transition from solute-free to drag-
controlled conditions is described by Gall et al. [107] in a model and experiments on commercially pure Ni with various amounts of S as solute, which was deformed in compression at temperatures 650-1200 °C at a strain rate of $10^{-3}$ s$^{-1}$. It showed that at lower concentration of S the transition temperature is nearly 850 °C with a clear change from drag-controlled to solute-free conditions. On the other hand, at very high S content there is no distinct transition point; although the linear relationship shows slightly lower mobility than that for solute-free condition.

The alloying elements in fcc materials and alloyed steels usually precipitate as MX, where M is the alloying element and X is C, N, O, S or a combination of these elements. Nb atom has a strong affinity to form carbonitrides or carbides rather than oxides [115]. The formation of NbC or NbCN is given as follows [108]:

$$Nb + n(C,N) = Nb(C,N)n \tag{2.8}$$

Where, n is taken as 1 or less. The equilibrium constant K is calculated from Eq. (2.9):

$$K = \frac{a_{Nb(C,N)n}}{a_{Nb}a_{(C,N)}^n} \tag{2.9}$$

Where, for pure compound activity $a_{Nb(C,N)n} = 1$ and for dilute solution the activities $a_{Nb}$ and $a_{(C,N)}$ are proportional to their concentration in solution.

The equilibrium constant is associated with the free enthalpy of formation $\Delta G^0$ and the entropy $\Delta S^0$ and the enthalpy $\Delta H^0$ [108] as

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 = -RT\ln K \tag{2.10}$$

Therefore the solution for precipitates,

$$\ln a_{Nb}a_{(C,N)}^n = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \tag{2.11}$$

Replacing the activities with the concentration of Nb and C, the solubility product equation gives as:-

$$\log[Nb][C + 12N/14] = 2.26 - 6770/T \tag{2.12}$$

When the reheating temperature is greater than the absolute temperature in the above equation then all the Nb, C and N atoms are in the solution. At lower reheating temperature, the equilibrium amount of $[Nb][C + 12N/14]$ is given by Eq. (2.12) and the supersaturation ratio $K_s$ at absolute temperature of T is given by Dutta et al. [111] as:
This supersaturation ratio predicts the ‘driving force’ such as the free enthalpy change for the precipitation to occur. The value is relatively high for undeformed austenite, but it is much lower for the deformed materials due to readily available nucleation sites [111].

Particles, whether precipitated or dispersed (undissolved during reheating), interact with dislocations in two different ways; either the dislocations cut the particles or dislocations bow around the particles. When the particles are coherent, small and soft, dislocation may cut the particles [2]. However, depending on the strength of the particles and the rate of boundary/dislocation motion, smaller hard precipitates could also interact with boundaries and dislocations in three different ways as demonstrated in Figure 2.20: (i) the particles pin the boundaries or dislocations, (ii) the interface or dislocation is dragging the precipitates and (iii) the dislocation breaks away leaving a loop around the particles [116-118].

![Figure 2.20 The interaction of particles with boundary or dislocations [117].](image)

The grain boundaries intersect with the particles and increase the GB length by distortion around the particles. When the GBs meet with the particles, the boundaries tend to curve along the circumference of the particles. These moving
grain boundary leads to introduce Zener dragging effect as some part of the GB disappears during intersection (Figure 2.21).

![Diagram of grain boundary and particle interaction](image)

Figure 2.21 The Zener dragging force on grain boundary by a particle [119].

The surface tension of the bowing grain boundary is $\gamma$, and the force to separate the boundary from particle of radius $r$ is given as [120]:

$$F = \pi \gamma r$$  \hspace{1cm} (2.14)

The process of interaction of GBs and particles expends energy through creating new boundary area and restrict the motion of grain boundaries [119]. Another kind of pinning force arises during deformation when a dislocation interacts with a particle of radius $r$. The pinning force can be written as:

$$F = \tau b \lambda$$ \hspace{1cm} (2.15)

where, $\tau$ is applied stress, $b$ is the Burgers vector and $\lambda$ is the spacing between particles along the dislocation line. If the particles can sustain the force $F$ then the dislocations bow around the particles leaving the dislocation loops on by-passing. The shear stress or Orowan [116] stress required for this process is:

$$\tau_0 = \alpha \frac{Gb}{\lambda}$$ \hspace{1cm} (2.16)

Where, $\alpha$ is material constant and $G$ is the shear modulus. Inter-particle spacing of particles plays a crucial role in impeding the movement of dislocations and in multiplication of dislocations by loops formation. This mechanism of
dislocation bowing around the particles and bypassing them in such manner was first proposed by Orowan [116] and is illustrated in the Figure 2.22. The mechanism increases the strength of material and dislocation density during deformation of materials.

Figure 2.22 The Orowan mechanism of dislocation interaction with particles (a) bowing of a dislocation around particles and (b) dislocation passing through particles leaving a loop [116].

In the case of the Orowan mechanism, the loops are formed around the precipitates on the slip planes. However, if the precipitates are strong and large and they exert a force on the dislocation, which is able to initiate the cross-slip of the part of dislocation line on another plane, a different type of loops will be formed around the particles, so called prismatic loops (Figure 2.23 and Figure 2.24) [118]. One or two prismatic loops can be originated when screw components of dislocations move into the cross-slip plane and then recombine. The repetition of the prismatic loop formation process produces rows of prismatic loops or it could merge with further Orowan bowing mechanism [117, 121].
Figure 2.23 Dislocation motion past hard particle (a) without or (b-d) with cross-slip showing Orowan and prismatic loop formation [118].

Figure 2.24 Transmission electron micrographs of prismatic loops formed by cross-slip on outer Orowan loop in Cu-30% Zn-0.22% Al₂O₃, taken from [117].

Another type of precipitates known as strain-induced precipitates (SIPs) is formed during or immediately after the deformation. These SIPs preferentially form on dislocation walls, microbands, shear bands and grain boundaries [17, 19-21] as shown in Figure 2.25. The dynamic recovery and recrystallisation are both retarded due to the presence of precipitates at nodes, microbands, shear bands and boundaries [111]. At early stage of deformation, the small SIPs hinder the motion of dislocations.
and GBs and increase the flow stress and delay the process of recovery. However, the particle size at a later deformation stage or during inter-pass holding gradually increases and the restricting force of the boundaries and dislocations are reduced \[111\]. However, the force to retard recovery and recrystallisation by SIP is higher than the solute drag effect. The solute drag is taking place when there is solute in the matrix and the solute dragging effect controls by the diffusivity of solute and SIP acts at lower temperature than the solute drag.

Figure 2.25 Bright-field TEM micrographs of strain induced precipitation of NbC on microbands during deformation of Ni-30Fe alloys at 925 °C to \(\varepsilon = 0.2\) and holding for 30 s \[17\].

The influence of particles on DRX could be summarised as \[6\]; (i) the stored energy hence driving force of recrystallisation may increase as small particles impede dislocation motions, (ii) large particles stimulate nucleation due to large deformation heterogeneities \[54\] and (iii) closely spaced particles exert pinning force on the mobility of low angle and high angle boundaries, as well as dislocations known as Zener drag \[22\]. The initial two aspects tend to accelerate recrystallisation process while the last one retards recrystallisation kinetics. The transition from accelerated to retarded DRX is mainly based on the ratio of volume fraction of particles \((F_V)\) and radius of particles \((r)\) and the retardation occurs when \(F_V/r\) is larger than \(0.2 \mu m^{-1}\) \[122\]. If the value of \(F_V/r\) is less than this value, DRX process is promoted even though particle size is smaller than that for PSN \[6\]. The extent of softening by DRX depends on the size and inter-particle spacing of second phase
particles if all other parameters remain the same, which was first demonstrated by Doherty et al. [123]. The recrystallisation process is significantly retarded or even sometimes completely halted in the presence of closely spaced second phase particles [59, 109, 110, 124, 125], although large particles could enhance the process by PSN. However, the most effective size of particles in retarding DRX is reported to be 100 nm or less, which produces a substantial pinning force [126, 127].

2.6 Summary

This chapter outlined the present understanding on the effect of processing parameters on DRX, the microstructure and micro-texture evolution during DRX and the influence of particles on DRX. The critical points of this review are abridged as follows:

The effect of processing parameters on DRX is clearly understood and suggests that DRX readily take place at higher temperatures and lower strain rates. However, the influence of processing parameters on DRX and on deformation activation energy of Ni-30Fe-Nb-C model alloys need to elucidate further as these alloys have been developed recently to observe high temperature austenite microstructure behaviour. In addition, SRX was studied in Ni-30Fe-Nb-C alloys [16], but there is limited literature on the effect of Nb on hot deformation behaviour of Ni-30Fe [17, 18, 128].

Dynamic recrystallisation of low to medium SFE fcc materials is originated by nucleation at prior grain boundaries by bulging of pre-existing GBs through SIBM mechanism. It is widely accepted that the initial DRX grains decorate the old grain boundaries as a necklace layer. However, there is a dispute regarding the evolution of a necklace layer: whether the successive necklace layers are developed by repetitive nucleation of new layers at DRX grains/deformed matrix interface or by the combination of progressive necklace layer growth and the irregular formation of new DRX nuclei inside deformed grains at higher strain. Thus, experimental validation of these two hypotheses during deformation in DRX temperature region is required. The investigation of DRX progression with strain using the advanced characterisation techniques, such as electron backscattering diffraction and transmission electron microscopy, will be able in providing insight into operating mechanisms at later stages of DRX.
The study of texture development during hot deformation is incomplete and requires further investigation, as reports in the literature suggest either randomised DRX texture or the one with a strong cube component. Thus, EBSD on a set of samples strained to different levels could be used to track the evolution of micro-texture during DRX of a Ni-30Fe-Nb-C model alloy.

The DRX flow behaviour is a function of alloying elements concentration. The Nb addition in model alloys studied so far is restricted to 0.1 wt. % or less. The effect of high Nb content and the presence of relatively coarse NbC on deformation microstructure development and DRX in model alloys have not been addressed yet. It is well established that large size particles stimulate DRX nucleation process by preferentially nucleating DRX grains at incoherent interfaces. However, there is a lack of experimental evidence on how PSN evolves during DRX, what is the minimum particle size which can induce the DRX nucleation. The high resolution transmission electron microscopy could be useful to elucidate the detailed mechanism of PSN.
Chapter 3: Experimental and Analytical Procedures

The initial processing of materials, chemical composition and solubility curve of niobium carbide (NbC) are described in Section 3.1. Dynamic recrystallisation is the prime softening mechanism in the present study, thus determination of non-recrystallisation temperature (Tnr) is crucial to design hot deformation schedules and this is outlined in Section 3.3. The schematic of different TMP schedules carried out in Gleeble-3500 thermo-mechanical processing simulator in this project are described in Section 3.3.2. The details of microstructure characterisation techniques and analytical procedures used in current investigations are given in Sections 3.4 and 3.5, respectively.

3.1 Chemical composition and production of alloys

The chemical compositions (wt. %) of studied alloys with NbC dissolution temperature and solidus temperature are given in Table 3.1. The main elements are Ni and Fe with various amounts of Nb and C. The content of trace elements is listed underneath the Table 3.1. Hereafter, depending on the amount of Nb added, the alloy are designated as L (low), M (medium) and H (high), with designation L₀ used for the alloy without Nb. The dissolution temperatures of NbC particles and solidus temperatures were calculated by Zurob et al. [129].

Table 3.1 Chemical compositions in wt. % of model alloys. Dissolution and solidus temperature for the model alloys.

<table>
<thead>
<tr>
<th>Designation</th>
<th>Ni</th>
<th>Fe</th>
<th>Nb</th>
<th>C</th>
<th>Dissolution temperature (°C)</th>
<th>Solidus temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy L₀</td>
<td>70</td>
<td>29.9</td>
<td>-</td>
<td>0.10</td>
<td>-</td>
<td>1430</td>
</tr>
<tr>
<td>Alloy L</td>
<td>69.7</td>
<td>29.8</td>
<td>0.331</td>
<td>0.040</td>
<td>976</td>
<td>1424</td>
</tr>
<tr>
<td>Alloy M</td>
<td>69.2</td>
<td>29.7</td>
<td>0.851</td>
<td>0.114</td>
<td>1196</td>
<td>1391</td>
</tr>
<tr>
<td>Alloy H</td>
<td>68.8</td>
<td>29.7</td>
<td>1.420</td>
<td>0.187</td>
<td>1355</td>
<td>1355</td>
</tr>
</tbody>
</table>

Trace elements: Al-0.007, Cr-0.07, Mn-0.013, N₂-0.0003, P-0.0014, S-0.004, Si-0.024, Ti-0.008
The model alloys with nominal composition 70Ni-30Fe-(0.33-1.42)Nb-(0.04-0.187) C wt.% were cast at McMaster University, Canada [129] as a 45 kg ingot, reheated to 1250 °C for 24 hours to break down the as-cast structure and then hot rolled in 6 passes from 60 mm to 30 mm. Thereafter an intermediate solution treatment was undertaken at 1350 °C for 30 minutes followed by hot rolling in 4 passes to 15 mm thick plate and immediately water quenched. Both first and second hot rolling stages were completed at temperatures > 1050 °C. The 15 mm plates were then subjected to a final solution treatment at 1225 °C for ~20 hours.

Figure 3.1 The schematic diagram of the original rolling schedule to produce 15 mm thick plates of model alloys (except Alloy L0) after casting.

Then, the hot rolled plates were machined into 20 mm × 15 mm × 10 mm in rolling direction, normal direction and transverse direction respectively to perform further thermo-mechanical processing in Gleeble-3500 TMP simulator.

3.2 Solubility of NbC

The solubility of NbC and NbCN was extensively studied in different steels [108, 115, 130-132] and the data for these steels are used to calculate the solubility of NbC in present model alloys. The estimation of solubility temperature is based on the available data on Nb alloyed steels, as (i) the lattice parameter for fcc iron is ~0.357 nm [133] is quite similar to the lattice parameter (~0.356 nm) of Ni-30Fe system [44] and (ii) the single phase austenite matrix in model alloys is similar to
that in steels at a high temperature. The solubility of Nb in austenite for stoichiometric NbC is given by Eq. (3.1):

\[ \text{NbC} = [\text{Nb}]^Y + [\text{C}]^Y \]  

Equilibrium constant \( K_S \) is:

\[ K_S = \frac{a_{\text{NbC}}}{a_{\text{Nb}}a_{\text{C}}} \]  

Activity of pure compound is one; and for dilute solution activity of Nb and C is equivalent to the concentration of these elements. The solubility of NbC in model alloys is estimated using the equation proposed by DeArdo et al. [115]:

\[ \log([\text{Nb}][\text{C}]) = 3.36 - \frac{7167}{T} \]  

The concentration of \([\text{Nb}]\) and \([\text{C}]\) in Eq. (3.3) is in atomic percentage. The concentration of Nb and C are calculated from the Eq. (3.3) at different temperatures. It is generally assumed that NbC has the stoichiometric ratio of Nb and C and Eq. (3.3) strictly applies to the equilibrium condition [115]. However, the solubility of NbC calculated by Nordberg et al. [130] is very close to the one obtained by DeArdo et al. [115], despite being assumed in the former case the composition of niobium particle to be NbC\textsubscript{0.87}. The solubility temperature (ST) of NbC in model alloys as a function of concentration of Nb and C is shown in Figure 3.2. Nose temperature (NT) is calculated from the ST by the relationship, \( NT = ST - (150 \degree \text{C} - 160 \degree \text{C}) \) proposed by Wang et al. [104]. The weight percent of Nb and C in solution at different temperatures is obtained from correspondingly left and right Y-axes in Figure 3.2b. Table 3.2 summarises the solubility temperature calculated based on Eq. (3.3), nose temperature and the wt. % of Nb and C in solution for all three alloys at 1250 °C.

Table 3.2 Solubility temperature and nose temperature with wt. % of Nb and C in solution for alloy L, M and H at 1250 °C reheating.

<table>
<thead>
<tr>
<th></th>
<th>Solubility temperature °C</th>
<th>Nose temperature °C</th>
<th>Nb in solution at 1250 °C</th>
<th>C in solution at 1250 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy L</td>
<td>1200</td>
<td>1050</td>
<td>0.33</td>
<td>0.040</td>
</tr>
<tr>
<td>Alloy M</td>
<td>1500</td>
<td>1350</td>
<td>0.40</td>
<td>0.051</td>
</tr>
<tr>
<td>Alloy H</td>
<td>1725</td>
<td>1575</td>
<td>0.40</td>
<td>0.051</td>
</tr>
</tbody>
</table>
Figure 3.2 (a) The solubility curve used to calculate Nb in solution in Ni-30Fe alloys from DeArdo et al. [115]. (b) Amount of solute Nb and C in Ni-30Fe solid solution as function of temperature based on Wang et. al. [104] method.

The NbC dissolution curves with respect to mole fraction of NbC in studied alloys are shown in Figure 3.3.

Figure 3.3 The solubility curve of studied alloys calculated by Zurob et al. [129].

The dissolution temperature of NbC obtained from Thermo-Calc is remarkably lower than the measurement using thermodynamic calculation. DeArdo et al. [115] summarised the techniques used in determination of solubility product
and concluded that Eq. (3.3) is very reasonable to predict NbC solubility product. Thus, the data calculated using Eq. (3.3) is used for discussion of the results in this research.

3.3 Thermo-mechanical Processing in Gleeble-3500 Simulator

The hot deformation of samples was conducted in a Gleeble-3500 TMP simulator in a hydrawedge mode; the samples were subjected to plane strain compression. All the control panels, deformation chamber and the loading unit of Gleeble-3500 are shown in Figure 3.4. The Gleeble-3500 system is fully integrated with control of thermal and mechanical testing combined with an array of powerful processors and post processing software. The deformation and simulation can be performed from room temperature to 1700 °C in high level vacuum (10^{-4} Pa). The direct resistance heating of the specimen provides the precise control of the temperature and can heat the specimen as high as 10,000 °C/s. An accurate control of the cooling rate can also be achieved by Gleeble-3500 as high thermal conductivity grips hold the specimen. Additionally, the auxiliary quench tank in the system offers a high cooling rate above 10,000 °C/s at the sample surface. At the end of each test the result is loaded into Origin software and further analysis of recorded data can be carried out. However, the correctness of the tests can also be checked by instant preliminary interpretation of the data.
The following steps were used to prepare the samples for testing in the Gleeble-3500 TMP simulator. The specimens were polished to remove roughness in the surface. Hereafter, a K-type thermocouple was spot welded in the centre of each specimen to measure in-situ process temperature. Thin layers of tantalum sheet (0.1 mm) were attached on both sides of the sample to prevent C diffusion to the sample followed by another layer of graphite foil that provides lubrication. The schematic of a sample positioned between the anvils is shown in Figure 3.5, where rolling direction is perpendicular to the compression axis. All the TMP experiments were conducted with the same orientation of samples in Gleeble-3500 TMP simulator.

The selection of reheating temperature is an essential parameter to dissolve the precipitated NbC. The upper limit for reheating temperature is the liquidus temperature (1430 °C) in alloy L₀ and the liquidus temperature is decreased with increase in Nb and C contents and finally reaches 1355 °C in alloy H. On the other hand, the holding force of anvils on the sample also has a significant influence on the selection of solution treatment temperature. Solution treatment at 1300 °C is the most preferable to dissolve the maximum amount of NbC. But softening of the samples during prolonged holding at 1300 °C causes automatic deformation and the samples get sticked to the anvils making it almost impossible to accomplish the second deformation. Therefore, maximum reheating and roughing temperature was chosen to be 1250 °C in most of the experiments. However, high reheating temperature also leads to grain coarsening. Thus, in the case of alloys L₀ and L to
study the effect of processing parameters on DRX, reheating temperature of 1200 °C was selected in order to minimise the grain coarsening.

3.3.1 Determination of the non-recrystallisation temperature

The non-recrystallisation temperature (Tnr) has been measured to assist in the design of further TMP schedules for model alloys. Hot rolling is associated with two critical temperatures; during cooling, the first one is the non-recrystallisation temperature (Tnr) where SRX is not happening at a specified holding time, the second critical temperature is known as the recrystallisation stop temperature (Tr) at which recrystallisation is entirely stopped at a given hold time. It is frequently observed that Tr is about 75 °C below Tnr [134-136]. Dynamic or static recrystallisation could occur depending upon the applied strain when deformation was conducted above Tnr. Thus, for the studies of DRX it is essential to determine the Tnr in order to select the appropriate temperatures for deformation. Multiple hit tests were conducted using alloys L, M and H according to the schedule depicted in Figure 3.6. The specimens were initially heated to 1250 °C and held for 300 s, then cooled at 3 °Cs⁻¹ to 1150 °C where multiple deformations were started with a strain of 0.1 in each step at a rate of 0.1 s⁻¹. The deformation is continued at descend of 25 °C until the temperature is reached to 700 °C then the samples were water quenched.

![Figure 3.6](image-url) The schematic diagram for determination of non-recrystallisation temperature (Tnr).

A representative true stress-true strain curve of the multiple deformation tests for alloy H is shown in Figure 3.7a. Tnr can be indicated by the change of slope on a
mean flow stress vs temperature plot [137-139]. The values of Tnr of the model L, M and H alloys are listed in Table 3.3. As expected, the higher the Nb content in the alloy, the higher the Tnr.

Table 3.3 Determined recrystallisation stop temperature (Tnr) (°C) values for alloys L, M and H.

<table>
<thead>
<tr>
<th>Alloy chemistry</th>
<th>Alloy H</th>
<th>Alloy M</th>
<th>Alloy L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tnr</td>
<td>1040 ± 15</td>
<td>1000 ± 15</td>
<td>925 ± 15</td>
</tr>
</tbody>
</table>

Figure 3.7 The procedure for determination of Tnr: (a) an example of true stress vs true strain curve of multiple hit test in alloy H and calculated Tnr for (b) alloy H, (c) alloy M and (d) alloy L.
3.3.2 Thermo-mechanical processing schedules

TMP schedules were designed to conduct the tests in the Gleeble-3500 TMP simulator based on the reheating temperature and Tnr. The reheating and roughing temperatures in the study of the effect of Nb and processing parameters in alloys L and L₀ is lower than that in all other experiments as these alloys have the lowest amounts of Nb which results in fast grain growth (Figure 3.8). This TMP schedule includes: heating at a rate of 10 °Cs⁻¹ to 1200 °C, holding for 180 s to dissolve NbC precipitates, then cooling to 1175 °C (roughing deformation temperature) at 3 °Cs⁻¹ followed by deformation to ε = 0.3 at 0.1 s⁻¹. Hereafter, the samples were cooled at 3 °Cs⁻¹ to the final deformation temperature (925 °C, 1000 °C, 1075 °C or 1150 °C) where they were either water quenched or subjected to a plane strain compression of 1.2 at 0.01 s⁻¹, 0.1 s⁻¹ or 1 s⁻¹ strain rates followed by immediate water quenching.

Figure 3.8 Schematic diagram to study the hot deformation behaviour of alloys L and L₀ (presented in Chapter 4).

All other tests on alloys L, M and H were carried out using the same 1250 °C reheating and roughing temperature. Moreover, the later studies were focused on the effect of Nb content on precipitation behaviour and substructure development at elevated temperatures, as well as on the evaluation of softening kinetics and mechanisms.
Furthermore, insight into the evolution of dynamic recrystallisation microstructure and micro-texture in alloy L was studied using the schedule shown in Figure 3.9: the samples were heated to 1250 °C at a rate of 5 °Cs⁻¹, held for 300 s to dissolve any NbC precipitates followed by roughing deformation of ε = 0.3 at 0.1 s⁻¹ strain rate at the same temperature. Thereafter, the specimens were cooled to 1075 °C at 3 °Cs⁻¹ to perform the final deformation to strains of ε = 0, 0.23, 0.35, 0.68, 0.85 or 1.2 at 0.1 s⁻¹ strain rate followed by immediate water quenching.

Figure 3.9 Schematic diagram to investigate the evolution of hot deformation microstructure and micro-texture in alloy L (Ni-30Fe-0.331Nb-C) (presented in Chapter 5).
Finally, the study of influence of NbC particles size and distribution on dynamic and post dynamic recrystallisation of alloys L, M and H was conducted using the TMP schedule depicted in Figure 3.10: heating at a rate of 5 °Cs⁻¹ to 1250 °C, holding there for 300 s followed by a roughing deformation of \( \varepsilon = 0.3 \) at a strain rate of 0.1 s⁻¹. Then, the samples are cooled at a rate of 3 °Cs⁻¹ to a final deformation temperature of 1075°C and deformed to \( \varepsilon = 1.2 \) at a strain rate of 0.1 s⁻¹ and subsequently held for 0 s, 30 s and 300 s before water quenching. The samples were also quenched at different stages of processing, as shown in Figure 3.10, for microstructure observations.

![Figure 3.10 Schematic diagram to study the effect of NbC precipitates on dynamic and metadynamic recrystallisation (presented in Chapter 6).](image)

### 3.4 Microstructure characterisation

#### 3.4.1 Sample preparation

There is a variation of strains through the thickness of the Gleeble samples due to friction and flow of material between the sample and anvils. In a plane strain compression test, the deformation is nearly zero at contact point of anvils and specimen and gradually increases to a maximum load at the centre of the sample. Thus, extreme care was taken to choose the middle section of the deformed samples.
for microstructure characterisation using all selected techniques. The schematic of one Gleeble sample with conventional PSC coordinates (black coordinate) and conventional rolling coordinates (red coordinate) is presented in Figure 3.11. All analysis using optical microscopy and scanning electron microscopy (SEM) including EBSD were carried out on CD-ND (compression direction-normal direction) plane, whereas CD-TD (compression direction-transverse direction) plane was used in TEM examination according to PSC coordinate convention.

Figure 3.11 The cutting direction for optical and SEM microscopy of Gleeble samples and planes of specimen with respect to compression axis. The black coordinates represent PSC convention and the coordinates in red represent rolling convention.

3.4.2 Optical microscopy and scanning electron microscopy

The deformed specimens were cut into two in the middle of their long direction as shown in Figure 3.11 using Struers Accutom-50. Half of the specimen was hot mounted in Struers CitoPress-20 using 25 ml of 100% polyfast resin at 180 °C with a pressure of 24.8 MPa. Polyfast resin was used to ensure better conductivity in SEM. Mounted specimens were polished in Struers, Tegrapol-21 automatic polisher following the steps listed in Table 3.4. The polished samples were etched with a solution of FeCl₃ (5%), HCl (25%) and H₂O (70%) for 10-15 s.
Table 3.4 Sample preparation steps in Tegrapol-21.

<table>
<thead>
<tr>
<th>Step</th>
<th>Plane Grinding (PG)</th>
<th>Fine Grinding (FG)</th>
<th>Diamond paste (DP1)</th>
<th>DP 2</th>
<th>Oxide polishing (OP)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface</td>
<td>SiC-500</td>
<td>MD-Largo</td>
<td>MD-DAC</td>
<td>MD-Plus</td>
<td>MD-Chem</td>
</tr>
<tr>
<td>Suspension</td>
<td>15 µm</td>
<td>3 µm</td>
<td>1 µm</td>
<td>OP-S</td>
<td></td>
</tr>
<tr>
<td>Lubricant</td>
<td>Water</td>
<td>Red</td>
<td>Green</td>
<td>Green</td>
<td></td>
</tr>
<tr>
<td>rpm</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
</tr>
<tr>
<td>Force (N)</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>Time (s)</td>
<td>2</td>
<td>5</td>
<td>3</td>
<td>5</td>
<td>10</td>
</tr>
</tbody>
</table>

Optical microscopy was carried out on a Leica DMR microscope equipped with Leica Grain Expert software. Average grain size was measured by Grain Expert software considering line intercepts method according to ASTM E112 standard to find the equivalent circle diameter and the data were exported to Microsoft Excel software for further analysis. The average grain size and recrystallised fraction (based on optical microscopy image) were determined by analysing a minimum of 6 optical micrographs in Leica Grain Expert software.

SEM was carried out on a JEOL JSM-7001F field emission gun scanning electron microscope (FEGSEM) equipped with a Nordlys-II(S) EBSD detector and an 80 mm² X-Max energy dispersive spectroscopy (EDS) detector which interface with the Oxford Instruments AZtec 2.0 Oxford software suite. The chamber of the FEGSEM console is connected with a turbo molecular pump to quickly reach high vacuum level ($10^{-4}$ pa). The acceleration voltage is varied from 20 kV to 5 kV based on the sample chemistry and types of analysis. The secondary electron (SE) images were acquired to identify the morphology and density of particles at 5 kV and at a working distance of 15 mm to identify Nb-rich particles. In SE imaging the detector collects the secondary electrons emitted from atoms that stimulated by electron beams. Nearly 300-700 particles in each TMP conditions were manually measured from high resolution SE images to determine an average particle size and the distribution of these particles. The chemistry of the particles were examined by the EDS where characteristic X-ray peaks were produced by incoming electrons.
impingement. At least, 20-25 particles in each TMP condition from different SE images were studied to define the chemistry of particles using AZtech EDS software.

3.4.3 Electron backscattering diffraction

In EBSD studies conventional rolling coordinates are used as RD (rolling direction)-ND (Normal direction)-TD (Transverse direction) to represent the texture and microstructure. As plane strain compression simulates rolling, the use of rolling system of coordinates is convenient to discuss grains orientation and micro-texture. The RD-ND planes of specimens for EBSD were mechanically polished using 1200 and 2000 SiC grinding papers. Thereafter, the samples were electropolished on a Struers Lectropol-5 using an electrolyte of 73% methanol + 10% butoxyethanol + 9% distilled water + 8% perchloric acid at 17 V, ~0.52–0.56 mA, 17 °C for 180 s. EBSD was conducted at 15 kV, ~5 nA and 15 mm working distance. A step size of 1 μm or 0.5 μm was used to acquire maps for all deformation conditions which covered an area around 2.4 × 1.2 mm² to 1.2 × 0.6 mm² respectively. Additional maps with a step size of 10 μm were acquired at ε = 0, 0.23 and 0.35 to obtain statistically representative texture information. Post-processing of EBSD maps was done using HKL-Channel 5 software. Wild orientation spikes from maps were cleaned by filling in zero solution via extrapolation up to four neighbours.

To remove artifacts from the maps, a minimum spatial resolution of three times the step size was maintained constantly. In all EBSD maps, subgrain structures are defined by misorientations (θ) ≥ 2° to reconstruct the maps. Consequently, LAGBs are defined as 2° ≤ θ < 15° misorientations whereas the total high-angle grain boundaries (THAGBs) comprise HAGBs (15° ≤ θ ≤ 57.5°) and TBs. The first order annealing TBs are defined as Σ3 = 60° (111) while second order twins are defined by Σ9 = 38.9° (101). The maximum tolerance of the misorientation angle (Δθ) from the exact axis-angle relationship was identified by the Palumbo–Aust criterion (i.e. Δθ ≤ 15° Σ−5/6) [140] yielding a tolerance limit of 6° for Σ3 and 2.4° for Σ9, respectively. For analysis of micro-texture, the orientation distribution functions (ODFs) of φ2 = 0°, 45° and 65° sections were depicted using Bunge’s notation after exporting EBSD data to JTex [141].

Internal misorientation within grains/subgrains is an indication of stored energy, and is described by either: (i) the grain average misorientation (GAM) which
is defined by the average misorientation between adjacent pixels in a grain/subgrain [142], or (ii) the grain orientation spread (GOS), comprising long range variations measured by average misorientation of all pixel pairs in a grain/subgrain [142]. GOS criterion is used in present analysis to distinguish recrystallised and unrecrystallised fractions of EBSD maps. Grains having GOS > 2° are ascribed as unrecrystallised while grains with GOS < 2° are considered recrystallised [142, 143].

For the whole maps and for two distinct subsets of recrystallised and unrecrystallised grains, the intragranular local misorientation between each pixel and its surrounding neighbourhood (in a 3 × 3 matrix) was calculated and the mean value was assigned to that pixel [144]. Misorientations over 2° were omitted to repudiate the misorientations related to subgrain boundaries. The relative frequency of the intragranular local misorientation distribution was fitted to a log-normal (L-N) probability distribution function as:

\[ f(\theta_{\text{local}}) = \frac{1}{w\theta_{\text{local}}\sqrt{2\pi}} \exp\left(-\frac{(\ln\theta_{\text{local}})^2}{2w^2}\right) \] (3.4)

Where, w and \( \theta_c \) are the width and median of the L-N distribution of \( \theta_{\text{local}} \). The associated mean (\( \theta_m \)) and standard deviation (\( \mu_\theta \)) are determined as follows:

\[ \theta_m = \theta_c \exp\left(\frac{w^2}{2}\right) \] (3.5)

\[ \mu_\theta = \theta_c \exp\left(\frac{w^2}{2}\right) \sqrt{\exp(w^2 - 1)} \] (3.6)

3.4.4 TEM

For TEM, thin slices of 0.25 mm - 0.3 mm were cut from the middle of CD-TD plane using Struers Accutom-50 at a feed rate of 0.02 mms\(^{-1}\). To obtain TEM thin foils, the slices were carefully polished to ~70 µm with 2000 and 4000 SiC papers followed by twinjet electropolishing in a Tenupol-5 at -35 °C using solution containing 10% perchloric acid in methanol at a voltage of 20 V. TEM characterisation was carried out in a probe-corrected JEOL ARM200F operating at 200 kV, with a cold field emission gun. Inner and outer collection angles were 50 and 180 mrad respectively for obtaining high-angle annular dark field (HAADF)
images, while 11 mrad collection angles was used to acquire bright field (BF) images. Both images were obtained with a dwell time of 38 μs, and a probe current of 35 pA. The thickness of the samples was calculated using electron energy loss spectrum (EELS) acquired by energy filtered TEM (EFTEM) images [145]. The sample thickness is expressed as follows:

\[ t = \lambda \ln \frac{I_t}{I_0} \]  

(3.7)

Where, \( t \) is the thickness of the specimen, \( \lambda \) is the average mean free path for these low-energy losses, \( I_0 \) is integrated intensity at zero loss peak and \( I_t \) is the total spectrum integral including \( I_0 \).

It is a challenge to precisely characterise strain induced precipitates at dislocations in conventional TEM bright-field as dislocation strain fields tend to obscure the precipitate images [17]. Generally moiré fringe imaging was used to investigate SIPs, particularly very fine precipitates on dislocation walls, microbands or HAGBs [17, 145, 146]. Moiré fringe is defined as mismatch between interfaces in diffraction of crystal lattices that might have distinct spacing and orientation from two overlaying crystals [17, 145]. Fine particles located in the matrix and having a distinct orientation relationship readily construct moiré fringe. Therefore, during a two-beam imaging condition relating to the Bragg diffraction on a certain crystal lattice plane is set for matrix, a spontaneous diffraction parallel to lattice plane arises from contiguous particles. Translational moiré pattern with fringes parallel to these diffracting planes has been formed during interference of these two diffracting lattice planes with different spacing [17, 145]. High resolution lattice imaging technique [145] was used to study the type of interfaces between NbC particles and the matrix. In this technique, a periodic fringe image represents a difference in contrast that arises due to differences in the phase of the electron waves scattered through a thin specimen allowing elucidation of lattice defects.

### 3.5 Calculation of dynamic recrystallisation fraction

All true strain-true stress curves obtained during Gleeble simulations were cleaned and fitted using a polynomial function in Table curve software and Qtiplot software. The critical strains which indicate the initiation of DRX were determined from the first derivaties of the flow curves and the apperance of critical point was
confirmed by plotting second derivatives vs true stress curve [73, 147] as shown in Figure 2.9. The parameters critical (εc) and peak (εp) strains were obtained from the first and second derivatives of the flow curve and the maximum softening (εm) was found by Stewart et al. [87] method, which is depicted in Figure 3.12.

![Figure 3.12 Determination of maximum softening (εm) strain from work hardening rate vs strain curve [87].](image)

These determined values were used in calculating Zener-Hollomon parameters, deformation activation energy and the dynamically recrystallised fraction. The deformation activation energy and Zener-Hollomon parameters are calculated using the method proposed by Sellars et al. [148].

\[
Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = A_0 (\sinh \sigma_p)^n
\]  

(3.8)

where Z is Zener-Hollomon parameters, \( \dot{\varepsilon} \) is the strain rate, \( \sigma_p \) is peak stress, T is absolute temperature in K, R is the universal gas constant (8.314 JK\(^{-1}\)mol\(^{-1}\)), Q is the deformation activation energy and \( A_0, \alpha, n \) are the constants which are independent of temperature. The value of n was determined by a natural logarithmic plot of \( \sinh(\alpha\sigma_p) \) vs strain rate, then Zener-Hollomon parameters and deformation activation energy were determined.

Finally, the fraction recrystallised during DRX was estimated based on an Avrami type model [87] using DRX parameters in the following equation:
\[ X_{\text{DRX}} = 1 - \exp \left[ -0.693 \left( \frac{\varepsilon - \varepsilon_c}{\varepsilon_m - \varepsilon_c} \right)^2 \right] \]  

(3.9)

Here, \( X_{\text{DRX}} \) is the fraction dynamically recrystallised, \( \varepsilon \) is the applied true strain, \( \varepsilon_c \) is the critical strain for initiation of DRX and \( \varepsilon_m \) is the strain for the maximum softening. Additionally, the dynamically recrystallised fractions were derived from optical microstructure using the morphological differences of deformed and recrystallised grains and compared with the calculated ones.
Chapter 4: Hot Deformation Behaviour of Ni-30Fe-C and Ni-30Fe-Nb-C Model Alloys

This Chapter includes the experimental results and discussion on the effect of processing parameters (temperature and strain rate) and the addition of Nb on the dynamic recrystallisation and on the deformation activation energy of alloys L and \( \text{L}_0 \). The schematic of thermo-mechanical processing used in this investigation is given in Figure 3.8 and described in Section 3.3.2. Critical and peak strains and stresses were determined in the temperature range of 925 °C-1150 °C at strain rates of 0.01 s\(^{-1}\), 0.1 s\(^{-1}\) and 1 s\(^{-1}\), as discussed in Section 4.1.1. Work hardening and softening behaviours are addressed in Section 4.1.3. In addition, optical metallography is used for microstructure observation and calculation of grain size distribution. The Zener-Hollomon parameter and the deformation activation energy values are presented in Section 4.1.4. Finally, the volume fraction of dynamically recrystallised grains was determined using an Avrami type model and presented in Sections 4.1.5 and 4.2.5. The effect of alloying elements on the deformation activation energy is discussed.

4.1 Results

4.1.1 Flow curve analysis

Figure 4.1 shows the flow curves of alloys with (alloy L) and without Nb (alloy \( \text{L}_0 \)) addition at different deformation temperatures and three strain rates. In general, the flow curves in both model alloys at strain rate of 0.1 and 0.01 s\(^{-1}\) deformed in the temperature range of 1075 – 1150 °C indicate the typical DRX flow curve with a distinct stress peak followed by steady state flow stress. The stress peak, however, became broader and an increase in flow stress has been observed (Figures 4.1 a, b) with a decrease in the deformation temperature and an increase in the strain rate and/or the addition of Nb. The curves above 1000 °C temperature exhibited a single peak flow curve and reached the steady state condition, while steady state stress was not reached in the samples that were deformed at 1000 °C at 1 s\(^{-1}\) and 0.1 s\(^{-1}\) strain rates for both alloys.
Figure 4.1 Flow curves of model alloys (a, c, e) alloy L and (b, d, f) alloy L_0 after a plane strain compression with ε = 1.2 at different deformation temperatures and strain rate of (a, b) 1 s^{-1} (c, d) 0.1 s^{-1} and (e, f) 0.01 s^{-1}.
There is a clear rising trend of flow stress of both alloys with an increase in strain rate or a decrease in deformation temperature. It is also evident that the steady state conditions are more readily achieved for the L₀ alloy compared to the L alloy. The work hardening and softening are different for each flow curve and will be discussed later. Nearly continuous work hardening flow curves (Figures 4.1 a, c, e) were seen at a deformation temperature of 925 °C at 1 s⁻¹, 0.1 s⁻¹ and 0.01 s⁻¹ strain rates in alloy L, which is especially noticeable at higher strain rates by the continuous increase of stress with strain.

Figure 4.2 shows an example of the determination of the critical stress and strain from flow curves at 1000 °C and for all three strain rates of alloy L. The clear inflection point in the work hardening curve indicates the critical point and the distinct minimum in second derivatives plot confirms this point. Similarly, the above parameters were determined for all studied conditions and are summarised in Table 4.1.

![Figure 4.2](image_url)

Figure 4.2 Determination of the critical point (a) first derivatives and (b) second derivatives of stress strain curves obtained from plane strain compression of 1.2 at a deformation temperature of 1000 °C for L alloy.
Table 4.1 Measured critical stress, strain and peak stress, strain for alloy L and alloy L₀ for different strain rates (s⁻¹) and temperatures.

<table>
<thead>
<tr>
<th></th>
<th>Strain rate</th>
<th>925 °C</th>
<th>1000 °C</th>
<th>1075 °C</th>
<th>1150 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>L</td>
<td>L₀</td>
<td>L</td>
<td>L₀</td>
</tr>
<tr>
<td>Critical strain</td>
<td>1</td>
<td>0.46</td>
<td>0.27</td>
<td>0.21</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>0.36</td>
<td>0.22</td>
<td>0.19</td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>0.21</td>
<td>0.16</td>
<td>0.13</td>
<td>0.11</td>
</tr>
<tr>
<td>Critical stress (MPa)</td>
<td>1</td>
<td>278</td>
<td>224</td>
<td>193</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>221</td>
<td>148</td>
<td>149</td>
<td>105</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>155</td>
<td>107</td>
<td>98</td>
<td>76</td>
</tr>
<tr>
<td>Peak strain</td>
<td>1</td>
<td>0.80</td>
<td>0.56</td>
<td>0.67</td>
<td>0.35</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>0.69</td>
<td>0.44</td>
<td>0.45</td>
<td>0.28</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>0.55</td>
<td>0.36</td>
<td>0.29</td>
<td>0.25</td>
</tr>
<tr>
<td>Peak stress (MPa)</td>
<td>1</td>
<td>297</td>
<td>233</td>
<td>206</td>
<td>167</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>248</td>
<td>162</td>
<td>160</td>
<td>115</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>163</td>
<td>113</td>
<td>111</td>
<td>81</td>
</tr>
</tbody>
</table>

Figure 4.3 represents the dependence of peak strain and peak stress on strain rate and temperature. It is evident from Table 4.1 and Figure 4.3 that critical and peak stresses and strains decreased with an increase in the deformation temperature or a decrease in strain rate, but the rate of change was different. A nearly linear relationship was obtained for the temperature versus peak strain/peak stress (Figures 4.3a, c). By contrast, strain rate vs peak strain/peak stress (Figures 4.3b, d) shows an initial steep rise in peak stress/strain values and then a steady rise after reaching a transition strain rate.
Effect of (a, c) temperature on peak stress and strain for a deformation strain rate of 0.01s⁻¹ and (b, d) strain rate on peak stress and strain for a deformation temperature of 1075 °C for (a, b) alloy L and (c, d) alloy L₀.

4.1.2 Optical microscopy and scanning electron microscopy

The roughing flow curve and some representative optical microstructures from 1150 °C and 1000 °C before the final deformation are shown in Figure 4.4 for both alloys. The flow curve of alloy L displays almost continuous work hardening whereas the curve for alloy L₀ shows the peak and then starts to decline. The grain size after solution treatment at 1200 °C of the alloy L is 174 ± 80 µm compared to 130 ± 83 µm in the alloy L₀. The difference in the solution treated grain size is associated with the initial production of alloys: the L alloy was hot rolled while the L₀ alloy was cold rolled and annealed. This difference in the initial grain size also affects the DRX process during deformation as it was reported by Sakai et al. [63] that finer starting microstructure accelerates DRX process. The average grain size after roughing and before final deformation of alloy L is 100 ± 40 µm, 114 ± 45 µm, 135 ± 38 µm and 145 ± 53 µm at 1150 °C, 1075 °C, 1000 °C and 925 °C, respectively (Figures 4.4 d, e) while the corresponding average grain size of alloy L₀ is 60 ± 45 µm, 66 ± 37 µm, 72 ± 43 µm at 1150 °C, 1075 °C and 1000 °C, respectively (Figures 4.4 b, c).
Figure 4. Representative optical microstructures of alloys (b, c) L₀ and (d, e) L after roughing at 1175 °C with 0.3 strain and quenched before deformation temperature of (b, d) 1150 °C and (c, e) 1000 °C.

Figure 4.5 depicts the microstructures of alloy L after final deformation. The average recrystallised grain sizes for both alloys are listed in Table 4.2 and the distribution of DRX grain size at a different deformation condition is shown in Figure 4.6. The microstructures at 925 °C at all strain rates (Figures 4.5 d, h, l)
consisted primarily of elongated grains with very few bulging regions and a small number of DRX grains at the boundaries of deformed grains. At 1000 °C, only for 1 s\(^{-1}\) strain rate (Figure 4.5c) the microstructure shows mainly equiaxed grains along with annealing twins and a small fraction of deformed grains indicating incomplete recrystallisation. All other microstructures (Figures 4.5 a, b, e, f, g, i, j, k) were fully recrystallised with some grains containing annealing twins. Similar microstructural development has been noticed for alloy L\(_0\) at a deformation temperature range of 1000-1150 °C. It can be seen from Figure 4.5 that with an increase in deformation temperature and/or a decrease in strain rate, the recrystallisation proceeds to completion. As expected, however, the DRX stops below a certain temperature. As can be seen from Figures 4.5 d, h and l, only partial recrystallisation took place at 925 °C.

![Figure 4.5 Microstructures of L alloy after ε=1.2 at a strain rate of (a-d) 1 s\(^{-1}\), (e-h) 0.1 s\(^{-1}\), (i-l) 0.01 s\(^{-1}\) and deformation temperature of (a, e, i) 1150 °C, (b, f, g) 1075 °C, (c, g, k) 1000 °C and (d, h, l) 925 °C.](image-url)
Table 4.2 Average recrystallised grain size (µm) after final deformation of alloys L and L₀.

<table>
<thead>
<tr>
<th>Deformation temperature</th>
<th>Alloys</th>
<th>1 s⁻¹</th>
<th>0.1 s⁻¹</th>
<th>0.01 s⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Alloy L</td>
<td>31.5 ± 23</td>
<td>40.1 ± 24</td>
<td>52.3 ± 40</td>
</tr>
<tr>
<td></td>
<td>Alloy L₀</td>
<td>35.3 ± 22</td>
<td>42.2 ± 26</td>
<td>56.8 ± 43</td>
</tr>
<tr>
<td>1150 °C</td>
<td>Alloy L</td>
<td>19.4 ± 9</td>
<td>21.5 ± 9</td>
<td>28.6 ± 22</td>
</tr>
<tr>
<td></td>
<td>Alloy L₀</td>
<td>23.2 ± 16</td>
<td>26.1 ± 14</td>
<td>32.3 ± 19</td>
</tr>
<tr>
<td>1075 °C</td>
<td>Alloy L</td>
<td>14.2 ± 8</td>
<td>17.3 ± 9</td>
<td>19.2 ± 9</td>
</tr>
<tr>
<td></td>
<td>Alloy L₀</td>
<td>19.1 ± 11</td>
<td>21.1 ± 11</td>
<td>25.1 ± 14</td>
</tr>
<tr>
<td>1000 °C</td>
<td>Alloy L</td>
<td>19.4 ± 9</td>
<td>21.5 ± 9</td>
<td>28.6 ± 22</td>
</tr>
</tbody>
</table>

Figure 4.6 Grain size distribution after final deformation; (a, c) for different temperature at 0.01 s⁻¹ strain rate and (b, d) for different strain rate at a deformation temperature of 1150 °C of alloys (a, b) L and (c, d) L₀.
Figure 4.6 illustrates the grain size distributions of alloys L and L₀ at 0.01 s⁻¹ strain rate as a function of deformation temperature and at 1150 °C as a function of deformation strain rate. At 1000 °C, in alloy L the peak of recrystallised grain sizes is in the range of <30 µm while at 1150 °C a significant fraction of coarse grains >80 µm is also present resulting in a bimodal distribution (Figure 4.6 a, b). The distribution of grain size changes in a similar manner with decreasing strain rate. This relationship is also true for the L₀ alloy (Figures 4.6 c, d). The recrystallised grain size increased rapidly with an increase in deformation temperature and a decrease in strain rate.

4.1.3 Characterisation of work hardening and softening behaviour

The work hardening rate during hot deformation is determined by the overall contributions of the hardening and softening processes taking place. Figure 4.7 represents the work hardening rate of both alloys at different temperatures at strain rates of 0.01 s⁻¹ and 1 s⁻¹. The first inflection point on the curve is the critical strain which is higher for lower deformation temperatures. The most interesting point in Figure 4.7 for alloy L is the region of the negative work hardening rate which is reached for samples deformed at all temperatures at 0.01 s⁻¹ strain rate (Figures 4.7 a, c) while at 1 s⁻¹ rate, a negative work hardening rate was observed at 1150 °C and 1075 °C and barely at 1000 °C, but it remains positive at 925 °C. In L₀ alloy, instead, at all three deformation temperatures (Figure 4.7d) the work hardening rate exhibits negative work hardening. However, the work hardening curve quickly crosses the zero hardening with an increase in temperature and a drop in deformation strain rate. The rate of work hardening is a strong function of both deformation temperature and strain rate as the work hardening curve shifts with a change in processing parameters as seen in Figure 4.7.
Figure 4.7 Work hardening rate curves in alloys (a, b) L and (c, d) L₀ at deformation temperature of 925-1150 °C and strain rate of (a, c) 0.01 s⁻¹ and (b, d) 1 s⁻¹.

4.1.4 Interrupted strain observations

The evolution of DRX in alloy L as a function of strain was studied by interrupted quenching along the flow curve at 1075 °C with 0.1 s⁻¹ strain rate (Figure 4.8a). It can be seen from Figure 4.8b, at ε = 0.23 only a few DRX grains are nucleated at grain boundaries and the boundaries (Figure 4.8b) of deformed grains become serrated. Nearly 28% DRX grains (Figure 4.8c) were found at ε = 0.35, whereas nearly 69% have been dynamically recrystallised at ε = 0.68. DRX is nearly completed at ε = 0.85 reaching over 90% and producing a microstructure of predominantly equiaxed grains (Figure 4.8d). The creation of equiaxed grains is seen
to start from the pre-existing grain boundaries at $\varepsilon = 0.23$. The fraction dynamically recrystallised in the microstructure steadily increased with strain.

![Flow curve of alloy L at a deformation temperature of 1075°C and 0.1 s$^{-1}$. Optical micrographs for intermediate strain of (b) 0.23 (arrows indicate serrated boundary), (c) 0.35, (d) 0.68 and (e) 0.85.](image)

Figure 4.8 (a) Flow curve of alloy L at a deformation temperature of 1075°C and 0.1 s$^{-1}$. Optical micrographs for intermediate strain of (b) 0.23 (arrows indicate serrated boundary), (c) 0.35, (d) 0.68 and (e) 0.85.

### 4.1.5 Zener-Hollomon parameter ($Z$) and deformation activation energy ($Q$)

The combined effect of temperature and strain rate can be represented by the Zener-Hollomon parameter [148]. Rearranging of Eq. (3.9) will give:

$$Z = \dot{\varepsilon} = A_0 \exp \left( -\frac{Q}{RT} \right) (\sinh \alpha \sigma_p)^n$$ (4.1)

$$\dot{\varepsilon} = A (\sinh \alpha \sigma_p)^n$$ (4.2)

$$A = A_0 \exp \left( -\frac{Q}{RT} \right)$$ (4.3)

where $\dot{\varepsilon}$ is strain rate, $Q$ is the deformation activation energy, $T$ is absolute temperature in Kelvin, $R$ is the universal gas constant ($8.314 \text{ JK}^{-1}\text{mol}^{-1}$), $\sigma_p$ is the peak stress; $\alpha$, $A_0$ and $n$ are material dependent constants. Figures 4.9 a, c illustrate
the determination of $n$ by a power law fitting using the experimentally obtained values of the peak stress under different TMP conditions and Figure 4.9 b, d shows the deformation activation energy in alloys $L_0$ and $L$.

Figure 4.9 Determination of (a, c) $n$ from sinh($\alpha\sigma_p$) vs strain rate plot and (b, d) the deformation activation energy ($Q$) from lnA vs -$1/RT$ plot for alloys (a, b) $L$ and (c, d) $L_0$.

The best fit of the $(\text{sinh } \alpha \sigma_p)^n$ curve to the experimental data was obtained for $\alpha = 0.008$ MPa$^{-1}$ for both alloys. The calculated value of $n$ is 4.45 and 4.40 in alloys $L$ and $L_0$ respectively. The value of $\alpha$ for the studied alloys is slightly lower compared to that for a high strength low alloy steel (0.012), Nb-microalloyed steel (0.011) and stainless steel (0.012), while the value of $n$ is in agreement with the values reported for various grades of carbon steels (4.2-5.1), Nb-microalloyed and
stainless steels (4-5) [30, 149-151]. The value of A can be determined by substituting \( n = 4.45 \) or 4.40 and \( \alpha = 0.008 \) in Eq. 4.2. The deformation activation energy \( Q \) can be determined by taking natural logarithm on both sides of Eq. (4.3):

\[
\ln A = \ln A_0 + \left( -\frac{Q}{RT} \right)
\]

(4.4)

In the \( \ln A \) vs \((-1/RT)\) plot (Figures 4.9 b, d), the slope of the straight line is \( Q \). The deformation activation energy for the studied alloys \( L \) and \( L_0 \) was calculated to be \( 419 \pm 9 \) KJ/mol and \( 362 \pm 3 \) KJ/mol, respectively.

In the current study, the Zener-Hollomon parameter for all deformation conditions has been determined by substituting the deformation activation energy of \( 419 \) KJ/mol for alloy \( L \) and \( 362 \) KJ/mol for alloy \( L_0 \) in Eq. (3.8). Critical and peak stresses are strongly dependent on \( Z \) (Figure 4.10). These dependences have been described by the power law relationships as \( \sigma_p = BZ^m \) [152] where \( B \) and \( m \) are the material dependent constants. The power law equations of alloys \( L \) and \( L_0 \) are listed in Table 4.3.

The relations between the critical and peak stresses and strains are: \( \sigma_c = 0.83\sigma_p \) and \( \varepsilon_c = 0.67\varepsilon_p \) in alloy \( L \) and \( \sigma_c = 0.65\sigma_p \) and \( \varepsilon_c = 0.67\varepsilon_p \) in alloy \( L_0 \). These relationship is in good agreement with the values obtained in other materials [86, 153, 154]. The value of the exponent for all equations (Table 4.3) is in the range of 0.14 to 0.22, which is in the range (0.11 to 0.21) in Nb-microalloyed steels and pipe line steels [30, 149]. The critical stress in alloy \( L \) is nearly \( 0.83\sigma_p \) comparing to \( 0.65\sigma_p \) in the \( L_0 \) alloy, which indicates faster work hardening in the former alloy during the start of deformation process due to the presence of Nb via solid solution strengthening.

Table 4.3 The power law relationships between Zener-Hollomon parameters (Z) and peak (\( \sigma_p \)) or critical (\( \sigma_c \)) stresses and peak (\( \varepsilon_p \)) or critical (\( \varepsilon_c \)) strains in two sets of model alloys.

<table>
<thead>
<tr>
<th></th>
<th>( \sigma_p )</th>
<th>( \sigma_c )</th>
<th>( \varepsilon_p )</th>
<th>( \varepsilon_c )</th>
</tr>
</thead>
<tbody>
<tr>
<td>alloy ( L )</td>
<td>( 0.6654Z^{0.1473} )</td>
<td>( 0.5537Z^{0.1498} )</td>
<td>( 0.0009Z^{0.1637} )</td>
<td>( 0.0006Z^{0.1736} )</td>
</tr>
<tr>
<td>alloy ( L_0 )</td>
<td>( 0.8609Z^{0.1618} )</td>
<td>( 0.5620Z^{0.1733} )</td>
<td>( 0.0003Z^{0.2207} )</td>
<td>( 0.0002Z^{0.2058} )</td>
</tr>
</tbody>
</table>
Figure 4.10 Effect of Zener-Hollomon parameter (Z) on (a, c) critical and peak stress and (b, d) critical and peak strain for (a, b) L and (c, d) L₀ alloys.

It has been observed (Figure 4.10) that both peak and critical stresses and strains increased with an increase in Z but the rate of change for critical and peak stresses and strains is different. Critical and peak stresses increased more rapidly than critical and peak strains with an increase in Z. This increase in stress and strain with Z is associated with a decrease in the dislocation and grain boundary mobility. The stress also increased due to presence of solute atoms that significantly controls the mobility of grain boundaries and dislocations. The boundary migration and dislocation motion is faster at lower values of Z [6] (i.e. at lower strain rates and
higher deformation temperatures), which leads to a decrease in the stress and strain required to initiate DRX. At high Z values, critical and peak stress (Figure 4.10c) are getting closer because of the limited amount of softening at high strain rates or low temperatures.

4.1.6 Fraction of dynamically recrystallised grains

Using parameters from the flow curves analysis, the volume fraction of the dynamically recrystallised microstructure can be calculated using Eq. 3.9 and data listed in Table 4.1. The values of strain for the maximum softening rate [62] were determined from the first derivatives of the flow curve (Figure 4.7) for both L and L₀ alloys. Figure 4.11 illustrates the fraction of dynamically recrystallised grains calculated by Eq. 3.9 for all strain rates and temperatures. It is obvious that the curve shifts to the right with a decrease in deformation temperature or an increase in strain rate indicating that longer time and larger strain are required in order to reach the same DRX fraction. Complete dynamic recrystallisation has been observed for all three strain rates (1 s⁻¹, 0.1 s⁻¹ and 0.01 s⁻¹) at deformation temperatures of 1075 °C and 1150 °C (Figures 4.5 a b, e, f, i and j) for the alloy L. However, the percentage of its recrystallisation at 1000 °C depends on the strain rate; 100% DRX was observed at strain rates of 0.1 s⁻¹ and 0.01 s⁻¹ while only 80% of DRX was completed for 1 s⁻¹ strain rate. On the other hand, in the case of alloy L₀, fully recrystallised microstructures have been observed except 1000 °C, 1s⁻¹ deformation condition.

The strain rate also has a considerable influence on the beginning of DRX and the DRX fraction. The fraction of recrystallised grains at different strains can be easily calculated using the curves shown in Figure 4.11. For example, to illustrate the effect of strain rate on DRX, the percentage of dynamic recrystallisation of alloy L was determined to be (at 0.3 strain and deformation temperature of 1150 °C) 93%, 40% and 12% at a strain rate of 0.01, 0.1 and 1 s⁻¹, respectively. The calculated fraction recrystallised for alloy L from DRX curve, for different strains at 1075 °C and at 0.1 s⁻¹ strain rate is 5% at 0.23, 28% at 0.35, 86% at 0.65 and 97% at 0.85 which showed a good agreement with the recrystallised fractions measured from optical micrographs (Figures 4.8 b, c, d, e).
Figure 4.11 Evolution of DRX fraction of L and L₀ model alloys at different deformation temperatures and strain rate of (a) 0.01 s⁻¹ (b) 0.1 s⁻¹ and (c) 1 s⁻¹.
4.2 Discussion

4.2.1 Flow behaviour

The flow stresses for alloy L are higher than those for alloy L₀, and this is more pronounced at the highest strain rate. This is due to the competition between work hardening, dynamic recovery and dynamic recrystallisation. The existence of a stress peak confirmed the initiation of DRX at 1000 °C at all three strain rates (Figures 4.1 a, b, c) for alloy L but the absence of a steady state stress region on the flow curves indicates incomplete DRX. In the case of alloy L₀, however, the flow curves showed full recrystallisation (Figures 4.1 d, f) except for the highest strain rate (Figures 4.1b). In contrast, the flow curves obtained by Beladi et al. [29] for a model Ni-30Fe without Nb and C showed a lower flow stress and an earlier initiation of DRX with considerable softening at 1000 °C even at the highest strain rate (1 s⁻¹). The reasons for this are: (i) the deformation mode was torsion which considerably changes the state of applied stress in comparison to the compression used in the present study, (ii) the absence of Nb and C solute atoms which implies that there are no these obstacles to dislocations and boundary mobility and (iii) the amount of deformation is much higher in the Ni-30Fe alloys (ε = 6) compared to those used in the present study (ε = 1.2). Generally, flow stress for the Ni-30Fe-Nb-C alloy was observed to be higher than that for Ni-30Fe-C, Ni-30Fe model alloys [13], plain carbon steels [28], pipeline steel [30], 304 stainless steel [5] and microalloyed steels [155] deformed in uniaxial compression using the same deformation conditions. Additions of Nb and C increased the strength of the model Ni-30Fe alloy [5, 26]. It can also be seen that the peak and critical stress values increase when Nb is present as an alloying element. The increase in critical stress is attributed to the quicker raise of dislocation density as Nb solute impede the motion of dislocations.

4.2.2 Grain structure

The average grain size obtained before final deformation increases with a decrease in quenching temperature for both alloys. This grain coarsening happens due to a longer time required to attain a lower final deformation temperature from the roughing temperature of 1175 °C. For example, it takes only 8.3 s to reach 1150 °C from the roughing deformation temperature, while approximately 83 s is needed
to reach 925 °C. Finer grain sizes in alloy L₀ compared to the alloy L are due to the occurrence of dynamic and possibly metadynamic recrystallisation, as well as finer starting microstructure.

The bimodal distribution of grains is clearly visible (Figure 4.6) together with a faster development of twins (Figures 4.5 a, j) especially at higher deformation temperatures and slower strain rates. The annealing twins are growing faster during dynamic recrystallisation [156-158] at the highest strain rate where the time of deformation is about 120 s. Similarly to our observations, the more pronounced growth of twins at higher deformation temperatures and lower strain rates during hot deformation was also reported [159, 160]. The growth of recrystallised grains is more pronounced at higher temperature (Figure 4.6 and Figures 4.5 a, e, i) due to higher grain boundary mobility and at lower strain rate, due to the availability of longer time for boundary migration. The recrystallised grains grow slower in alloy L than in alloy L₀ due to the presence of Nb.

Formation of strain-induced NbC is well documented for Nb-containing microalloyed steels [111, 112, 154, 161-163] and for Ni-30Fe-Nb-C alloy [4]. Taking into account a high Nb content in the studied here alloy, strain-induced precipitation at temperatures below 1000 °C would be expected. In addition, the deformation temperatures are higher (except 925 °C) than the NbC dissolution temperature in alloy L [27]. However, solubility calculation using the data in steels, the solubility temperature of NbC in alloy L is around 1200 °C (Figure 3.2). Thus the formation of NbC on cooling before deformation is expected but the results in Chapter 6 in TEM did not reveal any NbC particle in alloy L. Therefore, the observed stress-strain behaviour and grain structure variations may be attributed to the effect of solute drag, solute atom clusters and possibly at lower deformation temperature (925 °C, 1000 °C) to very fine particles (<2 nm). The latter needs to be verified by TEM studies (see Chapter 6).

4.2.3 Work hardening and softening behaviour

It has been noticed that the strain at which the maximum softening rate is achieved shifts to higher values as temperatures decrease and strain rates increase. The latter could be explained by the dominance of the work hardening process over softening at this temperature, as less than 5-10% dynamic recrystallisation occurred
at 925 °C for all strain rates (Figues 4.5 d, h, i and Figure 4.7). This behaviour correlates with 925 °C being very close to the recrystallisation stop temperature \( T_{nr} \) [128]. On the other hand, the former is due to the dominance of dynamic recovery and recrystallisation over work hardening. For alloy L\(_0\), instead, at all three deformation temperatures (Figure 4.7d) the work hardening rate exhibits negative work hardening. The difference in the work hardening behaviour of the two alloys is associated with presence of Nb in alloy L.

Nb has a pronounced effect on the work hardening rate: first, as Nb atoms are larger than those of Ni and Fe (0.143 nm vs 0.125 nm and 0.126 nm), substitutional Nb atoms create a strain field in the matrix, this lattice strains impede the dislocation motion leading to the solute drag effect [23-25] and second, because Nb may form clusters and/or nano-sized NbC particles, resulting in dislocation pinning [39-43]. Thus, the interaction of the dislocations with the above obstacles leads to a faster work hardening rate in the alloy L compared to the alloy L\(_0\) one. However, zero work hardening rate indicates that there is a balance between the hardening and softening processes, whereas the negative work hardening rate is the clear evidence of the dominance of the latter, e.g. active DRX.

The rate of dynamic recrystallisation slows down with a decrease in deformation temperature as well as an increase in strain rate. The behaviour of flow curves such as peak stress, work hardening rate and time to reach steady state condition is also a function of the deformation temperatures and strain rates. There are a number of reasons for this. (i) At a low temperature and at a high strain rate the rate of DRV is slow which leads to a higher flow stress. Simultaneously, the decrease in temperature and time available limits grain boundary motion and results in the delayed onset of recrystallization. (ii) The influence of solute Nb is remarkably high in restricting the process of DRX [99]. The evolution of recrystallised grains at a higher strain rate is delayed due to the lack of time available for grain boundary motion.

### 4.2.4 Effect of alloying elements on deformation activation energy

Table 4.4 summarises the deformation activation energy calculated by different researchers for various steels and Ni alloys [99, 154, 164-171] in ascending order of activation energy values. As can be seen from Table 4.4, the activation
energy is strongly influenced by the number of alloying elements and their amounts. An increase in alloying element content has been reported to raise the deformation activation energy, except for C which decreases it due to its faster diffusion [99, 149, 172]. Carbon interacts with vacancies both electrostatically and by strain interactions and this leads to a decrease in the activation energy [173]. The activation energy also increases with the addition of more alloying elements (Table 4.4). For example, pure Fe has activation energy of 272 KJ/mol whereas the value increases to 278 KJ/mol for C-Mn steel and to 347 KJ/mol for C-Mn-Nb steel.

Table 4.4 Deformation activation energy for different C-Mn steels, stainless steels and Ni based alloys.

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>Q (KJ/mol)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Fe</td>
<td>272</td>
<td>[99, 171]</td>
</tr>
<tr>
<td>Steel: 0.11C-0.24Si-1.23Mn-0.041Nb</td>
<td>288</td>
<td>[171]</td>
</tr>
<tr>
<td>Steel: 0.11C-0.24Si-1.32Mn-0.093Nb</td>
<td>294</td>
<td></td>
</tr>
<tr>
<td>Steel: 0.36C-1.42Mn</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Steel: 0.35C-1.41Mn-0.044Nb</td>
<td>278</td>
<td>[170]</td>
</tr>
<tr>
<td>Steel: 0.055C-1.42Mn-0.036Nb</td>
<td>360</td>
<td>[170]</td>
</tr>
<tr>
<td>Steel: 0.1C-1.42Mn-0.035Nb</td>
<td>325</td>
<td>[154]</td>
</tr>
<tr>
<td>304SS: 19Cr-9Ni-0.22Mo</td>
<td>393</td>
<td></td>
</tr>
<tr>
<td>304SS: 18Cr-8Ni-0.27Mo</td>
<td>410</td>
<td>[166]</td>
</tr>
<tr>
<td>304SS: 18Cr-11Ni-0.02Mo</td>
<td>410</td>
<td></td>
</tr>
<tr>
<td>316SS: 17Cr-11Ni-2.92Mo</td>
<td>460</td>
<td></td>
</tr>
<tr>
<td>Pure Ni</td>
<td>278</td>
<td>[164, 165]</td>
</tr>
<tr>
<td>Ni base: 69.7Ni-30Fe-0.1C</td>
<td>362</td>
<td>this work</td>
</tr>
<tr>
<td>Ni base: 60Ni-10.05Fe-28.88Cr-0.038C</td>
<td>380</td>
<td>[169]</td>
</tr>
<tr>
<td>Ni base: 69.7Ni-29.8Fe-0.331Nb-0.04C</td>
<td>419</td>
<td>this work</td>
</tr>
<tr>
<td>Ni base: 64Ni-3Fe-22Cr-14W-2Mo-5Co-0.1C</td>
<td>456</td>
<td>[168]</td>
</tr>
<tr>
<td>Ni base: 61Ni-21Cr-10W-5Mo-2Al-1Ti</td>
<td>496</td>
<td>[167]</td>
</tr>
</tbody>
</table>

The increase in activation energy in Nb microalloyed steel can be attributed to both solid solution and precipitation strengthening mechanisms. The nature and number of alloying elements, as well as their concentration in steels and Ni based alloys significantly change the SFE of austenite [174-176] which alters the activation
energy of these materials. As could be seen from Table 4.4, the total amount of alloying is 2-3% in C-Mn steels, which is associated with 278-288 KJ/mol deformation activation energy with the higher value related to the larger number of alloying elements [26, 55, 54].

On the other hand, the alloying in stainless steel is 26-31% leads to the 390-460 KJ/mol values for activation energy. The higher the amount of alloying elements, the deformation activation energy is higher (Table 4.4, [50]). Similar trend is reported for Ni-based alloys, a 60Ni-10.05Fe-28.88Cr-0.038C alloy with the total alloying amount of ~40% and three alloying elements has activation energy of 380 KJ/mol [53], whereas a much higher activation energy of 496KJ/mol was reported [51, 52] for 61Ni-21Cr-10W-5Mo-2Al-1Ti alloy having nearly the same total alloying addition of 39% but five alloying elements. Thus, the activation energy for austenitic stainless steels and Ni-based superalloys is much higher than that for C-Mn steels due to higher numbers and amounts of the alloying elements. Although the alloying level is low in high strength low alloy steels and tool steels, these steels exhibit high deformation activation energy due to higher diversity of solute atoms contributing to solid solution strengthening [172] and particles contributing to the precipitation strengthening.

4.2.5 Influence of processing parameters on dynamic recrystallisation evolution

The motion of dislocations is greatly accelerated at high temperatures, which leads to an earlier initiation and completion of DRX. Dislocation climb in different glide planes is much easier at elevated temperatures and the grain boundary mobility is also higher at high temperatures, making DRX easier. For example, the DRX fraction is only 22 % at the deformation temperature of 1000 °C, compared to 40 % at 1075 °C and 77 % at 1150 °C in alloy L deformed to ε = 0.4 at 0.1 s\(^{-1}\) strain rate. Strain rate also significantly alters the DRX mechanism such that earlier initiation of DRX and higher DRX fraction is obtained at the same temperature with a lower strain rate (Figure 4.11). The DRX fraction in alloy L\(_0\) after ε = 0.3 at 1000 °C is 0.6 for 0.01 s\(^{-1}\) strain rate and DRX fraction is about 0.1 for 1 s\(^{-1}\) strain rate at the same deformation strain and temperature. This is because duration of deformation time is 30 s at 0.01 s\(^{-1}\) rate comparing to 0.3 s during 1 s\(^{-1}\).
The fraction recrystallised calculated by Eq. (3.9) at 925 °C, \( \varepsilon = 1.2 \) is nearly 95 %, 84 % and 60 % at strain rate of 0.01 s\(^{-1}\), 0.1 s\(^{-1}\) and 1 s\(^{-1}\) respectively. However, the actual fraction recrystallised determined using optical microscopy is markedly lower than these values, as micrographs showed that only 5-10% have been dynamically recrystallised (Figures 4.5 d, h and l). The data obtained from the direct microstructure observation is considered to be a more realistic one compared to the estimation using the theoretical Eq. (3.9). The fraction of dynamic recrystallisation via Eq. (3.9) has also been estimated for different materials, such as microalloyed steels [177], hypereutectoid steels, 304 stainless steels [87] and API-X70 steels [30]. Similar to our results, in all these studies, the calculated DRX curves showed nearly complete dynamic recrystallisation even if the deformation temperature is below the recrystallisation stop temperature (\( T_{\text{nr}} \)), which is contradictory to the general theory of dynamic recrystallisation and microstructure observations. It should be pointed out that the slope of the DRX curve at 925 °C in Figure 4.11 is not only less steep than that at higher temperatures, but the start and finish of DRX are significantly delayed. As mentioned earlier in Section 4.1.4, a low mobility of dislocations and grain boundaries at lower temperature due to more pronounced pinning by solute atoms and precipitates reduces the kinetics of dynamic softening. Eq. (3.9) contains the strain corresponding to the maximum softening rate but as can be seen from Figure 4.7, the maximum softening rate for 925 °C conditions remains positive unlike the negative values for all other temperatures. Although at high temperatures DRX takes place, the simultaneous dynamic recovery and work hardening processes also operate, but to a lesser extent compared to DRX. Thus, near \( T_{\text{nr}} \), DRV is the dominant softening process after work hardening even for low and medium stacking fault materials whereas DRX is a major softening mechanism for the development of microstructure during deformation at higher temperatures [63]. However, at 925 °C the role of recovery and work hardening becomes more pronounced and the calculated value of \( \varepsilon_{\text{m}} \) is misleading due to the absence of DRX. The estimation of DRX fraction by Eq. (3.9) is difficult near \( T_{\text{nr}} \), because Eq. (3.9) considers the total softening including DRV as DRX fraction.

The grain refinement in both alloys L and \( L_0 \) can be observed from Table 4.2. A large standard deviation has been noticed after deformation at a high temperature or at a lower strain rate for both alloys. Fine grains are recrystallised at the
boundaries of coarse grains and grains are growing once DRX is completed. The alloy L has a slightly finer recrystallised grain size than that in the alloy L₀ despite the starting grain size of the former alloy being coarser. The recrystallised grains in alloy L₀ were also grown very rapidly as there is no Nb to impede the grain boundary motion. An average recrystallised grain size of 56 ± 43 µm was observed at a deformation condition of 1150 °C at 0.01 s⁻¹ with a starting grain size of 60 ± 45 µm in alloy L₀. By contrast, a final DRX grain size of 52 ± 40 µm (Figure 4.5i) was found for alloy L with an initial grain size of 100 ± 40 (Figure 4.4d) and deformed at 1150 °C at 0.01 s⁻¹ strain rate. It can also be seen that the initiation of DRX in the alloy L is delayed with respect to the alloy L₀, and the fraction which is dynamically recrystallised is less in the former one under identical TMP conditions. This means that Nb is the main influential alloying element to restrict the grain growth and DRX in L alloy. Thus, Nb as an alloying element has a threefold effect: (i) Nb segregates to grain boundaries and reduces the mobility of high angle grain boundaries, which delays the DRX process [106]; (ii) it restricts the motion of dislocations by interaction with Nb as solute, with Nb clusters; (iii) at lower deformation temperatures, it may provide the obstacles to boundary migration in the form of fine strain-induced NbC precipitates [114, 178].

4.3 Conclusions

The flow curves of alloys L and L₀ exhibited typical DRX behaviour except at 925 °C. The flow behaviour, the evolution of DRX and its characteristics were examined as a function of different temperatures, strain rates and the addition of Nb. The following conclusions can be drawn:

- The critical stresses/strains and peak stresses/strains have increased with a decrease in deformation temperature or an increase in strain rate, as well as the addition of Nb.
- The correlations between the critical and peak values for both stress and strain were derived as: ε_c = 0.67ε_p for both alloys and σ_c = 0.83σ_p, σ_c = 0.65σ_p for L and L₀ alloys, respectively.
• The deformation activation energy was calculated by an Arrhenius-type equation and an increase in deformation activation energy has been seen with the addition of Nb from 362 KJ/mol to 419 KJ/mol.
• The fraction of dynamically recrystallised grains is directly proportional to the deformation temperature and has an inverse relationship to strain rate. The DRX process is delayed by the addition of Nb.
• The predicted volume fraction of DRX differed significantly from that determined by optical metallography for the deformation conditions at the temperature near $T_{nr}$. This was explained by the limitation of the equation used, which takes into account the fraction softened by dynamic recovery near $T_{nr}$ instead of dynamic recrystallisation.
Chapter 5: Insight into Microstructure and Micro-texture Evolution During the Dynamic Recrystallisation of Ni-30Fe-Nb-C Model Alloy

The evolution of microstructure and micro-texture during discontinuous dynamic recrystallisation (DRX) in Ni-30Fe-Nb-C (alloy L) model alloy subjected to interrupted strain of 0, 0.23, 0.35, 0.68, 0.85 and 1.2 in plane strain compression mode at 1075 °C was investigated using electron backscattering diffraction and transmission electron microscopy. The obtained results are presented in Section 5.1 and discussed in Section 5.2. The detailed nucleation mechanism of DRX grains at low to high strain is outlined in Section 5.1.4. The dominance of the Cube orientation in the recrystallised grains and unrecrystallised grains (comprising a mixture of primary deformed, recovered and secondary deformed ones) is highlighted in Section 5.1.5 and Section 5.2.2.

5.1 Results

5.1.1 Flow curve analysis

The true stress vs. true strain curve along with work hardening curve are shown in Figure 5.1a, where the red circles on the flow curve indicate interrupted strain points at which samples for EBSD and TEM studies were produced. The flow curve of alloy L deformed at 1075 °C, 0.1 s\(^{-1}\) shows a broad stress peak with continuous flow softening up to final strain of 1.2 without reaching the steady state condition. Flow curves with broad stress peak were reported previously in Ni-30Fe and Ni-30Fe-Nb-C model alloys subjected to PSC [15]. However, a typical DRX flow curve consists of a stress peak either narrow or broad before reaching a steady state condition [56, 63, 73, 147]. The absence of steady state is associated with a coarser starting grain size (181 ± 110 µm) in the current study than the initial grain size (114 ± 40 µm) of alloy L used in previous thermo-mechanical schedule (Chapter 4) [179], where the steady state stress was observed at similar hot deformation conditions. In accordance with Fernández et al. [154], decreasing the starting grain size promotes the DRX process and assists in reaching the steady state condition earlier during hot deformation.
Figure 5.1 (a) Flow curve and work hardening curve of alloy L after 1.2 strain at 1075 °C, the red circles on flow curve are the points of interrupted strain experiments and (b) recrystallised fraction with respect to strain calculated from EBSD and empirical equation.

The WH rate decreases rapidly up to a transition point where the WH curve deviates from initial WH rate and the deviation point is the indication of critical strain for DRX. Then, the work hardening rate decreases very slowly and stays in the negative work hardening zone up to \( \varepsilon = 1.2 \). Figure 5.1b depicts the evolution of DRX fraction with strain as estimated from Avrami-type Eq. (3.9). Critical, peak and maximum softening strains were obtained from the fitted first and second derivatives of the flow curve (Chapters 3 and 4). These values were used in Eq. (3.9). The calculated DRX fraction shows increase up to \( \varepsilon = 1.2 \) with a DRX fraction of \( \sim 0.92 \).

5.1.2 Microstructure characterisation

The EBSD inverse pole figure (IPF) maps and band contrast maps are delineated respectively in Figure 5.2 and Figure 5.3 at ascending order of strain. The IPF maps segmented into recrystallised and unrecrystallised fractions are shown in Figure 5.4. The initial microstructure in non-deformed, \( \varepsilon = 0 \) condition (Figures 5.2a and 5.3a) consists of equiaxed grains of 90 ± 75 µm (with considering TBs as boundaries) and 181 ± 110 µm (without TBs as boundaries).
Figure 5.2 EBSD inverse pole figure maps at strains of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85, and (f) 1.2. LAGBs = grey, HAGBs = black, 60° (111), Σ3 TBs = red lines.
Figure 5.3 EBSD band-contrast maps at strains of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85, and (f) 1.2. LAGBs = blue lines, HAGBs = thick black lines and 60° (111), Σ3 TBs = red lines.
The microstructure at $\varepsilon = 0$ condition contains a large fraction of $\Sigma 3$ annealing TBs and a smaller fraction of $\Sigma 9$ TBs (Figure 5.3a). The large fraction of $\Sigma 3$ TBs produced a sharp peak at $60^\circ$ in the misorientation distribution profile (Figure 5.5a) and comprises ~41% of grain boundary area fraction (Figure 5.5b). Secondary annealing TBs of $\Sigma 9$ are also seen at $38.9^\circ$ (Figure 5.5a) which contribute only ~6.3% of boundary area fraction in the microstructure. This low fraction of $\Sigma 9$ could be explained by the limited impingement of $\Sigma 3$ boundaries as the former is formed by the twin reaction: $\Sigma 3 + \Sigma 3 = \Sigma 9$ \[180\]. At $\varepsilon = 0.23$, the microstructure mostly consists of deformed, slightly elongated grains with a few small grains along the GBs (Figures 5.2b, 5.3b and 5.4 a, f).

It is clear from the EBSD band contrast maps that the initial grains become more elongated perpendicular to the compression direction and the LAGBs are preferentially seen within the prior grains, (Figure 5.3b). These low angle boundaries are built as a network with an increase of deformation strain from 0.35 to 0.85 and gradually the LAGBs (blue) are replaced by equiaxed grains with HAGBs (black lines) which is depicted in Figures 5.3 c-e. The detailed analysis of how these recrystallised grains originate is illustrated in Section 5.1.4. An increase in the amount of LAGBs in pancaked grains is also seen from $\varepsilon = 0.23$ to 0.85 (Figures 5.3, b-e). A few recrystallised grains are readily observable along the original grain boundaries at $\varepsilon = 0.23$ (Figures 5.2 b and 5.3b). It is even clearer from Figure 5.4a, in which only recrystallised grains are present as a result of segmentation. At $\varepsilon = 0.35$ (Figures 5.2 c, 5.3c and 5.4c), the deformation is apparent and a clear necklace of recrystallised grains decorate the original grain boundaries (Figures 5.4 c, d). At $\varepsilon = 0.68$, 0.85 and 1.2, the microstructure comprises a significant fraction of recrystallised grains as seen in Figures 5.2 d-f and Figures 5.4 e, g, i.
Recrystallised

(a)

(b)

Unrecrystallised

(c)

(d)

(e)

(f)

(g)

(h)
Figure 5.4 EBSD inverse pole figure maps of (a, c, e, g, i) recrystallised and (b, d, f, h, j) unrecrystallised fractions at (a, b) 0.23, (c, d) 0.35, a clear necklace layer delineates the pre-existing grain boundaries (e, f) 0.68, (g, h) 0.85 and (i, j) 1.2 deformation strain.

The change in misorientation distribution as a function of strain is depicted in Figure 5.5a. With increasing strain, there is initially an increase in the LAGBs area fraction from $\varepsilon = 0$ to $\varepsilon = 0.23$ followed by a continuous decrease up to 0.68 and then plateauing (Figure 5.5b). The latter is associated with an increase in HAGBs and annealing TBs. On the other hand, a reduction in the HAGB, THAGB, $\Sigma 3$ and $\Sigma 9$ is observed at $\varepsilon = 0.23$ followed by gradual increase at higher strains (Figure 5.5b).
Figure 5.5 (a) Misorientation distribution as a function of strain with typical misorientation axis distributions in crystal coordinate system for Σ3 and Σ9 angular range, (b) grain boundary area fraction as a function of deformation strain, (c) the initial grain size is measured before any deformation and average DRX grain size at different strain with and without TBs as grain boundaries, (d) the area fraction Σ3 annealing TBs of cube and non-cube DRX grains.

The overall grain refining process can be seen from Figure 5.5 c, where the grain size of ε = 0 condition, drastically reduces already after 0.23 strain as a result of DRX and the DRX grain size remains almost constant at higher strains. Although a high fraction of recrystallised grains is found at higher strain leading to microstructure refinement. The average recrystallised grain sizes regarding TBs as
GBs are measured to be $10 \pm 9 \, \mu m$, $13 \pm 12 \, \mu m$, $13 \pm 11 \, \mu m$, $12 \pm 9 \, \mu m$ and $14 \pm 10 \, \mu m$ at strain of 0.23, 0.35, 0.68, 0.85 and 1.2, respectively.

The DRX grains were divided into two subsets of cube and non-cube components. The area fraction of cube and non-cube $\Sigma 3$ annealing TBs in DRX grains is given in Figure 5.5d, where grains exhibiting non-cube orientations have a higher area fraction of $\Sigma 3$ TBs than those of cube orientation.

The large disparity between the recrystallised fractions estimated from Eq. (3.9) and EBSD-based segmentation (Figures 5.1b and Figures 5.4 a, c, e, f, i) can be linked to the differences in their underlying assumptions. Eq. (3.9) overestimates the dynamically recrystallised fraction as it cumulatively calculates the DRX fraction and does not account for secondary deformation with increasing strain. On the other hand, the GOS threshold criterion returns an apparent underestimate as it strictly accounts for the recrystallised grains at any given strain (Figures 5.4 a, c, e, f, i). Consequently, grains that nucleated at lower strains and underwent secondary deformation are not part of the recrystallised fraction at higher strains (Figures 5.4 d, f, h and j). It follows that the estimates obtained from the two methods cannot be expected to match each other and as such, should not be compared at all.

5.1.3 Intragranular local misorientation and substructure

The intragranular local misorientation is analogous to the geometrically necessary dislocation (GND) density such that local areas of large intragranular misorientations correspond to higher densities of GNDs and allude to strain heterogeneity [143, 144]. The intragranular local misorientation maps are presented in Figures 5.6 a-e with the frequency distribution ($\theta_{\text{local}}$) and mean intragranular local misorientation ($\theta_m$) shown in Figures 5.7 a, b, respectively for all strain levels.

The intragranular local misorientation of deformed grains is increased with the extent of deformation, as expected. At $\varepsilon = 0.23$, the high local misorientation along existing GBs (Figure 5.6a) represents higher strain accumulation in these regions. At higher strains of 0.35 to 1.2, the fraction of equiaxed grains with low internal local misorientation increases and substructure appears in the grain interior of some deformed grains. However, at $\varepsilon = 1.2$, some of the equiaxed grains show higher level of local misorientation than other equiaxed grains. The change in the relative frequency distribution of the intragranular local misorientation angle with
increasing strain is given in Figure 5.7a. Expectedly, with increasing strain, the relative frequency distribution of $\theta_m$ ($\theta_m$ calculated from Eq. (3.5)) moves towards higher intragranular local misorientation values along with a larger standard deviation ($\mu_0$ calculated from Eq. (3.6)).

Figure 5.6 Intragranular local misorientation maps at strain of (a) 0.23, (b) 0.35, (c) 0.68, (d) 0.85 and (e) 1.2. Black lines are HAGBs, high local misorientation is delineated by green area, reds are twin.
Figure 5.7 Intragranular local misorientation of (a) DRX grains at different strains and (b) mean intragranular local misorientation of recrystallised, unrecrystallised subsets and full maps as a function of true strain.

The mean intragranular local misorientation value ($\theta_m$) of the various subsets as a function of strain is depicted in Figure 5.7b. The $\theta_m$ values increase sharply up to 0.35 strains in all subsets. The mean intragranular local misorientation in unrecrystallised subset rises rapidly while a slight increase in $\theta_m$ values in DRX subsets and full maps is observed up to 0.85 strain. At 1.2, the mean intragranular local misorientation plummets in all subsets as the fraction of DRX grains exceeds 0.65. Nonetheless, the unrecrystallised subset also contains a noticeable amount of recrystallised grains undergone deformation (secondary deformed grains) which gives a lower value of $\theta_m$.

Since step sizes of 1µm (for $\varepsilon = 0, 0.23, 0.35, 0.68$ and $0.85$) and 0.5 µm (for $\varepsilon = 1.2$) were used for EBSD mapping and we imposed a minimum spatial resolution of three times the step size, the smallest quantifiable microstructural feature is either 3 or 1.5 µm, respectively. Considering that the above feature sizes may be too large to elucidate some microstructural aspects, TEM was employed to help delineate substructures and early nucleation events.

The typical substructure development in grains at $\varepsilon = 0$ to 1.2 is shown in Figure 5.8. At $\varepsilon = 0$, a few scattered dislocations within the grains are observed (Figure 5.8a). At $\varepsilon = 0.23$, the dislocation density is relatively low and some dislocations are forming the tangles (Figure 5.8b), while clear tangles and dislocation
cells are seen at $\varepsilon = 0.35$ (Figure 5.8c). Apart from the abovementioned dislocation arrangements, the TEM micrographs in Figures 5.8 b, c also show coarsely spaced dislocation walls.

Figure 5.8 (a) TEM bright-field image using two beam with $[111]$ excited condition at $\varepsilon = 0$. TEM bright-field images where beam direction close to $[01\bar{1}]$ zone axis, showing dislocation cell walls and sub-structure development during DRX at (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85, and (f) 1.2. The inset in (f) shows the diffraction pattern with $[01\bar{1}]$ zone axis. The white arrows in the micrographs (b-f) indicate the dislocation walls and subgrain boundaries while the opposite arrows in the top right inset show the deformation direction.

Subgrains are formed at $\varepsilon = 0.68$ strains as dislocations are arranged in arrays and create LAGBs (Figure 5.8d). With further increase in strain to 0.85, cell blocks are subdivided into dense dislocation walls (DDWs), whereas increase in dislocation density is observed deep inside of some other grains. At $\varepsilon = 1.2$, most of microstructure is dynamically recrystallised and the development of substructure somehow similar to the one observed at $\varepsilon = 0.23$. DRX grains are visible with appearance of cell structure at grain boundaries and new subgrain formation (Figure
5.8f). Thus at higher strains the substructure can be divided into two categories; (i) elongated cells with DDWs containing high dislocation densities slightly recovered at later stages in continuously deformed grains and (ii) growing recrystallised grains subjected to deformation displaying isolated dislocations, arrays of dislocations and subgrains.

Figures 5.9 a-c illustrate high density of GNDs near pre-existing GBs and TBs that can be correlated to high dislocation density near GBs in TEM micrographs (Figures 5.9 d-f). From Figure 5.9a, it is clear that local misorientation maps portray a very similar to TEM images (Figures 5.9d and 5.8b) concentration of defects at grain boundaries due to accommodation of inhomogeneity of plastic deformation, as well as their high concentrations in the areas behind the bulged boundaries and near triple junctions, with limited substructure development within the grains at \( \varepsilon = 0.23 \). At \( \varepsilon = 0.35 \), although there is still a higher misorientation near the grain boundaries, it becomes higher within the deformed grain interior with formation of GND network and lower misorientations within the clearly defined cells. This again correlates very well with TEM observations of dislocation network development and subgrain formation with dislocation-free interior (Figures 5.8c and 5.9e). At \( \varepsilon = 0.68 \), together with further development of subgrain structure, the interior of deformed grains become more strained with higher local misorientation (Figure 5.9c) but also there was presence of small strain-free DRX grains, whereas some coarser DRX grains, which formed earlier, exhibit an increase in local misorientation due to secondary deformation. TEM image in Figure 5.9e also shows an increase in dislocation density but a more uniform distribution of dislocations within the interior of deformed grains coupled with subgrain formation. Thus, despite a limited resolution and inability to demonstrate the behaviour of statistically stored dislocations, local misorientation maps still provide a representative picture of the evolution of defect substructure with strain, especially in such a coarse grain structure.
Figure 5.9 (a) Zoomed-in area of EBSD local misorientation maps of (a) 0.23, (b) 0.35 and (c) 0.68 marked by red rectangle in Figures 4.6 a, b and c respectively. TEM bright-field image, beam direction close to [011̅] zone and samples deformed to (d) 0.23, (e) 0.35 and (f) 0.68. The white arrows in EBSD maps showed high intragranular local misorientation areas while in TEM red arrows indicate the arrays of dislocations and dislocations cells.
5.1.4 Nucleation and progression of dynamic recrystallisation

The nucleation mechanism during DRX at lower strain is illustrated in Figures 5.9 and 5.10. At low strains, the dominant nucleation mechanism is associated with the bulging of grain boundaries. It is accompanied either by the formation of dislocation networks, dislocation cells, sub-boundaries or annealing twins (Figures 5.9 a, b and 5.10 a, b). From Figure 5.10a, it is seen that nucleation at original boundaries occurred either via sub-boundary completion or by formation of annealing twins behind the bulge.

The new DRX grains are mainly originated at pre-existing GBs (Figures 5.10 a-c) although the boundaries of these DRX nuclei are not yet fully developed to HAGBs. The TBs that exist in the microstructure before plastic deformation may also provide additional nucleation site and DRX nuclei are observed to invent from those TBs by subgrain formation (Figure 5.10 d). The nucleation on TBs is occurred when the original GBs are exhausted by first necklace layer of new DRX nuclei [6]. A selected area diffraction pattern (SAD) along the [01Ī]T || [01Ī]M zone axis (where T and M represent the twin and the matrix respectively) confirms the twin and further validates its the presence by the dark field image using [200] spot of twin lattice (Figures 5.10 e, f).
Figure 5.10 (a) EBSD band contrast maps, thick black lines are HAGBs, blue lines are LAGBs and red lines are first order TBs and (b-d) STEM bright field images at $\varepsilon = 0.23$, the white arrows in EBSD and STEM micrographs depict the origination of new DRX nuclei at pre-existing boundaries (a, b, c) and twins (a, d) by subgrain formation. (e) A SADP along $[01\bar{1}]$ zone showing both twin and matrix lattices and (f) DF image of the twin in (d) using $\bar{2}00$ spot of twin lattice.
Nucleation at higher strain (≥ 0.68) was found to be simultaneous at the pre-existing boundaries (see smaller DRX grains at coarser DRX grains/matrix interfaces in Figures 5.11a-d) as well as in the interior of elongated deformed grains with well-developed subgrain structure, as seen from the representative EBSD band contrast maps in Figures 5.11 a, b. It should be also noted that 2-3 necklace-like DRX grains layers are formed by this stage of deformation at pre-existing boundaries (Figures 5.11c, d). The IPF maps in Figures 5.11c-d clearly show the difference in orientation of intragranular nuclei from the matrix. In Fig. 5.11c, the deformed grain has orientation close to cube one, whereas the nucleus formed by rotation towards [210]. Similarly in Fig. 5.11d, the nucleus of [001] orientation forms in ~[112] oriented matrix. This confirms nucleation of DRX by rotation of subgrains formed within the interior of deformed to high strains (>0.68) grains as their LAGBs absorbing the dislocations from the interior. For example, the subgrains indicated by the 2nd and 3rd arrows from the left in Figure 5.11a display an intermediate stage of development compared to the nucleus identified in Figure 5.11c by the white arrow: they have less orientation deviation from the matrix and only one HAGB with other boundaries still remained LAGBs. The interior DRX nuclei or DRX subgrains are shown in Figures 5.11 e, f at 0.68 and 0.85 respectively, where new dislocation-free grains continuously evolve from subgrains. Therefore, the nucleation of DRX grains at GBs and TBs occurs at all strain levels, however at higher strains new DRX grains are also formed by rotation of subgrains with LAGBs into nuclei with HAGBs.
5.1.5 Development of dynamic recrystallisation texture

The ideal texture components in fcc materials are represented schematically on the $\varphi_2 = 0^\circ$, $45^\circ$ and $65^\circ$ ODF sections in Figure 5.12 and listed in Table 5.1.

Figure 5.12 Schematic representation of important texture components in fcc metals and alloys.
<table>
<thead>
<tr>
<th>Texture component</th>
<th>Symbol</th>
<th>Euler angles</th>
<th>Miller indices</th>
<th>Fibre</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cube (C)</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Cube-RD (C-RD)</td>
<td>0</td>
<td>22</td>
<td>0/90</td>
<td>0</td>
</tr>
<tr>
<td>Cube-ND (C-ND)</td>
<td>22</td>
<td>0</td>
<td>0/90</td>
<td>0</td>
</tr>
<tr>
<td>Cube Twin (CT)</td>
<td>27/63</td>
<td>48</td>
<td>27/63</td>
<td>0</td>
</tr>
<tr>
<td>Goss (G)</td>
<td>90</td>
<td>90</td>
<td>45</td>
<td>110</td>
</tr>
<tr>
<td>G/B</td>
<td>74</td>
<td>90</td>
<td>45</td>
<td>110</td>
</tr>
<tr>
<td>Brass (B)</td>
<td>55</td>
<td>90</td>
<td>45</td>
<td>110</td>
</tr>
<tr>
<td>Rotated Goss (Rt-G)</td>
<td>0</td>
<td>90</td>
<td>45</td>
<td>011</td>
</tr>
<tr>
<td>Rotated Cube (Rt-C)</td>
<td>0/90</td>
<td>0</td>
<td>45</td>
<td>001</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>90</td>
<td>35</td>
<td>45</td>
<td>112</td>
</tr>
<tr>
<td>S</td>
<td>59</td>
<td>37</td>
<td>63</td>
<td>123</td>
</tr>
</tbody>
</table>

Table 5.1 Euler angles and Miller indices for common texture components in fcc metals and alloys [181].

Due to the coarse grain size at $\varepsilon = 0$, the ODF shown in Figure 5.13 was calculated from an EBSD map acquired with a step size of 10 $\mu$m in order to obtain statistically representative texture information. Alternatively, the ODFs shown in Figures 5.14-5.16 were calculated from the EBSD maps shown in Figures 5.2 (full map) and 5.4 (recrystallised and unrecrystallised fractions) which were acquired with step sizes of 1 $\mu$m (for $\varepsilon = 0, 0.23, 0.35, 0.68$ and 0.85) and 0.5 $\mu$m (for $\varepsilon = 1.2$). The initial micro-texture at $\varepsilon = 0$ condition mainly comprises of cube RD ($\{013\}<100>$) and weak spread between cube ND ($\{001\}<310>$) and cube orientations, though the intensity of these texture components is rather weak (Figure 5.13a).

In addition to the former orientations, the texture of the full map at $\varepsilon = 0.23$ and 0.35 also comprises spread near the B and Cu orientations and an intensity split near Rt-G as seen in Figures 5.14a and b, respectively. At $\varepsilon \geq 0.68$, the C orientation becomes dominant and intensifies with increasing strain. This is accompanied by weaker intensities along the $\alpha$-fibre and the Cu and $\sim$S orientations (Figures 5.14 c-e).
From the onset of strain, the recrystallised texture shows a continuous strengthening of the C orientation such that it dominates the final DRX texture at $\varepsilon = 1.2$ (Figure 5.15). A weak intensity spread is also seen along the $\alpha$-fibre and near the $\sim S$ and CT orientations (Figures 5.15 c-e), although DRX is associated with a high twin fraction. The unrecrystallised texture at $\varepsilon = 0.23$ returns intensity spreads along $\Phi$ near the B and Cu orientations and an intensity split around Rt-G (Figure 5.16a). At $\varepsilon = 0.35$, increasing deformation lead to stronger intensities along the $\alpha$-fibre accompanied by orientation splitting at $-\phi_1 = 45^\circ$, $\Phi = 45^\circ$, $\phi_2 = 0^\circ$ (Figure 5.16b). At $\varepsilon \geq 0.68$, the orientations of the unrecrystallised textures (Figures 5.16 c-e) mirror those of the full map (Figures 5.14 c-e) with a dominant and intensifying C orientation along with weaker intensities for the $\alpha$-fibre and the Cu and $\sim S$ orientations.
Figure 5.14 $\phi_2 = 0^\circ, 45^\circ$ and $65^\circ$ ODF sections of full maps at strain of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85 and (f) 1.2 with a step size of 1 µm. Contour levels = $2\times$
Figure 5.15 $\varphi_2 = 0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of recrystallised subsets at strain of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85 and (f) 1.2 deformation strain. Contour levels = $2\times$
Figure 5.16 $\varphi_2 = 0^\circ$, 45$^\circ$ and 65$^\circ$ ODF sections of unrecrystallised subsets at strain of (a) 0, (b) 0.23, (c) 0.35, (d) 0.68, (e) 0.85 and (f) 1.2 deformation strain. Contour levels = $2^\times$. 
5.2 Discussion

2.1 Microstructure evolution

The intragranular local misorientation maps (Figure 5.6) only reflect the GNDs content while the TEM technique captures all types of dislocations comprising GNDs and statistically stored dislocations (SSDs) [182]. The alloy L has a medium SFE of 50-60 mJ/m² [129], the main softening mechanism is DRX which occurs by nucleation and growth. It is widely accepted that the initiation of DRX takes place at inhomogeneities in the microstructure present before deformation such as: GBs, TBs, second phase particles or at those induced by deformation, such as microbands and shear bands [6]. Higher GNDs in the vicinity of the pre-existing grain boundaries in EBSD intragranular local misorientation maps (Figure 5.6a and Figures 5.9 a-c) implies a higher strain accumulation due to greater incompatibilities. This area of higher strain gradient is designated by higher dislocation densities in TEM micrographs as seen in Figure 5.9d. This differential dislocation pressure across the grain boundaries leads to their bulging [56]. The aforementioned DRX bulging phenomenon is in line with the initiation of DRX grains by SIBM mechanism proposed by Beck and Sperry [78] based on optical microscopy observations and by Bailey and Hirsh based on TEM work [55]. Besides, the pre-existing TBs (annealing twins present in the microstructure before deformation) acts as DRX nucleation site as those TBs also contribute in increasing pre-existing grain boundary area. The DRX nuclei on TBs are only observed on the pre-existing annealing twins (Figures 5.10 d, f) that is in great agreement with the literature and Figures 2.14 c, d [6, 84, 85].

The bulged regions of pre-existing GBs are clean and bounded by newly formed LAGBs, HAGBs or Σ3 twin boundaries (Figures 5.10 a-d). This nucleation mechanism is in agreement with the classical DRX nucleation phenomenon [25, 64, 66]. This is also in a good agreement with twin formation in copper bicrystals [183]. However, Miura et al. [183] reported predominant formation of twin boundaries (Σ3) behind the bulges, while in the present study the fraction of DRX nuclei developed by sub-boundary completion is substantially higher than that of being completed by twin boundary (Figure 5.10 a). The deformation temperature and strain rate play a critical role in the development of new grains from bulged boundaries. The bulged
portion of the GBs is accompanied by the formation of twin boundaries at low strain rate and/or high temperature or sub-boundaries at high strain rate and lower temperature [56, 76]. Here, the bulged boundaries expand via sub-boundary as well as twin completion as the experiments are conducted in the range of moderate temperature and strain rate (1075 °C, 0.1 s⁻¹).

The nucleation at high strain at DRX grains/deformed matrix interface and at TBs is exactly the same as the one at low strain, however a more well-developed subgrain structure and additional DRX nuclei were formed in grain interiors as shown in Figure 5.11. The formation of nuclei within the deformed grains results from the continuous development of subgrains, rotation of which (Figures 5.11 a, b) leads to the formation of DRX nuclei with HAGBs [184]. This type of nucleus at high strain was also observed during hot compression of 304 SS, although the deformation temperature was close to non-recrystallisation temperature [25], while our experiments were performed well above the non-recrystallisation temperature.

There is an agreement on the initial stage of DRX grains development via the formation of necklace layer at the prior GBs and TBs [84, 86]. However, the main disagreement is regarding the completion of the DRX either by the consecutive necklace layer expansion [84] or by the independent development of DRX grains [84, 148]. The current study indicates that the initial necklace layer of DRX grains decorates the pre-existing grain boundaries (Figures 5.4 b, g) and DRX necklace layer evolves consecutively by expanding initial necklace layer by nucleation of new DRX grains at the interface with the deformed matrix (Figure 5.4 e), as well as the appearance of nuclei inside the deformed grains at ε = 0.68 and above (Figures 5.5 c-e and h-j, Figure 5.10). However, the earlier formed DRX grains again accumulate strain (Figure 5.6 d, e) leading to the repetition of the described above processes and microstructure refinement.

The increasing area fraction of Σ3 boundaries during DRX (Figure 5.5 b and Figures 5.4 a, b) is in agreement with the trends typically observed for nickel and copper alloys [180]. From the EBSD map (Figure 5.9 a), it is clear that annealing twins originated during DRX grains nucleation; where new twin related grains are nucleated at the bulged grain boundary. The fraction of first order twins (Σ3) is significantly higher than that of the second order twins (Σ9). The low fraction of Σ9 could be ascribed by the limited impingement of Σ3 boundaries as the former is formed by the twin reaction: Σ3 + Σ3 = Σ9 [180]. The observed decrease of Σ9
boundaries is ascribed to the reversion of $\Sigma 3$ boundaries by the reaction of $\Sigma 3 + \Sigma 9 = \Sigma 3$, instead of developing $\Sigma 27$ TBs [92].

5.2.2 Development of micro-texture

The deformation texture consists of orientations around B, G/B, G, S and Cu in a descending order of the intensity similar to earlier investigation in Ni-30Fe [26, 95] alloys. The strong DRX cube texture (Figures 5.15 c-e), has not been reported previously, except only in one instance, where Guria et al. [97] observed a pronounced cube DRX texture during PSC of 316 SS.

A strong cube texture is typically observed during annealing of cold rolled medium to high SFE fcc materials; such as copper nickel and aluminium, undergoing SRX [185-187]. The cube orientation during SRX is explained by classical oriented nucleation phenomenon, where cube orientation predominantly nucleates more than other orientations as well as favourable growth relationship of cube orientation of $40^\circ \langle 111 \rangle$ with the S component that form during rolling [188].

It is clearly visible in Figures 5.15 a, b that a weak cube orientation exists at early stage of DRX, which indicates that the portion of DRX grains may have cube orientation. However, this C orientation is rather weak up to $\varepsilon = 0.35$ because the fraction of DRX grains is only around ~0.15. The origin of C orientation is due to preferential nucleation of low Taylor factor C grains [189]. The DRX texture evolution during PSC of Ni-30Fe alloy was reported [13, 95, 190] as weak C texture. The distinct difference in DRX texture development can also be attributed to the lower DRX fractions in Refs. [13, 95, 190] comparing to the much higher DRX fraction (~0.7 at $\varepsilon = 1.2$) in the current study.

Generally, the theory proposed to explain high rate of cube nucleation during SRX [188] might also be applicable in the case of DRX as Ridha and Hutchinson [191] pointed out that C oriented grains deformed by PSC might be more susceptible to recovery since their active slip systems have orthogonal and thus elastically non-interacting, Burgers vectors [188]; as a result of which they exhibit low stored energy. The cube oriented DRX grains are favourably nucleated from the cube bands or from cube oriented substructure and subgrains. Moreover, the increase of C oriented nucleation of DRX grains at higher strains yields a pronounced DRX texture.
and the systematic twinning reaction also contributes to strengthened cube texture at the later stages of recrystallisation.

The possible explanations of the intense C orientation in DRX may be obtained from the literature [12, 24, 181, 191, 192]; the nucleation of DRX is occurred by SIBM which yielded a strong C texture [12] by bulging of original cube grains and retaining the C orientations during formation of new DRX grains. Additionally, it was reported using slip trace analysis in single crystal aluminium in PSC, that activation of non-octahedral slip systems e.g. slip systems other than \( \{111\} (110) \) (mainly on \( \{110\} \)) systems is accountable for a strong cube component [24, 181]. This idea of non-octahedral slip response leading to the formation of intense cube orientation was later on supported by Perocheau et al. [192]. Thus, it is believed that in the present study the cube oriented DXR texture may be originated from the activation of non-octahedral slip. However, this requires further analysis. Furthermore, solute drag effect by Nb solid solution in the studied alloy may restrict the excessive grain rotation which is typically responsible for the scattered DRX texture at high temperature. Although DRX is associated with a high twin fraction, the weak spread of CT component in the texture is due to the higher area fraction of twins in the grains with non-cube orientation than the ones with cube orientation as shown in Figure 5.5d.

5.3 Summary

Microstructure and texture evolution in alloy L in the dynamic recrystallisation temperature range has been extensively investigated in plane strain compression from critical strain (0.23) to significantly high strain (1.2). The results showed that:

- Discontinuous nucleation of new grains is primarily observed at the pre-existing boundaries (GBs, TBs) and DRX grain/matrix interfaces at all strain levels via bulging. However, new DRX nuclei with different from the matrix orientation were found to be formed in grain interior at a higher strain (≥0.68) possibly via subgrain rotation. The growth of these nuclei occurs according to the strain induced boundary migration mechanism.
- The evolution of DRX microstructure is continuous and begins with discrete nucleus formation at original GBs and TBs leading to the necklace-like layer appearance, then followed by successive layers formation together with
gradual development of substructure and isolated DRX nuclei in grain interior at higher strains. The earlier formed DRX grains again start to deform and DRX process continues at $\varepsilon = 1.2$ reaching $\sim 70\%$ completion.

- The cube texture strengthens with progress of DRX. Between $0.68 \leq \varepsilon \leq 1.2$ strain ranges, the texture of the unrecrystallised grain fractions was similar to that of the recrystallised grains; both of which are dominated by the cube ($\{001\}<100>$) orientation. The dominance of the cube orientation can be ascribed to its low stored energy and its orientation stability during secondary deformation.
Chapter 6: Effect of Nb on Dynamic and Post Dynamic Recrystallisation of Ni-30Fe-Nb-C Model Alloys

Dynamic and post dynamic recrystallisation behaviour of Ni-30Fe-Nb-C model alloys were studied by Gleeble-3500 thermo-mechanical processing simulator and characterised by optical microscopy, scanning electron microscopy and scanning transmission electron microscopy. The schematic representation of thermo-mechanical processing schedule for this investigation is showed in Figure 3.10 and described in Section 3.3.2. The effect of Nb content on the flow behaviour and microstructure of dynamic and post dynamic softening process is outlined in Section 6.1.1, 6.1.2 and 6.2.3. The influence of different size of particles on dislocations and grain boundary mobility is also described in Section 6.1.3 and 6.1.6. The formation of strain-induced precipitation and particle stimulated nucleation during dynamic recrystallisation is discussed in Section 6.1.5.

6.1. Results

6.1.1 Flow behaviour

The flow curves of alloys L, M and H during plane strain compression with $\varepsilon = 1.2$ at 1075 °C, 0.1 s$^{-1}$ strain rate and respective first derivatives and second derivatives curve are shown in Figures 6.1 a-c. The flow stress increases rapidly up to a critical point after which a parabolic shape is observed. The flow stress in all alloys decreased steadily after reaching the peak, but did not achieve the steady state condition in any alloy. Alloy H exhibits the maximum flow stress followed by alloy M and alloy L (Figure 6.1a). The rate of flow stress decrease after reaching the peak is the slowest in alloy H. The inflection point in $\theta-\sigma$ (first derivatives) plot indicates the critical point for DRX and the stress minimum in $-d\theta/d\sigma$ plot verifies the critical point (Figures 6.1 b, c). The calculated critical stress/strain is plotted with respect to wt. % (Nb + C) in Figure 6.1d. The critical stress increases continuously in alloys L to M to H, while critical strain steeply decreases from alloy L to M, then slightly rises in alloy H.
Figure 6.1 Flow curve of model alloys (a) after final deformation of 1.2 at 1075 °C at 0.1 s\(^{-1}\) strain rate. (b) First derivatives (work hardening rate), (c) second derivatives and (d) the relationship of critical strain and critical stress with wt. % (Nb + C).

6.1.2 Microstructure in optical microscopy

The distinct grain orientation differences between grains results in colour contrast in optical micrographs although the microstructure contains only the austenite phase. Grain structure of the alloys in all processing conditions is shown in Figure 6.2 and average recrystallised grain size is summarised in Table 6.1. After reheating to 1250 °C for 300 s (Figures 6.2 a-c), the grain size rapidly reduces from alloy L to M to H; an average grain size in alloy L is 500 ± 100 µm whereas the
average grain size in alloys M and H is 90 ± 20 µm and 40 ± 10 µm, respectively. The grain size of non-deformed 1075 °C condition is also a function of alloy chemistry (Figures 6.2 d-f) with alloy L having the coarsest grain microstructure (Table 6.1). The microstructures of alloy M and H after roughing deformation (Figures 6.2 e, f) reveal some deformed grains albeit the number of elongated grains is higher in alloy H than alloy M. As seen in Figures 6.2 a-f, a high fraction of annealing twins is associated with the equiaxed grains. Figure 6.3 depicts the variations in the average recrystallised grain size and recrystallised fraction during holding after deformation.

The grain structure in alloy L after final deformation with 0 s holding mostly comprises of equiaxed DRX grains of 14 ± 10 µm (Figure 6.2g). These DRX grains in alloy L are accompanied by high fraction of annealing twins. By contrast, a high area fraction of grains in alloys M and H displays pancaked structure with some DRX grains containing annealing twins. The majority of the equiaxed DRX grains in alloys M and H are located near the original grain boundaries (Figures 6.2 h, i). The average recrystallised grain size in alloys M and H is 11 ± 4 µm and 10 ± 6 µm respectively. The recrystallised fraction in alloy L at 0 s holding is ~0.65, which is significantly higher than fractions of ~0.24 in alloy M and ~0.23 in alloys H.

Holding for 30 s after deformation at 1075 °C leads to complete recrystallisation of alloy L with equiaxed grains of 39 ± 15 µm (Figure 6.2j), which have grown twice in size compared to those after 0 s holding. Grain growth has been noticed in alloy L during further holding to 300 s yielded a DRX grain size of 92 ± 20 µm (Figure 6.2 m). By contrast, the microstructure at 30 s holding of alloys M and H still comprises elongated grains (Figures 6.2 k, l), while the recrystallised grains increase in size by approximately 60 % (Table 6.1, Figure 6.3a). After holding for 300 s, the microstructure of these two alloys depicts analogous recrystallised fraction of about ~0.38, which is rather close to the recrystallised fraction of ~0.33 after 30 s hold (Table 6.1, Figure 6.3b). The increase in recrystallised fraction in all three alloys is associated with MDRX and DRX grain growth (Figures 6.2 h, i k, l, n, o).
Figure 6.2 Optical microstructure of (a, d, g, j, m) alloy L, (b, e, h, k, n) alloy M and (c, f, i, l, o) alloy H after (a-c) reheating at 1250 °C for 300 s, (d-f) roughing at 1250 °C, ε = 0.3 and quenched from 1075 °C and (g-o) final deformation of ε = 1.2 at 1075 °C and subsequent hold for (g-i) 0 s, (j-l) 30 s and (m-o) 300 s.
Table 6.1 Average grain sizes at reheating and non-deformed 1075 °C and average recrystallised grain sizes (µm) of alloys at ε = 1.2, 1075 °C and subsequent holding for 0 s, 30 s and 300 s.

<table>
<thead>
<tr>
<th></th>
<th>Average grain size (µm)</th>
<th>Average recrystallised grain size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Reheat 1250 °C for 300s</td>
<td>ε = 0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0 s</td>
</tr>
<tr>
<td>Alloy L</td>
<td>500 ± 100</td>
<td>144 ± 75</td>
</tr>
<tr>
<td>Alloy M</td>
<td>90 ± 20</td>
<td>45 ± 23</td>
</tr>
<tr>
<td>Alloy H</td>
<td>40 ± 10</td>
<td>30 ± 18</td>
</tr>
</tbody>
</table>

It is seen from Table 6.1, that as expected, the recrystallised grain size increases with holding time as so does the standard deviation. The higher value of standard deviation results from the presence of small recrystallised grains which formed by MDRX and the growth of previously formed DRX grains in alloys M and H, whereas this might be due to the preferential grain growth of different DRX grains in alloy L. The rate of recrystallised grain growth in alloy L is markedly higher than that in alloys M and H (Figure 6.3a).

Figure 6.3 (a) Average recrystallised grain size and (b) recrystallised fraction in alloys L, M and H after ε = 1.2 at 1075 °C followed by 0 s, 30 s and 300 s holding.
6.1.3 Particle analysis in scanning electron microscopy

The particles found in SEM are mainly round in shape though some large elongated and faceted particles have also been identified. The representative chemical analysis of a large particle (~2.5 µm), a small particle (~45nm) and matrix by EDS is shown in Figure 6.4. All examined particles in SEM are confirmed to be NbC based on EDS analysis, as seen in Figure 6.4c. There is no evidence of complex carbide or carbonitride even in the case of extremely large particles. However, there are weak iron and nickel peaks in EDS spectra of small particles (approximately 20-25 nm size), but these peaks are believed to originate from the matrix during acquisition due to a large spot size in SEM.

![Figure 6.4](image-url) Representative SEM images of (a) large and (b) small NbC particles with (c) EDS spectra for large and small size NbC particles and matrix.

The SEM micrographs of alloys M and H after all processing conditions are shown in Figure 6.5. The size and number density of particles greater than 20 nm (resolution limit of SEM) in these two alloys are listed in Table 6.2 and distribution of these particles is shown in Figure 6.6. In the reheating conditions, only few large particles are observed in alloy M (Figure 6.5a), while a mixture of large particles and some small particles are seen in alloy H (Figure 6.5b). The distribution of particles consists of multiple peaks in both alloys (Figures 6.6 a, b) despite the particle density in alloy H (132 x 10⁹ m⁻²) being much higher than that in alloy M (5 x 10⁹ m⁻²).
(Table 6.2). The SEM micrographs of \( \varepsilon = 0, 1075 \, ^\circ\text{C} \) conditions in both alloys reveal appearance of small particles (Figures 6.5 c, d) with wide particle distribution. In this condition the average particle size in alloys M and H is \( 224 \pm 200 \, \text{nm} \) and \( 268 \pm 200 \, \text{nm} \) respectively (Table 6.2). The particle number density in both alloys increased significantly to \( 190 \times 10^9 \, \text{m}^{-2} \) in alloy M and \( 577 \times 10^9 \, \text{m}^{-2} \) in alloy H.

![Alloy M](image1.png) ![Alloy H](image2.png)

(a) Reheat at 1250 \( ^\circ \text{C} \)

![Alloy M](image3.png) ![Alloy H](image4.png)

(c) \( \varepsilon = 0, 1075 \, ^\circ\text{C} \)

![Alloy M](image5.png) ![Alloy H](image6.png)

(e) \( \varepsilon = 1.2, 0 \, \text{s hold} \)
Figure 6.5 SEM micrographs of (a, c, e, f, i) alloy M and (b, d, f, h, j) alloy H in (a, b) reheated to 1250 °C for 300 s, (c, d) non-deformed 1075 °C condition. The alloys deformed to ε = 1.2 at 1075 °C, then hold for (e, f) 0 s, (g, h) 30 s and (i, j) 300 s.

Table 6.2 Average particle size and particle number density in alloy M and H at different deformation conditions.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>( \mathbf{\text{Reheat 1250 \degree C}} )</th>
<th>( \mathbf{\varepsilon = 0} )</th>
<th>( \mathbf{\varepsilon = 1.2} )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \mathbf{\text{for 300s}} )</td>
<td>0 s</td>
<td>30 s</td>
</tr>
<tr>
<td>Alloy M</td>
<td>Number density, ( \left(10^3\right) \text{(m}^2\text{)} )</td>
<td>5 ± 2</td>
<td>190 ± 19</td>
</tr>
<tr>
<td></td>
<td>Particle size (nm)</td>
<td>337 ± 50</td>
<td>268 ± 200</td>
</tr>
<tr>
<td>Alloy H</td>
<td>Number density, ( \left(10^3\right) \text{(m}^2\text{)} )</td>
<td>132 ± 22</td>
<td>577 ± 31</td>
</tr>
<tr>
<td></td>
<td>Particle size (nm)</td>
<td>144 ± 150</td>
<td>224 ± 200</td>
</tr>
</tbody>
</table>
Figure 6.6 The particle size distribution in (a, c) alloy M and (b, d) alloy H. (a, b) after reheating and non-deformed 1075 °C condition and (c, d) after a final strain of 1.2 at 1075 °C and subsequent holding for 0 s, 30 s and 300 s.

In general, a significant number of particles is present in alloys M and H covering a wide sizes (from ~20 nm to 2 µm) after ε = 1.2, 0 s at 1075 °C (Figures 6.5 e, f). In this condition, the number density of particles proliferates in alloy M by about 7 times (1312/190 = 6.9) in comparison with the to ε = 0, 1075 °C condition, while in alloy H the number density raises marginally (Table 6.2). Moreover, in alloy M the average particle size is reduced by half of that in non-deformed 1075 °C condition becoming 127 ± 90 nm, whereas it reduces slightly from 224 ± 200 to 204 ± 150 nm in alloy H. The growth of particles occurs in both alloys during holding for
30 s and 300 s after deformation (Figures 6.5 d, i, e, j, Table 6.2) and conversely, the particle density decreases during holding.

### 6.1.4 Substructure development and interaction of particles with dislocations and boundaries

Based on TEM examination, the NbC particles are divided into two categories: (i) relatively small NbC particles (~10 – 400 nm), which interact with boundaries and dislocations whereas (ii) large NbC particles (> 400 nm) assisting in DRX nucleation. The volume fraction and number density of particles were not attempted to be estimated from the TEM study due to limited and localised appearance of NbC particles. No particles in alloy L are detected in SEM and TEM studies.

Figure 6.7 delineates the substructure development in all three alloys, whereas Figure 6.8 shows the interaction of particles with dislocations and boundaries in alloys M and H. Bright-field TEM micrographs reveal a few random dislocations inside the grains in the ε = 0 condition (Figures 6.7 a, c, e) in all alloys and few NbC particles in alloys M and H. Some of the remnant dislocations from roughing deformation form dislocation arrays. At ε = 1.2, alloy L displays increased dislocation density and formation of subgrains (Figure 6.7b), however dislocation networks are not visible. This could be due to the following reasons: (i) as there are no carbides to restrict the movement of dislocations, their re-arrangement and annihilation is much easier than in alloys M and H; (ii) some of the observed grains in alloy L may have been after second deformation, e.g. recrystallised and then again deformed, as fraction of DRX is ~0.65.

At ε = 1.2 a complex substructure is developed in alloys M and H which is shown in Figures 6.7 d, f and Figures 6.8 a-d. Dislocation density in alloys M and H markedly increases in deformed grains and dislocation cells and subgrains are formed. The subgrain boundaries is still diffuse in nature and have dislocations attached, as well as some dislocations remain in the subgrain interior. The deformation structure in deformed grains in alloys M and H is inhomogeneous with higher dislocation density and more pronounced dislocation entanglement in the areas where carbides are present; tangles of dislocations, dislocations pile-ups, as well as clearly defined dislocation cell networks are observed (Figures 6.8 b-d).
Figure 6.7 TEM bright-field images of NbC particles and substructure development obtained with the beam direction close to [01\bar{1}] zone in (a, b) alloy L, (c, d) alloy M and (e, f) alloy H at 1075 °C with a strain of (a, c, e) $\varepsilon = 0$ and (b, d, f) $\varepsilon = 1.2$. The white circles locate the position of NbC particles.
Figure 6.8 Bright-field TEM micrographs of the specimen deformed to 1.2 and beam direction close to [01̅1] zone axis showing the substructure and interaction of different size NbC particles with dislocations and (sub)grain boundaries in (a, c, e) alloy M and (b, d, f) alloy H. (e) The restriction of a dislocation array by a 40 nm NbC particle and (f) interaction with a subgrain boundary by 130 nm and 240 nm NbC particles. The white circles locate the position of NbC particles and the arrows in the top right corner indicate the compression direction.
However, the areas with fewer particles have lower density and less complex dislocations arrangements, mostly displaying subgrain structure. The re-arrangement of dislocations and formation of subgrains is clearly delayed due to the restriction of dislocation mobility by the particles, as seen in Figures 6.7 d, f. Furthermore, it is clear from Figures 6.8 a-d that small NbC particles (< 100 nm size) interact with dislocations and pin them. They also restrict grain boundary mobility, by exerting a significant pinning force, which is evident from boundary curvature in the vicinity of the particles (Figure 6.8c). Arrays of parallel dislocations form subgrain and the subgrain boundaries also pinned by NbC particles as seen in Figure 6.8e. By contrast, coarse particles (>200 nm) play a different role. Due to the inhomogeneity of plastic deformation in the matrix and of a hard particle, a stress field surrounds the coarse particle. To accommodate it, the geometrically necessary dislocations are formed in the vicinity of the particles leading to the increase in dislocation density around them (Figure 6.8b). In addition, the dislocations gliding in the matrix meet the barrier to their movement in the form of coarse, non-deformable NbC particles and form pile-ups or dislocation loops (Figure 6.8a) which also leads to the generation of extra dislocations at particle/matrix interface, e.g. increase in dislocation density in the matrix neighbouring the coarse NbC. As a result, formation of subgrains in many cases starts in these high dislocation density areas near coarse carbides, as discussed later.

6.1.5 Nucleation mechanisms during DRX

The detailed observation of nucleation phenomena during dynamic recrystallisation is shown in Figures 6.9 and 6.10. The zoomed in view of optical micrographs in L, M and H alloys are illustrated in Figures 6.9 a, c, e where it is clearly observed that the grain boundaries are serrated and the DRX grains are formed along the pre-existing grain boundaries. The DRX nucleation near grain boundaries is further elucidated from the bright-field TEM micrographs in Figures 6.9 b, d, f. The dislocations preferentially gather near prior grain boundaries leading to bulging of the pre-existing boundaries (Figures 6.9b). Sometimes DRX nuclei are seen to originate from triple junctions (Figure 6.9b). Additionally, some subgrains are observed in deformed grain interior (Figures 6.9 d, f) which may at a later stage develop into DRX nuclei as was shown in Chapter 5 for DRX nucleation in alloy L.
Figure 6.9 (a, c, e) Optical micrographs and (b, d, f) bright-field TEM images, the beam direction close to the [01\overline{1}] zone axis, of the classical DRX nucleation mechanism in (a, b) alloy L, (c, d) alloy M and (e, f) alloy H after a final strain of 1.2 at 1075 °C.
On the other hand, a special type of DRX nucleation is detected on large (> 400 nm) NbC particles in alloys M and H. Extensive investigation of large particles in TEM explicitly reveals the process of particle stimulated nucleation during DRX which is delineated in Figure 6.10. First, the dislocations preferentially pile up on the interface of larger particles forming tangles and loops (Figures 6.10 a, b), then these dislocation arrangements develop into dislocation cells and subgrains.

Figure 6.10 STEM images, the beam direction close to the [01̅1] zone axis, delineating particle stimulated nucleation in (a) alloy M and (b-d) H after ε = 1.2, 0 s at 1075 °C. The red numbers on the images are the misorientation angles of the boundaries and black arrows in the inset represent the compression direction. The misorientation angles in (c, d) were determined using CBED patterns.
The interiors of these subgrains are relatively clean with few 1-3 dislocations and the misorientation of still diffuse subgrain boundaries at first is low, around 0.5° to 3°. These subgrains are pre-cursors for DRX nuclei. Further stage of DRX nuclei development is seen in Figures 6.10 c, d, where the boundary misorientation has increased to 5°-9° with the boundaries becoming narrower and well-defined. Thus, it might be suggested from the above results that PSN process can start at a smaller particle size ~430-440 nm and this process is more advanced at large particles >800 nm, as higher density of dislocations was probably developed there during deformation. This clearly shows the mechanism of PSN during DRX of model alloys, which is reported for the first time in this study.

6.1.6 Particle shape evolution with size

In-depth analysis of GBs, dislocation tangles and DDWs was carried out to locate any very fine particles as depicted in Figure 6.11. However, the smallest particle found in all deformation conditions was of ~10 nm size.

![Bright field TEM micrographs obtained with the beam direction close to the [01\seen] zone axis in (a) alloy L, (b) alloy M and (c) alloy H after deformation of 1.2. The zoomed in view of dislocation network, arrays and subgrain boundaries in inset of each image shows the absence of strain induced precipitation.](image)

The diffraction pattern and fast Fourier transform (FFT) obtained from the particles confirm NbC crystal structure and also show a cube-on-cube orientation relationship, (001)\seen NbC || (001)\text{γ}, [001]\seen NbC || [001]\text{γ} with the matrix (Figures 6.12 a, d, e, h). On the other hand, SAD patterns from 155 nm and 250 nm particles show that the orientation relationship between a particle and the matrix is lost at this size.
range of particles (Figures 6.12 f, h). A clear moiré pattern of the particles from 10 nm to 46 nm size and representative images are depicted in Figures 6.12 a-d with corresponding FFT shown in the inset. The interfaces of these particles were investigated to detect the shape of the NbC particles. The finest 10 nm particle has a nearly elliptical shape with only one reasonably defined facet parallel to $\{111\}_{\text{NbC}} \parallel \{111\}_\gamma$ (Figure 6.13b). The 17 nm size NbC particle is bounded by $\{111\}_{\text{NbC}} \parallel \{111\}_\gamma$ facets and later on truncated by $\{001\}_{\text{NbC}} \parallel \{001\}_\gamma$ facets (Figure 6.12a). However, a 26 nm particle in alloy M after deformation shows an additional planar facet parallel to $\{001\}_{\text{NbC}} \parallel \{001\}_\gamma$ and a distinct hexagonal shape (Figure 6.12b). With a further increase in particle size, the morphology gradually transforms to 14 sided tetra-kai-decahedron (Figure 6.12 h, i), which was also reported for NbC in a Ni-30Fe-Nb-C model alloy subjected to uniaxial compression at 925 °C [18].
Figure 6.12 STEM observation of different size NbC particles for the samples deformed to $\varepsilon = 0$ or $\varepsilon = 1.2$ in (a-d, f) alloy M and (g, h) in alloy H: (a-c) Detailed bright-field micrographs of (a) 17 nm, (b) 10 nm and 26 nm and (c) 46 nm faceted particles with the Fast Fourier transformed (FFT) pattern in the inset of each image at $\varepsilon = 1.2$. (d) A 130 nm NbC particle and the corresponding selected area diffraction pattern ($\varepsilon = 0$). (e) Pattern indexing revealing the $[01\overline{1}]_{\text{NbC}}$ zone axis and cube-on-cube orientation relationship between the $\gamma$ and NbC lattices, DD denotes the double diffraction. (f) A 155 nm particle without an orientation relationship to the matrix. (g) A 70 nm faceted particle at $\varepsilon = 1.2$. (h) Bright-field STEM image of a tetra-kai-decahedral particle enclosed by $\{100\}_{\gamma\text{NbC}}$ and $\{111\}_{\gamma\text{NbC}}$ planar facets and the corresponding SAD patterns with $[01\overline{1}]_{\text{NbC}}$ zone axis at $\varepsilon = 0$. (i) Schematic representation of the tetra-kai-decahedral particle shape.
The lattice fringe imaging [145] method was used to determine the type of interfaces (coherent, semi-coherent and incoherent) between particles and austenite matrix. Some representative lattice fringe images are illustrated in Figures 6.13 and 6.14. Interestingly, NbC particles up to about 26 nm size apparently show full coherency with the matrix; the number of lattice fringes in the matrix and particle are the same, e.g. the lattice planes of NbC/γ interfaces meet perfectly (Figure 6.13 a-c). The explicit hexagonal interfaces of NbC/γ parallel to both {111} and {001} planes also display full coherency with the matrix (Figure 6.13 b, c).

Figure 6.13 STEM HAADF images of NbC/γ interfaces of the samples deformed to 1.2: (a) from 10 nm and (b, c) from 26 nm particles observed in alloy M, bright-field image shown at Figure 12 a, b and (d) from 46 nm particle of alloy H. The lattice fringes are marked on a red box and the white lines in all images are the approximate location of interfaces.

128
The measured lattice parameter of all size range (10-400 nm) NbC particles is \(~0.443\) nm. Thus, it is extremely unexpected to maintain coherency between 10-26 nm NbC particles and austenite matrix as the difference in lattice parameters is about \(24\%\) (\(~0.356\) nm in matrix and \(~0.443\) nm for NbC). The interface must not be coherent in such large NbC particle, thus there should be misfit dislocations in the interface. However, the lattice fringe analysis did not reveal any misfit dislocation at the interface. Therefore, either the presence of moiré pattern conceals the misfit dislocations or the interface of 10-26 nm size particles is not exactly edge on edge (Figure 6.13d). The additional misfit dislocation should be visible if the interfaces are edge on as seen in the case of large particles described in the next paragraph. Based on the above, it is suggested that the interfaces of 10-26 nm particles must be semi-coherent with the matrix with periodic distribution of misfit dislocations in the matrix in every five lattice planes.

When the NbC size exceeds \(~46\) nm, extra lattice fringes observed in the austenite matrix reveal the presence of extra crystal lattice planes (Figure 6.14a), indicating existence of misfit dislocations at the interface. These misfit dislocations interfaces accommodate periodically distributed additional atomic planes in the austenite matrix separated by four to five matching lattice planes. Thus, the NbC/\(\gamma\) interfaces become semi-coherent (Figure 6.14a). After an increase of NbC particles size to 150 nm, all interfaces between particles and matrix display distinct facets (Figure 6.14b) and still maintain semi-coherency. The analysis of lattice fringes of larger particles (>150 nm) by moiré pattern was attempted to determine the coherency among the interfaces. Unfortunately, the lattice fringe analysis was not possible at larger particles interface as the contrast between the particles and the matrix interface was blurry and did not produce any moiré pattern. However, the SAD patterns from the large particles in Figures 6.12 f, h indicate that these size range particles lost orientation relationships (cube-on-cube) with matrix. Moreover, it is also observed from Figures 6.8 b, d and Figure 6.10a that dislocations are gathered around large particles and form cell structure due to incoherency between particles and matrix interface. Therefore, the interfaces of the NbC particles above 150 nm are believed to be incoherent with the austenite matrix.
Figure 6.14 STEM HAADF and corresponding lattice images of NbC/γ interfaces in alloy H at ε = 0: (a) two types of interfaces (facets and oblique) on a 67 nm particles (b) The interfaces of \{100\}_{\gamma}^{\text{NbC}} and \{111\}_{\gamma}^{\text{NbC}} planar facets in a 150 nm particles. The locations of additional lattice planes are denoted by the red dotted lines in lattice images while the white dotted lines are the approximate location of the interface.
6.2. Discussion

6.2.1 Effect of the alloy composition and processing on NbC precipitation

The calculated solubility temperature (Section 3.2) in alloys M and H is higher than the melting point of Ni-30Fe alloy (nearly 1420 °C) [44]. Therefore, only 0.40 wt. % of Nb is in solution and the remaining Nb is present as NbC in alloys M and H after reheating to 1250 °C, while all Nb is in solution in alloy L due to solubility temperature being 1200 °C (Table 3.2). The latter is confirmed by SEM and TEM observations in the absence of any NbC particles in alloy L (Figures 6.5, 6.7 and 6.11). However, the maximum solubility of Nb and C at 1075 °C is nearly 0.20 (wt. %) and 0.025 (wt. %) respectively in Ni-30Fe matrix. Thus, a significant fraction of NbC precipitates after roughing and quenching from deformation temperature which is confirmed by an increase in particle number density (Table 6.2) in alloys M and H. However in alloy L, NbC particles were not observed even after deformation. The absence of NbC in alloy L in all conditions might be because the nose temperature (~1050 °C) is less than the deformation temperature (1075 °C) (Figure 3.2b). The precipitation of metal carbides is delayed if the cooling curve of thermo-mechanical processing does not cross the nose of the time-temperature-transformation curve [2]. Thus it is believed in the present situation that 300 s is not adequate to precipitate NbC in alloy L.

Most of small particles in alloy M are dissolved as the dissolution temperature is less than that of alloy H hence some large particles with few small particles have contributed to a very low number density as well as larger average particle size in alloy M after reheating (Table 6.2). A large standard deviation of the average particle size in alloy H is measured which possibly is due to its wide range of particle sizes. Moreover, there is a discernible difference in the solute content present in the particles of alloys M and H. At 1250 °C, 0.45 wt. % of Nb + 0.063 wt. % C forms the NbC particles in alloy M while 1.020 wt. % of Nb and 0.136 wt. % C contributes to the NbC particle formation as predicted from the solubility calculation (Table 3.2). This factor may lead to a higher number density of particles in alloy H than in alloy M.

The characterisation of different types of NbC particles is extremely complex because NbC particles may dissolve during cooling to 1075 °C and at the same time
particles may precipitate as the solubility temperature is above the reheating temperature of 1250 °C. Since the solubility decreases with a decrease in temperature, the particles are expected to form during cooling to deformation temperature.

Continuous precipitation during cooling to deformation temperature greatly increases the particle number density in both alloys M and H (Figures 6.6 c, d and Figures 6.6 a, b) in $\varepsilon = 0$, 1075 °C condition. The increase in number density of particles in alloy M is more pronounced than in alloy H which might be contributed by the presence of more new small precipitates in alloy M whereas both pre-existing and new particles constitute the particles distribution in alloy H. The average particles size in alloy M is almost half of the average particle size of alloy H due to a greater contribution of small precipitated particles in alloy M.

The trend of proliferation of particles number density in alloy M continues at $\varepsilon = 1.2$, 0 s as NbC particles are consistently forming from supersaturated solid solution, possibly strain-induced ones. By contrast, a steady increase in particle number density of alloy H can be ascribed to the precipitation of new particles accompanied by the growth of pre-existing particles. It is well established that the SIPs preferably nucleate on microbands or cell walls, dislocation arrays and dislocation tangles to reduce the strain energy by replacing dislocation sections [17, 19-21]. Most of the studies on strain induced precipitation were conducted at 900 – 950 °C [4, 111, 112, 162] with a strain rate of 1s$^{-1}$ [17, 18] or 10 s$^{-1}$ [4]. The strain induced precipitates grow very fast, the particles turn to 5 nm within 10 s holding after $\varepsilon = 0.2$ deformation at 925 °C as reported by Poddar et al. [17]. In the current study, the deformation temperature is 1075 °C at a lower strain rate of 0.1 s$^{-1}$. Thus, it is believed that SIPs might be precipitated in the early stage of deformation (e.g. at $\varepsilon = 0.1-0.3$) and progressively coarsen during further deformation in both alloys M and H. Therefore, the combined effect of higher deformation temperature, slower strain rate (longer time of 12 s) and higher deformation strain is responsible for the absence of very fine NbC precipitates (<10 nm). Besides, fast coarsening rate of NbC particles is also due to the contribution from accelerated pipe diffusion, as diffusivities of both Nb and C for pipe diffusion are faster than those for the bulk diffusion (e.g. the activation energy of pipe diffusion of Nb (210 KJ/mol) is lower than the activation energy of bulk diffusion (270 KJ/mol)) [4, 111-113]. However, it is difficult to say whether SIPs took place or not without following the evolution of
precipitation as a function of strain at this high temperature, as both coarsening of NbC and re-arrangement/annihilation of dislocations take place very fast. The average particles size in alloy M is almost half of the average particle size of alloy H due to a greater contribution of small precipitated particles in alloy M.

During holding for 30 s, the particle number density is slightly decreased in alloy H comparing to a drastic decline of number density to nearly half in alloy M. The coarsening process in alloy M, simultaneously leading to an increase in the mean particle size and a decrease in the particle number density. By contrast, the coarsening of precipitates was a constant feature during processing of alloy H due to the presence of pre-existing NbC particles and it is prevalent at the beginning of processing although precipitation of new particles is persistently occurring as the solid solution still contains a lot of solute elements. Further holding for 300 s increases the average particle size in both alloys M and H albeit small increase in particle number density in alloy H possibly by error in sampling. Therefore, in the high density particles condition and super saturated solid solution, the continuous precipitation process is probably taken over by the particle coarsening phenomena.

6.2.2. Precipitate and solute interaction with dislocations and boundaries

Solute elements have significant influence on the boundary migration and dislocation motion and even a small amount of solute atoms may reduce their mobility by several orders of magnitude [6]. At a high level of solute concentration, the diffusivity of solute atoms controls the kinetics of boundary movement. In the present study, alloys L, M and H have a very high concentration of solute in the solid solution. As the Nb atom has a higher atomic radius (0.145 nm) [193] than those of two matrix elements pure Ni (0.135 nm) and pure Fe (0.140 nm), it may introduce lattice strain in the solid solution and hinder the mobility of boundaries and dislocations. Moreover Nb as a solute atom has a very strong effect on retarding the movement of boundaries [194]. Although solute atoms might not pin the boundaries, they can reduce the driving force for DRX. However, the solute dragging effect is a strong function of temperature as the increase of temperature greatly enhances the diffusivity of the solute atoms [195], thus it is easier for recrystallisation to occur.

The lattice parameter of a NbC crystal is ~0.443 nm [196] which is greater than ~0.356 nm of Ni-30Fe matrix [47]. Thus NbC particles exert a significant
amount of force on the boundary by creating a large positive misfit between NbC precipitates and austenite matrix and the dislocations are repulsed by the force acting from the particles but during deformation form pile-ups and/or loops around particles. Therefore, the precipitates located at the boundaries and in the grain interior, as seen from Figure 6.8, impede the dislocation and boundary mobility.

It is well known that the coherent interface has a low interfacial energy (up to about 200 mJ m$^{-2}$), hence smaller NbC particles at early stage of precipitation tend to keep coherency with austenite matrix [197]. The investigation of strain induced NbC particles in Fe-30Ni-Nb-C alloy at 925 °C showed a rapid loss of coherency in only 5-8 nm particle size [17]. But 10 nm particle in present study is large enough to show extra planes of misfit dislocations in the matrix and these misfit dislocations are believed to be concealed in the moiré pattern. Otherwise, the matrix could not adjust ~24 % of misfit between matrix and particles as elastic strain. The semi-coherency of interfaces between matrix and particles remains up to 150 nm size particle. It is well accepted that the elastic strain energy increases with an increase in particles size and when the elastic energy at the interface becomes excessively high, extra misfit dislocations at the interface accommodate some of the elastic energy [197]. It can be concluded from the above discussion that the 10 nm particles are semi-coherent and NbC particles maintain semi-coherency up to 150 nm size, whereas above 150 nm the particles became incoherent with the matrix.

The prominent feature of the particles is their shape which changed from nearly elliptical to faceted. This change in shape and coherency of NbC particles based on the size is associated with gradual decrease in the influence of interfacial energy over elastic strain energy [198-200]. 10 nm NbC precipitate is nearly elliptical shape with barely identified $\{111\}$ facets (Figure 6.12b), which changes to tetrahedral with well-defined $\{111\}$ facets in 17 nm (Figure 6.12a) precipitate. Then distinct $\{001\}$ facets appear on hexagonal NbC particles (>26 nm) and finally the particles (>160 nm) are transformed to a tetra-kai-decahedron. This later change of the particle shape is associated with the lower boundary energy of $\{001\}$ facets which gradually develop into $\{111\}$ planar facets [199, 201]. The observed sequence of shape change is in line with the literature [17, 199].
6.2.3 Flow behaviour, softening mechanism and microstructure evolution

The flow stress increase in alloys M and H corresponding to the amount of solute elements present is associated with the density and shape of NbC particles and some influence from the solute atoms. The NbC particles present before deformation significantly restrict the mobility of dislocations and boundaries, which increases the flow stress from alloy L to alloy M to alloy H. Since, dislocation glide and climb is hindered as the particles obstacle the movement of dislocations, the rate of DRV via dislocation annihilation is slow. Thus the dislocation density at early stage of DRX process rapidly raised to critical dislocation densities to nucleate DRX grains which is evident by a decrease in critical strain (Figure 6.1) in alloys L to M to H. The presence of high density NbC particles in alloys M and H after deformation (Figures 6.5 c, h and Figures 6.7 d, f) limit the grain boundary and dislocation mobility hence the softening via DRX results in a broad stress flow curve without much drop in peak stress in those alloys.

The refined grain size in alloys M and H after reheating is due to restricted grain boundary movement by NbC at this high temperature (1250 °C) (Figures 6.2 a-c) leading to much finer grains compared to those in alloy L. Recrystallisation after roughing is also more pronounced in the microstructure with finer grains resulting in a finer grain size [63] at ε = 0, 1075 °C condition in alloys M and H than in alloy L (Figures 6.2 d-f). It is well accepted that finer starting grain size accelerates DRX process owing to a higher fraction of pre-existing grain boundaries as DRX initiates from prior grain boundaries [56]. In addition, the stored energy of plastically deformed fcc alloys containing second phase particles is higher than that of the same alloys without particles [6] and promotes the initial stage of DRX. On one hand, the rapid increase in stored energy leads to initiation of the DRX process in alloys M and H at lower strains than in alloy L. On the other hand, second phase particles and solute atom hinder the motion of dislocations and grain boundaries by pinning which delays the progress of DRX (Figures 6.8 a-d). The competition between softening and hardening is regulated by the precipitates size, their number density, particle distribution and solute Nb in solid solution.

Due to the rapid development of strain gradients in the vicinity of grain boundaries, the DRX process starts with the nucleation of new grains at prior grain boundaries by bulging [54, 56, 63, 148, 202]. This is in full agreement with the
discontinuous DRX nucleation phenomenon (Figure 6.9) and growth by the strain-induced boundary migration (SIBM) mechanism proposed by Beck and Sperry [78]. Unlike the classical DRX mechanism process where dislocations are mainly gathered in the vicinity of pre-existing grain boundaries due to higher incompatibilities, the high dislocation density configurations have also developed in grain interiors when a large number of relatively small size particles (<150nm) do exist (Figure 6.8 a-d). This leads not only to a higher work hardening rate and peak stress in alloys M and H compared to alloy L, but also to a less pronounced rate of softening.

Despite the operation of an additional nucleation mechanism during DRX of alloys M and H- PSN at NbC particles with >400 nm size as described earlier, DRX and correspondingly softening are significantly less pronounced in these alloys due to a high Nb content in solution and a high density of NbC particles. No microbands are seen in any of the studied alloys deformation at 1075 °C contrary to the reports on microband formation in model alloys deformed at 900 – 950 °C [5, 17, 56]. Faster dislocation mobility and rapid disintegration of microbands at elevated temperature might be the reason for their absence after final strain of 1.2 in the studied alloys [15]. The dramatic increase in particle number density in alloy M at ε = 1.2, 0 s is associated with a high supersaturation of Nb solute in the solid solution and possibly contribution from SIPs of NbC at the beginning of deformation and these particles readily coarsened to >10 nm size at the final stage of deformation. A large number density of NbC in both alloys, especially fine ones <100 nm, restricted the progress of DRX and thus not much decrease in flow stress was observed compared to alloy L (Figure 6.1a).

During holding after deformation, the microstructure in alloy L is completely recrystallised through MDRX as MDRX process is fast and does not need incubation time (Figure 6.2)). The rapid increase of recrystallised grain size in alloy L during further holding is due to negligible restriction of grain boundary mobility, although the solute Nb atoms exert a minor dragging force. Post deformation softening in alloys M and H is remarkably sluggish and only a small increase in recrystallised fraction occurred via MDRX and growth of pre-existing DRX grains. Nb is one of the most influential elements to retard dynamic recrystallisation by solute drag, strain induced precipitation or presence of precipitation before deformation [108, 109, 203]. Although, NbC particles above 400 nm size stimulate DRX nucleation, a high density of small NbC particles plays the dominant role by pinning the dislocations
and by retarding the high angle grain boundary mobility, by both fine Nb carbides and Nb solute atoms leading to the cessation of DRX and MDRX processes as well as the DRX grain growth. As more NbC and Nb in solute are present in alloy H, it displays the highest flow stress, the slowest softening and the finest grain size among the three studied alloys.

6.3 Conclusions

The extensive studies of alloys L, M and H using scanning transmission microscopy and scanning electron microscopy after thermo-mechanical processing in a Gleeble-3500 simulator were conducted and the results could be summarised as follows.

- The flow stress is increased from alloy L to M to H with an increase in Nb content. The critical strain for DRX is decreased rapidly from alloy L to alloys M and H due to the presence of adequate amount of NbC precipitates assisting the rapid work hardening.
- The DRX in alloy L is well progressed after $\varepsilon = 1.2$ while only ~0.25 fraction is achieved because the DRX is retarded by NbC precipitates in M and H alloys. During holding for 30 s, alloy L is completely recrystallised via MDRX whereas there is a little increase in DRX + MDRX fraction in alloys M and H. During further holding to 300 s, the recrystallised grains are grown in alloy L whereas the grain growth of DRX grains in alloys M and H is restricted.
- Particle stimulated nucleation was observed at coarse (>400nm size) NbC particles during DRX following the sequence of build-up of dislocations at NbC/$\gamma$ matrix interface→ formation of dislocation cells→ development of subgrains→ appearance of dislocation-free DRX grains.
- The NbC particles up to 150 nm show cube-on-cube orientation relationship with matrix $(001)_{NbC} || (001)_{\gamma}$, $[001]_{NbC} || [001]_{\gamma}$ . The morphology of NbC particles changes from elliptical shape to octahedral faceted to tetra-kai-decahedral by developing $\{001\}$ facets over $\{111\}$ facets. The interfaces of NbC particles tend to maintain semi-coherency up to 150 nm size while orientation relationship is lost above 150 nm size particles.
This study was aimed to investigate the dynamic recrystallisation phenomena and the interaction of particles and solute with dislocations and grain boundaries during hot deformation. The extensive literature review was conducted explicitly to summarise the existing research on hot deformation behaviour of austenite and to elucidate some of the unresolved issues. Several thermo-mechanical processing schedules were conducted in a wide dynamic recrystallisation processing window for a range of Nb alloying addition to Ni-30Fe model alloys. The investigation of the evolution of microstructure and micro-texture during hot deformation was conducted for the alloy with the lowest Nb content. This Chapter recapitulates the general conclusions, key contributions to the original knowledge and also suggests some potential directions for future study.

7.1 Conclusions

The following effects of processing parameters and Nb content on DRX flow behaviour during thermo-mechanical processing of Ni-30Fe model alloys were observed:

1. The flow curves in alloy L₀ readily exhibit stress peak with a distinct steady state region at 1150-1000 °C and 0.01-1 s⁻¹ strain rate. The stress peak on the flow curve of alloy L is broadened and unable to reach the steady stress state. Further increase in Nb content in alloys M and H (0.85 and 1.42 wt. %) produced a parabolic flow curve instead of a typical single peak DRX flow curve at 1075 °C and 0.1 s⁻¹ strain rate due to retarded dynamic recrystallisation process.

2. The flow stress increases with an increase in deformation temperature or a decrease in strain rate. The flow stress is trending upward with an increase in Nb content in model alloys, e.g. the flow stress in alloy L is higher than that in the alloy L₀. The critical and peak stresses and strains are seen increased with a decrease in deformation temperature and/ or a raise in strain rate, e.g. with an increase in the Zener-Hollomon parameter. The critical stress for DRX nucleation is decreased in alloys M and H with higher Nb content.
3. The correlations between the critical and peak values for both stress and strain are derived as: $\epsilon_c = 0.67 \epsilon_p$ in both L and L$_0$ alloys and $\sigma_c = 0.83 \sigma_p$ in alloy L and $\sigma_c = 0.65 \sigma_p$ in alloy L$_0$.

The evolution of dynamic recrystallisation microstructure and micro-texture and the influence of particles and solute Nb on the kinetics of DRX is summarised as:

1. Due to the inhomogeneity of plastic deformation there is an increase in dislocation density in the vicinity of grain boundaries subsequently leading to boundary serrations and bulging. The sub-boundary closure of the bulged region on grain boundary was the predominant mechanism of DRX nuclei formation, although sometimes annealing twins also formed behind the bulge. Formation of some DRX nuclei was also observed at pre-existing annealing twins. New DRX grains continuously consume elongated deformed grains and replace them by the refined, dislocation-free equiaxed grains. The fraction of dynamically recrystallised grains is directly proportional to the deformation temperature and inversely proportional to the strain rate. Although the DRX process is delayed by the presence of Nb solute in alloy L, semi-coherent NbC particles in alloys M and H significantly suppress the DRX. Only a recrystallised fraction of 0.25 was achieved in alloys M and H at the same deformation condition compared to fraction of 0.67 in alloy L after $\varepsilon = 1.2$.

2. Large NbC particles (> 400 nm size) promote nucleation of DRX grains by particle stimulated nucleation on these particles. The density of dislocations first increases near coarse particles, then they re-arranged to form subgrains with low angle boundaries. Gradually the interior of subgrains becomes free of dislocations, the low angle boundaries become sharp and their misorientation increases finally resulting in new DRX nuclei.

3. The recrystallisation process in L alloy is completed by metadynamic recrystallisation during 30 s holding after deformation and further holding to 300 s leads to DRX grain growth. However, M and H alloys show a limited metadynamic recrystallisation and the growth of equiaxed grains is sluggish in these alloys during holding after the deformation. This DRX and MDRX retardation behaviour in these alloys is associated with a large number of
NbC particles present which restrict the mobility of dislocations and grain boundaries.

4. The predicted volume fraction of DRX determined from experimental observations (optical microscopy and EBSD) is smaller, especially at a lower deformation temperature, than the DRX volume fraction calculated using the Avrami-type equation. The difference in volume fractions can be ascribed to the dominant DRV over recrystallisation at lower deformation temperatures. In addition, the continuous accumulation of dislocations in DRX grains during deformation led to underestimation of DRX fraction in EBSD examination.

5. Micro-texture measurement after interrupted plane strain compression tests at 1075 °C in alloy L returned a weak DRX texture at an early stage of recrystallisation. The strong cube texture dominates at the final stage of DRX as the C orientation possesses the lowest Taylor factor (or low stored energies) when calculated using octahedral slip systems for fcc materials. Annealing twins contribute to dynamic recrystallisation acting as DRX nucleation sites from the beginning of nucleation at ε = 0.23. The population of annealing twin boundaries rises with the progress of DRX to ~30% of the total grain boundaries area fraction. However, these annealing twins have preference for the formation in non-cube oriented grains leading to the relatively low density of cube-twin orientations.

6. The NbC particles in alloys M and H are observed in all deformation conditions and the changes during development of these particles were tracked. The NbC particles <150 nm size display the cube on cube orientation relationship to austenite matrix (001)_{NbC}|| (001)_γ, [001]_{NbC}|| [001]_γ while large particles >150 nm size have no orientation relationship with the matrix. The minimum detected particle size is close to 10 nm, which already has a {111} faceted interface. The smaller particles have {111} faceted interface while {001} facets gradually develop in larger NbC particles. Based on the literature, the formation of {111} facets at earlier stages is related to their close-packed character and lower mobility, whereas appearance of {001} facet is associated with their lower energy compared to that of {111} facets. This change the particles shape to octahedron to tetra-kai-decahedral. The
interfaces between NbC and austenite matrix maintain semi-coherency up to 150 nm size with additional planes in austenite matrix and misfit dislocations at the interface.

7.2 Contributions to original knowledge

The following key contributions have been made to further elucidate the DRX behaviour of low to medium SFE model alloys based on the extensive experiments in DRX temperature interval and using advanced experimental techniques:

- This is one of the first studies to provide the insight into the dynamic recrystallisation kinetics in model alloys with a wide range of Nb concentrations. These extensive studies suggest that for the same deformation conditions the DRX kinetics is impeded by the addition of Nb solute and the DRX and MDRX phenomena are significantly suppressed in the presence of a high density of semi-coherent NbC particles.

- The deformation activation energy is calculated by an Arrhenius-type equation and an increase in deformation activation energy has been noticed with the addition of Nb from 362 KJ/mol in alloy L₀ to 419 KJ/mol in alloy L. These values of deformation activation energy are in a good agreement with the existing literature data. Based on the extensive literature review and incorporating the present experimental results, it is suggested that the deformation activation energy during DRX is not only a function of the total amount of solute present but also of the number of solute elements. For the first time it is suggested that for the same alloying level, the wider the diversity of alloying elements the higher the deformation activation energy.

- The nucleation mechanism during DRX has been studied by advanced experimental techniques such as electron back scattering diffraction and high resolution TEM. The investigation by these two complementary techniques allows to obtain not only statistically valid information, but also to clarify the details of the early stage of DRX. At low strain, DRX nucleus results from the serration of pre-existing grain boundaries followed by low angle boundaries and twin boundaries enclosure of the bulged regions. Then the DRX nuclei form a necklace-like layer at grain boundaries. This is in agreement with the accepted viewpoint [86] on the discontinuous mechanism.
of DRX nucleation. However, a new insight into DRX nucleation at higher strains was obtained. In the case of alloy L, new DRX nuclei also emerge within the interior of deformed grains by the gradual development of dislocation cells into subgrains and finally into DRX grains. Although the DRX fraction in alloys M and H is about 0.25, DRX nuclei inside deformed grains have also originated. The particles in these two alloys impede the motion of dislocations leading to earlier achievement of the critical dislocation density for DRX nucleation.

- This is one of the first studies to describe the evolution of DRX microstructure using the interrupted experiments at different strains along the DRX flow curve. The origination of initial necklace-like layer is well-established and the present result also confirms this. However, the development of microstructure at high strain is accompanied by successive necklace layer expansion as well as cell and subgrain formation in deformed grains interior. Growth of DRX grains occurs via strain induced boundary migration.

- For the first time, the micro-texture development during DRX is studied using EBSD methodology which involved the segmentation of partially recrystallised maps into recrystallised and unrecrystallised fractions using a grain orientation spread criterion. At an early stage of deformation, the DRX texture is rather weak and mainly consists of cube \{001\}(100), cube-RD \{013\}(100) and cube-ND \{001\}(310) orientations. Cube oriented grains advantageously nucleate at an incipient stage of DRX and this cube orientation further sharpens with the progress of DRX, finally producing a strong cube oriented microstructure comprising both recrystallised and deformed after recrystallisation grains. The dominance of the cube orientation in the secondary deformed and recrystallised grains can be ascribed to its low stored energy and high orientation stability.

- The present study is the first to investigate the mechanism of particle stimulated nucleation during DRX in Ni-30Fe-Nb-C model alloys. Dislocations during hot deformation are generally accumulated around incoherent NbC particles/matrix interfaces hence the dislocation density in the vicinity of particles raises to a critical level to form cell structure and
nucleate new DRX grains. It was also discovered that PSN could start to form pre-cursor of DRX nuclei on smaller particles of ~400 nm.

- This is one of the limited numbers of studies to characterise different size NbC particles and their evolution with size. It confirmed that with a size increase the NbC particles change shape from nearly elliptical to octahedral with \( \{111\} \) facets and then to multi-faceted tetra-kai-decahedral by growing \( \{001\} \) facets on \( \{111\} \) facets. This process is dictated by both boundary energy and kinetics considerations. Furthermore, in depth crystallographic investigation of the NbC precipitate/matrix interface showed that the particles from ~10nm to ~150nm size maintain semi-coherent interfaces and the cube-on-cube orientation relationship with austenite.

### 7.3 Future directions

The dynamic recrystallisation behaviour of the model alloys has been studied by scanning electron microscopy and scanning transmission electron microscopy. Some aspects of future studies on this model alloy are given below:

In the current work the interrupted strain tests were performed only on the alloy L. Interrupted strain tests along the flow curve in alloys M and H to identify the effect of initial NbC presence on microstructure and micro-texture development are warranted.

It would be also advantageous to carry out deformation at higher temperature or at higher strain than the one used in the current study to obtain a higher recrystallised fraction in alloys M and H. As all attempts to find strain-induced precipitates in alloy L failed, different processing schedules could be designed for further work to elucidate strain-induced precipitation in this alloy.

The present study was conducted on austenitic model alloys to observe high temperature austenite microstructure preserved to room temperature. The texture observed in the current experiments is micro-texture albeit a coarse 10 \( \mu \)m step size used in EBSD for increased statistics. Bulk texture measurement in X-ray diffraction or by synchrotron could be beneficial to elucidate the evolution of bulk DRX texture.

It has shown in the results that the DRX kinetics is retarded in the presence of Nb solute in the Ni-30Fe-C matrix even in the absence of precipitated particles. The current characterisation techniques did not quantify the effect of solute atoms in the
matrix although the solute concentration is very high. The solute segregation at grain boundaries and solute clustering can be examined by atom probe tomography which might be useful to illustrate the retardation mechanism of DRX by solute atoms.

In the current work the initial stage of PSN was detected during DRX. It would be interesting to follow up on its progress during holding after deformation using both high resolution STEM and EBSD.
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155


