

THESIS FOR THE DEGREE OF DOCTORATE OF PHILOSOPHY

Dynamic and meta-dynamic recrystallization of Ni-based superalloy Haynes 282

Emil Eriksson



Department of Physics

CHALMERS UNIVERSITY OF TECHNOLOGY

Göteborg, Sweden 2021

Dynamic and meta-dynamic recrystallization of Ni-based superalloy Haynes 282
Emil Eriksson

© Emil Eriksson, 2022.

ISBN 978-91-7905-726-8

Doktorsavhandlingar vid Chalmers tekniska högskola

Department of Physics
Chalmers University of Technology
SE-412 96 Göteborg
Sweden
Telephone + 46 (0)31-772 1000

Cover illustration

The illustration on the cover shows the microstructural evolution of Ni-base superalloy as it progresses from consisting of large grains to fully recrystallized. Each map is acquired from different strains, where the upper most map is the original microstructure. Recrystallized grains are nucleated at grain boundaries, and progressively replace the original grains as deformation progresses. In the end, the microstructure is completely replaced and now consists of new, small grains.

Printed by Chalmers Reproservice
Göteborg, Sweden 2021

Abstract

Thermomechanical processes are a crucial manufacturing step because they can “reset” the microstructure, and set the starting point for all following steps. In turn, the microstructure can be used to tailor the mechanical properties of the material. It is therefore of great importance to understand how deformation parameters affect the resulting microstructure. The mechanism responsible for this “reset” of microstructures is recrystallization, where the thermal energy and internal stored energy drive the creation of new, deformation-free, grains at the expense of the deformed ones. However, recrystallization is a complex phenomenon affected by alloy composition, temperature, strain, strain rate etc.

In the work presented here, the dynamic, and meta-dynamic recrystallization mechanisms occurring in Ni-base superalloy Haynes 282 are investigated, both below and above the secondary carbide solvus temperature (1100 °C) at various strains, strain rates and post-deformation holding times. Discontinuous dynamic recrystallization, with a clear nucleation of grains at grain boundaries, was observed to be the dominating recrystallization mechanism. For strains up to 0.8 the increase in recrystallized fractions stemmed from nucleation of new grains, whereas for larger strains continued increase in recrystallized fractions was caused by grain growth. Particle stimulated nucleation, where MC carbides acted as nucleation sites, was also observed. Carbides located at grain boundaries did not affect the recrystallization progression significantly. During deformation, the strain rate was seen as the governing factor on the final microstructure, while temperature, strain and holding times were the dominating parameters affecting the meta-dynamic recrystallization. Larger strains led to shorter times to reach a fully recrystallized microstructure during a post-deformation hold. The average grain size also decreased with higher strains applied prior to a static hold.

Keywords: Ni-base superalloys, dynamic recrystallization, meta-dynamic recrystallization, EBSD, hot compression

Preface

The research work presented in this thesis was carried out at the Division of Microstructure Physics at the Department of Physics, Chalmers University of Technology, during the time period March 2018 - October 2022, under the supervision of Associate Professor Magnus Hörnqvist Colliander.

The funding for the present work has been provided by the Swedish Agency for Innovation (VINNOVA), through the Swedish National Aeronautical Research Program (NFFP) grant no. 2017-04863, in collaboration with GKN Aerospace Engine Systems AB.

List of appended papers

- I. *Dynamic and Post-Dynamic Recrystallization of Haynes 282*
Emil Eriksson, Magnus Hörnqvist Colliander
Metals 11 (2021): 122
- II. *The Effect of Grain Boundary Carbides on Dynamic Recrystallization During Hot Compression of Ni-Based Superalloy Haynes 282 below the Secondary Carbide Solvus*
Emil Eriksson, Joel Andersson, Magnus Hörnqvist Colliander
Metallurgical and Materials Transactions. A 53 (2022): 29
- III. *Dynamic recrystallization during hot compression of Ni-based superalloy Haynes282*
Emil Eriksson, Fabian Hanning, Joel Andersson, Magnus Hörnqvist Colliander
Submitted to Journal of Alloys and Compounds, in review
- IV. *Meta-dynamic recrystallization of Ni-based superalloy Haynes 282*
Emil Eriksson, Fabian Hanning, Joel Andersson, Magnus Hörnqvist Colliander
In manuscript.

My contributions to the appended papers

- I. I developed the sample matrix, performed the Gleeble experiments, the EBSD analysis and evaluated the data with the help of my supervisor, and was the lead author.
- II. I performed the Gleeble experiments, the EBSD analysis and evaluated the data, and was the lead author.

- III. I performed the Gleeble experiments, the EBSD analysis and evaluated the data, and was the lead author.
- IV. I performed the Gleeble experiments, the EBSD analysis and evaluated the data, and was the lead author.

Acknowledgements

My deepest gratitude goes out to my supervisor Dr. Magnus Hörnqvist Colliander for his tremendous support. I am grateful that you chose me as the student for this project, for the time you take and the patience you show during our discussions. Your supervision and guidance helps me to better tackle my results from logical, thorough and academic perspectives. I thank the people at GKN Aerospace AB, and Ceena Joseph as project supervisor especially. Your support and knowledge on superalloys has helped me a lot with my work. I also thank Uta Klement, Yiming Yao, Adrianna Lozinko and Xiaolong Li at the department of Industrial and Materials Science for access and help with sample polishing and handling the muffle furnace, Kjell Hurtig and Fabian Hanning at University West for their many hours of support with the Gleeble experiments and of course the staff at the Chalmers Material Analysis Laboratory who taught me to operate all electron microscopes I have used. A special thanks to Ola Löfgren for his quick help in regards to computer issues. I am also grateful for all my colleagues at my division, Eva Olsson Group and the CMAL staff who were there for discussions, and coffee breaks and that you created a friendly work environment that was encouraging during the more stressful times most PhD students experience at one point or another. A special thanks to Anand who taught me how to perform EBSD analysis, and Kristina who taught me the ropes when I started. I am grateful for my mother and late father who always supported my decisions in life and who continue to do so to their best extent. You of course hold the main responsibility in what kind of person I turned out to be, and I think (and hope) many agree that you did a good job of it. I also extend a thank you to my personal trainer, who helped me maintain a healthy body, after hours of sitting at a desk.

To my Teachers, Parents and Friends

Contents

1	Introduction	1
1.1	Background and motivation	1
1.2	Aim of the study	3
2	Microstructure and processing of Ni-base superalloys	5
2.1	Ni-base superalloys	5
2.2	Thermomechanical processing	10
2.3	Forging	11
3	Microstructure evolution during hot working	15
3.1	Work hardening and dynamic recovery	16
3.2	Recrystallization	19
3.2.1	Dynamic recrystallization	21
3.2.2	dDRX nucleation mechanisms	24
3.2.3	Meta-dynamic recrystallization	28
3.3	Recrystallization in Ni-base superalloys	31
4	Methods	35
4.1	Hot deformation	35
4.2	Microstructural investigation	42
4.2.1	Electron Channeling Contrast Imaging	43

4.2.2	Electron Backscattered Diffraction	44
5	Results	51
5.1	Initial structure	52
5.2	Mechanical response during deformation	52
5.3	Stress relaxation during hold	56
5.4	Dynamic recrystallization	58
5.4.1	Kinetics	61
5.4.2	Nucleation of recrystallized grains	62
5.4.3	Effect of deformation parameters	65
5.4.4	Effect of secondary phases	68
5.5	Meta-dynamic recrystallization	72
6	Conclusions & Outlook	83
6.1	Conclusions	83
6.2	Outlook	84
	Bibliography	87

Acronyms

AH adiabatic heating

APB antiphase boundary

γ_{APB} APB energy

BSE backscattered electrons

bct body-centered tetragonal

cDRX continuous dynamic recrystallization

CSL coincident site lattice

dDRX discontinuous dynamic recrystallization

DRV dynamic recovery

DRX dynamic recrystallization

EBSD electron backscatter diffraction

EBSP electron backscatter pattern

ECCI electron channeling contrast imaging

EDM electric discharge machining

EDS dispersive X-ray spectroscopy

fcc face-centered cubic

GB grain boundary

GNDs geometrically necessary dislocations

GOS grain orientation spread

GROD grain reference orientation deviation

HAGB high angle grain boundaries

hcp hexagonal close-packed

HT heat treatments

IPF inverse pole figure

KAM kernel average misorientation

K-M plot Kocks-Mecking plot

LAGB low angle grain boundaries
MAD mean angular deviation
mDRX meta-dynamic recrystallization
pDRX post dynamic recrystallization
PSN particle stimulated nucleation
 ε strain
SE secondary electrons
SEM scanning electron microscope
SF stacking fault
 γ_{SFE} stacking fault energy
SIBM strain induced boundary migration
 $\dot{\varepsilon}$ strain rate
SSS solid solution strengthening
SRX static recrystallization
 T temperature
TCP topologically close-packed
TEM transmission electron microscopy
TMP thermomechanical processing
WH work hardening
WQ water quenching
 X_{DRX} recrystallized fraction
 X_{mDRX} meta-dynamically recrystallized fraction
 X_{tot} total recrystallized fraction

CHAPTER 1

Introduction

1.1 Background and motivation

With an increasing understanding of the consequences of green house gas emissions, sectors like the aero engine and gas turbine industries are striving to increase the efficiency of the combustion process in order to reduce fuel consumption and pollution levels. This is today the main factor driving the development in the aeroengine industry. Although the goal is to develop emission-free solutions, those are too far into the future to not justify research

into minimizing emissions now. This can be achieved by increasing the temperature in the hot sections of aircraft engines and turbine generators. However, the materials employed today are at, or close to, their limits, so new or improved materials are needed. Therefore, extensive efforts are being made to develop materials that better can withstand the harsh environments occurring in the hot sections of jet engines.

Ni-base superalloys are well adapted to cope with these extreme conditions in terms of high temperatures, corrosive gases, high loads as well as high loading/unloading rates. They derive these properties from their microstructure, which is a result of complex chemistry and tailored, alloy-specific, processing routes. Haynes 282, developed by Haynes International, is a relatively new competitor to existing precipitation strengthened superalloys like Waspaloy and René 41. It has a slightly lower volume fraction of strengthening phase compared to these alloys, with its equilibrium fraction being around 20 % in a fully heat treated condition. The kinetics for nucleation of the strengthening phase is also slow compared to other alloys, which improves weldability and fabricability [1, 2]. Haynes 282, and other similar wrought Ni-base superalloys are shaped by thermomechanical processing (TMP), where the component is deformed at elevated temperatures. Operations like forging and hot rolling fall into the category of TMP, and is followed by heat treatments (HT). Fig. 1.1 illustrates the different TMP and HT steps that a wrought Ni-base superalloy can undergo before its microstructure is optimized.

The microstructure of a metal or alloy will control the mechanical properties. The HT sequence is therefore designed to precipitate secondary phases with optimal morphology for the intended application. Consequently, most previous research on the microstructure of Haynes 282 has been focused on the mechanical properties [3–8], heat treatments [9, 10] or microstructure stability and related effects [11, 12]. These aspects are important to investigate since after HT the processing is finished and the microstructure will be in its oper-

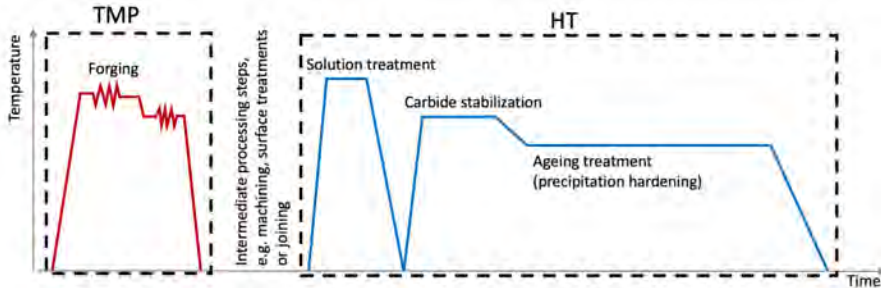


Figure 1.1: Illustration of the TMP and HT processing steps for a wrought Ni-base superalloy.

ational form. However, many mechanical properties are also strongly affected by the grain size. For example, a microstructure consisting of large grains show higher creep and fatigue crack growth resistance compared to a microstructure consisting of small grains. Small grains, on the other hand, improves the overall strength and low cycle fatigue properties, since dislocation motion is hindered with the increased grain boundary area.

However, for the above mentioned research, the material used was already in a forged condition, and from Fig. 1.1 we can see that forging (or hot rolling in case of sheets) is the initial step in the processing route. The TMP steps set the basis for the microstructure during subsequent processing, like HT, and thus affects the mechanical properties in the final state. Forging, or hot rolling, lead to recrystallization, which will be explained in detail in coming chapters, that 'resets' the microstructure through the formation of new grains. This can either occur during deformation if it is done at high temperature: dynamic recrystallization (DRX), or if a material is deformed at low temperatures and later annealed: static recrystallization (SRX).

During forging of industrial components at high temperatures recrystallization can be divided into two distinct phenomena: DRX which occurs *during* deformation, and meta-dynamic recrystallization (mDRX), which progresses statically afterwards if temperature is maintained and the component was not fully recrystallized from DRX. mDRX thus occurs e.g. between forging steps or during cooling of large components which cannot be quenched. Forging is therefore a process which can yield a 'new' microstructure with smaller grains than initially present. Considering Ni-base superalloys, that usually are deformed at high temperatures, the microstructure is often fully recrystallized after TMP due to DRX and mDRX. All subsequent annealing steps, HT in Fig. 1.1, are static which will only lead to growth of the present grains. So not only is forging the first step, it is also the step that defines the available property space for the coming steps.

Understanding the effect of forging on the resulting microstructure is therefore very important, but it is relatively un-researched ground for this particular alloy. Some research has been done [13–15] at a range of temperatures and strain rates. However, no study on the DRX nucleation or kinetics, the presence of secondary carbides, or the progression of mDRX have been made.

1.2 Aim of the study

This thesis will consist of two main themes, the DRX and mDRX part of hot deformation of Ni-base superalloy Haynes 282. The questions in the for-

mer concern the recrystallization phenomena and behaviour **during** deformation/forging. The latter, on the other hand, will yield insights into the meta-dynamic part, i.e. when the temperature is maintained **after** deformation. The following research questions are considered in each theme:

- Dynamic recrystallization
 - When does nucleation/recrystallization start, and how does the microstructure evolve with continued strain?
 - How does temperature and strain rate affect the microstructure?
 - What role do primary and secondary carbides have on the dynamic recrystallization behaviour?
- Meta-dynamic recrystallization
 - How does the microstructure evolve with time when held statically (without progressive deformation) at target temperature?
 - How do parameters like prior strain and temperature affect the evolution of the microstructure?

CHAPTER 2

Microstructure and processing of Ni-base superalloys

2.1 Ni-base superalloys

Superalloys have been defined as "alloys developed for high service temperatures" [16, 17]. They show the highest mechanical stability in applications with heavy loads, high temperatures (up to 0.8 of the melting temperature, T_m [18]) and corrosive environments. What makes Ni-base superalloys so good at maintaining their mechanical properties at these extreme conditions is a meticulous tailoring of the present phases, made possible by the multitude of alloying

elements and TMP followed by HT. This complex combination of chemistry and processing results in a microstructure consisting of multiple strengthening phases. Below follows a brief explanation of these, and it should be mentioned that for each alloy only some of these phases are present, depending on the alloying elements and their content.

Austenitic γ phase This is the main phase present in Ni-base superalloys, constituting the matrix in which other phases are present. The γ phase consists of a solid solution where Ni and elements of similar atomic radii occupy the face-centered cubic (fcc) lattice positions in a random manner. Common elements found in γ , beyond Ni, are Co, Fe, Cr, Mo, W, Ti and Al [17, 19]. Due to the slight differences in atomic radii, local elastic strains are created in the lattice providing solid solution strengthening (SSS). These strains hinder dislocation movement which strengthens the alloy. Some elements also contribute to a lowering of the stacking fault energy (γ_{SFE}) which further hinder dislocation movement by obstructing their ability to cross slip [17]. Additional positive effects from the alloying elements present in γ is Cr forming protective scales on the surface which hinders diffusion of corrosive elements into the alloy improving corrosion resistance, and Al forming oxides at high temperatures which improves the oxidation resistance. Elements with a large difference in the atomic radii, relative to Ni, have also been shown to contribute to the SSS by forming atomic clusters [20]. Additions of refractory elements like Mo and Nb into the alloying mix has been shown to increase the solvus temperature of the γ' phase [17], which is described in the following section.

Intergranular phases

Gamma prime (γ'): The most common type of strengthening precipitate is the γ' phase, which can be found in alloys like Waspaloy, Rene 42 and the alloy covered in this thesis, Haynes 282. It forms with the addition of Ti and Al into an ordered $L1_2$ structure with a nominal chemical composition of $Ni_3(Al,Ti)$. In γ' strengthened alloys, it is the γ' precipitates that give the highest strengthening contribution, which increases with temperature up to 800 °C [17, 21, 22]. This strengthening arises from coherency strains due to slight lattice differences between γ and γ' , and antiphase boundary (APB) formation during cutting of a particle by a dislocation. A second dislocation is required to shear the γ' particle to restore the order, and thus, dislocations become coupled via the APB, and will move in pairs, so called superdislocations. However, due to the repelling forces between them, they will be separated by a distance defined by the balance between APB energy (γ_{APB}) and elastic interactions. If the size of γ' is smaller than the spacing between the dislocations pair, i.e. only one dislocation can be inside the γ' particle, weak coupling

occurs and the shear stress, τ_w , required to shear a precipitate is given as [23]

$$\tau_w \propto \gamma_{APB}^{3/2} \sqrt{\frac{rf}{G}}, \quad (2.1)$$

where r is the radius of γ' and f is the volume fraction and G the shear modulus of γ' . If the size of γ' becomes larger than the superdislocation spacing, and both dislocations can be inside the particle simultaneously, the pair becomes strongly coupled, and the shear stress is instead given as [23]

$$\tau_s \propto \sqrt{\frac{\gamma_{APB} G f}{r}}. \quad (2.2)$$

As can be seen in Eq. (2.1) and Eq. (2.2) for the weakly coupled dislocations the shear stress increases with r and decreases in the strongly coupled case. If the size increases even further, dislocations will bend around the particles instead of shearing them, so called Orowan looping, which decreases the strengthening even further. The highest contribution to strength is therefore at a particle size where transition from weakly to strongly coupled dislocations occur [22, 24].

The γ' precipitates form coherently with the γ matrix and with a relatively small lattice mismatch between the phases, 0-2% [21]. The thermal stability is increased with a smaller mismatch, making γ' grow slower at operating temperatures, which can increase the lifetime prior to Orowan bowing occurring. However, a larger mismatch improves the hardening contribution from the precipitates. For some high fraction γ' alloys, large, primary γ' at grain boundaries are also employed during forging to control the grain size. Otherwise it is purposefully nucleated and grown during HT employed post the TMP steps. The amount of γ' can vary from a few percent to over 60 % for different polycrystalline superalloys and can go up to 80 % for single crystal alloys. A higher γ' percentage improve mechanical properties, but complicate processing. Alloys with γ' over 45 % are very hard to deform both at low and high temperatures [18].

Gamma double prime (γ''): This phase is created by the addition of Nb where it, together with Ni, forms a precipitate with an ordered body-centered tetragonal (bct) crystal structure with nominal chemical composition Ni_3Nb [25]. As for γ' , the strengthening effect arises from APB formation and coherency strains. Its morphology is in the form of discs due to anisotropic misfit [22]. The misfit is larger compared to γ' so the coherency strengthening

contribution is greater. γ'' is metastable and can during high temperature operations undergo a phase transformation into the stable form called δ phase, described below, which is detrimental for the mechanical properties.

Carbides: Primary carbides and/or borides are usually intragranular phases and are typically of the type MC. Ti and W are common metallic elements forming MC [3]. These particles are unavoidable as they nucleate during solidification. Their size, number and morphology are affected by the solidification conditions [26]. The small amount and inhomogeneous distributions of MC carbides means that they do not contribute to any mechanical properties positively or negatively. However, they have been shown to hinder grain growth and due to their brittle nature they can be the region for crack initiation and propagation [27].

Topologically close-packed (TCP) phases: Three distinct precipitates fall into the category of TCP phases. These are σ (tetragonal), μ (rhombohedral) and Laves (hexagonal). All are undesirable as they lead to an overall decrease of ductility and mechanical properties. They also deplete the γ matrix of elements contributing to the SSS and decrease the creep resistance [28].

Intergranular phases

Delta (δ): As mentioned, the δ phase is the stable form of γ'' and has a orthorhombic crystal structure. The δ phase nucleates at grain boundaries and has a plate-like morphology [29]. However, globular shape has also been observed [17]. Its chemical composition is similar to γ'' (Ni_3Nb) [30]. During service, a low amount of δ improves the dwell fatigue and notch sensitivity. It is often also nucleated prior to TMP in Nb containing alloys, which is then performed below the δ solvus temperature to receive further control over the recrystallization process. However, it can also lead to the creation of microvoids during deformation [31].

Eta (η): This phase has a hexagonal close-packed (hcp) crystal structure and chemical composition Ni_3Ti and can be both undesirable or desirable. In some Ni-base superalloy, like Allvac 718+, it is used as an grain boundary strengthening particle instead of δ phase [32]. However, it can also appear as γ' decomposes into η after long heating times, and it can also form in alloys where the Ti to Al ratio becomes too high [33].

Grain boundary carbides: There are primarily two kinds of grain boundary (GB) carbides, M_{23}C_6 , rich in Cr, and M_6C , mainly containing Mo [34]. These carbides are nucleated during HT post TMP in order to improve the creep resistance by hindering GB sliding. Due to the high Cr content in M_{23}C_6 they can deplete the Cr content from γ around GBs, which lowers corrosion resis-

Table 2.1: Summary of phases in Ni-base superalloys.

Phase	Crystal structure	Chemical composition	Main elements	Intra- or intergranull
γ	fcc (disordered)		Ni, Co, Fe, Cr, Mo, W	N/A
γ'	fcc (L1 ₂)	Ni ₃ (Al,Ti)	Ni, Al, Ti	Intra
γ''	bct (D0 ₂₂)	Ni ₃ Nb	Ni, Nb	Intra
MC	cubic	(Ti,Ta,Nb,Hf)C	Ti, Ta, Nb, Hf, C	Intra
δ	orthorhombic (ordered Cu ₃ Ti)	Ni ₃ Nb	Ni, Nb	Inter
η	hcp (D0 ₂₄)	Ni ₃ Ti	Ni, Ti	Intra
M ₂₃ C ₆	complex cubic	(Cr,Fe,W Mo) ₂₃ M ₆	Cr, C	Intra
M ₆ C	fcc	Mo ₆ C	Mo, C	Intra
σ	tetragonal	FeCr, FeCrMo, CrCo	Fe, Cr, Mo, Co	Inter
μ	rhombohedral	(Fe,Co) ₇ (Mo,W) ₆	Fe, Co, Mo, W	Inter
Laves	hexagonal	Fe ₂ Nb, Co ₂ Ti, Fe ₂ Ti	Fe, Co, Ti, Nb	Inter

tance locally. The GB carbides nucleate at random high angle grain boundaries (HAGB) i.e. GBs with high interfacial energy. When they nucleate, their orientations make them coherent with one of the grains, and they grow mainly into the other grain [35].

A summary of the above described phases is shown in Table 2.1 which gives information on chemistry, structure and location.

Fully treated Haynes 282 will contain the following phases: γ , γ' , primary MC (mainly intragranually) and secondary M₂₃C₆ and M₆C carbides (mainly intergranually). The morphology of γ' varies with annealing time, beginning with smaller, spherical γ' as seen in Fig. 2.1(a) to later adopt a cuboidal shape, Fig. 2.1(b). However, due to its solvus temperature being 997 °C for Haynes 282 [2], it is not present during TMP as these processes are performed at higher temperatures. It is nucleated and grown during HT. Because this thesis only investigates the TMP processing, the material will contain no γ' . All three type of carbides, MC (1), M₆C (2) and M₂₃C₆ (3) are shown in Fig. 2.1(c). Secondary carbides are found at GBs and have a solvus temperature of around 1100 °C [36]. MC carbides are large and blocky, and in Haynes 282 the metallic elements constituting the carbide are Ti and Mo [36]. Their solvus temperature is very high, making it impossible to dissolve them. They do not affect the mechanical properties, but during compression they have been seen to act as nucleation sites for recrystallization through a process called particle stimulated nucleation (PSN) [37]. During service, it is also suggested that they aid the formation of GB carbides, M₂₃C₆, by breaking down and supplying extra C according to reaction given in Eq. (2.3).



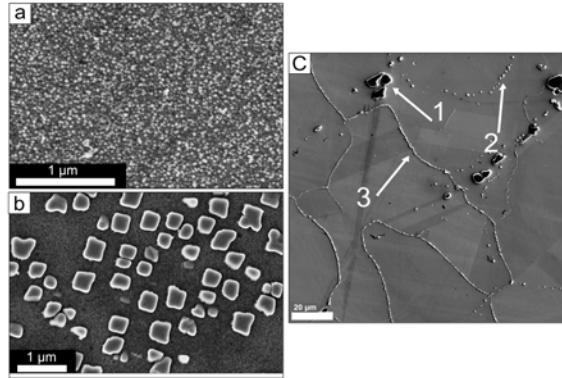


Figure 2.1: Phases that nucleate in Haynes 282. (a) and (b) show γ/γ' with different morphologies achieved with increased annealing time and temperature. Courtesy of Nitesh Raj Jaladurgam. (c) Displays the carbides where (1) highlights primary MC , (2) secondary M_6C and (3) secondary M_{23}C_6 carbides.

2.2 Thermomechanical processing

In a metallurgical sense, TMP refers to manufacturing processes where metallic materials are heated and shaped from basic materials to high quality components [38]. Due to their high strengths, alloys like Ni-base superalloys show low formability at low temperatures and cold working is therefore unviable, as shaping cannot be achieved without fracture.

A typical TMP procedure for wrought Haynes 282 and similar Ni-base superalloys is presented in Fig. 2.2. The initial step is soaking (heating) of the billet in order to dissolve unwanted precipitates and to obtain a homogenous temperature throughout the piece. Directly after soaking, when the material is hot, it is forged to its desired shape through compressive forces. During forging the combination of stored energy (dislocations) and thermal energy leads to DRX. Multiple forging steps can be necessary to reach the desired shape, and in order to maintain temperature, the piece can be soaked between forging steps as well. Industrially forged pieces can range in mass from a couple of kg to metric tons, and therefore fast quenching is impossible. As mentioned in Chapter 1 recrystallization can continue through mDRX during intermediate soaking or slow cooling.

After the TMP is complete, multiple HT follow. The first, solutionizing, is performed at a relatively high temperature, typically at or slightly above the secondary carbide solvus, and is employed to dissolve all precipitates (except MC) that may have formed during cooling in the TMP steps. If desirable, grain growth is also achievable during the solutionizing step. In the 1st ageing

step, GB carbides are nucleated and during the 2nd ageing step γ is nucleated and grown together with the carbides.

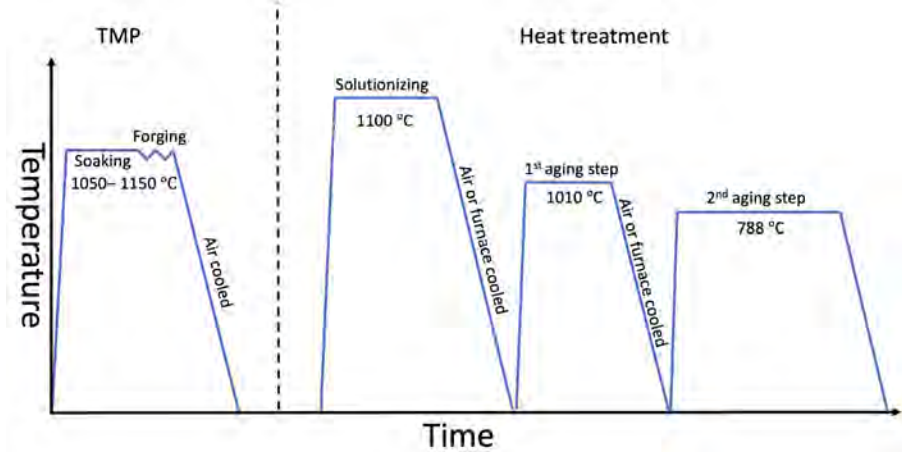


Figure 2.2: Standard TMP and HT procedure for Ni-base superalloys. Shaping is done at elevated temperatures via e.g. forging. Heat treatment show the three annealing steps to primarily nucleate and grow precipitates to the desired size. The indicated temperatures are typical for processing of Haynes 282.

The microstructural evolution during TMP will be a result of the applied deformation parameters. strain (ϵ), strain rate ($\dot{\epsilon}$), temperature (T), holding times between forging steps and cooling rate will all contribute to the recrystallization behaviour. And the resulting microstructure will in turn determine the mechanical properties. Understanding how each deformation parameter affects the microstructural evolution is therefore extremely important.

2.3 Forging

Forging is a process in which a piece is shaped by compressive forces, and is one of the oldest know techniques for metalworking. Forging can be performed either at room temperature, cold forging [39, 40], at elevated temperatures but below the recrystallization temperature, warm forging, [40], or at temperatures around $0.75T_m$ where DRX occurs during deformation, hot forging [41]. For large scale productions, forging is a cost-effective material processing process. An initial piece, called billet, is heated until the temperature is homogenous throughout the volume and placed between two dies, Fig. 2.3, that compress the billet to the desired shape. The forces applied are high enough to induce plastic deformation to shape the component, increasing the stored energy, in

the form of dislocations, which drives the change of the materials microstructures. This in turn changes the mechanical properties. It is therefore of utmost importance to understand how each metal/alloy is controlled by each forging parameter. Forging thus offers control both over shape and structure, with minimal loss of material making it a cost-effective process for large scale production.

In the case of Ni-base superalloys they can only be subjected to hot forging (if at all) due to their high strength at low temperatures [42]. The most common type of forging operations are open- and closed die forming, and ring rolling.

Open die forging: This is suitable as an initial forging operation for the creation of disks. The method is presented in Fig. 2.3(a). The dies are open and the upper die is connected to a hydraulic press or hammer [43]. The finished component is called a pancake.

Closed die forging: For components with a more complicated geometry a closed die is needed. The closed die acts like a mold while a hammer or hydraulic press presses the metal/alloy to fill all cavities of the closed die. Excess material, called flash, is ejected from smaller openings in the dies. This process is more expensive compared to open die forming because the dies need to be machined to get their desired shapes. The principle is showed in Fig. 2.3(b).

Ring rolling: In order to create e.g. large casings for jet engines with often complex geometries, ring rolling is a suitable forging procedure. The initial preform is disc shaped with a hole in the middle. It is then rotated with rolls applying pressure to shape the spinning piece, Fig. 2.3(c).

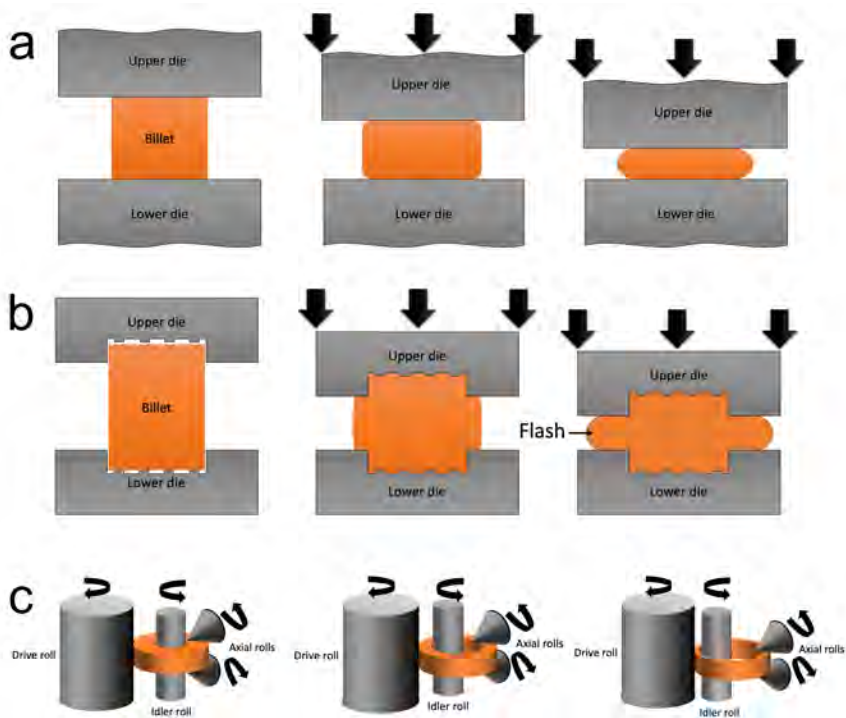


Figure 2.3: The most common forging operations for Ni-base superalloys. (a) open die forming, (b) closed die forming, (c) ring rolling.

CHAPTER 3

Microstructure evolution during hot working

In this chapter the theory of dynamic recrystallization will be explained. However, before arriving at that point, an explanation on what happens during deformation prior to, or without, DRX is given as this plays an important role for the DRX behaviour.

3.1 Work hardening and dynamic recovery

In order to understand the progression of plastic deformation in metallic material, it is preferable to begin by mentioning the defects that affect the plasticity. These can be divided into groups ranging from 0D (zero dimension) to 3D.

0D defects: Vacancies, substitutional atoms and interstitials fall into this category. Vacancies increase the rate of diffusion, which can aid recovery. Substitutional atoms and interstitials interact with dislocations and can segregate to stacking faults which decreases the γ_{SFE} .

1D defects: Dislocations are 1D, or line, defects and are the most important defects to consider. They are the main source by which a metallic material is able to deform plastically. A dislocation represents a discontinuity between a part of the lattice that has sheared and a part that has not [44].

2D defects: 2D defects consist of boundaries between grains (GB, or APB) or different phases (phase boundaries), and stacking faults. Boundaries hinder dislocation movement and stacking faults decrease the recovery rate.

3D defects: As mentioned in Chapter 2, precipitates affect the deformation of metals. Voids and cracks also fall into this category.

In order to keep the explanation on a relevant level with regards to this thesis the following sections of this chapter will assume a single-phase material, i.e. the volume fractions of secondary phases and precipitates are small and we assume no effect from potential cracks and pores.

Plastic deformation in a crystalline material is achieved via sliding of the atomic planes. It was not until 1934 that what caused this shearing was the movement of dislocations [45–47]. In metallic materials, dislocations usually move on the most densely packed atomic planes and in the direction of the highest atomic densities. These are called the slip planes and slip directions respectively, together they define the slip system. For a fcc material these are the $\{111\}$ planes and $\langle 110 \rangle$ directions. The last component that needs to be known in order to fully explain a dislocation's movement is the length of a single step. This is called the dislocation's Burgers vector, $\mathbf{b}$ and in an fcc material it is equal to

$$\mathbf{b} = \frac{a}{2}[110], \quad (3.1)$$

where a is the lattice spacing. At high temperatures, dislocations can also climb from one slip plane to another.

During plastic deformation, new dislocations are constantly generated through different sources, most commonly Frank-Reed sources [48], increasing the dislocation density ρ_d and, in turn, the stored energy, E , in the material according to Eq. (3.2)

$$E \sim 0.5\rho_d Gb^2, \quad (3.2)$$

where G is the shear modulus and b the magnitude of the Burgers vector. Metallic materials especially can contain a very large amount of dislocations of up to 10^{12} cm/cm³ in a heavily deformed metal [49]. Dislocations exert an elastic strain in their vicinity [50] hindering the movement of other dislocations as their strain fields interact. As a consequence, when a metal is plastically deformed, the large increase in ρ_d hinders dislocation movement, i.e. plasticity, making it harder to further deform it. This is called work hardening (WH) and stems from interactions of large dislocations groups throughout the volume [48, 51].

To understand the effects of dislocation density evolution on the work hardening, we consider a simple model proposed by Kocks and Mecking [52, 53]. While this is an oversimplification it serves to demonstrate the underlying concepts. Assuming a uniaxial deformation, the kinetics of the plastic strain rate, $\dot{\varepsilon}_p$, resulting from thermally activated dislocation motion, is expressed by a power law equation

$$\dot{\varepsilon}_p = \dot{\varepsilon}_o \left(\frac{\sigma}{\hat{\sigma}} \right)^m, \quad (3.3)$$

where $\hat{\sigma}$ is an internal variable giving the state of the material. $\dot{\varepsilon}_o$ and m are material parameters. $\hat{\sigma}$ is dependent on multiple variables, however, if considering continuous monotonic straining only one variable, namely ρ_d , can be considered [54] and $\hat{\sigma}$ can be expressed as

$$\hat{\sigma} = M\alpha Gb\sqrt{\rho_d}, \quad (3.4)$$

where M is the average Taylor factor, and α a numerical constant. The proportionality between $\hat{\sigma}$ and $\sqrt{\rho_d}$ comes from the assumption that glide resistance for dislocations is given by Gb/L , where L is the average obstacle spacing. Assuming a single phase material, the only obstacles present are dislocations (the grain size $\gg$ dislocation spacings so GBs can be disregarded). This gives

$$L = \frac{1}{\sqrt{\rho_d}}. \quad (3.5)$$

Eq. (3.3) and Eq. (3.5) show the direct link between ρ_d and resistance to plastic flow. However, with progressive deformation, when ρ_d becomes large, other mechanisms will start to compete with WH.

One of these is named dynamic recovery (DRV) which acts as a softening mechanism. During DRV moving dislocations will start to either arrange themselves into lower energy structures like sub-boundaries and low angle grain boundaries (LAGB), or annihilate each other upon meeting. These mechanisms lower ρ_d , and thereby increase the ability for other dislocations to glide and climb. WH and DRV will occur simultaneously, and the change in ρ_d with continued strain will have a positive and negative contribution. In the Kocks-Mecking model, this is expressed as

$$\frac{d\rho_d}{d\varepsilon^p} = M (k_1 \sqrt{\rho_d} - k_2 \rho_d), \quad (3.6)$$

where k_1 is a material parameter characterizing the rate of dislocation generation and k_2 the loss of dislocations. The first term in Eq. (3.6) describes WH while the second handles DRV, the latter being thermally activated and thus rate-dependent, which can be explicitly included as

$$k_2 = k_{20} \left(\frac{\dot{\varepsilon}^p}{\dot{\varepsilon}_o^*} \right)^{-1/n}, \quad (3.7)$$

where k_{20} is a material constant while n and $\dot{\varepsilon}_o^*$ are dependent on temperature.

If deforming with a constant strain rate, Eq. (3.6) can be integrated and the strain hardening behaviour can be expressed with the Voce equation [54],

$$\frac{\sigma - \sigma_s}{\sigma_i - \sigma_s} = \exp \left(- \frac{\varepsilon^p - \varepsilon_i^p}{\varepsilon_{tr}} \right), \quad (3.8)$$

where σ_i and ε_i^p are the stress and plastic strain at some initial point i , σ_s is the saturation stress, given by

$$\sigma_s = M \alpha G b \left(\frac{k_1}{k_{20}} \right) \left(\frac{\dot{\varepsilon}^p}{\dot{\varepsilon}_0} \right)^{1/m} \left(\frac{\dot{\varepsilon}^p}{\dot{\varepsilon}_0^*} \right)^{1/n}, \quad (3.9)$$

and ε_{tr} is a parameter describing the rate at which σ is approaching σ_s , which is expressed as

$$\varepsilon_{tr} = \frac{\sigma_s}{\Theta_{II}}, \quad (3.10)$$

where Θ_{II} is the hardening rate during stage II hardening (stage I is only occurring in single crystals [55]) and given by

$$\Theta_{II} = \frac{1}{2} M^2 \alpha G b k_1 \left(\frac{\dot{\varepsilon}^p}{\dot{\varepsilon}_0} \right)^{1/m}. \quad (3.11)$$

Differentiating the stress σ in Eq. (3.8) with respect to the plastic strain, ε^p , the strain hardening coefficient, Θ , will become

$$\Theta = \frac{\partial \sigma}{\partial \varepsilon^p} = \Theta_{II} \left(1 - \frac{\sigma}{\sigma_s} \right) \quad (3.12)$$

From Eq. (3.11) it can be seen that the strain hardening becomes Θ_{II} as σ approaches 0. The stress-strain response and the strain hardening for a material described by the Kocks-Mecking model is shown in Fig. 3.1. At σ_i , i.e. the start of plastic deformation, only WH is acting on the material, and the strain hardening is Θ_{II} . When DRV initiates, stage III hardening starts and the hardening rate decreases linearly, Fig. 3.1(b), until eventually the saturation stress, σ_s is reached i.e. where WH and DRV reach equilibrium. The illustrated plot in Fig. 3.1(b) is often referred to as a Kocks-Mecking plot (K-M plot) and is often employed to find values of Θ_{II} and σ_s from experimental data where σ_s is not reached.

3.2 Recrystallization

DRX is the process where new grains are created and grow at the expense of the deformed grains in order to reduce the stored energy during high temperature deformation ($> 0.5T_m$ [56]). Recrystallization should not be confused with recovery. Both are softening processes, i.e. they lower the overall energy, but recovery is a rearrangement and loss of dislocations, while recrystallization is dominated by long range motion of grain boundaries [48, 55, 57]. This distinction is important because this means that recrystallization replaces the

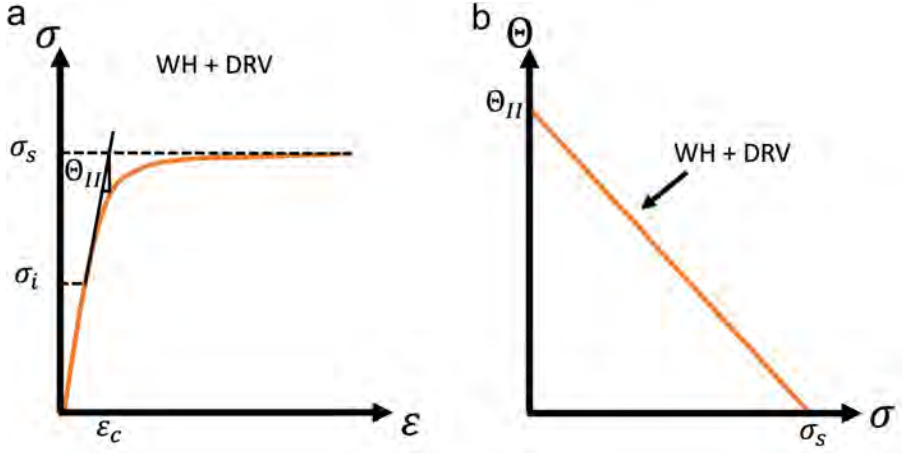


Figure 3.1: (a) Illustration of stress-strain response for a material where WH and DRV are in play. (b) Illustration of a K-M plot for the same material.

deformed microstructure with a new, strain free one, whereas recovery reduces the stored energy by a lowering of ρ_d , while no new HAGBs are created during DRV [58].

It should be mentioned that the naming convention of the different recrystallization phenomena are not consistent throughout literature. The main cause of this is the complexity of the field. Therefore, this following list aims to enlighten the reader of how the different phenomena will be defined in this thesis and also how each phenomena will be abbreviated henceforth

SRX: A recrystallization process where the energy contributions in terms of supplied strain and thermal energy are separated in time. Is often a process occurring during annealing of a previously cold worked material.

Dynamic recrystallization (DRX): A recrystallization process where both mechanical and thermal energy are supplied together, i.e. during TMP. This process is further divided into two categories, namely:

Discontinuous dynamic recrystallization (dDRX): Occurs mainly at grain boundaries in low-to-medium γ_{SFE} materials and can initiate at relatively low strains. A clear nucleation, and growth (by high angle grain boundary movement) of recrystallized grains is evident. Illustrated in Fig. 3.3(b-d). This will be the main mechanism covered, and therefore DRX and dDRX are used interchangeably throughout the text unless otherwise specifically stated.

Continuous dynamic recrystallization (cDRX): Occurs mainly in

high γ_{SFE} materials at large strains during deformation. It is called continuous since there is no clear initiation stage. New HAGB are formed by progressive rotation of sub-grains. Illustrated in Fig. 3.3(e-g).

Meta-dynamic recrystallization (mDRX): Occurs immediately after deformation (if the temperature is maintained) in low-to-medium γ_{SFE} materials that have undergone an incomplete dDRX. It occurs through growth of existing DRX grains and has no incubation time.

Post dynamic recrystallization (pDRX): Occurs through continued growth of recrystallized grains after complete recrystallization if temperature is maintained. Since there is virtually no more stored energy in the form of dislocations, this process lowers the internal energy by decreasing grain boundary area. In literature the term pDRX is often used interchangeably with mDRX.

Like DRX, SRX can also progress through a continuous or discontinuous mechanisms and pDRX is the equivalent to the so called "secondary recrystallization" often mentioned for SRX. The two main topics of this thesis are dDRX and mDRX, however other items on the list above will be mentioned.

3.2.1 Dynamic recrystallization

Both dDRX and cDRX fall into the category of DRX, however, they differ much from each other as mentioned above. The material parameter that has the highest influence on whether dDRX or cDRX will be the dominating mechanism is the γ_{SFE} [56–60]. For fcc materials, dislocations move on the $\{111\}$ planes in the $\langle 110 \rangle$ directions with a Burgers vector given in Eq. (3.1). However, it can be energetically favourable for a dislocation to split into two partials with a stacking fault (SF) in between, so called Shockley partials. This is shown in Fig. 3.2 illustrating a close packed (111) plane where the initial dislocation, $\mathbf{b}_1$ splits into $\mathbf{b}_2$ and $\mathbf{b}_3$ with Burgers vectors $a/6\langle 121 \rangle$. A SF is caused by the first partial and restored by the passing of the second partial. The energy of a SF increases with its width. However, the partials exert repelling forces on each other, increasing the energy with decreasing d . This results in an equilibrium spacing between partial dislocations according to [58]

$$d = \frac{Ga^2}{24\pi} \frac{1}{\gamma_{SFE}}. \quad (3.13)$$

At elevated temperatures dislocations can also move from one (111) plane to

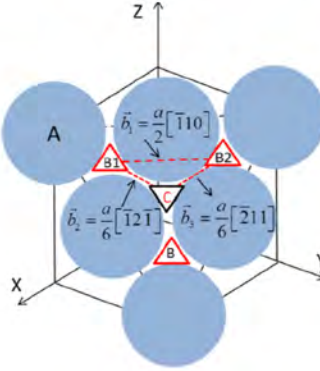


Figure 3.2: A close-packed plane in a fcc material including Burgers vectors for full and partial dislocations [58].

another via either climb or cross-slip [44]. These added modes of movement are important for the processes of DRV. However, if a dislocation has split into partials, it cannot move via climb or cross-slip. In order to do so, the partials have to recombine, which becomes progressively more unlikely with increased d . Therefore, in low γ_{SFE} materials, the rate of DRV is significantly lower compared to a material with a high γ_{SFE} .

Another hindrance to dislocations movement are HAGB due to their amorphous nature. During deformation (in a low γ_{SFE} material) dislocations move through a grain until they reach a GB, where they will pile up. This creates sufficiently high, localized, gradients of ρ_d at HAGB where new grains nucleate via dDRX, as illustrated in Fig. 3.3(b). With continued deformation these new grains grow, consuming the more strained microstructure, while also experiencing an increase in ρ_d [61]. Fig. 3.3(c). At a high enough strain, or after a post deformation hold, the entire microstructure is replaced with new grains, Fig. 3.3(d). When deforming to large strains, the grains that nucleated early could have accumulated enough strain to drive a second generation of recrystallization [62, 63]. cDRX on the other hand, occurs at high strains in materials where DRV is prominent. The arrangement of dislocations into sub-structures, Fig. 3.3(e), progressively evolves, increasing the misorientation in these cell like structures with a continuous supply of dislocations. Eventually, these sub-structures become LAGB, Fig. 3.3(f), and later HAGB and the microstructure is replaced with new grains, Fig. 3.3(g).

In fcc materials, many of the HAGB that form during recrystallization are, or become, coincident site lattice (CSL) boundaries [64, 65]. The main form of these is the $\Sigma 3$ type boundary, called twin boundaries. They form both during nucleation and growth of grains through different processes. One way of twin

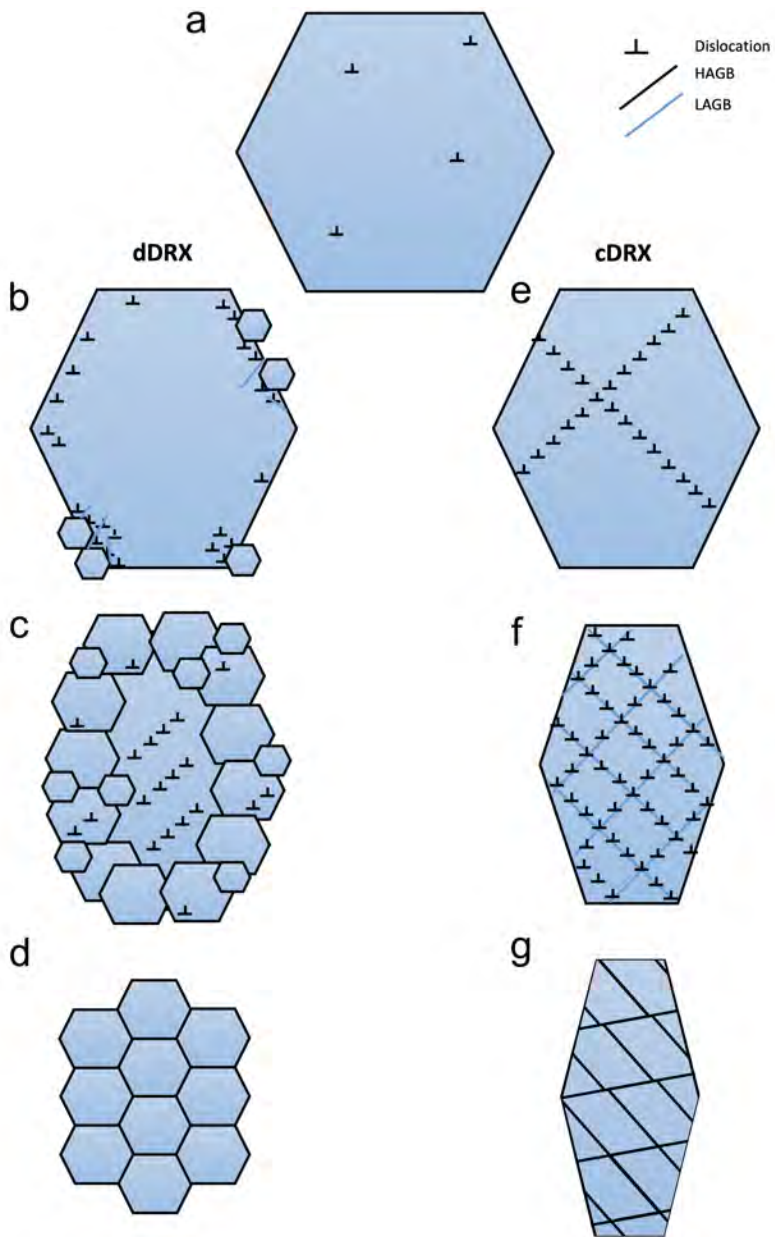


Figure 3.3: Illustration of dDRX to the left and cDRX to the right.

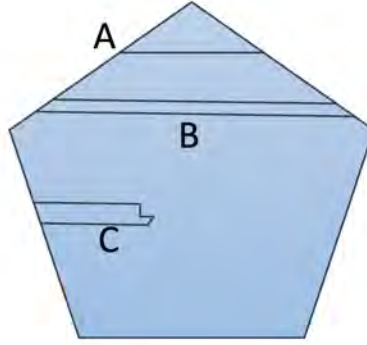


Figure 3.4: Different kinds of annealing twins. A and B are created as SF during HAGB movement. C are a result of SF terminated by partial dislocations.

creation is called growth accident formation where the stacking sequence of $\{111\}$ planes experiences a SF during growth, i.e. the ordering of the crystal planes becomes ..ABCABACABC.. [66]. They usually form at triple junctions, see A in Fig. 3.4. These boundaries have a very low energy compared to random HAGB. Another mechanism for describing the formation of annealing twins are stacking faults terminated by partial dislocations, C in Fig. 3.4.

3.2.2 dDRX nucleation mechanisms

As mentioned, dDRX is a discontinuous process with clearly separated nucleation and growth stages. An illustration of the stress-strain response, and corresponding K-M plot is shown in Fig. 3.5(a) and (b) respectively. It is generally accepted that nucleation initiates at a critical strain, ε_c [58, 67], which corresponds to an inflection point in Fig. 3.5(b). This decreases the WH rate seen in both the stress-strain and K-M plot. This progresses up until the peak strain, ε_p , which is the strain corresponding to the highest measured stress. After this point, the softening from DRX outweighs the WH and the flow stress decreases, and the strain hardening becomes negative, Fig. 3.5(b). The material is 100 % recrystallized when the saturation stress, σ_{ss} is reached, after which hardening and softening reach equilibrium.

Multiple different nucleation mechanisms that can generate recrystallized grains have been proposed. One question that arose early in the field was what gave certain "embryos" the energy advantage of others to develop into sub-grains and later into recrystallized grains [57].

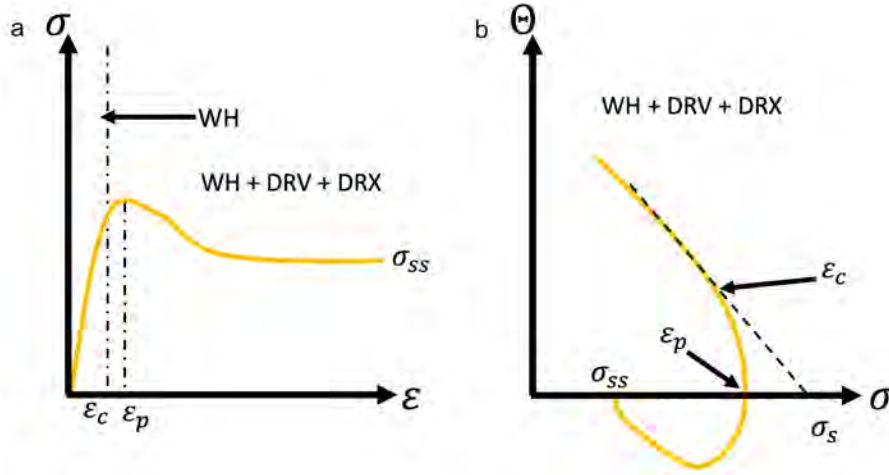


Figure 3.5: (a) Illustration of stress–strain response for a material where WH, DRV and dDRX occur. (b) Illustration of a K-M plot for the same material.

Strain induced boundary migration (SIBM)

This theory was first proposed by Beck and Sperry [68] and is today an accepted nucleation mechanisms in dDRX [55]. During deformation the GB will slide and shear, creating serrations which hinders further sliding [56, 58], Fig. 3.6(a). These serration accumulate large amounts of dislocations, locally increasing ρ_d , creating sub-boundaries, and making parts of the GB bulge into one of the grains creating a dDRX nucleus [69] as shown in Fig. 3.6(b). This new grain, containing relatively low ρ_d , can grow into the deformed grain even though this introduces an increase in energy due to an increase in HAGB surface area since the energy decrease of replacing a strained microstructure with an unstrained one is higher. Dislocations will accumulate and create a random HAGB or a CSL boundary through a SF, that will cut off the recrystallized grain from the parent, as seen in Fig. 3.6(c.1) and (c.2) respectively. Since the nucleus is created from a part of a deformed grain, there will be an orientation dependency between them [70].

Subgrain growth/coarsening

Another nucleation mechanisms that can occur during dDRX is subgrain growth, or sometimes also called subgrain coarsening [70–72]. When dislocations accumulate at GB recovery via arrangement and dislocation annihilation

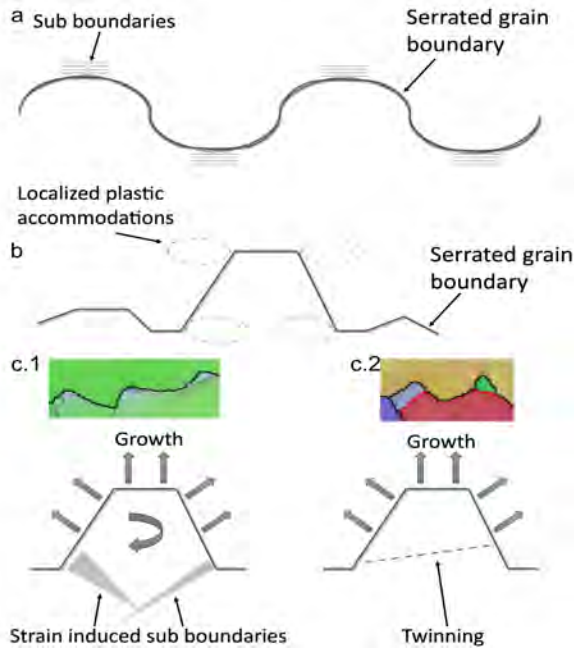


Figure 3.6: SIBM nucleation where (a) illustrates a sheared, serrated GB with sub boundaries accumulating, (b) bulging of GB, showing dDRX nucleus with localized plastic strain accumulation. Nucleus is cut off from parent by either (c.1) strain induced sub boundary (gray lines) or (c.2) twinning (red lines). Redrawn after [58].

will occur, though to a lesser extent compared to materials with high γ_{SFE} . This creates cell like structures that become gradually more misoriented eventually becoming LAGB. Some of these subgrains will grow at the expense of others. During this growth the boundaries will consume dislocations from the strained microstructure, increasing their misorientation. Eventually the subgrain will be surrounded by a HAGB and a nucleus is formed. Subgrain growth occurs in a heterogeneous fashion where some subgrains will grow faster than others. Only a small fraction of the created subgrains will become DRX nuclei [71].

Particle stimulated nucleation (PSN)

Secondary phases can act as nucleation sites for dDRX grains, so called particle stimulated nucleation (PSN), which has been observed for e.g. cementite par-

ticles in a low-carbon steel [73], at NbC carbides in a Ni-30%Fe-Nb-C alloy [74] and at carbides in a Ni-base superalloy [75]. Humphreys [76] concluded that PSN occurs at pre-existing sub boundaries within the locally higher strained region and nucleation is in the form of rapid sub boundary migration. This process end when the deformed region is consumed. During deformation, the stress field generated around the particles is generally higher than the rest of the matrix, where the strain incompatibility of the two phases can act as dislocation sources. However, the secondary particle has to be sufficiently large, $>1 \mu\text{m}$. Smaller particles have been shown to instead pin dislocations so sub boundaries around the particles are not developed [74].

Recrystallization kinetics

The recrystallization kinetics can either be determined by microstructural analysis on dedicated tests to varying strains where the recrystallized fraction (X_{DRX}) is measured at each strain. This process is time consuming, and instead, X_{DRX} can be calculated by the stress-strain data, as proposed by Laasraoui and Jonas [77]

$$X_{DRX}(\varepsilon) = \frac{\sigma_{DRV}(\varepsilon) - \sigma(\varepsilon)}{\sigma_s - \sigma_{ss}} = \frac{\Delta\sigma(\varepsilon)}{\sigma_s - \sigma_{ss}}. \quad (3.14)$$

In Eq. (3.14) $\sigma_{DRV}(\varepsilon)$ is the stress if the only softening mechanism was DRV [78, 79] (orange line in Fig. 3.7) and $\sigma(\varepsilon)$ the measured stress (yellow line). $\Delta\sigma(\varepsilon)$ is the difference between the two.

The kinetics of dDRX can then be described with the Avrami equation shown in Eq. (3.15) [80]

$$X_{DRX}(\varepsilon) = 1 - \exp\left(-k \left[\frac{\varepsilon - \varepsilon_c}{\varepsilon_p}\right]^n\right) \quad (3.15)$$

where k and n are material constants. Since they are dependent on the deformation parameters. In order to generalize k and n they are often expressed as

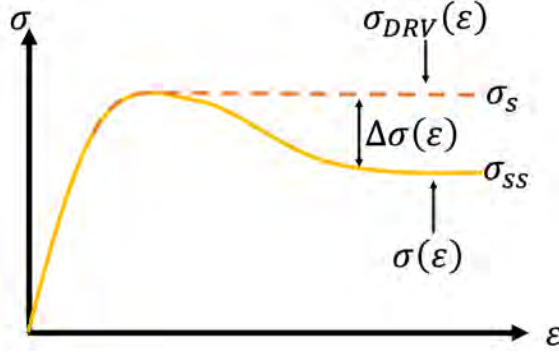


Figure 3.7: Evolution of stress with strain for a material that only softens via DRV (dashed orange line) and one that experiences both DRV and DRX (solid yellow line).

functions based on the Zener-Holloman parameter, Z , given as Eq. (3.16),

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right), \quad (3.16)$$

where Q is the activation energy and R the universal gas constant [81].

3.2.3 Meta-dynamic recrystallization

mDRX is often investigated via multi-pass compression tests [82–84], in order to understand the microstructural change during time between hits. It is defined as the continued progression of recrystallization of a microstructure that is not fully recrystallized, which is kept at the target temperature after deformation [85, 86]. Recrystallized grains continue to grow into the remaining deformed microstructure. It initiates immediately, i.e. has no incubation time, which is what separates mDRX from SRX where recrystallized grains have to nucleate and then grow in an, often cold worked, material subjected to high temperatures, or pDRX which is the pure grain growth in order to reduce GB area. The rate of mDRX is determined by the velocity, v , of HAGBs of the recrystallized grains as they move into the deformed microstructure, and is affected by the driving pressure, p , i.e. the differences in stored energy between the recrystallized and deformed grains, and the HAGB mobility, ω according to Eq. (3.17) [85]

$$v = p\omega, \quad (3.17)$$

where, see Eq. (3.2),

$$p = \Delta E = \frac{1}{2}Gb^2\Delta\rho_d, \quad (3.18)$$

with $\Delta\rho_d$ the difference is dislocations densities between the recrystallized and deformed grains and

$$\omega = \frac{\omega_0}{T} \exp\left(-\frac{Q_\omega}{RT}\right). \quad (3.19)$$

where Q_ω the activation energy for HAGB mobility and ω_0 is a material constant.

The supplied thermal energy ensures that the HAGB mobility is high, leading to fast diffusion of atoms from the GB into the recrystallized lattice and from the deformed lattice into the GB. The fraction of the remaining deformed microstructure which has been replaced through mDRX, X_{mDRX} , during hold can be determined by the following equation [85]

$$X_{mDRX} = \frac{X_{tot} - X_{DRX}}{1 - X_{DRX}}, \quad (3.20)$$

where X_{tot} is the total recrystallized fraction, X_{DRX} the dDRX fraction at the end of deformation.

Effect of initial microstructure and deformation parameters

There are many factors which affect the DRX progression. Firstly looking at the material itself, alloying content is a large contributor. The chemistry will affect the γ_{SFE} which in turn determine the ability for dislocations to climb and cross slip. Different alloying elements also affect which secondary phases will exist. They in turn can also affect the DRX behaviour either via pinning of dislocations (hindering DRX progression) or by acting as nucleation sites (promoting it).

The initial grain size will affect the dDRX progression by changing the amount of GB area where nucleation takes place, [58, 87, 88]. Dehghan-Manshadi et al. [87] investigated the effect on initial grain size on DRX for a 304 austenitic

steel. They found that for a more fine grained material the strain required to reach full DRX was lower compared to a coarse grained material, with nucleation appearing more at triple junctions compared to at HAGBs. At large strains, the average grain size will reach similar sizes independent on the initial size [58, 63].

Temperature also plays an important role since both DRV and HAGB mobility are thermally dependent. Fig. 3.8 shows the typical appearances and changes in the stress-strain response for materials with low γ_{SFE} , where (a) illustrates different temperature and (b) different strain rates. The critical strain marked is given as the onset of DRX, which leads to a decreased WH rate. It has also been shown that decreasing the temperature increases both the peak stress and peak strain [57, 89]. Since dDRX contains thermal elements [56], a higher temperature helps drive the recrystallization leading to larger grain sizes [90]. Deforming at higher temperatures also increases the occurrences of other thermal processes like diffusion of atoms and dislocations movement, the measured flow stress decreases, Fig. 3.8(a), [91, 92].

With an increase in strain rate, Fig. 3.8(b), the generation of dislocations is faster, leading to a higher ρ_d , while time for DRV decreases, leading to a higher WH, and the peak stress occurs at larger strains due to the faster deformation [91]. Increasing the strain rates also leads to a higher adiabatic heating (AH) [93]. This is the explanation for the faster softening rate seen at higher strain rates, Fig. 3.8(b). This increase in thermal energy helps soften the material by a higher diffusion rate of vacancies and mobility of dislocations [13]. It also increases the mobility of HAGBs increasing growth rate of DRX grains which softens the material. Pradhan et al. [94] showed an illustration of the DRX kinetics from strain rate during deformation, where two processes were in play. At low strain rates, the dominating process was the time for grain boundary mobility. The low strains accumulated here led to fewer nucleated grains but larger grain sizes. For high strain rates the roles were reversed. There was not much time for grains to grow, but the large strains led to high nucleation rates. Intermediate strain rates did not benefit any process and the kinetics were slower.

Regarding mDRX it has been seen that temperature, strain and time are the main parameters that affect the resulting, fully recrystallized, microstructure [87, 95, 96]. Strain rate has an effect on the initial (first seconds) mDRX progression, as showed by Nicolaÿ et al [97]. A higher strain rate introduces both a larger ρ_d in the deformed grains, as well as a higher temperature due to the AH, which will lead to a faster mDRX progression at the initial stages.

Dehghan-Manshadi et al. [87] investigated the mDRX behaviour of a 304 austenitic steel with varying initial (before deformation) grain sizes and found that for the if held at temperature after deformation, the fine grained material

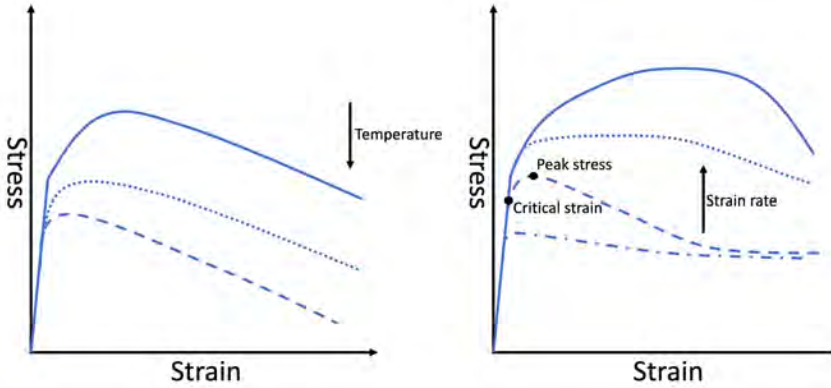


Figure 3.8: Illustration of stress–strain response with varying (a) temperature and (b) strain rate.

showed faster mDRX kinetics, however after a long holding time (1200 s) the microstructure were very similar.

3.3 Recrystallization in Ni-base superalloys

Ni-base superalloys generally have a low to medium γ_{SFE} [98–100], and the primary mode of dynamic recrystallization observed is dDRX [91, 93, 101]. However some suggest that cDRX also can occur at lower deformation temperatures and higher strain rates [93, 101–103], as well as at the initial stages of deformation to later be replaced by dDRX [89]. Multiple dynamic recrystallization phenomena has been seen for different materials [58]. Guo et al. [89] showed that deforming Inconel 625 at 900 °C and 0.1 s^{-1} did not lead to any softening after peak stress was reached, instead a behaviour similar to Fig. 3.1 was observed. They also argued that cDRX was the primary mode of recrystallization for low strains at low temperatures while for higher temperatures dDRX was the primary mechanisms for all investigated strains.

Zhang et al. [104] investigated the dynamic recrystallization mechanisms in a Ni-base superalloy and found that for their deformation parameters (1010 °C–1210 °C, and a strain rate of 0.1 s^{-1}) the flow stress decreased with increasing temperature. The onset of DRX was found to occur at smaller strains with increasing temperature due to the higher dislocation mobility. Zhang et al. [105] also investigated the evolution of recrystallization with strain. They found that DRX had occurred at a strain of 0.1. The increase in recrystallized fraction was most intense between strains of 0.1 and 0.3 where the fraction

increased from 0.05 to above 0.5. Interestingly, when comparing with the average grain sizes of recrystallized grains the largest increase occurred between strains of 0.3 and 0.5 where the average size increased from 6 to 15 μm . This is a clear indication of dDRX where grains first nucleate, and then grow.

Azarbarmas et al. [101] investigated Inconel 718 at multiple strains (0.2, 0.4, 0.7), strain rates ($0.001\text{-}1\text{ s}^{-1}$) and temperatures ($950\text{-}110\text{ }^{\circ}\text{C}$). They showed a similar mechanical response as the one illustrated in Fig. 3.8. The flow stress decreased with increasing temperature and decreasing strain rate, and the peak stress occurred at lower strains with decreasing strain rates. They also found that nucleation occurred through twin boundaries "cutting off" the grain boundary bulges, as illustrated in Fig. 3.6(c.2). These nucleated grains then lost their $\Sigma 3$ characteristics with continued applied strain and became random HAGB. The $\Sigma 3$ boundaries that were present for larger strains were twins created during growth of the recrystallized grains, A and B in Fig. 3.4.

Deforming at high temperatures and low strain rates reduces the observed flow softening prior to steady state flow. Kumar et al. [100] showed that when deforming at $1150\text{ }^{\circ}\text{C}$, with a strain rate of 10^{-4} s^{-1} a steady state in the stress-strain curves were achieved without a flow softening, indicating that DRV has a more prominent role in these deformation regimes, which was attributed to the long deformation times. The increase in flow softening at higher strain rates, up to 1 s^{-1} , was explained by the increase in AH which softened the material. This effect was also shown by D'Souza et al. [106] for deformation at $1140\text{ }^{\circ}\text{C}$ with a strain rate of 0.1 s^{-1} as well as Zhang et al. [93] who measured the critical strain rate to 1 s^{-1} when deforming at $1110\text{ }^{\circ}\text{C}$.

During dynamic recrystallization of Ni-base superalloys, the recrystallized fraction often display a minima at intermediate strain rates. Kumar et al. [100] showed this phenomena in their study, where the lowest DRX fraction was for a strain rate of 0.01 s^{-1} when deforming at $1150\text{ }^{\circ}\text{C}$ to a total strain of 0.7. The reason for this, Nicolaÿ et al. [97] argued, was that for higher strain rates the increase in DRX fraction was actually mDRX during the quench delay. Deforming faster led to both an increase in temperature and dislocation density. With faster strain rates, the relative portion of mDRX of the total time for recrystallization, DRX + mDRX, was larger, since the quench delay is the same independent on deformation parameters. The strain rate this minima occurs increases with decreasing deformation temperature. Since mDRX is a thermally driven process the added driving force from AH at higher strain rates could be necessary lower deformation temperatures.

Qiao et al. [95] investigated the mDRX for a PM Ni-Co-Cr alloy at varying hold times (0-45 s), strain rates ($0.001\text{-}1\text{ s}^{-1}$), strains ($\sim 0.1\text{-}0.4$) and temperatures ($1120\text{-}1080\text{ }^{\circ}\text{C}$). Their result did not show a prior strain dependency of the size of recrystallized grains. Their microstructure also contained γ' , which

they showed affects the pDRX progression, both by acting as nucleation sites during hold as well as pinning of moving GB.

Zouari et al. [96] have also investigated mDRX in Inconel 718 proposed that temperature did not have a large effect on the mDRX progression, but the main parameter was the differences in stored energy between recrystallized and deformed grains. Zouari et al. [107] have also performed an in-situ annealing electron backscatter diffraction (EBSD) investigation on the mDRX progression of Inconel 718 deformed via torsion. Their results showed that for low initial strains, new grains nucleated during annealing, suggesting mDRX and SRX both being in effect at the same time. For higher strains prior to annealing, mDRX was the only mechanisms seen. Another interesting detail in the study was a comparison of mDRX progression during the in-situ annealing and simply maintaining temperature after deformation and investigating the microstructure ex-situ. They showed that the average size of recrystallized grains was similar, but the kinetics were 3-4 times faster for the in-situ annealing. Their reason was the potential of progressive mDRX during cooling and heating between annealing steps (~ 100 °C) and a larger area investigated for the ex-situ samples, which could introduce slower kinetics due to the fact that the stored strain and temperature will decrease with distance from the centre. However, the heating was done via induction for the torsion tests, where the sample was later held, and the heating for the in-situ annealing was done resistively. Differences in heating methods have been shown the yield different recrystallization kinetics [108]. The fact that the in-situ annealing was performed on a surface, while the mDRX occurring when maintaining the temperature after deformation occurred in the bulk of the sample, could also have yielded different kinetics.

Tang et al. [85] investigated mDRX for a Ni-base superalloy at temperatures (950-1100 °C), strain rates ($0.01-1 \text{ s}^{-1}$) and holding times (0-60 s). They showed that the $\Sigma 3$ boundary fraction could be promoted by an increase in strain rate and temperature as these parameters increased the GB mobility during hold. They also showed that for high holding temperatures the fraction of $\Sigma 3$ boundaries could decrease due to grain growth. The growing grains could consume smaller grains nucleated via twinning.

CHAPTER 4

Methods

4.1 Hot deformation

Haynes 282 was supplied as discs with a diameter of 152 mm (6 inches) and thickness of 15 mm cut from a billet. From these disks, samples for hot compression testing were prepared by electric discharge machining (EDM) of a circle at half the radius of the disks, to avoid any significant differences in microstructure between samples. The finished samples were in the form of cylinders with dimensions $\varnothing 8$ mm and 12 mm in length. An illustration is

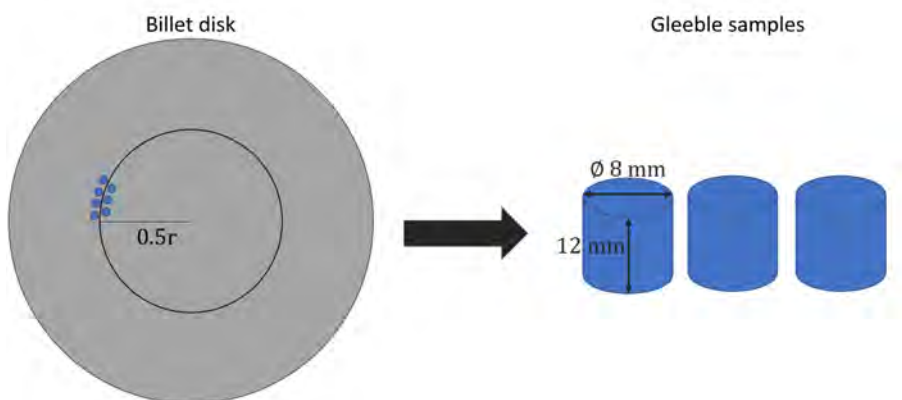


Figure 4.1: Illustration of where the Gleeble samples were machined from the billet.

shown in Fig. 4.1. The disks were flat milled to create good parallelity between the upper and lower side.

During forging of industrial components large variations of the deformation parameters exist throughout the volume, making it difficult to control and capture the actual T , $\dot{\epsilon}$ and ϵ . In order to reduce these uncertainties, dedicated tests on small samples were performed instead of investigating actual forged components. The T , $\dot{\epsilon}$, ϵ and hold time that were investigated were chosen in order to mimic real operations. Another aspect that was taken into consideration was the soaking done on real components. Due to their size, they are often heated for 30-60 minutes, before they are deformed, in order to get an even temperature throughout the whole piece. Depending on the soaking temperature, grain boundary carbides can either nucleate or be dissolved and grain growth can occur. These possible changes in the microstructure were also taken into account. Soaking is performed right before forging, but in order to save time, here soaking was done by placing batches of samples into a preheated furnace for 30 minutes and water quenching (WQ) them. The quick cooling during WQ, and rapid subsequent heating before deformation, hindered any changes to the microstructure.

The equipment used to simulate forging conditions was a Gleeble 3800. The Gleeble as a system offers a wide range of variations in deformation procedures like temperature, heating and cooling rate, $\dot{\epsilon}$ and ϵ . Samples can also be held statically at temperature before and after deformation making it possible to simulate processes like cold deformation with post annealing, or soakings prior to deformation.

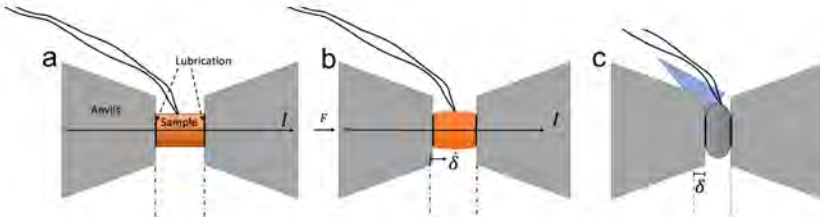


Figure 4.2: Illustration of the deformation procedure in a Gleeble. (a) the sample is placed between two anvils and heated resistively via a current I . Temperature is measured by thermocouples spot welded to the sample. Lubrication, in the form of graphite discs, is applied between sample and anvils. (b) The sample is deformed by movement of one anvil with a deformation rate of δ . (c) At the end of the deformation, heating is turned off and water is sprayed in order to quickly quench the sample and "freeze" the microstructure.

An illustration of how a compression test is performed is shown in Fig. 4.2. The sample was mounted between two anvils, Fig. 4.2(a). Heating was done resistively by applying a current, I , through the sample monitored with a thermocouple spot welded at half the gauge length. In order to reduce friction, lubrication, in the form of graphite sheets, was used between anvils and sample. Different kinds of lubricating media exists [97, 109]. During deformation, Fig. 4.2 (b), one anvil moved towards the other with a given speed, $\dot{\delta}$, compressing the sample. Temperature was still monitored and eventual corrections were made by the heating system to hold the temperature as stable as possible. When the compression was finished, Fig. 4.2(c), the current was turned off and the sample was WQ in order to "freeze" the microstructure. If the sample was subjected to a static hold after compression, the anvils were kept at the end positions while the current would still be driven through the sample in order to keep it at the target temperature. WQ initiated after the specified hold time was reached.

During compression, the Gleeble system registers the force, F , in kN, the displacement, δ , of the anvils in mm, the time in s and temperature in $^{\circ}\text{C}$ from thermocouples spot welded to the sample. For compression, with the assumption of homogenous deformation, the true stress-strain values can then be calculated with the following equation for the strain

$$\varepsilon = \ln \left(\frac{L_0 + \Delta L}{L_0} \right), \quad (4.1)$$

where L_0 is the initial sample length and ΔL the displacement. The stress is

calculated according to

$$\sigma = \frac{F}{A_0} \times \left(\frac{L_0 + \Delta L}{L_0} \right), \quad (4.2)$$

where F is the force, A_0 the initial area and the second term handles the increase in area during compression.

However, when analysing Gleeble data, some consideration have to taken into account. One issue is that deformation parameters are not homogenous throughout the volume. Fig. 4.3(a) shows a FEM simulation of the distribution for the effective ε . The largest ε will occur in the central region and corners of the sample, and be practically zero along the edges in contact with the anvils due to friction. This is not only the case for ε but also T and $\dot{\varepsilon}$ [72]. Because of the aforementioned contact with the anvils, which are water cooled, large amounts of thermal energy is dissipated making the ends much cooler than the middle creating temperature gradients larger than 100 °C [110, 111]. Due to this, the real deformation parameters cannot be easily known, and the evaluation of the microstructural data based on deformation parameters can only be nominal.

Another issue regarding temperature is that around 90 % of the supplied mechanical energy from compression is converted to thermal energy [112, 113], heating the sample. This AH increases with $\dot{\varepsilon}$ [114]. With faster deformation rates, more energy is supplied and converted and it also has less time to dissipate. When this AH is registered, the heating system in the Gleeble will try to compensate and the consequence of this can vary. Fig. 4.3(b) shows the T history with ε for three different $\dot{\varepsilon}$. For $\dot{\varepsilon} = 0.05 \text{ s}^{-1}$ (solid line) a slight increase in temperature from AH is seen at low strains, but since the sample is deformed slowly, the heating system can manage to bring the temperature back down to the target (1080 °C). Increasing $\dot{\varepsilon}$ to 0.5 s^{-1} gives a T history as seen in the dashed line if Fig. 4.3(b). The AH is more severe, but when the system tries to compensate by lowering the supplied current, it overshoots and the temperature falls below the target during deformation. Increasing the $\dot{\varepsilon}$ yet further, to 5 s^{-1} , the temperature increase from AH becomes so pronounced that even though the Gleeble's heating system completely turns off (seen as the temporary dip) the temperature continues to rise, dotted line in Fig. 4.3(b). Recalculating the stress to compensate for the AH at higher $\dot{\varepsilon}$ is sometimes employed [13, 62], however, thus method also assumes homogeneous deformation, which has been stated is not the case.

The third issue arises in the data. The second factor in Eq. (4.2) handles the increase in area of the sample during deformation. However, this assumes

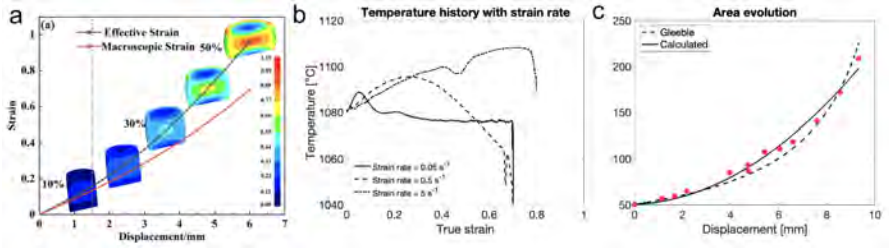


Figure 4.3: (a) FEM simulation of the distribution of strain and the difference between the specified and actual strain with increasing displacement. Courtesy of [95]. (b) The temperature history with strain for 0.05 s^{-1} (solid), 0.5 s^{-1} (dashed) and 5 s^{-1} (dotted). (c) Dashed line shows area evolution according to the second factor in Eq. (4.2). Circles are measured area of samples after deformation and the solid line a polynomial fit to these points.

homogeneous deformation, i.e. that the sample maintains a perfect cylindrical shape throughout the entire deformation. This is of course not the case, as friction between the sample and anvils is impossible to reduce to zero, even with lubrication, and as mentioned, the temperature is not uniform. As a consequence the sample will take on a barreled shape. The area increase is shown in Fig. 4.3(c) where the dashed line show the area increase with displacement according to Eq. (4.2). Meanwhile, the red marks are measured areas of deformed samples with the solid line being a polynomial fit for these. Initially the areas agree quite well, however, Eq. (4.2) then underestimates the areas and at high displacements (corresponding to a strain of about 1.25) starts to overestimate the area compared to areas of actual samples.

One more issue is the quench delay, i.e. the time between the end of deformation and water hitting the sample so that it cools. Nicolaÿ et al. [97] have shown that at high strain rates, the large dislocation density and high adiabatic heating results in mDRX directly after compression if quenching is not immediately initiated. At strain rates of 1 s^{-1} and higher, if the quench delay is around 2 s, more than half of the resulted DRX fraction could be from mDRX and not dDRX. This becomes an issue if it is the dynamic part of recrystallization that is investigated.

All these issues will be present to varying degrees in all Gleeble tests, however, they are seldom taken into consideration within the available literature.

In this research these issues have been tackled in the following ways. The inhomogeneous deformation parameters are impossible to disregard and will always be present in hot compression. Therefore all results presented in the following chapter and papers can and should only be regarded as nominal. All microstructural investigations have also been conducted on a small area

in the central volume of each sample, where the real deformation conditions are highest. To tackle the AH, all dedicated investigations have only been performed with the lowest strain rate, 0.05 s^{-1} , in order to minimize AH. For tests at higher $\dot{\epsilon}$ no compensation for AH were made. This decision was based on the fact that models compensating for AH also assume a homogeneous deformation, which is not the case. These corrections, therefore, introduce more uncertainties into the data. Having a low strain rate was also partly a way to tackle the quench delay. With slower deformation speeds, the relative time of the quench delay compared to the total time for recrystallization (compression + quench delay), was lowered. In order to lower the time of the quench delay, even pipe between the water tank and sample chamber was filled with water prior to deformation. This eliminated the time for the pressure to drive water between the tank and chamber, effectively reducing the time to only the processing speed of the Gleeble switching from compression to the quenching sequence, around 0.2 s.

In order to answer the questions posed in Chapter 1 four experimental matrices were developed and are shown in Table 4.1. Here, the complete history is shown for each sample after machining from the billet piece. Experiment matrix 1 aimed to give a general idea of the DRX and mDRX behaviour for Haynes 282 for different T and $\dot{\epsilon}$. In experiment matrix 2, samples were soaked at $1120 \text{ }^{\circ}\text{C}$ and deformed at $1080 \text{ }^{\circ}\text{C}$, i.e. above and below the secondary carbide solvus temperature. These were compared with samples containing GB carbides in order to determine if these had any effect on the DRX. Experimental matrix 3 was designed to investigate the DRX progression with strain. And finally, with experimental matrix 4 the mDRX kinetics was investigated.

Table 4.1: Experimental matrices.

Experiment matrix 1				
Soak temperature [°C]	Def. temperature [°C]	Strain rate [s ⁻¹]	Strain	Hold time [s]
1060	1060	0.05	0.8	0
1060	1060	0.05	0.8	90
1060	1060	0.5	0.8	0
1060	1060	0.5	0.8	90
1060	1060	5	0.8	0
1060	1060	5	0.8	90
1080	1080	0.05	0.8	0
1080	1080	0.05	0.8	90
1080	1080	0.5	0.8	0
1080	1080	0.5	0.8	90
1080	1080	5	0.8	0
1080	1080	5	0.8	90
1120	1120	0.05	0.8	0
1120	1120	0.05	0.8	90
1120	1120	0.5	0.8	0
1120	1120	0.5	0.8	90
1120	1120	5	0.8	0
1120	1120	5	0.8	90
Experiment matrix 2				
Soak temperature [°C]	Def. temperature [°C]	Strain rate [s ⁻¹]	Strain	Hold time [s]
1120	1080	0.05	0.8	0
1120	1080	0.05	0.8	0
1120	1080	0.05	0.8	0
Experiment matrix 3				
Soak temperature [°C]	Def. temperature [°C]	Strain rate s ⁻¹	Strain	Hold time [s]
1080	1080	0.05	0.1	0
1080	1080	0.05	0.15	0
1080	1080	0.05	0.2	0
1080	1080	0.05	0.4	0
1080	1080	0.05	0.6	0
1080	1080	0.05	0.8	0
1080	1080	0.05	1.25	0
1080	1080	0.05	1.5	0
Experiment matrix 4				
Soak temperature [°C]	Def. temperature [°C]	Strain rate [s ⁻¹]	Strain	Hold time [s]
1080	1080	0.05	0.2	5
1080	1080	0.05	0.2	15
1080	1080	0.05	0.2	30
1080	1080	0.05	0.2	60
1080	1080	0.05	0.2	120
1080	1080	0.05	0.4	5
1080	1080	0.05	0.4	15
1080	1080	0.05	0.4	60
1080	1080	0.05	0.4	120
1080	1080	0.05	0.6	5
1080	1080	0.05	0.6	15
1080	1080	0.05	0.6	60
1080	1080	0.05	0.6	120

4.2 Microstructural investigation

All of the microstructural investigations have been done in a scanning electron microscope (SEM). A SEM uses electrons instead of photons as the illuminating particles. The incoming beam is generated by the electron gun and focused with magnetic lenses. When electrons interact with sample three types of signals are generated: backscattered electrons (BSE), secondary electrons (SE) and X-rays. The SE are electrons knocked out from the outer shells of the sample atoms by the incoming beam. These give topological contrast because different surface features allows varying amounts of SE to escape [115]. BSE are electrons from the beam being scattered back towards the electron gun by interactions with atomic nuclei, and give compositional information (or Z-contrast) because heavier elements generate more BSE [115]. In terms of energies, BSE often have an energy close to the electron beam as the interaction with the nucleus is elastic (but they still experience some inelastic interactions in the sample). Under special conditions, they can also give orientation information, which will be explained in the following sections. The SE have a much lower energy than BSE.

X-rays are generated as a result of electron-electron interactions where the incoming beam either excite or knock out an electron from an inner shell. In order to reduce the energy, the excited electron de-excites, or an electron from an outer shell jumps down to fill the hole left by the knocked out electron. In both cases, this de-excitation generates an X-ray with an energy that is element specific. Analysing X-rays, dispersive X-ray spectroscopy (EDS), generated in an SEM therefore gives elemental information.

Due to the inhomogeneous deformation parameters, only the area/volume at the centre of the sample was investigated. In order to access this region, all samples were cut parallel to the compression direction, Fig. 4.4(a) and the center of the exposed cross section is the region of interest, Fig. 4.4(b). However, this sawing introduced deformation in the near surface region that had to be removed in order to perform the microstructural investigation with the methods explained below. The deformation was removed by encasing the samples in conductive Bakelite and followed by a grinding/polishing procedure described in Table 4.2.

During the investigation of sample matrix 4 a Gatan PECS II system was also available, where instead of polishing with diamond and silica the surfaces were etched with an argon ion beam. The benefits of this is that ions do not introduce any artefacts in the form of scratches which is a potential risk when polishing with diamond particles. The samples that were polished in the PECS II were ground with the same procedure as previously, Table 4.2, to grinding paper P4000. They were later placed in the PECS and etched with a



Figure 4.4: (a) Deformed sample with the cutting plane illustrated. (b) The revealed cross-section of a cut, ground and polished sample with the region of interest marked with the rectangle.

Table 4.2: Grinding and polishing procedures.

Grinding procedure				
Grinding paper	Time [min]	Force [N]	Rotation	Rotation speed (head/bottom) [RPM]
P320	1	25	Co-rotation	150/300
P500	1	25	Co-rotation	150/300
P800	1	25	Co-rotation	150/300
P1200	2	15	Co-rotation	150/300
P2000	5	10	Co-rotation	150/300
P4000	6	10	Co-rotation	150/300

Polishing procedure				
Diamond particle size [μm]	Time [min]	Force [N]	Rotation	Rotation speed (head/bottom) [RPM]
3	12	10	Co-rotation	150/300
1	12	10	Co-rotation	150/300
0.25	12	10	Co-rotation	150/300

Oxide polishing				
Oxide polishing solution	Time [min]	Force [N]	Rotation	Rotation speed (head/bottom) [RPM]
OP-U	15	10	Counter-rotation	150/300

6 keV beam voltage with an incident angle ranging between 8-10° for 80 min rotating at a speed of 3 RPM.

4.2.1 Electron Channeling Contrast Imaging

Electron channeling contrast imaging (ECCI) is an imaging mode in the SEM for capturing the microstructure of a crystalline material. Due to differences in crystallographic orientations electrons will channel into the sample with varying depths. If the channelling reaches high depths, as illustrated in Fig. 4.5(a), the amounts of generated BSE that leave the sample are low. On the other hand, if the channelling is shallow, more BSE able to escape and be detected are generated, Fig. 4.5(b). The difference in contrast therefore is a direct result of the channelling depth, where deeper penetration causes lower contrast. The optimal conditions for ECCI are an electron beam as parallel as possible, with highest possible current, while maintaining a high spatial resolution to minimize the escape area of the BSE [115]. ECCI is a surface sensitive technique (10-100 nm) since increased penetration depth give rise to more

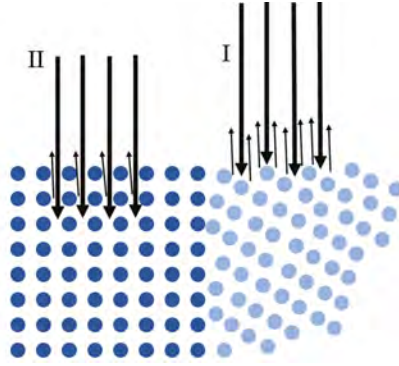


Figure 4.5: Basic illustration of the mechanism giving rise to ECCI

scattering events which destroys the parallel electron beam. It should also be noted that the actual contrast changes between different grains are rather low compared to other phenomena causing BSE, so it is generally necessary to decrease brightness close to zero and maximize the contrast for the SEM image.

Three ECCI images are shown in Fig. 4.6, all are of Haynes 282. Fig. 4.6(a) shows a recrystallized surface after a post deformation hold for 90 s prior to quenching. Each grain has relatively homogeneous contrast due to the low amount of internal dislocation structures rotating the grains. Fig. 4.6(b) shows a microstructure consisting of large deformed grains, which vary in contrast due to dislocations, and smaller recrystallized ones homogeneous in contrast. Fig. 4.6(c) aims to show the importance of a deformation free surface. Here, preparation induced structures are present which give rise to artefacts visible as darker lines. Because the grains are large, and otherwise relatively free from deformation, they are still easily distinguishable in the image but it is easy to see that preparation induced structures can complicate image interpretation. ECCI does not give any orientational information, only differences are seen as variations in contrast. And Z contrast is still the dominating mechanisms. As can be seen from Fig. 4.6, the white dots in (a) are from carbides rich in Cr or Mo and in (c) where C rich MC carbide are the darkest in the image.

4.2.2 Electron Backscattered Diffraction

With EBSD crystallographic information can be acquired, but compared to other techniques, like XRD, this information can be mapped spatially. EBSD

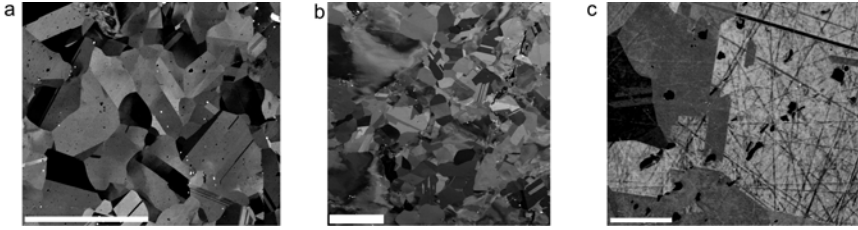


Figure 4.6: ECCI images showing (a) a well polished, fully recrystallized, surface and (b) a surface with both deformed and recrystallized grains. (c) Artefacts from a bad polishing procedure. Scalebar is 50 μm .

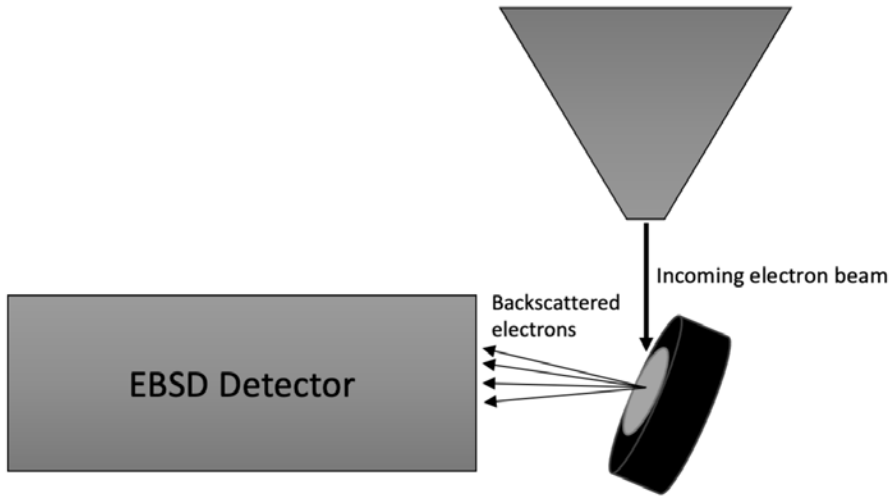


Figure 4.7: Illustration of an EBSD-setup in an SEM with the sample tilted 70° . The BSEs are then scattered into a phosphorescent screen marked as detector.

requires a large angle between the surface normal of the sample and the incoming electron beam so a tilting of 70° is often employed. The BSE are then technically being scattered forward so the detector is placed in front of the sample. An illustration of the setup is shown in Fig. 4.7. The detector consists of a phosphorescent screen with a CCD camera behind it that captures the illumination of the screen. In order to reduce noise from low energy SE a thin metallic layer is added on the phosphor which these electrons cannot penetrate. When the incident beam enters the sample surface, a portion of electrons will scatter inelastically losing a small amount of their energy, making

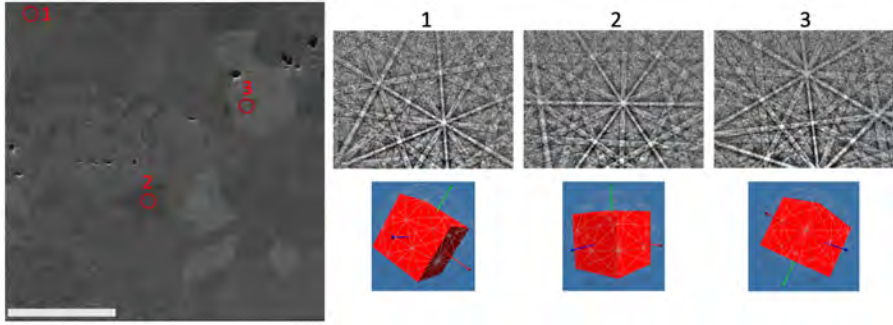


Figure 4.8: Leftmost: SE image with three positions marked. 1-3 the EBSD for each position marked in SE image and the corresponding crystallographic orientation of the grains at each position.

some of them satisfy the Bragg condition,

$$\lambda = 2d_{hkl} \sin \theta_{hkl}, \quad (4.3)$$

where λ is the electron wavelength, θ_{hkl} is the Bragg diffraction angle and d the interplanar spacing. If the condition in Eq. (4.3) is satisfied electrons are diffracted [115–117]. For each plane (hkl) electrons will diffract into two so called Kossel cones with angles of $90-\theta_{hkl}$ and $90+\theta_{hkl}$. Due to the small θ_{hkl} , and proximity of the detector, these two cones appear as parallel (Kikuchi) lines on the phosphorescent screen. A corresponding pair of lines is called a Kikuchi band and a bands angular width is equal to two times the Bragg angle and the corresponding plane spacing can be determined by Eq. (4.3) [117, 118]. Kikuchi bands are shown in Fig. 4.8 where (a) is a SE image with three points marked and the corresponding detector image for each point shown in (b – d). As can be seen, there are multiple bands at each point and they intersect at zone axes corresponding to actual crystallographic directions within the lattice. The set of these Kikuchi bands are called an electron backscatter pattern (EBSP), and from this the crystallographic orientation of the crystal can be determined at that specific spot.

EBSD data analysis

The EBSD acquired at each point is transformed via a Hough transformation where each line is translated into a point in the 2-D Hough space. This makes

it easier for a detection and indexing software to correctly index each pixel [118]. From the Hough transform the orientation, $\mathbf{o}$, is determined and stored as three Euler angles, $\mathbf{o} = (\phi_1, \Phi, \phi_2)$ [119]. The Euler angles are determined by rotating a crystal from an initial reference orientation until it coincides with the measured orientation. For each pixel the Euler angles and a quality metric called mean angular deviation (MAD) is stored. In order to plot and analyse the captured microstructures, the data first needs to be cleaned up and the grains reconstructed. Software used in the work here was CHANNEL 5 and MTEX [120], the latter being an extension in Matlab. In order to recreate grains and clean up some noise a size threshold (in pixels) and misorientation threshold (in degrees) are chosen. If two pixels have an orientation difference larger than the misorientation threshold, they are considered to belong to different grains, and a HAGB is drawn between them. However, to disregard pixels that are just noise, a grain cannot be smaller than the size threshold specified. The data can further be cleaned up by applying some filter. For the work here, the size threshold of grains were set to 5 pixels, while the misorientation was set to either 10 or 15°. A Kuwahara filter was used in order to clean the data.

Euler angles are not often used for orientation representation since small differences in orientations can result in sharp colour changes [119]. Instead, orientations are visually represented with inverse pole figure (IPF) colouring, where each orientation is assigned a colour according to a legend [115]. Each pixel is then coloured depending on which crystal direction is assigned with a certain reference direction ($\mathbf{X}, \mathbf{Y}, \mathbf{Z}$) in the sample coordinate system, e.g. parallel to the sample surface normal in the sample coordinate system. IPF plots for all three reference directions in the specimen coordinate system are shown in Fig. 4.9(a – c), with the corresponding colour map, or IPF legend shown in Fig. 4.9(d).

With EBSD it is also possible to investigate orientation gradients and internal substructures created during deformation. It should be noted that only dislocations that causes rotations of the lattice can be 'seen' (said with quotations because EBSD cannot capture dislocations, only the rotations they cause) and these are called geometrically necessary dislocations (GNDs). In this thesis, visualization of deformation present in a grain was mainly made with three different methods. The first one is called grain orientation spread (GOS) and it is calculated with Eq. (4.4).

$$\text{GOS} = \frac{1}{N} \sum_{i=1}^N \omega(o_i, \Theta), \quad (4.4)$$

ω is the misorientation between the orientation at i , o_i and the average orientation of the grain Θ . where o_i is the orientation at each pixel i and Θ is the average orientation of the grain. ω represents a function that determines the misorientation between two pixels as $o_i^{-1}o_j$ and takes the lowest measured misorientation accounting for symmetry.

GOS results in one value per grain, the average misorientation from the mean orientation, and this value is given a color based on the used color map. A representation is shown in Fig. 4.9(e) and from the attached color map one can determine that the average misorientation from the mean for this grain is around 6° . GOS gives a good and quick indication of the amount of deformation present in each grain, since a heavily deformed grain will result in high misorientations from the average and yield high GOS values. Because it also gives one value per grain it is often used to separate recrystallized and deformed grains [105], by selecting a GOS threshold below which grains are assumed to be recrystallized.

To determine how the deformation is distributed inside a grain, a grain reference orientation deviation (GROD) map can be used. Here, each pixel gets an independent color based on how much the orientation in that pixel deviates from the average orientation of the grain. The equation for calculating the GROD value is shown in Eq. (4.5).

$$GROD_{i,j} = \omega(o_{i,j}, \Theta) \quad (4.5)$$

where $o_{i,j}$ is the orientation at the pixel located on position i,j , Θ is the mean orientation of the grain containing $o_{i,j}$. A GROD plot is shown for a grain in Fig. 4.9(f). It is now seen that the lower part of the grain has rotated furthest from the mean orientation.

In order to obtain even more local information regarding the strain distribution, one can use a kernel average misorientation (KAM) plot. A kernel, grid of 3×3 , 5×5 etc pixels, is chosen and the misorientation of the central pixel to its neighbors in the kernel is determined. If there is a large misorientation, the KAM value will be high, according to Eq. (4.6).

$$KAM_{i,j} = \frac{1}{\|N(i,j)\|} \sum_{(k,l) \in N(i,j)} \omega(o_{i,j}, o_{k,l}) \quad (4.6)$$

where $N(i,j)$ is the set of pixels constituting a kernel, $\|N(i,j)\|$ is the number of pixels, ω is the misorientation between the orientations at the pixel in the centre of the kernel $o_{i,j}$ and the neighbouring pixels $o_{k,l}$. A KAM plot is shown

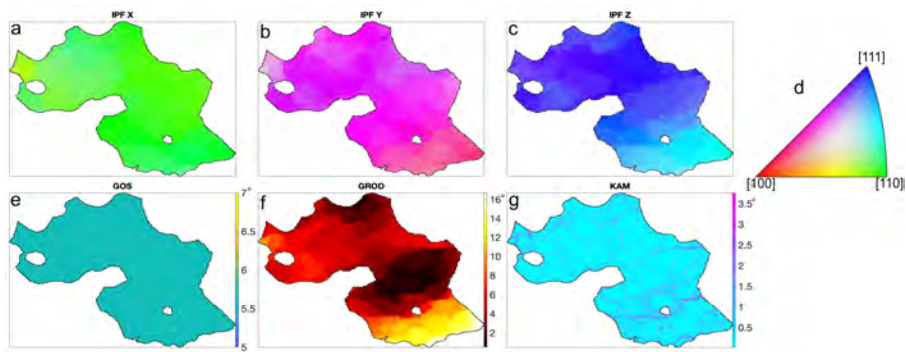


Figure 4.9: Illustration of how EBSD data can be visualized for a grain. (a – c) IPF maps for X, Y and Z directions respectively (legend in (d)). (e) The grain with the colour given by the GOS value. (f) GROD map and (g) KAM map.

in Fig. 4.9(g) and here all subgrain boundaries are visible.

Identification of recrystallized grains

An important aspect of the work here was of course to determine which grains were recrystallized and which were deformed, and this can be done in multiple ways. One is by a grain size threshold, since the nucleated recrystallized grains are smaller than the deformed and EBSD software can determine grain size. Another one is, as mentioned, a GOS threshold. However, setting a definitive parameter or value is challenging and good practice should be to manually investigate each map and consider the deformation procedure employed. For example, Fig. 4.10 shows two maps where the DRX has been determined by a size (equivalent diameter) threshold (a), and based on a GOS value typically used [105], which was 2° . As can be seen, the amount of recrystallized grains vary significantly. The suggested reason for this is that even recrystallized grains can have internal substructures, as will be shown in the results, Chapter 5, leading to increased GOS values. On the other hand, the size of recrystallized and deformed grains are easier to differentiate between. This is also shown in Fig. 4.10(c,d) where (c) shows the calculated DRX fraction with increasing GOS and (d) with increasing grain size. A clear "cut-off" value is seen in (d) from where the DRX fraction stops increasing with grain size, while no such value is seen for GOS. This map shows a sample that has only been subjected to DRX. If mDRX is to be investigated, where recrystallized grains have been given time to grow, both deformed and recrystallized grains could have similar sizes. On the other hand, the recrystallized grains have low

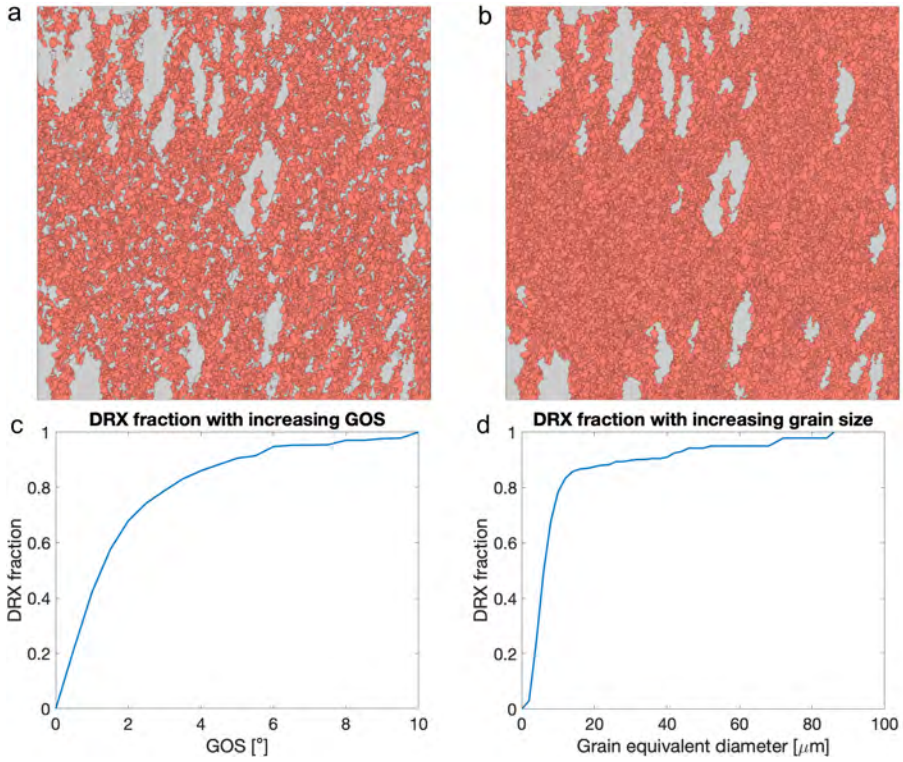


Figure 4.10: Maps of a microstructure where DRX grains are coloured red and deformed grey. In (a) a GOS threshold of 2.5° was used and in (b) the separation was based on a size threshold of $16\text{ }\mu\text{m}$. (c) DRX fraction as a function of GOS and (d) grain diameter.

internal structures so here GOS can prove a more viable separation parameter.

CHAPTER 5

Results

The work presented in this thesis evaluates the microstructural evolution of Haynes 282 during hot compression with different thermomechanical parameters. The mechanical data received from the Gleeble tests was correlated with the microstructural data obtained from the tested specimen by SEM, ECCI and EBSD. It is covered in four papers, each of answers the following questions, see table Table 5.1 posed in Chapter 1.

Table 5.1: Short summation of which papers covered each research question.

Question	Paper			
	I	II	III	IV
When does nucleation/recrystallization start, and how does the microstructure evolve with continued strain?			X	
How does temperature and strain rate affect the microstructure?	X			
What role do primary and secondary carbides have on the dynamic recrystallization behaviour?		X		
How does the microstructure evolve with time when held statically (without progressive deformation) at target temperature?				X
How do parameters like prior strain and temperature affect the evolution of the microstructure?	X			X

5.1 Initial structure

SE images for the initial, billet, structure is shown in Fig. 5.1(a). The present phases, except γ , are intergranular MC carbides, black, as well as secondary carbides, white. No particles are located at grain boundaries. After soaking below secondary carbide solvus temperatures (1100 °C) $M_{23}C_6$ and M_6C carbides are nucleated and grown at grain boundaries, as can be seen after soaking for 1060 °C, Fig. 5.1(b) and 1080 °C, Fig. 5.1(c). When soaking above the solvus temperature no carbides are nucleated, and eventual carbides have been dissolved, Fig. 5.1(d).

IPF maps are shown in Fig. 5.2, where (a) shows the as received sample, (b) after soaking at 1060 °C, (c) 1080 °C and (d) 1120 °C. Each map spans an area of 2.5×2.5 mm with a step size of 7 μm . The corresponding pole figures to each map show a random texture for all samples. Average grain sizes from each map were 150 μm , Fig. 5.2(a), 137 μm , 5.2(b), 177 μm , 5.2(c), and 179 μm , 5.2(d). Soaking for 30 minutes at any temperature did not alter the microstructure from the as received structure significantly, so it can be safe to assume that each sample had a similar structure when being subjected to hot compression.

5.2 Mechanical response during deformation

Fig. 5.3(a) shows the stress-strain curve up to a strain of 1.5, at 1080 °C and 0.05 s^{-1} . This strain was calculated to the the stress where 100 % DRX would be achieved, explained more thoroughly in Section 5.4.1. Initially the material experienced only WH up to a strain of around 0.1, which was determined to be ε_c . At this point DRX was initiated and the hardening rate started to

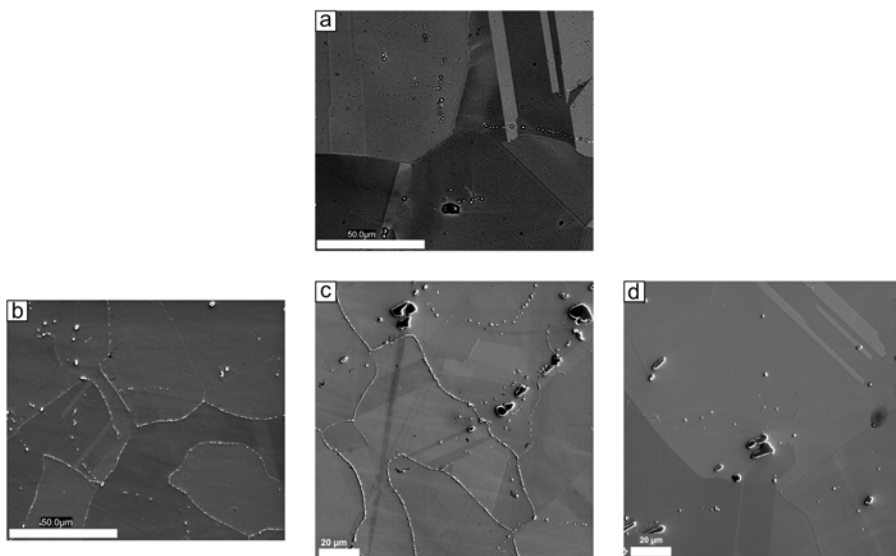


Figure 5.1: SE images of (a) the initial billet structure and the microstructure after (b) soaking at 1060 °C, (c) 1080 °C and (d) 1120 °C.

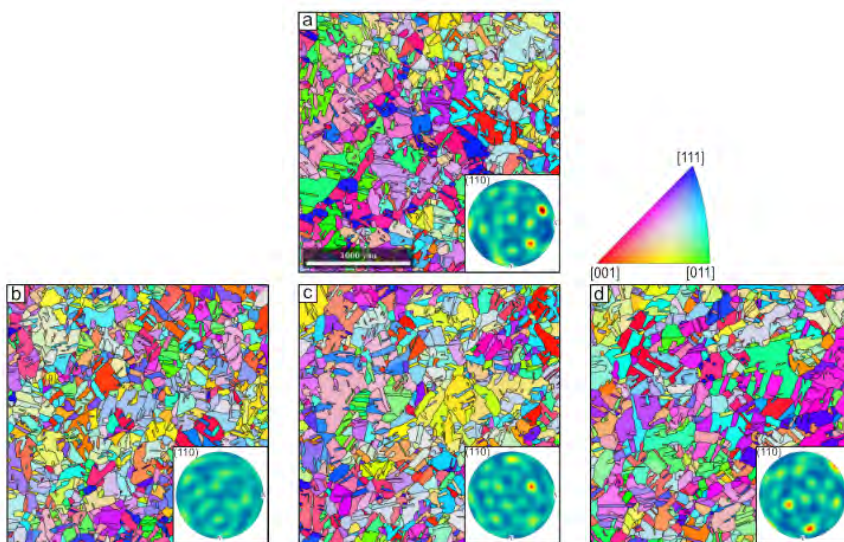


Figure 5.2: Inverse pole figure maps and pole figures for (a) as received structure, (b) soaked at 1060 °C for 30 min, (c) soaked at 1080 °C for 30 min and (d) soaked at 1120 °C for 30 min.

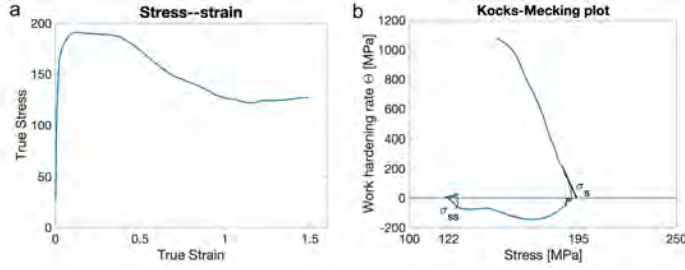


Figure 5.3: (a) Stress–strain curve for a sample compressed to a strain of 1.5. (b) Corresponding K-M plot.

decrease up to the peak strain, ϵ_p , after which the material softened due to DRX being the dominant mechanisms affecting the flow stress. The softening progresses until a plateau is reached. From the data, this seem to occur at a strain of around 1.1. After this point, the steady state is reached. Fig. 5.3(b) shows the K-M plot derived from the stress–strain data. From this σ_s and σ_{ss} can be approximated to be around 195 and 122 MPa respectively.

Stress–strain curves for different combinations of deformation parameters are shown in Fig. 5.4(a–c). Firstly, by increasing the temperature the flow stress decreases. More thermal energy also increases the recovery rate, making the impact from DRV more pronounced, even though DRX is still the dominant softening mechanism. An increase in strain rate, on the other hand, leads to both an increase in ρ_d from the faster deformation, but also a decrease in time which decreases DRV. Even though the dislocation density increases with faster deformation, ϵ_p shifts to higher strains. Shi et al. [13] have also investigated Haynes 282 during hot compression and their results agree well with the data presented here. They achieved steady state stress at lower strains and temperatures than here, but for a strain rate of 0.01 s^{-1} which means that their compression to a strain of 0.7 lasted 5 times as long a compression with the lowest strain rate here. They also argue that a higher temperature promotes both a higher nucleation rate and grain boundary mobility.

Fig. 5.5 shows the temperature evolution with deformation for samples deformed at different strain rates. For the lowest strain rate, Fig. 5.5(a), the slight increase in temperature from adiabatic heating does not cause a severe increase in temperature and the heating system can stabilize it after a couple of seconds. For the intermediate strain rate, Fig. 5.5(b), the heat generated during deformation is higher, and the response from the heating system led to the temperature falling below target at a strain of about 0.65, and continued to decrease until WQ was initialized. For 5 s^{-1} , Fig. 5.5(c), the generated adiabatic heating was so high that the temperature continued to increase even though the Gleeble tried to compensate. Unfortunately, this is seldom re-

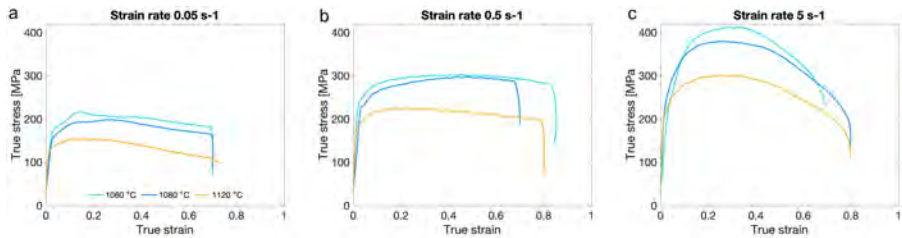


Figure 5.4: Stress-strain curves of samples deformed at 0.05 s^{-1} , 0.5 s^{-1} and 5 s^{-1} at temperatures $1060 \text{ }^{\circ}\text{C}$ (blue lines), $1080 \text{ }^{\circ}\text{C}$ (yellow lines) and $1120 \text{ }^{\circ}\text{C}$ (red lines).

ported in the literature. Instead, only the maximum temperature is plotted, as shown for this data in Fig. 5.5(d), in order to establish the adiabatic heating, or calculated temperature-curves neglecting heating system response are reported. This of course yields some information, for example, it can be seen that with increased strain rate, more heat is generated heating the samples. For the highest strain rate, temperatures rose with more than $20 \text{ }^{\circ}\text{C}$, which technically placed the $1080 \text{ }^{\circ}\text{C}$, 5 s^{-1} samples to above the secondary carbide solvus temperature, even though it was for a very short time. However, as can be seen in Fig. 5.5 the temperature should be investigated throughout the whole deformation procedure since the varying degrees of adiabatic heating lead to different temperature histories, and there are strain rates where the temperature will not only increase, but also decrease, like for the investigated strain rate of 0.5 s^{-1} . The Gleeble system was programmed based on time, so for when deforming at 5 s^{-1} , the heating did turn off before deformation was complete, due to the extra time needed for acceleration and deceleration. This is visible as the "jump" in temperature for one point, Fig. 5.5(c), however, the temperature continued to increase due to AH.

Before forging, soaking is required to get a homogeneous temperature throughout the entire volume. From Fig. 5.1(b,c) it is seen that a 30 minute soak below the carbide solvus introduces a clear carbide structure. As a consequence, Haynes 282 forged below $1100 \text{ }^{\circ}\text{C}$ the GB will contain carbides. Fig. 5.6 shows the stress-strain curves for dedicated tests in order to investigate the potential role of carbides on DRX. Expanding on sample matrix 1, three additional tests were made for which soaking was instead done at $1120 \text{ }^{\circ}\text{C}$ and compressed at $1080 \text{ }^{\circ}\text{C}$. These results are shown as the dark blue lines in Fig. 5.6(a-c). The general trend is that the materials seems to have a slightly lower flow stress without GB carbides, however this is more a result of variations in the Gleeble system. Deforming at identical parameters, with similar microstructure, will still introduce variations in the mechanical data received from the Gleeble. For example, Fig. 5.6(d) shows the temperature vs time 1 s before deformation initiates and during deformation for the samples shown in Fig. 5.6(b). For the

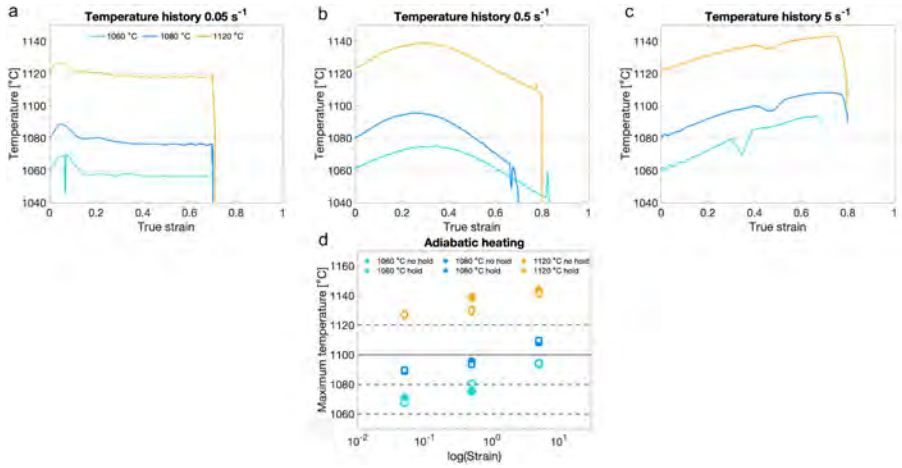


Figure 5.5: Temperature evolution during deformation for samples at a strain rate of (a) 0.05 s^{-1} , (b) 0.5 s^{-1} and 5 (c) s^{-1} at temperatures $1060 \text{ }^{\circ}\text{C}$, $1080 \text{ }^{\circ}\text{C}$ and $1120 \text{ }^{\circ}\text{C}$. (d) Shows the highest measured temperature for samples quenched directly after deformation (filled symbols) or held prior to quenching (empty symbols). The horizontal dashed lines show the target temperatures. The solid horizontal line indicate the carbide solvus.

sample without GB carbides, the temperature starts to increase and this could be the cause for the higher temperature, which would result in the lower flow stress. The variations shown in the stress–strain data in Fig. 5.6(a–c) for samples deformed at $1080 \text{ }^{\circ}\text{C}$ with carbides (yellow) and without (blue) are smaller than variations seen between two nominally identical samples deformed with the same parameters.

5.3 Stress relaxation during hold

The stress–strain data during deformation to strains of 0.2, 0.4 and 0.6 at $1080 \text{ }^{\circ}\text{C}$, 0.05 s^{-1} are shown in Fig. 5.7(a). The stress relaxation during a 120 s hold for the same samples are shown in Fig. 5.7(b). The vertical lines in Fig. 5.7(b) indicate the holding times investigated, namely 5, 15, 60 and 120 s in order to investigate the progression of mDRX. The graph in Fig. 5.7(a) also shows the aforementioned variation in data for samples deformed at similar temperatures and strain rates. Ideally, the curves would follow each other perfectly, and just stop at different strains. All three investigated strains are larger than the peak strain 0.15 which indicates that DRX has initiated and progressed in all samples prior to hold. Looking at the stress during hold, Fig. 5.7(b), it drops

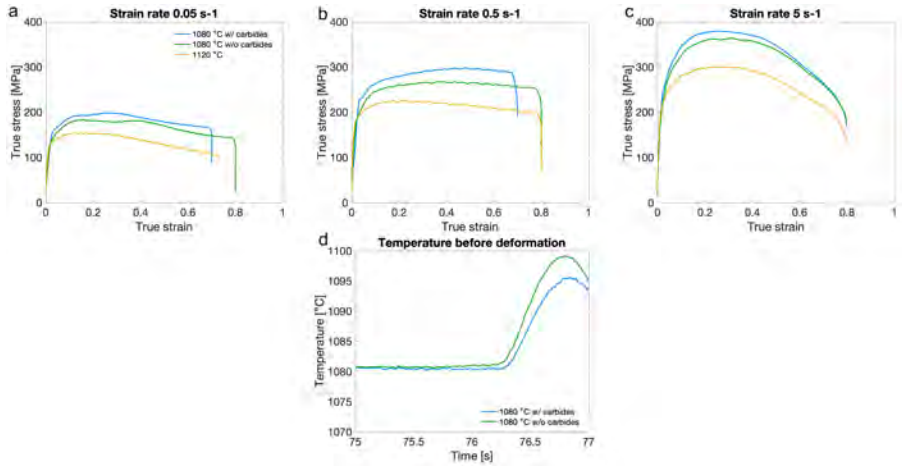


Figure 5.6: Stress–strain curves for samples deformed at 1080 °C with GB carbides (yellow), 1080 °C without carbides (blue) and 1120 °C (red). (a) Strain rate of 0.05 s⁻¹, (b) 0.5 s⁻¹ and (c) 5 s⁻¹. (d) Shows the temperature history right before, and during the start of the deformation.

quickly and a steady value of between ~40 MPa is reached for all samples. This steady value is both somewhat lower and also reached in a shorter time with increasing prior strain but the effect is not pronounced. Fig. 5.8 shows the stress evolution during the 90 s hold for samples from sample matrix 1 with (a) samples deformed at 0.05 s⁻¹ at varying temperatures, (b) 0.5 s⁻¹ and (c) 5 s⁻¹. The cause of the oscillating behaviour seen for temperatures 1060 °C and 1080 °C are likely the result of sub-optimal PID control settings of the Gleeble. However, it can be seen that all samples reach similar steady state stress here as well, and that temperature seems to have the largest contribution to the behaviour.

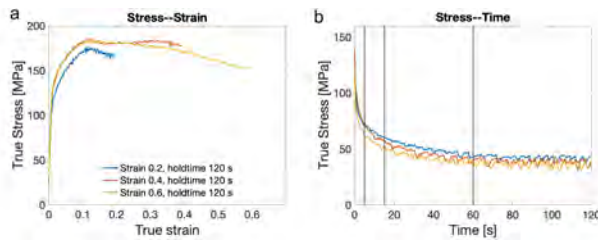


Figure 5.7: (a) Stress–strain curves during deformation for samples deformed to strains of 0.2, 0.4 and 0.6 at 1080 °C and 0.05 s⁻¹. (b) The stress evolution during hold for 120 s after deformation.

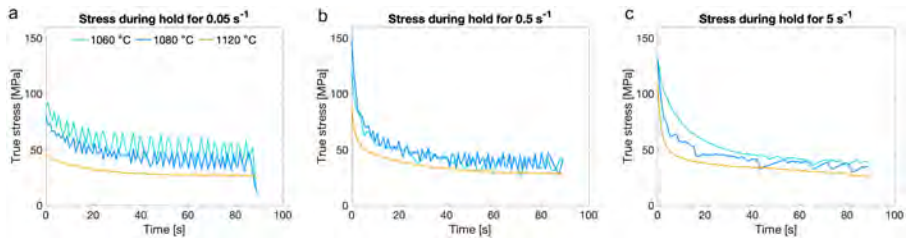


Figure 5.8: Stress relaxation during hold time after deformation for samples deformed with a strain rate of (a) 0.05 s^{-1} , (b) 0.5 s^{-1} and (c) 5 s^{-1} .

5.4 Dynamic recrystallization

As mentioned, the onset of DRX is typically proposed to occur at a critical strain, ε_c , which is seen as a decrease in work hardening rate in the stress-strain response, or as an inflection point in a K-M plot. However, this is not actually the case, since the onset of would have a negligible effect on the stress-strain behaviour. Instead, the decrease in WH would occur somewhat after the initiation of DRX. With continued deformation, more grains are nucleated and eventually a peak strain, ε_p is reached after which the material softens. In this regime the lowering of ρ_d due to DRX and DRV is larger than the generation of dislocations via WH. After softening a steady-state stress is reached, and here softening and hardening are in equilibrium. In regards to the microstructure, in this regime, recrystallized grains become so deformed that new generations of DRX incurs. Due to this, the average grain size with progressive deformation remains rather constant [58].

In order to investigate the microstructural evolution from the 'initiation' to the steady state, a number of samples were deformed to varying strains and then water quenched. In order to isolate the effect of only DRX, and minimize contribution from other parameters, the temperature and strain rate were set to relatively low values, $1080 \text{ }^\circ\text{C}$ and 0.05 s^{-1} in order to reduce adiabatic heating, and the quench delay was managed to get down to 0.2 s . From previous stress-strain curves at similar deformation parameters the peak strain was determined to be around 0.15 , Fig. 5.3(a) and Fig. 5.4(a). So the strains chosen for this investigation were set to 0.1 , 0.15 , 0.2 , 0.4 , 0.6 and 0.8 . Later, additional two samples were deformed to strains of 1.25 and 1.5 . The parameter used to separate recrystallized and deformed grains were based on the equivalent diameter of grains. The threshold values for each strain is shown in Table 5.2.

The microstructures of these samples are shown in Fig. 5.9. Here recrystallized grains are coloured red and deformed in grey. For strains $0.1 - 0.2$ Fig. 5.9(a-c), the MC carbides are coloured in black. Since MC carbides have a similar crys-

Table 5.2: Grain size thresholds chosen for separation between recrystallized and deformed grains for samples deformed to varying strains.

Cut off size	Strain							
	0.1	0.15	0.2	0.4	0.6	0.8	1.25	1.5
	13	13	13	16	16	16	22	22

tal structure to the γ matrix, they were indexed as this phase during EBSD acquisition, even if a dedicated file with their crystal structure information loaded as a separate phase, they were more often than not still indexed as γ . During compression, they flow with the γ matrix and do not become strained. This means, that independent of if the separation between recrystallized and deformed grains are done via a size or a GOS threshold, they will be determined as recrystallized grains. Therefore, for the three strains in Fig. 5.9(a–c) they have been manually excluded to reduce the error of any calculations of DRX fractions. This was done by overlaying the EBSD map over the SE image, where the carbides are easy to identify, see Fig. 5.1(a,c,d), and removing those grains, based on their grain-id, in Mtex. For higher strains, the amount of DRX grains were deemed large enough so that any eventual MC carbide included in the recrystallized set did not induce any significant error to the DRX fraction. Even at the lowest strain, Fig. 5.9(a) some recrystallized grains are visible. The critical strain, ε_c , was therefore chosen to 0.1, however, this slight overestimation of ε_c will not have a significant effect on the fit. With higher strains more recrystallized grains appeared at original random HAGB. Lower energy GB, like $\Sigma 3$ boundaries did not act as initial nucleation sites, which has been shown in other research as well [121]. Instead they first had to become random HAGB by serration with continued compression. The rate of DRX accelerated after a strain of 0.2, i.e. after the peak stress was reached. The clear "necklaces" of recrystallized grains around deformed ones is a typical indication that dDRX is the dominating DRX mechanisms [56]. Another evidence for this is seen in Fig. 5.10. Here, the pole figures for orientations $\parallel \langle 110 \rangle$, for deformed grains in the upper row and recrystallized in the lower, are shown. During deformation, the original grains become textured, rotating towards a $\langle 110 \rangle$ fibre texture. If a recrystallized grain has nucleated via e.g. SIBM there is a higher probability that its orientation will be similar to that of the deformed grain [58, 70], which is observed in the pole figures for recrystallized grains in Fig. 5.10. The texture is not as strong as for the deformed grains.

As can be seen in Fig. 5.11, the average grain size during recrystallization does not increase much with larger strains up to 0.8. Instead, what seems to drive the DRX fraction is continued nucleation of new grains. At larger strains, this shifts and instead growth of grains is what causes continued increase in DRX fraction. Here it is also evident that after the peak stress, the DRX progression

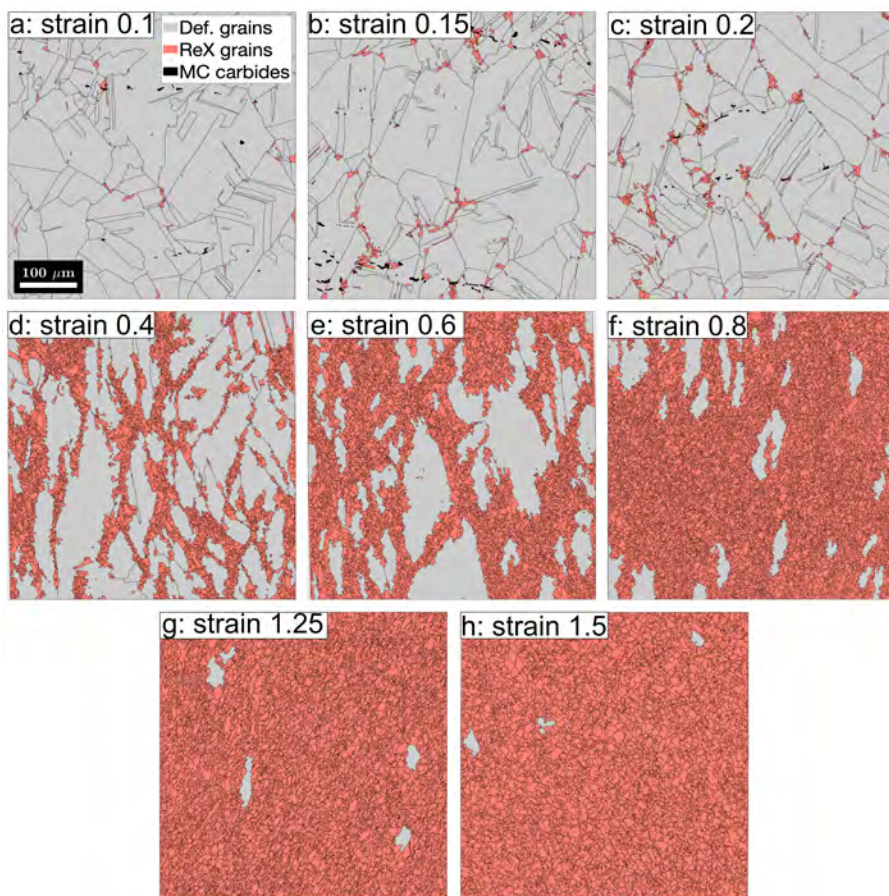


Figure 5.9: Microstructure evolution with progressive strain. The microstructures are captured at strains 0.1, 0.15, 0.2, 0.4, 0.6, 0.8, 1.25 and 1.5. The deformation parameters were 1080 °C, 0.05 s⁻¹. Recrystallized grains are colored red and deformed gray.

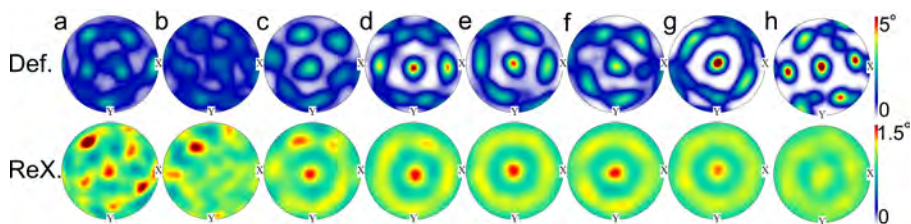


Figure 5.10: Pole figures for the deformed (upper row) and recrystallized grains (lower row). (a) Are the pole figures for strain 0.1, strain 0.15 and (c-h) 0.2–1.5 respectively.

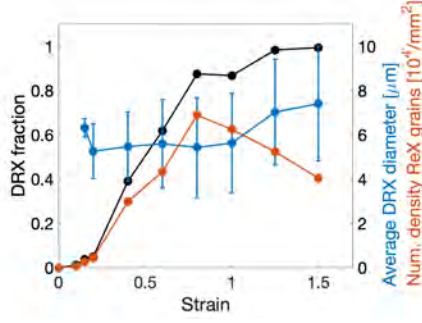


Figure 5.11: DRX fraction, average grain size of recrystallized grains and number density of recrystallized grains for samples deformed to varying strains.

accelerates significantly.

5.4.1 Kinetics

From the microstructural evaluation with increasing strain, the kinetics based on the Avrami equation Eq. (3.15), here written out again for the reader's convenience,

$$X_{DRX} = 1 - \exp \left[-k \left(\frac{\varepsilon - \varepsilon_c}{\varepsilon_p} \right)^n \right] \quad (5.1)$$

could be determined. k and n are calculated by rewriting Eq. (5.1) into

$$\ln(-\ln(1 - X_{DRX})) = n \ln \left(\left(\frac{\varepsilon - \varepsilon_c}{\varepsilon_p} \right) \right) + k \quad (5.2)$$

Fig. 5.12(a) shows the $\ln(-\ln(1 - X_{DRX}))$ vs $\ln \left(\frac{\varepsilon - \varepsilon_c}{\varepsilon_p} \right)$ plot, Eq. (5.2), and the fitted line. Here the orange line and filled circles shows a fit for strains ranging from 0.1 to 1.5. The values of k and n were 0.16 ± 0.10 and 1.56 ± 0.11 respectively. Inserting these values into Eq. (5.1) yielded a kinetics curve for the DRX shown as the orange line in Fig. 5.12(b) with the filled circles being the measured DRX fractions. This suggested a fully recrystallized microstructure at a strain of 1.5. In order to validate this, two additional samples were compressed to a strain of 1.25 and 1.5. The procedure was redone and the fits with these new samples included is shown as the dashed

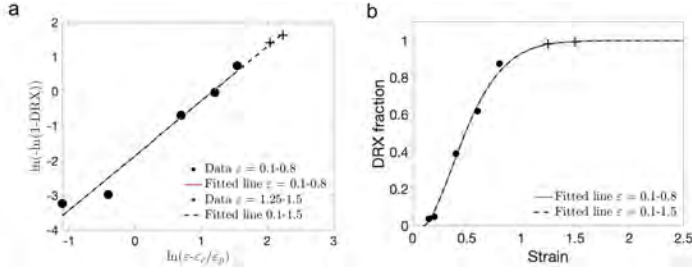


Figure 5.12: Fitting of the Avrami exponents. (a) Graph of Eq. (5.2) in order to calculate k and n based on the measured DRX fractions. (b) The fit of Eq. (5.1). Black circles corresponds to measured DRX fractions for strains between 0.1 to 0.8. Crosses the measured DRX fractions for additional strains of 1.25 and 1.5.

lines in Fig. 5.12(a,b). The fit gave a good estimation and it can be determined that a fully recrystallized microstructure was achieved at a strain of 1.5.

5.4.2 Nucleation of recrystallized grains

The primary mode of recrystallization for all investigated parameters is dDRX. A clear necklace structure, a result of new grains nucleating at grain boundaries of deformed grains can be seen, Fig. 5.9. This is reasonable since Haynes 282 has a relatively low stacking fault energy, being estimated to $\sim 28 \text{ mJm}^{-2}$ by Polkowska et al. [122].

Fig. 5.13(a,b) show grain boundary serrations/bulging indicated by black arrows. Bulging occurs in both "directions", i.e. in the upper part of Fig. 5.13(a,b) the left grain bulges into the right and below the opposite occurs. This indicates that the strains inside deformed grains are localized and that it is this localization of strain that drives nucleation, and not the overall stored energy of the grains. The white arrows in Fig. 5.13(b,e) show two regions with localized high strain in deformed grains. The white rectangles in Fig. 5.13(a,b) show nucleation through the evolution of a subgrain structure, which is analogous to how cDRX operate. However, cDRX usually occurs uniformly throughout an entire grain, and not just at the grain boundaries [58], and at larger strains than investigated here. It has, however, been suggested that the initial stages of cDRX can be similar to dDRX [123], and therefore occur in low γ_{SFE} materials as well, though localized to grain boundaries. Nucleation by twinning was also observed. White rectangles in Fig. 5.13(c-e), more specifically marked with black arrows in Fig. 5.13(d), show nucleated grains that have been cut off from their parent grain by $\Sigma 3$ boundaries.

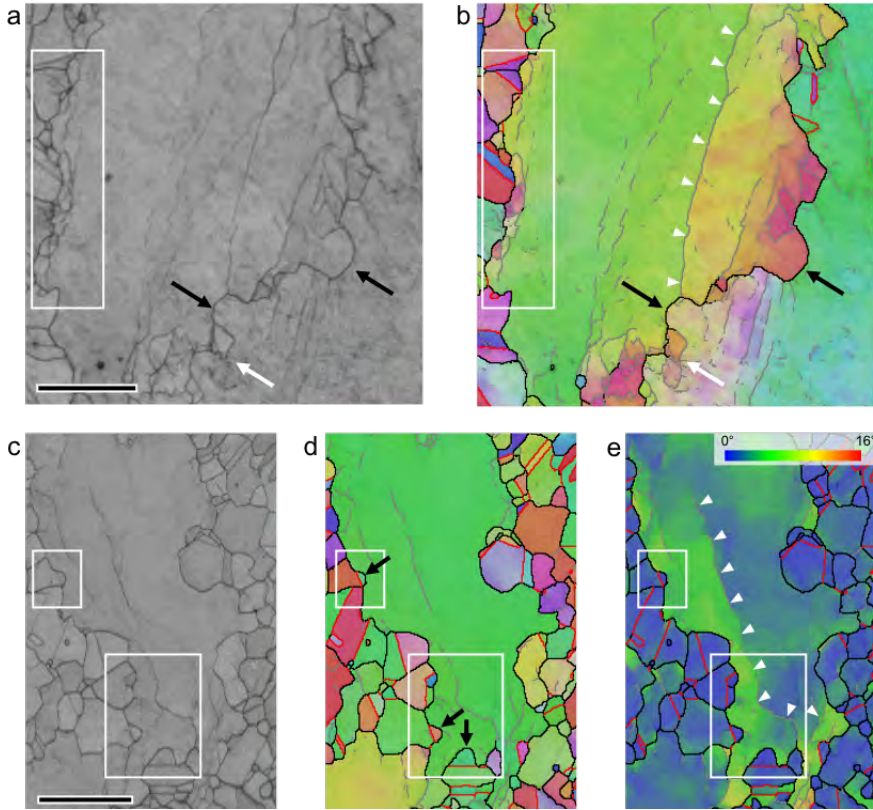


Figure 5.13: Nucleation phenomena occurring at grain boundaries of deformed grains for the sample deformed at 1080 °C, 0.05 s⁻¹. (a, b) Show the band contrast and IPF map of a region where nucleation occurs by serration/bulging of grain boundaries (black arrows), and subgrain rotations (white rectangle and white arrow). (c - e) Show band contrast, IPF and GROD map respectively of another region. Here nucleation occurs by a grain nucleating by being cut off with a twin (black arrows). Two long misorientation fronts seen as a LAGB are marked by white arrow heads in (b, e). Scale bars are 20 μm .

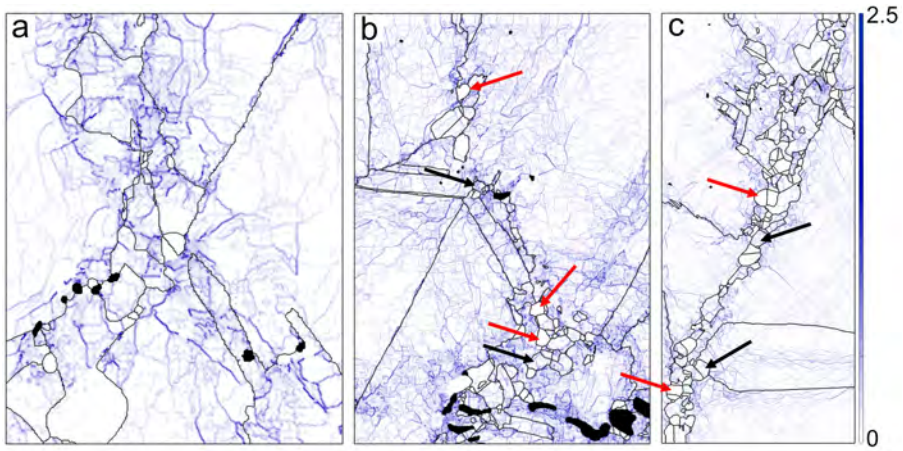


Figure 5.14: KAM maps of magnified regions in the maps in Fig. 5.9 for (a) $\epsilon = 0.1$, (b) $\epsilon = 0.15$ and (c) $\epsilon = 0.2$. Note that the misorientation range in the KAM maps are 0–2.5°, so local misorientations above this value will not be seen. MC carbides are marked in black. Black arrows points at small grains with substructures, while red arrows point to grains with similar size without internal structures.

Another interesting finding was that many small grains present in the microstructure showed an extensive amount of low misorientation sub-structures, even at low strains. Fig. 5.14 shows three maps acquired with higher magnifications on samples deformed to a strain of 0.1, Fig. 5.14(a), 0.15, (b) and 0.2 (c). All of these maps show a part of the central region of the samples, shown in Fig. 5.9(a–c). Zhang et al. [124] showed a similar behaviour where many small grains had internal structures. However, this was for higher strains and their initial microstructure was also partly recrystallized and contained γ' during deformation. They stated that the cause for recrystallized grains to contain substructures was most probably an effect of DRX grains that nucleated early also eventually became deformed. For the low strains shown here, where all recrystallized grains should be relatively recently nucleated via dDRX this appears unreasonable. Especially when the sub-structures are not uniform. Grains of similar size, suggesting they have nucleated at the same time, either show no, or clear sub-structures. There is no correlation between size and misorientations present. Instead, what has been suggested is another type of "nucleation" process. The argument is that during compression, parts of deformed grains are sheared off from the parent, by a creation of a HAGB. The process is unknown, and a transmission electron microscopy (TEM) study would perhaps yield some insights, however the process does not seem to involve a progressive accumulation of dislocations. The sheared off piece inherits the deformed structure of the parent.

5.4.3 Effect of deformation parameters

IPF maps of the microstructure acquired after deformation with varying temperature and strain rate are shown in Fig. 5.15. Deformation at supersolvus 1120 °C, Fig. 5.15(g-i), yielded a microstructure with larger recrystallized grains and fewer remaining deformed ones compared to deformation at sub-solvus temperatures, Fig. 5.15(a-f). Another observation is that deformation at 1120 °C also yielded similar microstructures for all three strain rates. Deformation at sub-solvus temperatures, on the other hand, resulted in microstructure more affected by strain rate, where deformation at the intermediate strain rate, Fig. 5.15(b,e) showed much smaller grains compared to the other strain rates.

This is further showed in Fig. 5.16 where grain size distributions of recrystallized grains for each sample quenched directly after deformation is shown in Fig. 5.16(a-c) for each strain rate respectively. The separation between deformed and recrystallized grains were made with individual size thresholds, which are shown in Table 5.3. Note that grain diameters are presented on a $\log_{10}$ -scale in order to better visualize the distributions of the smaller grains present in 1060 °C and 1080 °C in Fig. 5.16(b). Increased deformation temperature led to larger grains visible in histograms, Fig. 5.16(a-c) as well as in 5.16(d), which shows the average grain diameter and standard deviation for recrystallized grains. Comparing the sub-solvus temperatures (1060 °C and 1080 °C) for each strain rate in Fig. 5.16(d) the average grain size and standard deviation are very similar for samples deformed at these temperatures at the lowest strain rate 0.05 s^{-1} . With increasing strain rate, the average grain size increased for samples compressed at 1080 °C compared to 1060 °C, which is attributed to the higher adiabatic heating. The lowest average diameter was measured during compression at 0.5 s^{-1} for both temperatures. For 0.5 and 5 s^{-1} , grain growth mainly occurs during mDRX at the quench delay (the deformation time was approximately 1.4 and 0.4 s when compressing at 0.5 and 5 s^{-1} while the quench delay was around 2 s). Because the temperature is lower at this stage for the strain rate 0.5 s^{-1} (due to the undershoot in temperature it was actually below the deformation temperature, Fig. 5.5(b)) grain growth was not as efficient. At the lowest strain rate, 0.05 s^{-1} , the deformation time is long and the temperature stabilize quickly, Fig. 5.5(a). It was shown that deforming at 0.05 s^{-1} , for a strain up to 0.8 grain nucleation, and not growth, was the main factor increase the DRX fraction, Fig. 5.11. However, deforming at 1120 °C showed larger recrystallized grain sizes, Fig. 5.16(a,d) suggesting that temperature has an effect on the grain size. Deformation at 1120 °C also indicated that the response from the adiabatic heating is not as impactful as when deforming at lower temperatures. The average grain size and spread was very similar for 1120 °C, 0.5 and 5 s^{-1} , but larger for 0.05 s^{-1} .

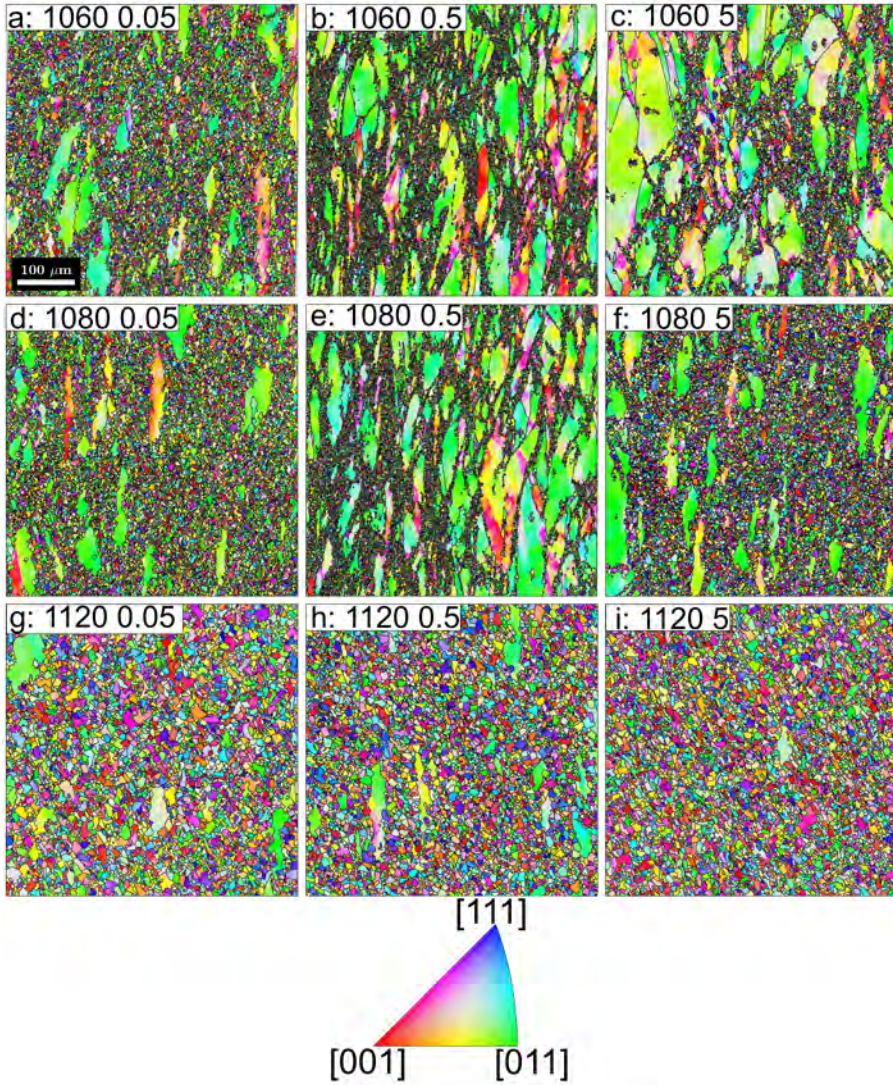


Figure 5.15: IPF maps from samples WQ directly after deformation at varying temperatures (1060 °C, 1080 °C and 1120 °C) and strain rates (0.05 s⁻¹, 0.5 s⁻¹ and 5 s⁻¹).

For a quantitative analysis, recrystallized vs deformed grains were separated based on a equivalent diameter cut off size, specific for each sample (see discussion in Chapter 4). These cut off diameters, for samples quenched directly after deformation, are presented in Table 5.3. The DRX fractions for each sample are presented in Fig. 5.16(e). The upper and lower limits of the error

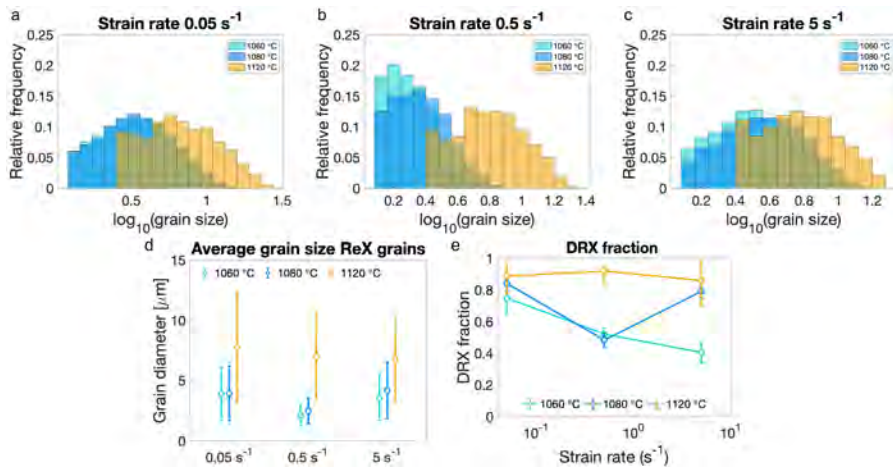


Figure 5.16: Grain size statistics from samples WQ directly after deformation. (a - c): grain size distribution of recrystallized grains, where (a) shows deformation with strain rate 0.05 s^{-1} , (b) 0.5 s^{-1} and (c) 5 s^{-1} . (d) shows the average grain size with standard deviation and (e) the recrystallized fraction for each sample.

Table 5.3: Grain diameter thresholds chosen for separation between recrystallized and deformed grains.

	Strain rate		
Temperature	0.05 s^{-1}	0.5 s^{-1}	5 s^{-1}
1060 °C	12 μm	7 μm	10 μm
1080 °C	15 μm	7 μm	15 μm
1120 °C	25 μm	21 μm	18 μm

bars presented indicate the DRX fraction of the DRX cut off grain diameter was set to 1.5 and 0.8 of the cut off diameter shown in Table 5.3. The DRX fraction decreased with increasing strain rate for samples deformed at 1060 °C, while deformation at 1080 °C showed a "V"-shaped behaviour with the lowest DRX fraction occurring after deformation at 0.5 s^{-1} . As mentioned by Nicolaÿ et al. [97], this "V"-shape is often referred to as the critical strain rate, above which the dDRX is said to be accelerated. However, they argue that it is mDRX during the quench delay, and not dDRX, that is accelerated due to the higher adiabatic heating. With increase in strain rate the deformation time decreases while the quench delay remains constant, leading to the fraction of time the microstructure undergoes dDRX compared to mDRX decreases. Deformation at 1120 °C, showed both the highest DRX fraction of the investigated temperatures at all three strain rates, and showed practically the same DRX fraction for all strain rates.

The temperature effect on dDRX is also shown by Shi et al. [13] who investigated Haynes 282. Increasing temperature led to both increase in recrystallized fraction and grain size, attributed to the higher diffusion rate at higher temperatures, which increased the HAGB mobility of recrystallized grains. Shi et al. also reported the lowest DRX progress at a strain rate of 1 s^{-1} . This could be a result of the Gleeble system undershooting the temperature, as it did for strain rate 0.5 s^{-1} presented in this work, Fig. 5.5(b). Their study did not show the temperature evolution during deformation, so a clear correlation cannot be drawn that the similar responses are due to the Gleeble.

Gardner et al. [14] did both single, and multipass compression tests of Haynes 282, all at a temperature of $1100 \text{ }^{\circ}\text{C}$, and their stress-strain data for the single pass test showed flow softening, reaching steady state at a strain of 0.8 during deformation with a strain rate of 0.2 s^{-1} . It should be noted that their tests were performed in a hydraulic deformation rig with a furnace, making the temperature much more stable during deformation since the heating was not controlled based on the samples temperature. Gardner et al. [14] also saw complete recrystallization of their microstructures. However, they cooled their samples through air cooling. Most likely, mDRX occurred here, consuming the deformed grains present right after deformation.

5.4.4 Effect of secondary phases

As mentioned in Chapter 1, the resulting microstructure will determine the final mechanical properties, and for certain applications, there is an interest in producing a microstructure consisting of smaller grains. As has been shown in the previous sections, deforming at lower temperatures yields smaller recrystallized grains. These will lay the foundation for what is an achievable microstructure after all subsequent HT steps. However, if industrial forging will be performed below the carbide solvus ($1100 \text{ }^{\circ}\text{C}$), there is no escaping the fact that grain boundaries will contain carbides, Fig. 5.1(b,c). Since DRX primarily occurs at original GB, understanding any potential effects the carbides can have on DRX is important.

During the initial 30 min soak at sub-solvus temperatures ($1060 \text{ }^{\circ}\text{C}$, $1080 \text{ }^{\circ}\text{C}$), carbides nucleated and grew at the grain boundaries, as is shown in Fig. 5.17. Black arrows in Fig. 5.17(b), and white arrows in 5.17(c) show small round carbides on a random HAGB. It has been shown that M_{23}C_6 carbides have different morphologies based on the interfacial energy [125], and in a random HAGB they form with an orientation similar to one of the grains, and continue to grow into the other grain. However, neither ECCI nor SE give information

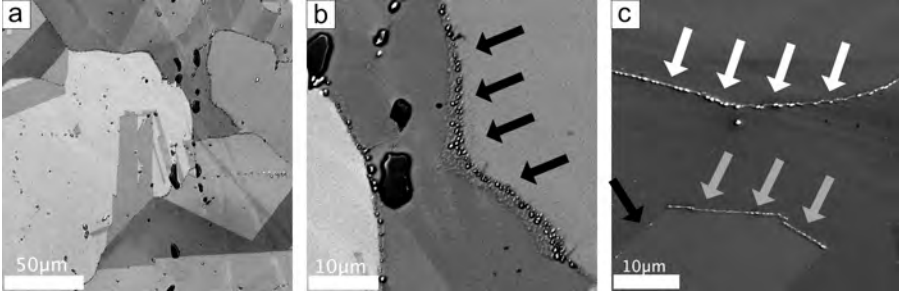


Figure 5.17: Grain boundary carbides morphology in Haynes 282 soaked at 1080 °C for 30 minutes with a subsequent WQ. (a) And (b) are ECCI images also capturing difference in grain orientation. (c) is a SE image showing different morphologies of carbides for different HAGB.

Table 5.4: Grain size thresholds chosen for separation between recrystallized and deformed grains.

Subset	Strain rate		
	0.05 s^{-1}	0.5 s^{-1}	5 s^{-1}
Soak 1080 °C, def 1080 °C	15 µm	7 µm	15 µm
Soak 1120 °C, def 1080 °C	15 µm	8 µm	15 µm

of the misorientation angle like EBSD, and a correlation has not been made to establish the angles for these particular boundaries. Gray and black arrows in Fig. 5.17(c) show smaller carbides on an incoherent $\Sigma 3$ boundary and an absence of carbides on a coherent $\Sigma 3$ boundary due to their low interfacial energy.

The microstructures for samples deformed at 1080 °C with GB carbides are shown in Fig. 5.18(a–c), i.e. the samples soaked at 1080 °C in order to nucleate GB carbides prior to deformation, see Fig. 5.1(c). Fig. 5.18(d–f) show the microstructures after deformation at 1080 °C for samples soaked at 1120 °C, i.e. no GB carbides are present, see Fig. 5.1(d). Upon first glance no indication that the presence of carbides affect the resulting microstructure was observed.

This is further supported by Fig. 5.19(a–d), where grain size distributions in the recrystallized fractions (defined by cut-off grain sizes given in table 5.4) appear practically identical for samples deformed at each strain rate both with and without carbides. Samples deformed without carbides show a marginal shift to a distribution of larger grains. However, since the shift was small and the temperature of the samples without carbides were slightly higher during deformation, as previously described, Fig. 5.6(d), the difference cannot be conclusively assigned to a material response.

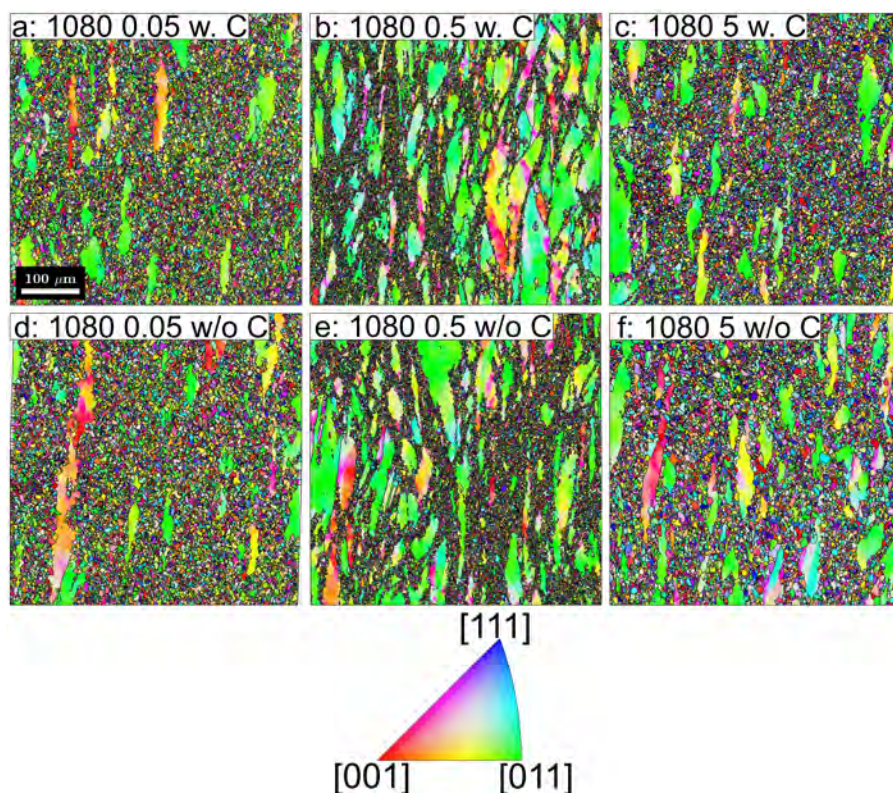


Figure 5.18: IPF maps of samples deformed at 1080 °C with (top row) and without (bottom row) grain boundary carbides. Acquired from the centre of each deformed sample. Increasing strain rate from left to right. Legend on bottom gives information on crystallographic orientation and colour.

Fig. 5.20 shows ECCI images of different areas of a compressed sample, that has been etched in order to reveal the carbides. Fig. 5.20(a,b) were acquired from areas contained in the IPF map shown in Fig. 5.18(a) while Fig. 5.20(c,d) show areas closer to the sample edges, where the strain was less and the area is not as recrystallized. The white ellipses in Fig. 5.20(a,b) contains lines of grain boundary carbides that now lie both intra- and intergrainally through the recrystallized grains, presumably in lines that were grain boundaries prior to deformation. Arrows in Fig. 5.20(b) show a boundary between a deformed grain (left) and recrystallized region (right), with no visible carbides along it.

Going further out, Fig. 5.20(c,d) grain boundary carbides are present at grain boundaries, ellipse 1 and 2 in (c) while the ellipse in (d) shows a grain boundary where no carbides are seen. One thing to note is that the amount of carbides seem a lot less than for the non compressed, soaked pieces, Fig. 5.17. The

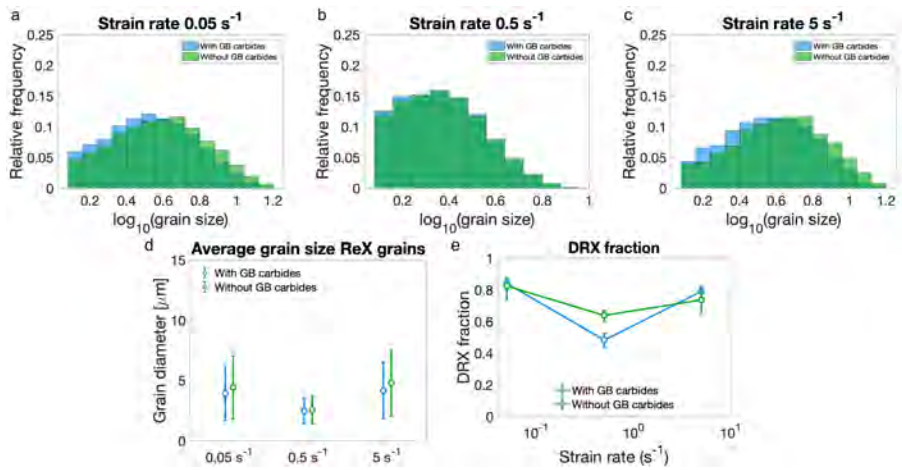


Figure 5.19: Comparison of grain statistics for recrystallized deformed at 1080 °C with or without grain boundary carbides, where (a - c) show grain size distributions after deformation with strain rate of (a) 0.05 s^{-1} , (b) 0.5 s^{-1} and (c) 5 s^{-1} . (d) Shows the average grain size with standard deviation and (e) recrystallized fractions for each sample.

reason for this is unknown. Deformation at 0.05 s^{-1} should not have raised the temperature to 1100 °C, so carbide dissolution should be caused by something else, either the applied strain, or due to DRX.

The conclusion in regards to the role of grain boundary carbides during deformation at sub-solvus temperatures is that no mechanical or microstructural differences observed could be confirmed to occur by changes in material properties, and are instead attributed to experimental variations. So if the carbides have any effect it is insignificant, and other parameters like temperature should instead be taken into consideration when deforming Haynes 282 below the carbide solvus temperature.

The last secondary phase present was the primary MC carbides. Due to their high melting temperature, they were present for all deformation conditions. Their contribution was another nucleation mechanisms, PSN, which was seen to occur frequently. Only MC particles were large and stable enough during material flow to accumulate the necessary ρ_d needed to drive nucleation. What differed between PSN and the other nucleation mechanisms was mainly two things. Firstly, it does not occur at grain boundaries but at MC carbides, often located intragranularly. Secondly, there was no clear "inheritance" of the $\{110\}$ || deformation direction, and no texture preference was observed. Fig. 5.21(b) shows the misorientation profiles between grains marked 1-4 in Fig. 5.21(a) with the lowest misorientation being almost 40°. The grains marked "C" in

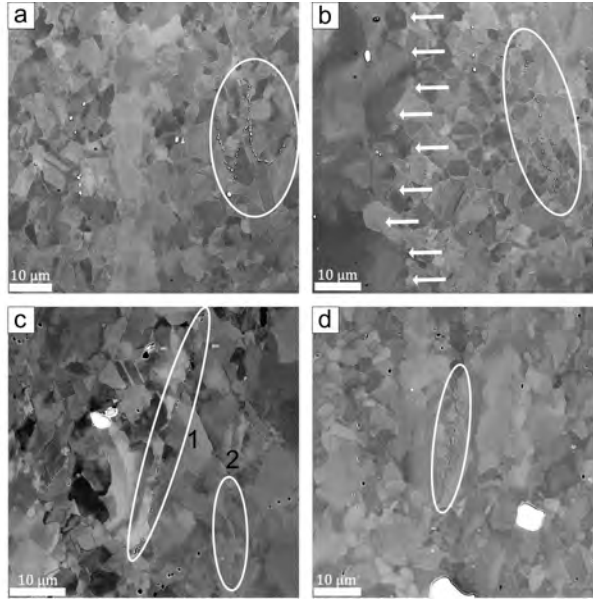


Figure 5.20: ECCI images captured from the sample deformed at 1080 °C, 0.05 s⁻¹ with grain boundary carbides present marked with ellipses. (a, b) are from the centre of the sample while (c, d) are in regions closer to the edge.

Fig. 5.21(a) has likely nucleated through PSN with a carbide either above or below the plane investigated. Fig. 5.21(c,d) show the KAM and GROD maps respectively showing the high local strains around the MC carbides. Fig. 5.21(e-g) clearly show recrystallized grains around MC carbides (in black). All similar sites, even where MC carbides were not seen, are likely associated with PSN. As the data presented is a planar cross-section, a carbide can be located right above or below the investigated surface.

5.5 Meta-dynamic recrystallization

Since industrial pieces are not, or cannot be, quenched quickly due to size or risk of cracking, they are instead typically air cooled and will maintain a high temperature for a time after deformation. It is therefore important to study mDRX and eventual pDRX that will occur during this time since recrystallization will continue post deformation. Here, theoretically, three processes may occur: (i) mDRX via the continued growth of DRX grains if there exists any deformed microstructure; (ii) pDRX via the continued growth of grains after

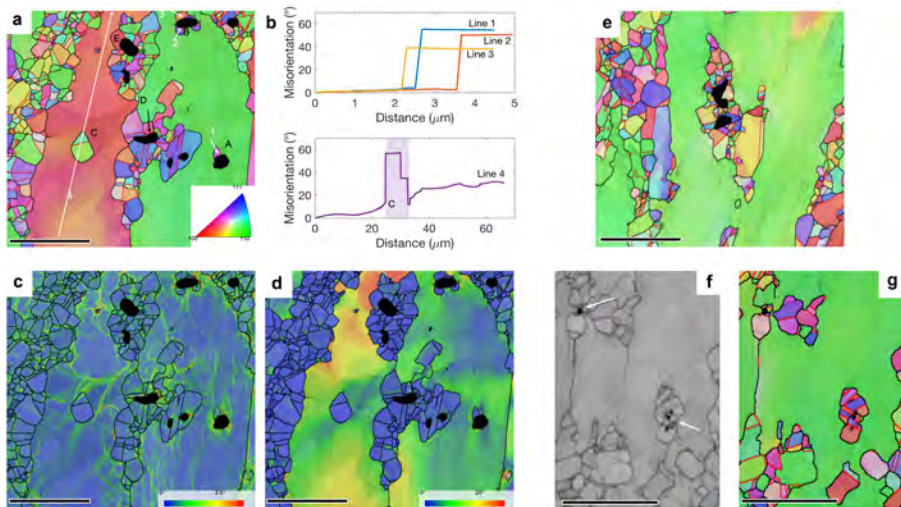


Figure 5.21: IPF maps with carbides marked in black. Clear nucleation and growth has occurred intragranually with carbides as nucleation sites.

complete recrystallization in order to reduce grain boundary area; and lastly (iii) SRX via the nucleation of new grains during the static hold.

The progression of mDRX for different prior strains is shown in Fig. 5.22. Here (a–e) are microstructures after holding for 0, 5, 15, 60 and 120 s after deforming to strain 0.2. Fig. 5.22(f–j) the same holding times but after a strain of 0.4 and (k–o) for a strain of 0.6. Recrystallized grains are colored in red and deformed in gray. Here, the separation of deformed and recrystallized grains were made with a size threshold for Fig. 5.22(a,f,k) (holding time of 0 s), while a GOS threshold of 2° was chosen instead of size for all other samples. However, some manual intervention was necessary. Some grains, specifically for long holding times, were marked as deformed. Upon further inspection many of these grains contained a LAGB while otherwise being free from internal structures. As the boundaries fell below the set HAGB limit (10°) they resulted in identification of single grains with high GOS values. For example, the KAM maps are plotted for the grain marked 1 in Fig. 5.22(e) and 2 in (o) are shown in Fig. 5.23. Even though both of these grains have a GOS value higher than 2° , it is clear that grain 1 has an internal structure indicating that it is, indeed, a deformed grains, whereas grain 2 is essentially free of internal structures, with the exception of the LAGB.

The mDRX kinetics was affected by prior strain. More stored energy and a larger recrystallized area fraction at time 0 increased the rate at which mDRX progressed. Fig. 5.24(a) shows the total recrystallized fraction from both DRX

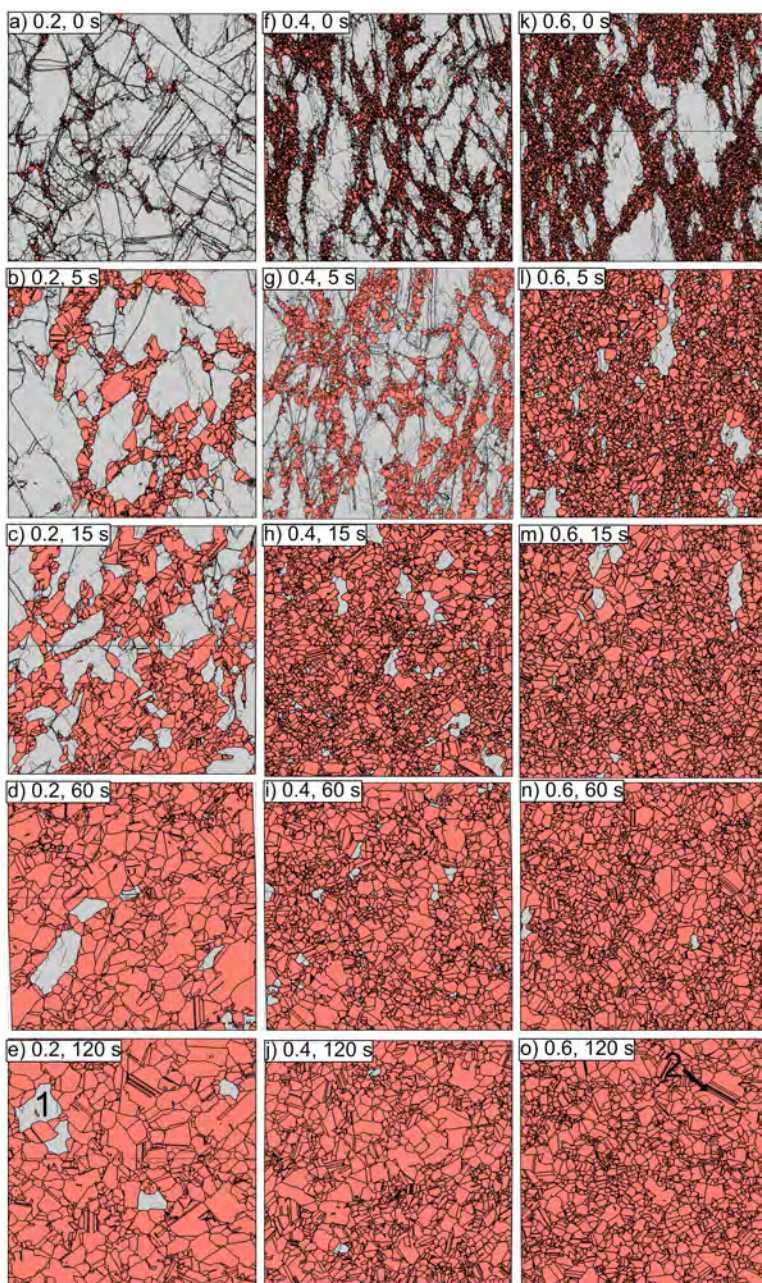


Figure 5.22: Maps of the mDRX progression for samples compressed to different strains (0.2, 0.4 and 0.6) and held for different times (0, 5, 15, 60, 120 s). Gray grains are deformed and red grains recrystallized, based on a GOS threshold of 2° .

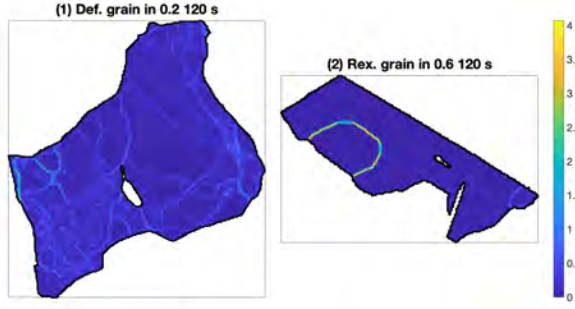


Figure 5.23: Differences in internal structures for grains both having a GOS value larger than 2° .

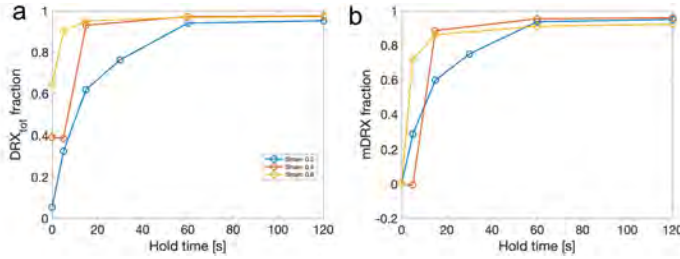


Figure 5.24: (a) Recrystallized fractions, determined by microstructural analysis, with holding times 0 (DRX), 5, 15, 60 and 120 s. (b) The mDRX fractions with hold times as calculated with Eq. (3.20).

and subsequent mDRX. Fig. 5.24(b) shows only the mDRX progression calculated according to Eq. (3.20). Here the steepness of the initial slopes increased with prior strain, i.e. mDRX progressed faster. The slight decrease of both the DRX_{tot} and mDRX fraction seen between 0 and 5 s holding times for strain 0.4 is a result of switching from size to GOS for separation. Most likely, some fraction of the recrystallized grains still contain sub-structures and therefore get a GOS value $> 2^\circ$. In order to receive a more complete picture of the kinetics for strain 0.2, an additional sample was deformed and held for 30 s. The measured recrystallized fraction (map is not shown) indicates that the rate of mDRX slows down somewhat after 15 s.

Fig. 5.25(a) shows the average grain size (equivalent diameter) for recrystallized grains at each holding time and strain. A lower prior strain led to a microstructure consisting of larger recrystallized grains during mDRX. Metzler et al. [15], on the other hand, showed that total strain (0.4 and 0.82) did not matter. However, they considered the average grain size both on the remaining deformed grains and recrystallized ones whereas here, the investigation only concerns the recrystallized grains. Fig. 5.25(b) shows the number

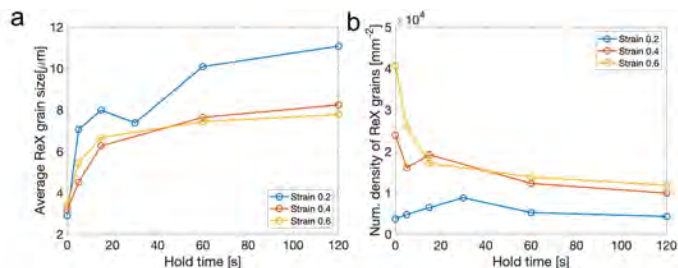


Figure 5.25: (a) Average recrystallized grain size as a function of holding time with varying prior strains. (b) Number density of recrystallized grains.

density of recrystallized grains. For samples deformed to a strain of 0.2, the number density of recrystallized grains increases with increasing holding times up to 30 s, after which it starts to slowly decrease. For samples deformed to the higher strains, 0.4 and 0.6, a rapid decrease in the number density of recrystallized grains was instead observed for holding times up to 30 s, and a slower decrease, similar to samples deformed to 0.2, for longer holding times. The increase in recrystallized grains seen for samples deformed to 0.2 up to 30 s of holding time could indicate SRX where completely new grains nucleate during the hold as suggested by Zouari et al. [107]. However, SRX is defined as both a nucleation and growth process, much like dDRX during deformation. Since a strain of 0.2 corresponds to ε_p for the current deformation parameters, 1080 °C and 0.05 s⁻¹, the material has already begun to recrystallize dynamically. Nuclei should therefore be present in the material already. Therefore, it is suggested that some nuclei present at original GB but they are either too small, or have an misorientation difference lower than the angular resolution of the EBSD setup (0.5°) and are therefore not seen. With holding time, these nuclei grow and become detectable. From Fig. 5.22(a–c) there still exists lengths of serrated HAGBs that has not been consumed by the DRX grains. The drastic decrease in recrystallized grains between 0–30 s for the higher strains suggests that during mDRX some recrystallized grains are consumed. This could stem from the fact that many DRX grains contained substructures, while others did not, see Fig. 5.14. During mDRX the grains with low internal structures could then consume both the deformed grains and recrystallized ones that had internal structures. Another possible explanation is that recrystallized grains coalesce, i.e. they rotate and receive a similar orientation, merging into one grain. Knipschildt [70] has argued that coalescence can occur between subgrains, here, however, two recrystallized grains are separated by a HAGB making coalescence less probable.

In regards to other deformation parameters, the microstructures after deforming to a strain of 0.8 but with varying temperature and strain rate are shown in Fig. 5.26. Here all microstructures are deemed 100 % recrystallized. The

color difference presented here is instead based on size where small grains (below an equivalent diameter of 10 μm for 1060 $^{\circ}\text{C}$ and 1080 $^{\circ}\text{C}$ samples and 20 μm for 1120 $^{\circ}\text{C}$ samples) are shown in red and larger grains in blue. It was noted that there exists a heterogeneous distribution of grain sizes where small grains seem to be present in groups and not uniformly distributed. A possible suggestion for this can be that during mDRX some recrystallized grains will be completely surrounded by other recrystallized grains, and some will border deformed ones, Fig. 5.15. Since the driving force for mDRX is to reduce the strain, the grains bordering a deformed microstructure will have a growth advantage, and these are the larger grains seen after 90 s. Another, not probable, suggestion could be SRX and the small grains have nucleated during the hold. However, nucleation in an un-strained microstructure would not occur. For a comparison similar maps were plotted for strain 0.2 and 0.6 hold time 120 s. These are shown in Fig. 5.27(a) and (b) respectively. There are fewer recrystallized grains that are completely surrounded by an unstrained, recrystallized, microstructure at the lowest strain, Fig. 5.22(a). For the higher strains, 0.6, Fig. 5.22(k), and 0.8, Fig. 5.15(d), on the other hand, few deformed grains remain. The majority of recrystallized grains are not in contact with a deformed grain. Looking at Fig. 5.27(a) no groups of small grains are visible. Instead, most of them are isolated and usually separated from a large grain with a $\Sigma 3$ boundary.

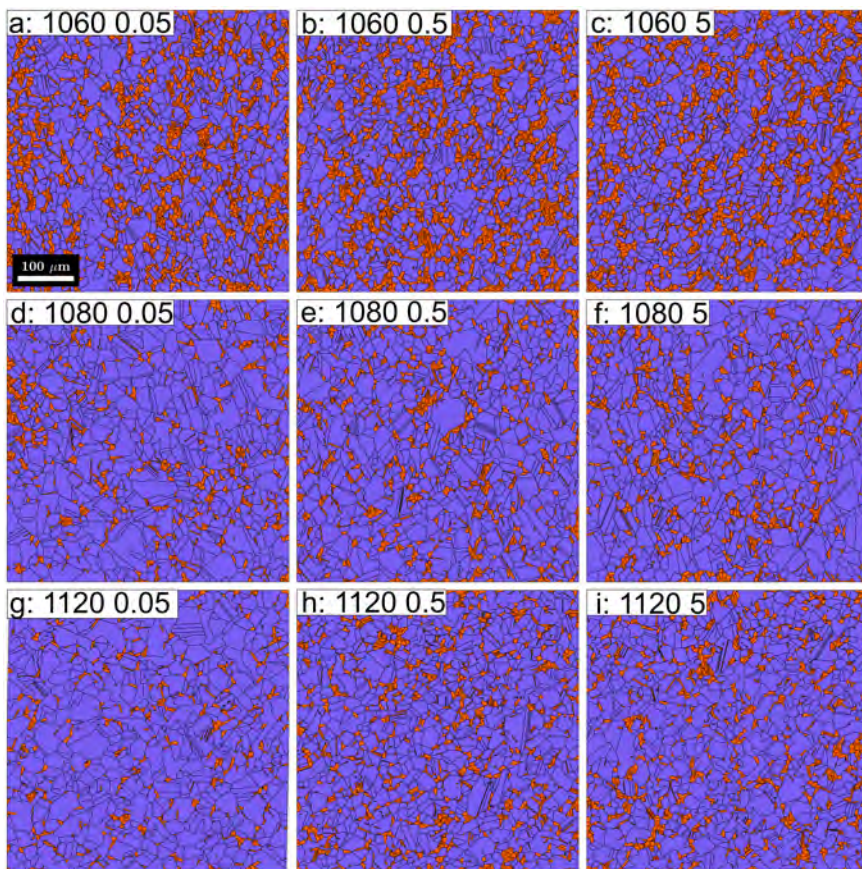


Figure 5.26: Maps of samples held for 90 s prior to WQ. Color is based on size where grains with equivalent diameters < 10 μm are red and larger ones are blue for (a-f). For (g-i) the cutoff diameter was instead 20 μm.

Another factor to consider for the maps in Fig. 5.26 is that effect of the strain rate seen for dDRX has disappeared. Looking at the grain size distributions presented for the hold samples in Fig. 5.28(a-c), and average grain sizes, Fig. 5.28(d), shows that only temperature seem to affect the resulting microstructure, with grain sizes increased with increasing temperature. This has also been shown in other work as well [15, 96]. Zouari et al. [96] also saw that the resulting microstructure after mDRX was not affected by previous $\dot{\epsilon}$ and higher temperatures led to larger grains for a fully recrystallized sample. Metzler et al. [15] investigated temperatures 1100 °C, 1150 °C and 1200 °C, and saw a larger average grain size after a 60 s hold for 1150 °C and 1200 °C than was seen here after 90 s at 1120 °C. They also found that the calculated velocity of grain growth was higher than expected, suggesting that residual

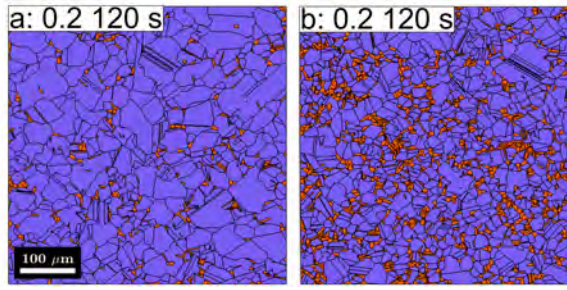


Figure 5.27: Maps of samples deformed to a strain of 0.2 (a) and 0.6 (b) and later held for 120 s. The color is based on size where grains with equivalent diameters $< 10 \mu\text{m}$ are red and larger ones are blue.

stored energy can contribute to an acceleration in grain growth at the initial stages, where deformed grains, are still present.

The microstructure became textured with a fibre $\langle 110 \rangle$ texture parallel to the deformation direction during compression. This can be seen in Fig. 5.29 which shows the textures for deformed and DRX grains from the samples WQ directly after deformation, as well as the texture of the complete microstructure for samples held at the deformation temperature for 90 s prior to WQ. The texture from the deformed grains, seen in the left most columns for each temperature in Fig. 5.29, was adopted by the DRX grains, middle column. This effect was

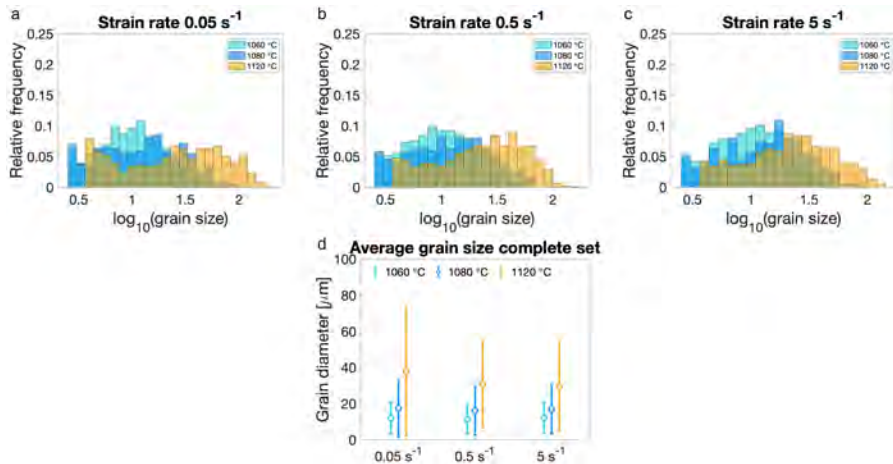


Figure 5.28: Comparison of grain statistics for grains from samples held for 90 s prior to WQ. (a - c) show grain size distributions after deformation and hold with strain rate of (a) 0.05 s^{-1} , (b) 0.5 s^{-1} and (c) 5 s^{-1} . (d) Average grain size with standard deviation for each sample.

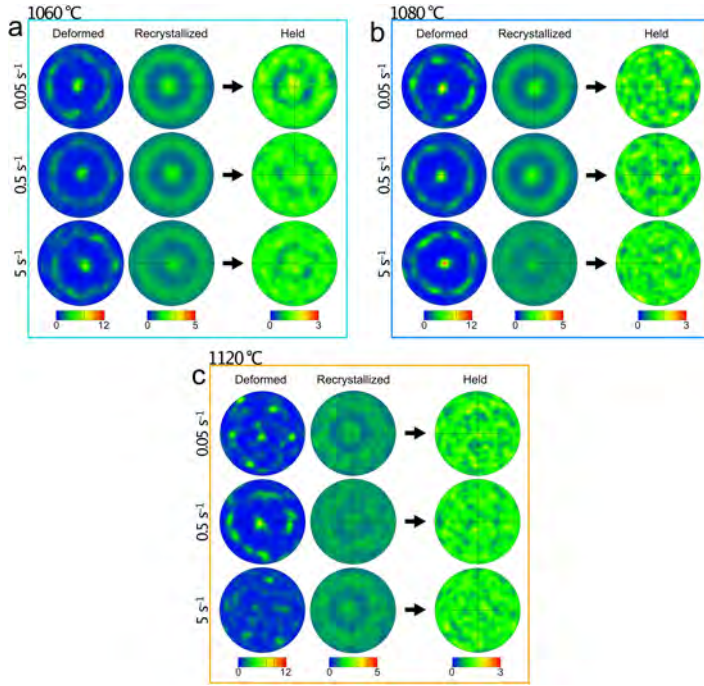


Figure 5.29: Texture evolution of Haynes 282 during deformation and post deformation hold. Pole figures in the $\langle 110 \rangle$ direction for deformed and recrystallized grains in samples WQ directly after deformation, and all grains for the held samples compressed at (a) 1060 °C (b) 1080 °C and (c) 1120 °C.

less pronounced with increasing strain rate where texture is more random for samples deformed at 5 s^{-1} . However, it is also weaker in samples deformed at 1120 °C, suggesting this may be temperature dependent, and the increase in temperature from the adiabatic heating at 5 s^{-1} is what causes this weakening for 1060 and 1080 °C. The $\langle 110 \rangle$ texture parallel to the deformation direction is not as clear in the deformed grains for samples deformed at 1120 °C most likely to the very low statistics. Comparing with Fig. 5.15, lowest row, the microstructures are almost completely recrystallized.

The texture became randomized during the 90 s hold, Fig. 5.29, and could be seen as completely random for 1080 °C and 1120 °C while sampled held at 1060 °C still showed some texture, though weaker than the recrystallized grains after deformation. This was attributed to the generation and growth of annealing twins that occurred during mDRX. The $\Sigma 3$ boundaries create 60° misorientation between the parent and twin. This is further portrayed in Fig. 5.30 showing the $\Sigma 3$ boundary fractions for samples held at 90 s prior to WQ. Fig. 5.30 show the fraction of $\Sigma 3$ boundaries for certain grain size

intervals. Due to statistics, the intervals get larger with increase in grain size, and each fraction is the average for that interval.

With increasing grain size, as the HAGBs move, new twins can also be created with grain growth due to stacking faults [64]. The twin boundary fraction was also seen to increase with temperature, being higher in samples deformed at 1080 °C and 1120 °C, also showing a steeper increase with size compared to 1060 °C. The randomization of the inherited texture on the recently recrystallized grains seen in Fig. 5.29 is attributed to the increase in $\Sigma 3$ boundaries with grain growth during the post deformation hold. On the other hand, investigating the length fraction of $\Sigma 3$ boundaries with holding time, Fig. 5.31(a), showed that independent of initial fractions after deformation the fraction of $\Sigma 3$ quickly (within 15 s) rose to around 0.45 and remained constant. Fig. 5.31(b) shows the $\Sigma 3$ length fractions with average recrystallized grain size for each sample held at varying times. The fraction initially increased with grain size, but saturated and remained constant at later stages. A similar observation was made in Fig. 5.30. The $\Sigma 3$ fraction first increased but eventually saturated at some grain size. However, temperature seemed to have an effect as higher deformation and hold temperatures led to increased $\Sigma 3$ densities. To note here is that technically, Fig. 5.30 and Fig. 5.31 show two different things. Fig. 5.30 shows the $\Sigma 3$ boundary fraction subsets with varying grain size in a microstructure after one holding time, whereas Fig. 5.31(b) shows the average $\Sigma 3$ fraction as a function of average grain size (time). In order to compare these two figures, the $\Sigma 3$ boundary length fraction was plotted vs the average grain size for the samples deformed to 0.8 at 1080 °C and held for 90 s, Fig. 5.30(d) and inserted into Fig. 5.31(b) seen as the black filled circle. The length fraction of $\Sigma 3$ boundaries agree well between studies. In fact, the average $\Sigma 3$ boundary length fractions for samples deformed at 1060 and 1080 °C and held for 90 s (corresponding to Fig. 5.30(a–f)) are presented in paper I. Each sample deformed at the same temperature showed similar values of the average $\Sigma 3$ boundary length fractions, however, it was lower for samples deformed at 1060 °C suggesting temperature has an effect on the $\Sigma 3$ boundaries. The discrepancy that the added sample in Fig. 5.31(b) (black circle) show a larger average grain size most likely stems from different step sizes used when acquiring EBSD data for this map was larger.

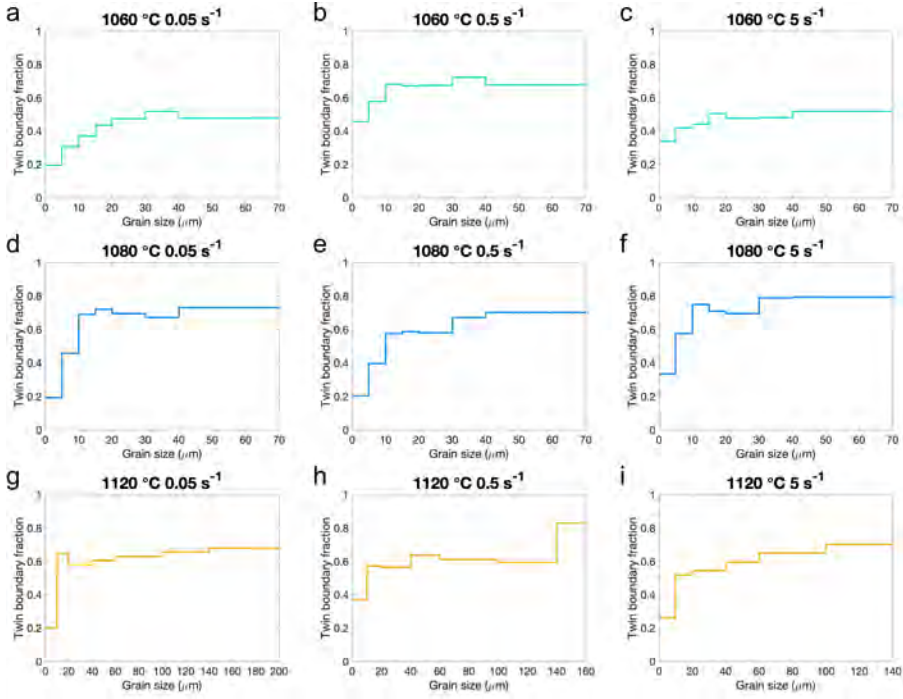


Figure 5.30: Twin boundary fraction with increasing grain size. (a - c) show stair graphs for the $\Sigma 3$ fractions for 0.05 s^{-1} , 0.5 s^{-1} and 5 s^{-1} respectively. (d - f) for $1080 \text{ }^{\circ}\text{C}$ samples and (g - i) for $1120 \text{ }^{\circ}\text{C}$ samples. Please note that values on the x-axis are not uniform since grains in $1120 \text{ }^{\circ}\text{C}$ samples were a lot larger.

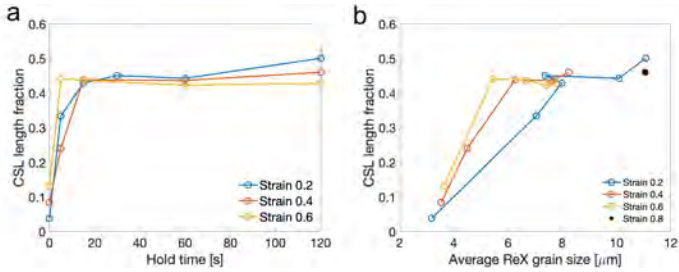


Figure 5.31: (a) $\Sigma 3$ boundary length fraction for the recrystallized microstructure at varying holding times. (b) $\Sigma 3$ boundary length fraction with average recrystallized grain size.

CHAPTER 6

Conclusions & Outlook

6.1 Conclusions

Based on the questions asked in Chapter 1 the main conclusions are given below.

1. *When does nucleation/recrystallization start, and how does the micro-structure evolve with continued strain*

Recrystallized grains were visible at strains as low as 0.1. Nucleation occurred primarily on original GB but also observed at MC carbides. For strains up to 0.8 recrystallization progressed via nucleation of new grains and growth of recrystallized grains occurred at larger strains.

2. How does temperature and strain rate affect the microstructure?

Compression above the caride solvus greatly increased the DRX fractions as well as the size of the recrystallized grains. Deformation at the two lower temperatures did not show significant changes to the resulting microstructures. At the lowest strain rate the adiabatic heating was relatively low and the Gleeble system quickly managed to stabilize the temperature. At the intermediate strain rate, the PID caused the temperature to undershoot, resulting in a lower DRX fraction and smaller grains. At the highest strain rate the large adiabatic heating accelerated and stored energy accelerated the mDRX during the quench delay resulting in a larger DRX fractions compared to the intermediate strain rate. The primary factor affecting the recrystallization behavior during DRX was therefore the strain rate.

3. What role do primary and secondary carbides have on the dynamic recrystallization behaviour?

The primary MC carbides were seen to act as nucleation sites, PSN. Recrystallization therefore also occurred intragranually instead of only intergranually. The grains nucleated via PSN did not show any inheritance of the orientation of the parent grain. Secondary carbides did not show any significant effect on the DRX process.

4 and 5. How does the microstructure evolve with time and prior deformation parameters.

The temperature is the governing factor for the resulting microstructure evolution during mDRX. The effect of strain rate seen during the dynamic part was erased after 90 s. A higher deformation temperature and smaller strain led to a larger grain size. A larger prior strain accelerated the mDRX kinetics, and the resulting microstructure consisted of smaller grains. All investigated parameters gave a similar length fraction of $\Sigma 3$ boundaries.

6.2 Outlook

Based on the observations of the microstructural changes of Haynes 282 during dynamic and meta-dynamic recrystallization presented in Chapter 5, the

following propositions would expand our understanding even further. The more thorough investigation on the dynamic and meta-dynamic recrystallization concerned only one strain rate and temperature, chosen so as to minimize contributions from e.g. adiabatic heating. A next step would therefore be to expand such studies to include more deformation parameters. There is still uncertainty regarding the mechanism which increased the amount of recrystallized grains during a post-deformation hold with a low prior strain. TEM analysis could reveal further details on this as eventual nuclei present on original HAGB not seen in EBSD could be observed. TEM could also allow observations of the dislocation structures at grain boundaries and around MC carbides, which would help understand the nucleation processes even further. A more detailed investigation on the substructures in recrystallized grains could help expand our theory that some small grains present at low strains are sheared of the deformed structure.

The next step would be an in-situ study on the recrystallization. With synchrotron radiation, capturing nucleation and growth of grains during deformation and hold could be possible. This would also avoid the unknown amount of mDRX that occurs during the quench delay. In the early chapters, a lot of focus was also put on the fact that the microstructure controls the mechanical properties. A future study on the correlation of microstructure designed by the investigated deformation parameters and the mechanical properties would therefore be of interest.

Bibliography

- [1] L. M. Pike. HAYNES® 282™ Alloy: A New Wrought Superalloy Designed for Improved Creep Strength and Fabricability. In *Volume 4: Cycle Innovations; Electric Power; Industrial and Cogeneration; Manufacturing Materials and Metallurgy*, pages 1031–1039. American Society of Mechanical Engineers, 2006.
- [2] L.M. Pike. Development of a Fabricable Gamma Prime Strengthened Superalloy. In *Superalloys 2008 (Eleventh International Symposium)*, pages 191–200. TMS, 2008.
- [3] Ceena Joseph, Christer Persson, and Magnus Hörnqvist Colliander. Influence of heat treatment on the microstructure and tensile properties of Ni-base superalloy Haynes 282. *Materials Science and Engineering: A*, 679:520–530, 2017.
- [4] Nitesh Raj Jaladurgam, Hongjia Li, Joe Kelleher, Christer Persson, Axel Steuwer, and Magnus Hörnqvist Colliander. Microstructure-dependent deformation behaviour of a low gamma prime volume fraction Ni-base superalloy studied by in-situ neutron diffraction. *Acta Materialia*, 183:182–195, 2020.

- [5] Shreya Mukherjee, Kaustav Barat, S. Sivaprasad, Soumitra Tarafder, and Sujoy Kumar Kar. Elevated temperature low cycle fatigue behaviour of Haynes 282 and its correlation with microstructure – Effect of ageing conditions. *Materials Science and Engineering A*, 762, 2019.
- [6] Kyeong-Yong Shin, Jin-Hyeok Kim, Mathieu Turner, Byeong-Ook Kong, and Hyun-Uk Hong. Effects of heat treatment on the microstructure evolution and the high-temperature tensile properties of Haynes 282 superalloy. *Materials Science and Engineering: A*, 751:311–322, 2019.
- [7] K Barat, M Ghosh, S Sivaprasad, Sujoy Kumar Kar, and S Tarafder. High-Temperature Low-Cycle Fatigue Behavior in HAYNES 282: Influence of Initial Microstructure. *Metallurgical and Materials Transactions A*, 49:5211–5226, 2018.
- [8] P. Zhang, Y. Yuan, H. Yin, Y. Gu, J. Wang, M. Yang, G. Yang, and X. Song. Tensile Properties and Deformation Mechanisms of Haynes 282 at Various Temperatures. *Metallurgical and Materials Transactions A: Physical Metallurgy and Materials Science*, 49:1571–1578, 2018.
- [9] Adelajda Polkowska, Wojciech Polkowski, Małgorzata Warmuzek, Natalia Cieśla, Grzegorz Włoch, Dariusz Zasada, and Robert M. Purgert. Microstructure and Hardness Evolution in Haynes 282 Nickel-Based Superalloy During Multi-variant Aging Heat Treatment. *Journal of Materials Engineering and Performance*, 28:3844–3851, 2019.
- [10] Sylvio Haas, Joel Andersson, Martin Fisk, Jun-Sang Park, and Ulrich Lienert. Correlation of precipitate evolution with Vickers hardness in Haynes® 282® superalloy: In-situ high-energy SAXS/WAXS investigation. *Materials Science and Engineering: A*, 711:250–258, 2018.
- [11] Fabian Hanning, Abdul Khaliq Khan, Joachim Steffenburg-Nordenström, Olanrewaju Ojo, and Joel Andersson. Investigation of the Effect of Short Exposure in the Temperature Range of 750–950 °C on the Ductility of Haynes® 282® by Advanced Microstructural Characterization. *Metals*, 9:1357, 2019.
- [12] K. A. Rozman, G. R. Holcomb, C. S. Carney, Ö. N. Doğan, J. J. Kruzic, and J. A. Hawk. Effect of 730 °C Supercritical Fluid Exposure on the Fatigue Threshold of Ni-Based Superalloy Haynes 282. *Journal of Materials Engineering and Performance*, 28:4335–4347, 2019.
- [13] Zhaoxia Shi, Xiaofeng Yan, Chunhua Duan, Cunjiang Tang, and Enxiang Pu. Characterization of the Hot Deformation Behavior of a Newly Developed Nickel-Based Superalloy. *Journal of Materials Engineering and Performance*, 27:1763–1776, 2018.

- [14] S Gardner, W Li, M Coleman, and R Johnston. The effects of thermo-mechanical history on the microstructure of a nickel-base superalloy during forging. *Materials Science and Engineering: A*, 668:263–270, 2016.
- [15] D. Metzler and M. G. Fahrmann. The Effect of Prior TMP on Annealed Grain Size in HAYNES 282 Alloy. In *8th International Symposium on Superalloy 718 and Derivatives*, pages 379–389. John Wiley & Sons, Inc., Hoboken, NJ, USA, dec 2014.
- [16] J.H. Westbrook. Chapter 48 Superalloys (Ni-base) and Dislocations — an Introduction. In *Dislocations in Solids*, pages 1–26. North-Holland, 1996.
- [17] Chester Thomas Sims, Norman F Stoloff, and William C Hagel. *Superalloys II*. Wiley, [revision] edition, 1987.
- [18] Jeremy E Watson. *Superalloys : Production, Properties and Applications*. Nova Science Publishers, Incorporated, New York, UNITED STATES, 2011.
- [19] Chester Thomas Sims, Norman F Stoloff, and William C Hagel. *Superalloys II*. New York : Wiley, cop. 1987, 1987.
- [20] Blaine Geddes, Hugo Leon, and Xiao Huang. Strengthening Mechanisms. In *Superalloys: Alloying and Performance*, chapter 3, pages 17–24. ASM International, 2010.
- [21] Blaine Geddes, Hugo Leon, and Xiao Huang. Phases and Microstructure of Superalloys. In *Superalloys: Alloying and Performance*, chapter 4, pages 25–56. ASM International, 2010.
- [22] Roger C Reed. *The Superalloys : Fundamentals and Applications*. Cambridge University Press, Cambridge, UNITED KINGDOM, 2006.
- [23] Nitesh Raj Jaladurgam. *Deformation mechanisms and load distribution in multi phase engineering materials*. PhD thesis, 2021.
- [24] Martin Heilmaier, Utje Leetz, and Bernd Reppich. Order strengthening in the cast nickel-based superalloy IN 100 at room temperature. *Materials Science and Engineering: A*, 319-321:375–378, 2001.
- [25] P.M. Mignanelli, N.G. Jones, E.J. Pickering, O.M.D.M. Messé, C.M.F. Rae, M.C. Hardy, and H.J. Stone. Gamma-gamma prime-gamma double prime dual-superlattice superalloys. *Scripta Materialia*, 136:136–140, 2017.
- [26] L Liu, F Sommer, and H Z Fu. Effect of solidification conditions on MC carbides in a nickel-base superalloy IN 738 LC. *Scripta Metallurgica et Materialia*, 30:587–591, 1994.

- [27] Ceena Joseph. *Microstructure evolution and mechanical properties of Haynes 282*. PhD thesis, Chalmers, 2018.
- [28] Juraj Belan. GCP and TCP Phases Presented in Nickel-base Superalloys. *Materials Today: Proceedings*, 3:936–941, 2016.
- [29] M. Sundararaman, P. Mukhopadhyay, and S. Banerjee. Precipitation of the δ -Ni3Nb phase in two nickel base superalloys. *Metallurgical Transactions A*, 19:453–465, 1988.
- [30] E. Andrieu, N. Wang, R. Molins, and A. Pineau. Influence of Compositional Modifications on Thermal Stability of Alloy 718. In *Superalloys 718, 625, 706 and Various Derivatives (1994)*, pages 695–710. TMS, 1994.
- [31] Y C Lin, Jiao Deng, Yu-Qiang Jiang, Dong-Xu Wen, and Guan Liu. Hot tensile deformation behaviors and fracture characteristics of a typical Ni-based superalloy. *Materials & Design*, 55:949–957, 2014.
- [32] Bilal Hassan and Jonathan Corney. Grain boundary precipitation in Inconel 718 and ATI 718Plus. *Materials Science and Technology*, 33:1879–1889, 2017.
- [33] S M Seo, I S Kim, J H Lee, C Y Jo, H Miyahara, and K Ogi. Eta Phase and Boride Formation in Directionally Solidified Ni-Base Superalloy IN792 + Hf. *Metallurgical and Materials Transactions A*, 38:883–893, 2007.
- [34] L. O. Osoba, A. K. Khan, and O. A. Ojo. Identification of Mo-based Precipitates in Haynes 282 Superalloy. *Metallurgical and Materials Transactions A*, 48:1540–1543, 2017.
- [35] H U Hong, B S Rho, and S W Nam. Correlation of the M23C6 precipitation morphology with grain boundary characteristics in austenitic stainless steel. *Materials Science and Engineering: A*, 318:285–292, 2001.
- [36] C Joseph, C Persson, and M Hörnqvist Colliander. Precipitation Kinetics and Morphology of Grain Boundary Carbides in Ni-Base Superalloy Haynes 282. *Metallurgical and Materials Transactions A*, 51:6136–6141, 2020.
- [37] Emil Eriksson and Magnus Hörnqvist Colliander. Dynamic and Post-Dynamic Recrystallization of Haynes 282 below the Secondary Carbide Solvus. *Metals*, 11:122, 2021.
- [38] Bert Verlinden, Julian Driver, Indradev Samajdar, and Roger D Doherty. *Thermo-Mechanical Processing of Metallic Materials*. Number Vol. 11 in Pergamon Materials Series. Elsevier Science, Amsterdam, 2007.

- [39] Vasim A. Shaikh and Lokesh Arvind Borse. A Review of Cold Forging. *INTERNATIONAL JOURNAL OF SCIENTIFIC RESEARCH*, 4, 2015.
- [40] Prashant Mangukia. Cold and Warm Forging. In *Cold and Hot Forging*, chapter 17, pages 211–235. ASM International, feb 2005.
- [41] M. Zhan, Z. Sun, and H. Yang. Modeling of Hot Forging. In *Comprehensive Materials Processing*, pages 441–493. Elsevier, 2014.
- [42] Matthew J Donachie and Stephen J Donachie. *Superalloys - A Technical Guide* (2nd Edition).
- [43] B. Aksakal, F. H. Osman, and A. N. Bramley. Analysis of Open die Forging. In *Proceedings of the Twenty-Ninth International Matador Conference*, pages 415–419. Macmillan Education UK, London, 1992.
- [44] Robert William Kerr Honeycombe. *The plastic deformation of metals*. Edward Arnold, 2. ed. edition, 1984.
- [45] E. Orowan. Zur Kristallplastizität. I. *Zeitschrift für Physik*, 89:605–613, 1934.
- [46] M. Polanyi. Über eine Art Gitterstörung, die einen Kristall plastisch machen könnte. *Zeitschrift für Physik*, 89:660–664, 1934.
- [47] G. Taylor. The mechanism of plastic deformation of crystals. Part I.—Theoretical. *Proceedings of the Royal Society of London. Series A, Containing Papers of a Mathematical and Physical Character*, 145(855):362–387, 1934.
- [48] Smallman R.E. and Ngan A.H.W. *Physical Metallurgy and Advanced Materials Engineering*. Elsevier, 2007.
- [49] Milton Ohring. *Engineering Materials Science*. Elsevier, 1995.
- [50] D Hull and D J Bacon. *Introduction to Dislocations*. Elsevier Science & Technology, Oxford, UNITED KINGDOM, 2011.
- [51] Quan Guo-Zheng. Characterization for Dynamic Recrystallization Kinetics Based on Stress-Strain Curves. In *Recent Developments in the Study of Recrystallization*, pages 61–88. InTech, feb 2013.
- [52] U. F. Kocks. Laws for Work-Hardening and Low-Temperature Creep. *Journal of Engineering Materials and Technology*, 98:76–85, 1976.
- [53] H. Mecking and U.F. Kocks. Kinetics of flow and strain-hardening. *Acta Metallurgica*, 29:1865–1875, 1981.
- [54] Yuri Estrin. Dislocation-Density-Related Constitutive Modeling. In *Unified constitutive laws of plastic deformation*, chapter 2. Academic Press, Inc., 1996.

- [55] M Hatherly, Anthony Rollett, F J Humphreys, and Gregory S Rohrer. *Recrystallization and Related Annealing Phenomena*. Elsevier Science & Technology, London, UNITED KINGDOM, 2004.
- [56] Taku Sakai, Andrey Belyakov, Rustam Kaibyshev, Hiromi Miura, and John J Jonas. Dynamic and post-dynamic recrystallization under hot, cold and severe plastic deformation conditions. *Progress in Materials Science*, 60:130–207, 2014.
- [57] R D Doherty, D A Hughes, F J Humphreys, J J Jonas, D.Juul Jensen, M E Kassner, W E King, T R McNelley, H J McQueen, and A D Rollett. Current issues in recrystallization: a review. *Materials Science and Engineering: A*, 238:219–274, 1997.
- [58] K. Huang and R.E. Logé. A review of dynamic recrystallization phenomena in metallic materials. *Materials & Design*, 111:548–574, 2016.
- [59] D.G. Cram, X.Y. Fang, H.S. Zurob, Y.J.M. Bréchet, and C.R. Hutchinson. The effect of solute on discontinuous dynamic recrystallization. *Acta Materialia*, 60:6390–6404, 2012.
- [60] Gangshu Shen, S. L. Semiatin, and Rajiv Shivpuri. Modeling microstructural development during the forging of Waspaloy. *Metallurgical and Materials Transactions A*, 26:1795–1803, 1995.
- [61] A.M Wusatowska-Sarnek, H Miura, and T Sakai. Nucleation and microtexture development under dynamic recrystallization of copper. *Materials Science and Engineering: A*, 323:177–186, 2002.
- [62] Jue Wang, Jianxin Dong, Maicang Zhang, and Xishan Xie. Hot working characteristics of nickel-base superalloy 740H during compression. *Materials Science and Engineering: A*, 566:61–70, 2013.
- [63] D.G. Cram, H.S. Zurob, Y.J.M. Brechet, and C.R. Hutchinson. Modelling discontinuous dynamic recrystallization using a physically based model for nucleation. *Acta Materialia*, 57:5218–5228, 2009.
- [64] G Gottstein. Annealing texture development by multiple twinning in f.c.c. crystals. *Acta Metallurgica*, 32:1117–1138, 1984.
- [65] Nathalie Bozzolo and Marc Bernacki. Viewpoint on the Formation and Evolution of Annealing Twins During Thermomechanical Processing of FCC Metals and Alloys. *Metallurgical and Materials Transactions A*, 51:2665–2684, 2020.
- [66] H Gleiter. The formation of annealing twins. *Acta Metallurgica*, 17:1421–1428, 1969.

- [67] Guo-Zheng Quan, Gui-Sheng Li, Tao Chen, Yi-Xin Wang, Yan-Wei Zhang, and Jie Zhou. Dynamic recrystallization kinetics of 42CrMo steel during compression at different temperatures and strain rates. *Materials Science and Engineering: A*, 528:4643–4651, 2011.
- [68] Paul A. Beck and Philip R. Sperry. Strain Induced Grain Boundary Migration in High Purity Aluminum. *Journal of Applied Physics*, 21:150–152, 1950.
- [69] Taku Sakai. Dynamic recrystallization microstructures under hot working conditions. *Journal of Materials Processing Technology*, 53:349–361, 1995.
- [70] Elisabeth Filippa Ferdinand Knipschildt. Nucleation of recrystallization. *Materials Science and Technology*, 38:1–15, 2022.
- [71] Dierk Raabe. Recovery and Recrystallization: Phenomena, Physics, Models, Simulation. In *Physical Metallurgy*, pages 2291–2397. Elsevier, 2014.
- [72] C. Kienl. *Hot forging of the nickel-base superalloy ATI 718Plus*. PhD thesis, University of Cambridge, 2019.
- [73] Longfei Li, Wangyue Yang, and Zuqing Sun. Dynamic Recrystallization of Ferrite with Particle-Stimulated Nucleation in a Low-Carbon Steel. *Metallurgical and Materials Transactions A*, 44:2060–2069, 2013.
- [74] Elena V. Pereloma, Parvez Mannan, Gilberto Casillas, and Ahmed A. Saleh. Particle stimulated nucleation during dynamic and metadynamic recrystallisation of Ni-30%Fe-Nb-C alloy. *Materials Characterization*, 125:94–98, 2017.
- [75] D.S. Weaver and S.L. Semiatin. Recrystallization and grain-growth behavior of a nickel-base superalloy during multi-hit deformation. *Scripta Materialia*, 57:1044–1047, 2007.
- [76] F.J. Humphreys. The nucleation of recrystallization at second phase particles in deformed aluminium. *Acta Metallurgica*, 25:1323–1344, 1977.
- [77] A. Laasraoui and J. J. Jonas. Prediction of steel flow stresses at high temperatures and strain rates. *Metallurgical Transactions A*, 22:1545–1558, 1991.
- [78] Lei Wang, Feng Liu, Hongyuan Chen, and Qiang Chi. Hot deformation characteristics and kinetics analysis for Ni-based corrosion resistant alloy. *Engineering Reports*, 3, 2021.

- [79] Markus Bambach, Irina Sizova, Sebastian Bolz, and Sabine Weiß. Devising Strain Hardening Models Using Kocks–Mecking Plots—A Comparison of Model Development for Titanium Aluminides and Case Hardening Steel. *Metals*, 6:204, 2016.
- [80] Xiao-Min Chen, Y.C. Lin, Dong-Xu Wen, Jin-Long Zhang, and Min He. Dynamic recrystallization behavior of a typical nickel-based superalloy during hot deformation. *Materials & Design*, 57:568–577, 2014.
- [81] Chi Zhang, Liwen Zhang, Wenfei Shen, Cuiru Liu, Yingnan Xia, and Ruiqin Li. Study on constitutive modeling and processing maps for hot deformation of medium carbon Cr–Ni–Mo alloyed steel. *Materials & Design*, 90:804–814, 2016.
- [82] Yang Cheng, Huayun Du, Yinghui Wei, Lifeng Hou, and Baosheng Liu. Metadynamic recrystallization behavior and workability characteristics of HR3C austenitic heat-resistant stainless steel with processing map. *Journal of Materials Processing Technology*, 235:134–142, 2016.
- [83] Dao-Guang He, Y.C. Lin, Ming-Song Chen, and Ling Li. Kinetics equations and microstructural evolution during metadynamic recrystallization in a nickel-based superalloy with δ phase. *Journal of Alloys and Compounds*, 690:971–978, 2017.
- [84] Dahai Liu, Jingdong Chen, Haorui Chai, Yihang Jiang, Zhi Li, Wenqing Qiu, and Zhenghua Guo. Study of meta-dynamic recrystallization behavior of GH5188 superalloy. *Journal of Materials Research and Technology*, 15:1179–1189, 2021.
- [85] Xuefeng Tang, Baoyu Wang, Hongchao Ji, Xiaobin Fu, and Wenchao Xiao. Behavior and modeling of microstructure evolution during metadynamic recrystallization of a Ni-based superalloy. *Materials Science and Engineering: A*, 675:192–203, 2016.
- [86] Gangshu Shen. Microstructure Modeling in Superalloy Forging. In *Cold and Hot Forging*, chapter 19, pages 247–255. ASM International, feb 2005.
- [87] A. Dehghan-Manshadi and P.D. Hodgson. Dependency of Recrystallization Mechanism to the Initial Grain Size. *Metallurgical and Materials Transactions A*, 39:2830–2840, 2008.
- [88] M. El Wahabi, L. Gavard, F. Montheillet, J.M. Cabrera, and J.M. Prado. Effect of initial grain size on dynamic recrystallization in high purity austenitic stainless steels. *Acta Materialia*, 53:4605–4612, 2005.
- [89] Qingmiao Guo, Defu Li, Shengli Guo, Haijian Peng, and Jie Hu. The effect of deformation temperature on the microstructure evolution of Inconel 625 superalloy. *Journal of Nuclear Materials*, 414:440–450, 2011.

- [90] A. Dehghan-Manshadi, M.R. Barnett, and P.D. Hodgson. Hot Deformation and Recrystallization of Austenitic Stainless Steel: Part I. Dynamic Recrystallization. *Metallurgical and Materials Transactions A*, 39:1359–1370, 2008.
- [91] Y.C. Lin, Xian-Yang Wu, Xiao-Min Chen, Jian Chen, Dong-Xu Wen, Jin-Long Zhang, and Lei-Ting Li. EBSD study of a hot deformed nickel-based superalloy. *Journal of Alloys and Compounds*, 640:101–113, 2015.
- [92] Qingmiao Guo, Defu Li, Haijian Peng, Shengli Guo, Jie Hu, and Peng Du. Nucleation mechanisms of dynamic recrystallization in Inconel 625 superalloy deformed with different strain rates. *Rare Metals*, 31:215–220, 2012.
- [93] Hongbin Zhang, Kaifeng Zhang, Haiping Zhou, Zhen Lu, Changhong Zhao, and Xiaoli Yang. Effect of strain rate on microstructure evolution of a nickel-based superalloy during hot deformation. *Materials & Design*, 80:51–62, 2015.
- [94] S.K. Pradhan, S. Mandal, C.N. Athreya, K. Arun Babu, B. de Boer, and V. Subramanya Sarma. Influence of processing parameters on dynamic recrystallization and the associated annealing twin boundary evolution in a nickel base superalloy. *Materials Science and Engineering: A*, 700:49–58, 2017.
- [95] Shichang Qiao, Yan Wang, Liangxing Lv, Gang Tan, Tianwei Cheng, and Jingjun He. Post-dynamic recrystallization behavior of a powder metallurgy Ni-Co-Cr based superalloy under different deformation conditions. *Journal of Alloys and Compounds*, 898:162961, 2022.
- [96] Meriem Zouari, Nathalie Bozzolo, and Roland E. Loge. Mean field modelling of dynamic and post-dynamic recrystallization during hot deformation of Inconel 718 in the absence of delta phase particles. *Materials Science and Engineering: A*, 655:408–424, feb 2016.
- [97] A Nicolaÿ, G Fiorucci, J.M. Franchet, J Cormier, and N Bozzolo. Influence of strain rate on subsolvus dynamic and post-dynamic recrystallization kinetics of Inconel 718. *Acta Materialia*, 174:406–417, 2019.
- [98] Joshua McCarley, Randolph Helmink, Robert Goetz, and Sammy Tin. Grain Boundary Engineering of a Low Stacking Fault Energy Ni-based Superalloy. *Metallurgical and Materials Transactions A*, 48:1666–1677, 2017.
- [99] Joshua McCarley, B Alabbad, and S Tin. Influence of the Starting Microstructure on the Hot Deformation Behavior of a Low Stacking Fault Energy Ni-based Superalloy. *Metallurgical and Materials Transactions A*, 49:1615–1630, 2018.

- [100] S S Satheesh Kumar, T Raghu, Pinaki P Bhattacharjee, G Appa Rao, and Utpal Borah. Strain rate dependent microstructural evolution during hot deformation of a hot isostatically processed nickel base superalloy. *Journal of Alloys and Compounds*, 681:28–42, 2016.
- [101] M. Azarbarmas, M. Aghaie-Khafri, J.M. Cabrera, and J. Calvo. Dynamic recrystallization mechanisms and twinning evolution during hot deformation of Inconel 718. *Materials Science and Engineering: A*, 678:137–152, 2016.
- [102] Y L Hu, X Lin, Y L Li, S Y Zhang, X H Gao, F G Liu, X Li, and W D Huang. Plastic deformation behavior and dynamic recrystallization of Inconel 625 superalloy fabricated by directed energy deposition. *Materials & Design*, 186:108359, 2020.
- [103] Jue Wang, Jian-Xin Dong, and Mai-Cang Zhang. Nucleation mechanisms of dynamic recrystallization for G3 alloy during hot compression. *Rare Metals*, 35:543–550, 2016.
- [104] Hongbin Zhang, Kaifeng Zhang, Shaosong Jiang, Haiping Zhou, Changhong Zhao, and Xiaoli Yang. Dynamic recrystallization behavior of a gamma prime-hardened nickel-based superalloy during hot deformation. *Journal of Alloys and Compounds*, 623:374–385, 2015.
- [105] Hongbin Zhang, Shengxue Qin, Huiping Li, Jie Liu, Yuting Lv, Yan Wang, Peng Zhang, Haiping Zhou, and Tao Wu. EBSD study of strain dependent microstructure evolution during hot deformation of a typical nickel-based superalloy. *Journal of Materials Research*, 34:321–334, 2019.
- [106] N D’Souza, W Li, C Argyrakis, G D West, and C D Slater. On the Evolution of Primary Gamma Prime Precipitates During High Temperature and High Strain Rate Deformation and Subsequent Heat Treatment in the Ni-Based Superalloy, RR1000. *Metallurgical and Materials Transactions A*, 50:4205–4222, 2019.
- [107] Meriem Zouari, Roland Logé, and Nathalie Bozzolo. In Situ Characterization of Inconel 718 Post-Dynamic Recrystallization within a Scanning Electron Microscope. *Metals*, 7:476, 2017.
- [108] A. Nicolaÿ, J. M. Franchet, J. Cormier, R. E. Logé, G. Fiorucci, J. Fausty, M. Van Der Meer, and N. Bozzolo. Influence of Joule Effect Heating on Recrystallization Phenomena in Inconel 718. *Metallurgical and Materials Transactions A*, 52:4572–4596, 2021.
- [109] A. Chamanfar, M. Jahazi, J. Gholipour, P. Wanjara, and S. Yue. Evolution of flow stress and microstructure during isothermal compression of Waspaloy. *Materials Science and Engineering: A*, 615:497–510, 2014.

- [110] D.J. Yu, D.S. Xu, H. Wang, Z.B. Zhao, G.Z. Wei, and R. Yang. Refining constitutive relation by integration of finite element simulations and Gleeble experiments. *Journal of Materials Science & Technology*, 35:1039–1043, 2019.
- [111] Erofil Kardoulaki, Jianguo Lin, Daniel Balint, and Didier Farrugia. Investigation of the effects of thermal gradients present in Gleeble high-temperature tensile tests on the strain state for free cutting steel. *The Journal of Strain Analysis for Engineering Design*, 49:521–532, 2014.
- [112] Yu Cao, Hongshuang Di, Jingqi Zhang, Jiecen Zhang, Tianjun Ma, and R D K Misra. An electron backscattered diffraction study on the dynamic recrystallization behavior of a nickel–chromium alloy (800H) during hot deformation. *Materials Science and Engineering: A*, 585:71–85, 2013.
- [113] Gangshu Shen. Temperature and Heat Transfer. In *Cold and Hot Forging*, chapter 6, pages 59–66. ASM International, feb 2005.
- [114] L. Zhou, C. Cui, Q.Z. Wang, C. Li, B.L. Xiao, and Z.Y. Ma. Constitutive equation and model validation for a 31 vol.% B 4 Cp/6061Al composite during hot compression. *Journal of Materials Science & Technology*, 34:1730–1738, 2018.
- [115] Joseph I. Goldstein, Dale E. Newbury, Joseph R. Michael, Nicholas W.M. Ritchie, John Henry J. Scott, and David C. Joy. *Scanning Electron Microscopy and X-Ray Microanalysis*. Springer New York, New York, NY, 2018.
- [116] Ralf Hielscher, Felix Bartel, and Thomas Benjamin Britton. Gazing at crystal balls: Electron backscatter diffraction pattern analysis and cross correlation on the sphere. *Ultramicroscopy*, 207:112836, dec 2019.
- [117] Nicolas Brodusch, Hendrix Demers, and Raynald Gauvin. *Field Emission Scanning Electron Microscopy*. Springer Singapore, Singapore, 2018.
- [118] Adam J. Schwartz, Mukul Kumar, Brent L. Adams, and David P. Field, editors. *Electron Backscatter Diffraction in Materials Science*. Springer US, Boston, MA, 2009.
- [119] G. Nolze and R. Hielscher. Orientations – perfectly colored. *Journal of Applied Crystallography*, 49:1786–1802, 2016.
- [120] F. Bachmann, Ralf Hielscher, and Helmut Schaeben. Texture Analysis with MTEX – Free and Open Source Software Toolbox. *Solid State Phenomena*, 160:63–68, 2010.

- [121] H. Miura, T. Sakai, H. Hamaji, and J.J. Jonas. Preferential nucleation of dynamic recrystallization at triple junctions. *Scripta Materialia*, 50:65–69, 2004.
- [122] Adelajda Polkowska, Sebastian Lech, and Wojciech Polkowski. The effect of cold rolling degree on microstructure, crystallographic texture and mechanical properties of Haynes® 282® wrought nickel superalloy. *Materials Science and Engineering: A*, 787, 2020.
- [123] Bingchao Xie, Baoyun Zhang, Hao Yu, Hao Yang, Qi Liu, and Yongquan Ning. Microstructure evolution and underlying mechanisms during the hot deformation of 718Plus superalloy. *Materials Science and Engineering: A*, 784, 2020.
- [124] Baoyun Zhang, Zhaotian Wang, Hao Yu, and Yongquan Ning. Microstructural origin and control mechanism of the mixed grain structure in Ni-based superalloys. *Journal of Alloys and Compounds*, 900, 2022.
- [125] Rui Hu, Guanghai Bai, Jinshan Li, Jingqing Zhang, Tiebang Zhang, and Hengzhi Fu. Precipitation behavior of grain boundary M₂₃C₆ and its effect on tensile properties of Ni–Cr–W based superalloy. *Materials Science and Engineering: A*, 548:83–88, 2012.