

CRANFIELD UNIVERSITY

CAITLIN GILROY-HIRST

MATERIAL STRENGTH EVOLUTION OF FCC METALS UNDER
HIGH STRAIN RATES

DEFENCE AND SECURITY

PhD

Academic Year: 2016 – 2020

Supervisor: Dr. Rachael Hazael

Associate Supervisors: Dr. David Wood, Prof. Jackie Akhavan

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ABSTRACT

Copper and stainless steel 21-6-9, two face-centred cubic (FCC) materials, were shock loaded in partially recovered uniaxial strain flyer-plate impact experiments at varying impact pressures (6 GPa - 16 GPa). These samples were collected post-impact via three differing recovery configurations: (1) standard or 'free boundary'; (2) partial shock recovery, i.e. using both 'momentum trapping' and 'soft recovery' techniques but with a flyer plate thickness of 10 mm to see the effects on partial tensile release in the rear spall plates of an established recovery target design; finally (3) a traditional full recovery comparison with a 2 mm flyer impact.

These samples were then analysed using a suite of analytical techniques including X-ray diffraction, optical microscopy, hardness testing and compression after impact testing, to observe the material deformation characteristics in the simplified partially recovered shock and release condition compared to the more complex 'standard' release wave situation. A control sample of a 'fully recovered' sample was also carried out for comparative purposes. The rear spall plates were modified on these partially-recovered samples to see the effect that this had on the tensile release waves and how this changed the material deformation properties overall.

Results for the stainless steel 21-6-9 demonstrated the ability of the partial shock recovery technique (hereafter called 'partial-recovery') to mitigate the reverberations compared to that of the standard samples. Upon analysis of the targets, it was observed that the dislocation density was generally lower for the partially recovered samples but varied with impact pressure for both the standard and recovered configuration. For the stainless steel recovery experiments an increasing dislocation

density from $4 \times 10^{15} \text{ m}^{-2}$ at 6 GPa to $6.5 \times 10^{15} \text{ m}^{-2}$ at 12 GPa was observed which then decreased to $5.5 \times 10^{15} \text{ m}^{-2}$ at 16 GPa.

The twinning density to grain ratio obtained from optical microscopy showed a linear increase ($y = 1.5345x - 8.3508$) with an R^2 value of 0.8295 from 7 GPa at 4 twins per grain to 17 twins per grain at 16 GPa. Twinning density is indicative of a successful recovery, where a successful recovery is a reduction in reverberations in the sample and in a full recovery is a 1D shock. The dataset both verifies the use and success of the recovery technique as well as demonstrating the materials characteristics under partial-recovery, while observing reverberation effects.

Keywords: shock physics, material deformation, fcc, bcc, stainless steel

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James Gilroy (1933- 2015)

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance (Statistical Test)
AWE	Atomic Weapons Establishment
BCC	Body-Centred Cubic
BCT	Body-Centred Tetragonal
EBSD	Electron Backscatter Diffraction
FCC	Face-Centred Cubic
FEA	Finite Element Analysis
HCP	Hexagonal Close Packed
Het-V	Heterodyne Velocimetry
IATA	International Air Transport Association
OFHC	Oxygen-Free High-Conductivity Copper
SFE	Stacking Fault Energy
TEM	Transmission Electron Microscopy
TWIP	Twinning-Induced Plasticity
VISAR	Velocity Interferometer System for Any Reflector
XRD	X-ray Diffraction

1 INTRODUCTION

Knowledge and understanding of the mechanisms and effects of high velocity impacts are vital to many different research areas; including aerospace, for example satellite or aeroplane protection, crashworthiness in the automotive industry, or armour systems, within the military field. These different research areas focus on the safety of either people or electronics and therefore it is vital to understand how these materials are affected by high velocity impact or shock wave interaction [1].

In the late 19th century, the initial theory of shock waves was developed by Rankine and Hugoniot, who investigated and derived the equations governing shock. Rankine's initial work developed the theory of thermodynamics, as prior to this heat transfer was presumed to be based on the Newtonian theory of sound [2].

In 1870 Rankine published 'On the Thermodynamic Theory of Waves of Finite Longitudinal Disturbance' [3], which outlined the theory of instant heat transfer based upon the ideal gas laws. The ideal gas laws can be related to a shock disturbance in a material as it flows hydrodynamically under a shock wave i.e. the material behaves as a fluid as the material is inertially confined, preventing lateral flow and building up high levels of stress [2]. Once these stress levels exceed the dynamic flow stress, the solid has no resistance to shear stress and flows as a fluid. Hugoniot worked on shock with relation to mechanics and ballistics, investigating high velocity impacts of artillery shells. His theory developed from Rankine's thermodynamic wave theory, which allowed for improvements in fluid flow study, and led to the Rankine-Hugoniot equations, which still underpin shock wave propagation today [4].

As well as ballistics, shock waves are crucial to planetary scientists, as they govern astronomical object collisions in space and how they aid in crater formation [5]. These features help us determine how our solar system and others formed and evolved. It could be argued that one of the first investigations into craters was carried out in 1897 and published as a book on 'The study of splashes' which used photography to look into the effects of a water droplet upon a body of water investigating the crater formation and shape [6]. The study was initially on a liquid before future work focused upon planetary impact craters of solids, which behaves in a fluid like manner due to the high strain rate, greater than 10^4 s^{-1} [5], [7].

Though the initial interest in shock waves was based upon gases and liquids, shock compression in solids became of interest during World War II, as it was required for the development of nuclear weapons during the 'Manhattan Project' [8]. A study was carried out by Taylor, on one of the atomic tests in New Mexico as part of the Manhattan Project [9]. During this experiment, he investigated the formation and propagation of the blast wave. By treating the mushroom cloud as 'a large bubble in water' using fluid dynamics in the calculations [9], it was found that high explosive and nuclear explosive sources buried in the ground could simulate shock wave cratering phenomena similar to meteor impact events at elevated strain rates of approximately 10^8 s^{-1} [5], [10].

One of the first modern papers on shock compression of materials published in 1954 was on the Equations of State for aluminium, copper and zinc [11]. An Equation of State defines how a material behaves under high strain rate loading $>10^4 \text{ s}^{-1}$, as opposed to quasi-static loading [2]. This initial work was carried out on behalf of the

U.S. Atomic Energy Commission [11] . In more recent years, shock compression of materials has related to the way shock waves interact with materials, as well as the way the microstructure of a material changes including: phase changes, dislocations, twinning, fracture, spall and fragmentation [1], [12]–[16].

A catalogue of the Equation of States (EoS) of different materials has been experimentally obtained by different institutions, where an EoS provides a description of how a material behaves under shock conditions. The largest material catalogue is provided by Los Alamos Shock Laboratory (LASL) [17]. Many different materials have been catalogued, including metals, alloys, ceramics, soil, rocks and minerals, polymers, composites, porous solids and explosives [18]–[22]. The applications of these materials are varied, as in the past, some of these applications are for military uses such as shaped charges [23]. However, more recently, as different modes of transport have developed and the importance of safety has evolved, an impetus upon material selection has been vital to ensure that the material will deform in a predictable manner [24].

For example, in the automotive industry cars must be designed in a safe way for both drivers and passengers, so material selection must be appropriate for the level of force created during an impact at strain rates of approximately 10^3 - 10^5 s⁻¹ [25]. In the EU this is governed by Euro-NCAP, which rate and reward car manufacturers for adding and improving safety features within their cars [26]. Some of these safety developments have included crumple zones, to absorb energy from an impact as well as considerations in vehicle size, weight and frame stiffness [26]. These developments

have led to a decrease in serious injuries by 67% in the years 2015-2018 than in 1980-1984 [26].

Similarities in safety improvements and design of vehicles are also shown in the aviation field. In the 2003 International Air Transport Association (IATA) safety report, it was found that 20% of accidents were caused by mechanical failures, compared to 100 years prior where 80% of accidents were caused by mechanical failure [27]. However, issues such as bird strike still persist and an appropriate material selection must be made both for impact issues while still being lightweight [28]. The most recent Boeing 787 Dreamliner, consists of composite, aluminium, titanium and steel [29]. The composite is 80% of the volume of the overall aeroplane but the titanium, aluminium and stainless steel are used in areas which need extra protection such as the fuel tanks or under the fuselage where high strain rate impacts from below are more likely, as they all have a high strength to weight ratio [30].

It would be incredibly costly to test different ratios of materials to build an aircraft therefore these costs for experimental safety testing across these different industries have led to an increased use of computer simulation methods such as Finite Element Analysis (FEA) to assess the properties of a design before manufacture [24]. The initial research linking structural analysis and continuum mechanics was developed by Clough, while working for Boeing on aircraft wings and developed by Turner, Wilson *et al.* at the University of California Berkeley [31]–[35]. FEA has also developed rapidly in the last 40 years, allowing designers to simulate object impacts more accurately than before in an economical way [36]. The increased use of computer modelling is dependent upon understanding a materials' behaviour on the microstructural level in

order to understand the deformation and potential failure or fragmentation of a material in a macroscopical way [37].

In order to obtain the material data to input into these computer simulations, various tests are carried out upon materials. Within the context of high velocity impacts and shock physics, gas guns can be used to obtain high strain rate data for different materials [38], [39]. However, when an impact occurs a shock wave travels through an object reflecting back and forth until it dissipates, causing many changes to the microstructure [40], [41]. However, it is impossible to predict which reflection caused which deformation characteristic. To simplify this further in the laboratory a ‘soft recovery’ technique is used, which can exist within the impact target itself or a capture technique in the target chamber; usually both.

The origins of recovery stem from the development of different dynamic experiments. These include high explosives¹, as previously mentioned with the crater impact work, as well as Hopkinson’s work in 1914, leading to the modern version of the split Hopkinson pressure bar developed with research from Kolsky, allowing for high strain rate experiments in the order of 10^2 to 10^4 s⁻¹ [42]–[44]. As the methods to induce dynamic compression of materials became more refined and regulated, it was desirable to recover the material afterwards. The recovery of the material allowed for analysis of the microstructure, as well as observation of stresses and strains in the material, which, at that time, could not be measured accurately throughout the material during the experiment. However, more recent developments allow for the use of manganin pressure gauges, relying upon piezoresistivity (change in the electrical

¹ An explosive which is more violent and reactive than gunpowder i.e. detonation as opposed to deflagration

resistivity of a material with applied external stress) to measure the stresses within the material [45]–[48]. There is also heterodyne velocimetry measurements (Het-V), which use laser interferometry to measure the particle velocity of the surface of a material [49]–[51].

One of the first experiments adopting a ‘recovery’ technique was on the ultimate yield strength of copper where the 1D zone from the material was analysed after a target was impacted by an explosively-driven copper flyer plate [52]. However, the material was ‘recovered’ by being collected after the experiment, not through a soft recovery technique i.e. no soft capture facility in the chamber of the gun nor target modifications. Nevertheless, shock waves will reflect in a tensile manner both laterally and longitudinally once they contact a boundary, causing both spallation (dynamic tensile failure) and plastic deformation caused by lateral release loading [53]–[55].

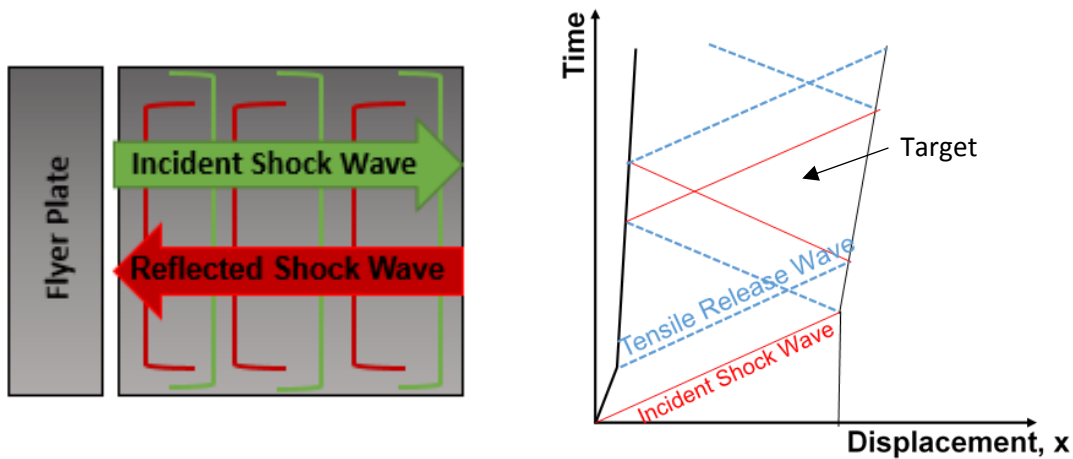


Figure 1: Diagram showing the simplified version of a shock wave entering a target and reflecting back and forth (left). X-t diagram showing the incident compressive shock wave entering a target and reflecting back as a tensile release wave (right).

Different techniques have been used to reduce these reverberations, which throughout this thesis shall be referred to as ‘soft recovery’. Kumar and Clifton used star-shaped flyers to reflect tensile releases away from the 1D recovery zone. Research has also

been completed, involving ‘momentum trapping’ targets, with multiple plates and rings to prevent shock reflection back into the recovery target, which has been developed over the last 30 years by researchers including Blumenthal, Llorca and Gray *et al.* [55]–[59]. As previously mentioned, FEA can be used to quickly and economically test materials and has been used in more recent years to assist with recovery target design. The most up to date designs having been developed by Bourne and Gray using Eulerian code and Vignevic *et al.* using Lagrangian code [41], [60], [61].

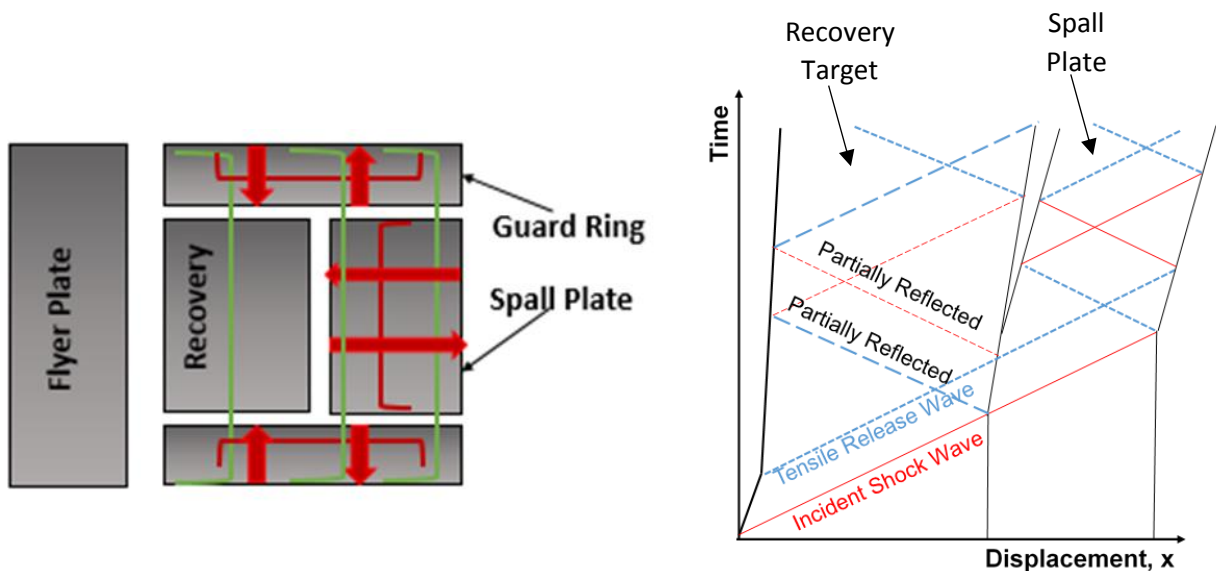


Figure 2: Diagram showing a simplified version of a shock wave entering a recovery target and the wave being reflected back through the spall rings but not into the recovery sample (left). X-t diagram showing the incident compressive shock wave entering a target and spall plate and reflecting back as a tensile release wave but mostly being (momentum) trapped within the spall plate (right).

Although these soft recovery techniques have been developed, very few materials have been tested and characterised using it. This project aims to develop and carry out soft recovery on two different metals, Oxygen-Free High-Conductivity (OFHC) copper and stainless steel 21-6-9 (21% Cr, 6% Ni, 9% Mn, an austenitic, face centred cubic (FCC) steel), and then analyse these metals using a range of techniques to gain an insight into their deformation mechanisms. OFHC copper has been selected as it

is a well characterised material and can be used for comparative purposes in the development of the soft recovery technique, used for both the recovery target itself and the recovery capture fixture in the target chamber of the gas gun [12], [17], [62]. Stainless steel 21-6-9, has been selected as it has multiple applications within aerospace, automotive, nuclear and construction industries [1], [63].

In conjunction with the experimental work and analysis, computer simulations using ANSYS® Autodyn will be used to predict behaviour and help to validate the recovery of the metals [64]. Post-shock, the materials will be analysed using optical microscopy, hardness testing, X-ray Diffraction (XRD), compression after impact (CAI) testing and Transmission Electron Microscopy (TEM) to interrogate changes in the microstructure of the OFHC copper and stainless steel 21-6-9. Once the materials analysis is completed, this information can be entered back into the computer simulations to help improve the library of data. The recovery technique can then be used on a wide range of FCC metal structures in future to create a more accurate and complete catalogue of materials to help future materials selection across multiple industries.

2 LITERATURE REVIEW

This project is based upon three main disciplines, shock physics, dynamic material response and hydrodynamic computer modelling. This literature review will provide an introduction to each of these topics as well as introducing the two main materials in the project, copper and stainless steel 21-6-9.

Initially, material structure will be introduced to provide a general understanding of the metal structure and behaviour, then shock and material response to shock effects will be explained and finally computer modelling and hydrocodes.

The computer modelling compliments the experimental work carried out, by aiding in the explanation of the plastic strain and shock effects. It also helps to predict future behaviour, though is limited by experimental data, time and pressure. The intricacy of deformation behaviour is significantly more complex and takes large amounts of computational power to represent an entire sample rather than a small subsection. This will be discussed in detail in Chapter 3.

2.1 Structure of a Metal

The microstructure of a solid dictates how that solid behaves macroscopically [41]. Metals specifically consist of a crystalline structure, which is a structure consisting of one single crystal (grain) or numerous grains [65]. The theory behind crystallographic structure was first developed by Haüy, Hessel, Bravais, Fedorov and Schönflies [66]–[70]. Haüy first discovered in 1784 that crystals cleaved along crystallographic planes and it was already known that the interfacial angles always had the same values, so he concluded that crystals were periodic and composed of polyhedral stacks, which

Haüy named *molecules intégrantes* [66]. From this research, Hessel concluded in 1830 that the restrictions in rotational axes governed by Haüy's laws resulted in 32 possible classes of crystal [67]. Bravais derived all 14 possible lattice symmetries in 1850 after Seeber and Delafosse independently determined that a crystal can best be described as discrete points, which are generated by translational operations [68]. Schönflies and Fedorov agreed upon a full catalogue of 230 space groups after it was realised that rotations and reflections of the lattice must be considered [69], [70].

A grain consists of all unit cells interlocking in the same way with the same orientation [65]. There are seven different geometries of crystal, these are: triclinic, monoclinic, orthorhombic, trigonal, tetragonal, hexagonal and cubic. Within the confines of this project, it is the latter two crystal systems, which are of importance and define how the metals being investigated behave.

The simplest form of a structure consists of a unit cell, which is the base building block of any crystalline material [65]. There are several types within each category of crystal. Most metals either have cubic or hexagonal systems, consisting of unit cells that are body-centred cubic (BCC), face-centred cubic (FCC) or hexagonal close packed (HCP), which will be discussed in detail below and are shown in Figure 3.

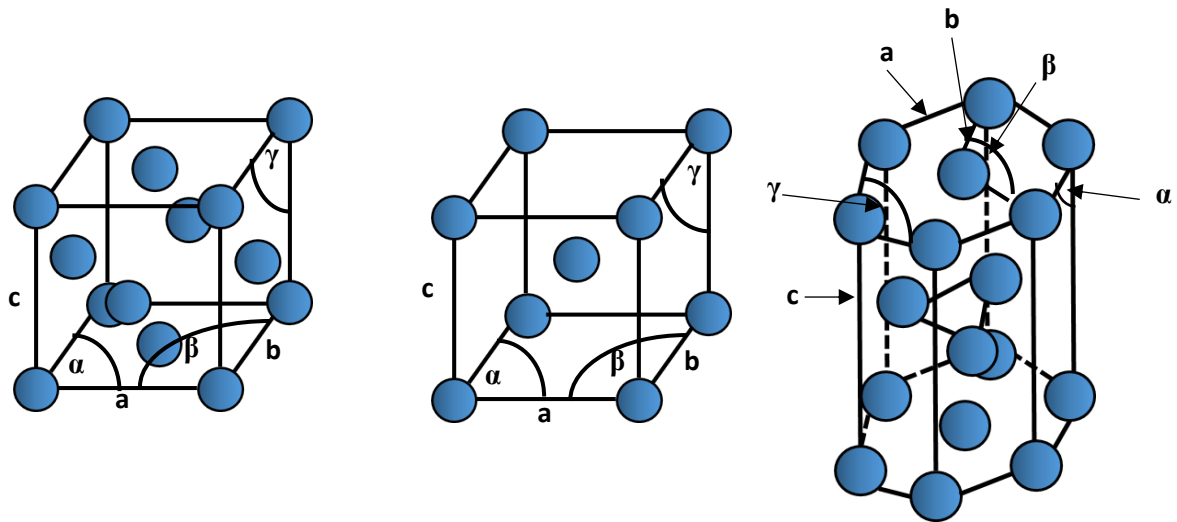


Figure 3: Left to right: Face-Centred Cubic (FCC), Body-Centred Cubic (BCC), Hexagonal Close Packed (HCP). Adapted from Callister [10]

All cubic crystal structures consist of $a = b = c$ with regards to their axial relationship and their interaxial angles are $\alpha = \beta = \gamma = 90^\circ$ [65]. A FCC crystal (e.g. austenitic steel) consists of a cubic structure with one atom in the centre of each face. The cubic edge length a and the atomic radius R are related through [65]:

$$a = 2R\sqrt{2}$$

Equation 1

A BCC crystal (e.g. niobium) also has a cubic structure but with one atom within the centre of the cube. The unit cell length a and the atomic radius R are related through [65]:

$$a = 4R\sqrt{3}$$

Equation 2

All hexagonal crystal structures consist of $a = b \neq c$ axially and their interaxial angles are $\alpha = \beta = 90^\circ$, $\gamma = 120^\circ$ [65]. One of the phases of iron, or more specifically martensite, the ϵ phase has a HCP structure.

More complex structures can exist however, under shock, for example, stainless steel 21-6-9, which can undergo a phase transformation from γ -Fe, an FCC structure, to α' -Fe (or martensite) which has a Body-Centre Tetragonal or BCT structure [1]. A phase transformation or change occurs when the crystalline structure of the lattice changes due to changes in energy, where a phase is a material that has the same crystalline structure, composition and properties everywhere under equilibrium conditions [71]. This structure is shown in Figure 4. Tetragonal crystal systems are cuboids in that $a = b \neq c$ and $\alpha = \beta = \gamma = 90^\circ$.

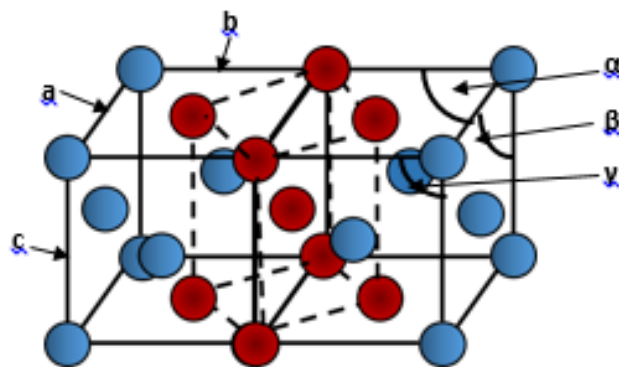


Figure 4: BCT martensite forming within a FCC lattice. Adapted from Callister [10]

Martensite cannot exist as a 100% pure phase as it is an interstitial structure where the BCT unit cell lies within two FCC unit cells. Constrained paraequilibrium occurs where the phase boundaries become stationary as no more martensite can be formed due to the specific size of α' -martensite being larger than austenite (FCC steel phase) [72].

FCC and HCP crystallographic systems are more common due to the stacking of the atoms, as they possess close packed planes unlike BCC crystals [65]. In FCC crystals the stacking order is ABCABC..., whereas in HCP metals, the stacking order is ABAB... Due to the ABC sequence in FCC crystals, stacking faults are very important their dislocation dynamics [65].

The addition of nickel and manganese in stainless steel 21-6-9 helps to inhibit the γ -Fe (austenitic phase) to α' martensitic phase transformation [73]. Martensite is undesirable, as when there is too much of the phase present in a steel, the steel becomes brittle and more likely to fracture, usually due to increased carbon content and changes in grain boundaries around martensite crystals increasing local stress [74].

There are two types of martensite α' -(BCT) and ϵ -(HCP) martensite. α' - martensite is applicable to this project because of the initial, nickel containing austenitic structure, whereas it's less likely for ϵ -(HCP) martensite to form [75]. Martensite formation can occur in one of two ways: either when a material is quenched or under pressure i.e. from shock or rolling [76].

Overall, all of these crystalline structures are examples of the basic building blocks of a metallic crystalline structure. To fully understand material strength and deformation, a background on dislocations; formation and propagation is required.

2.1.1 Dislocations

All crystalline materials, including metals, contain defects. These defects can change the properties of a material, in the case of alloying, these defects are intentionally created to manipulate how the material behaves [77]. There are three basic types of defect: point, linear and planar [65]. A point defect can be either an interstitial atom, a vacancy, a substitution impurity atom or interstitial impurity atoms i.e. atoms from another element [65]. A linear defect is where lines of atoms are irregularly arranged, these are also known as dislocations [65]. Planar defects are interfaces between ordered or homogeneous regions of a material; this includes external surfaces, grain boundaries and stacking faults [65]. These are most commonly found in close packed crystal structures including those of BCC, FCC and HCP. Stacking faults carry a given formation enthalpy per unit area, which is also known as stacking fault energy (SFE) [78]. The stacking fault energy of a material is the overall energy of the atoms based upon the stacking faults (or defects) within the material [79]. The lower the stacking fault energy; the more likely for twinning to occur (see Section 2.1.2).

Dislocations are a line defect within the crystal lattice of a material [80]. These dislocations can be either screw type or edge type. An edge type dislocation is one where there is an extra half plane of atoms within the lattice, called the dislocation line which in Figure 5 is perpendicular to the plane of the page [65].

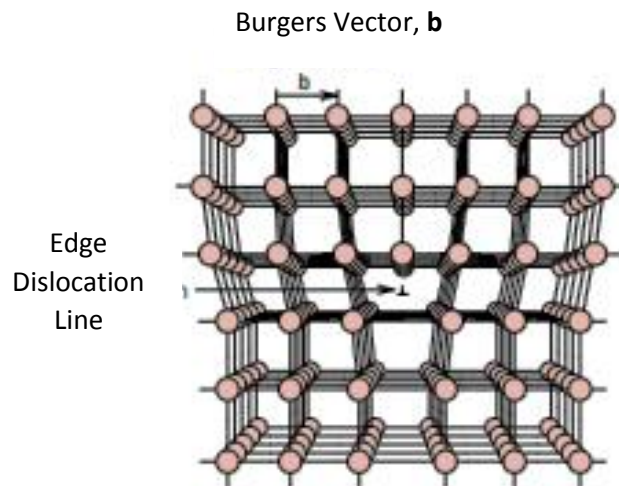


Figure 5: Atom positioning around an edge dislocation [65]

A screw type dislocation is caused by shear stress, where the upper front region of the crystal is shifted by one atomic step. In Figure 6 this atomic displacement occurs to the left hand side. Usually, dislocations are a combination of the two rather than purely one or the other. The Burger's vector indicates the direction and the extent to which the lattice has been displaced by the dislocation [81].

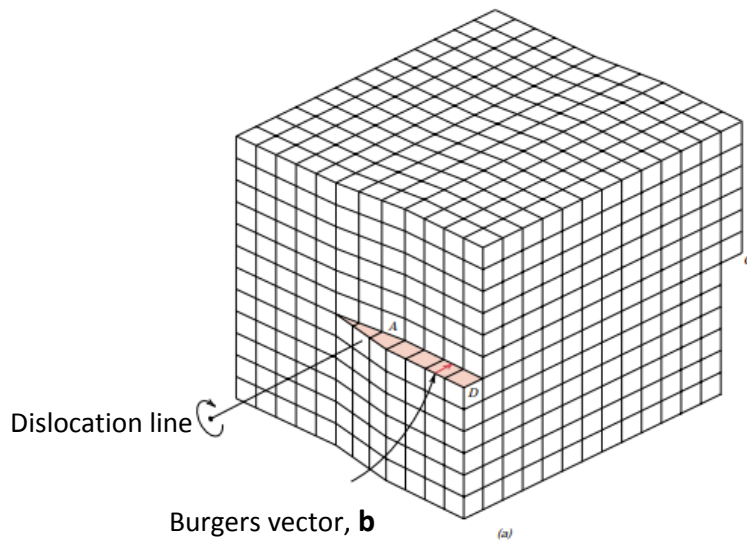


Figure 6: Example of a screw dislocation as shown from above [65]

When a shearing stress is applied to the lattice, which exceeds the elastic limit, atomic movements will occur which will cause the dislocation line to move through the lattice. The Peierls stress gives the minimum shear stress required to move a single dislocation of unit length in a perfect crystal [82]. The magnitude of the Peierls stress determines the ability of the lattice to resist dislocation motion – the lattice resistance in the absence of thermal activation [82]. Kosugi and Kino quantitatively derived a Peierls stress for an FCC metal at approximately $3 \times 10^{-5} \mu$, where μ is the shear modulus of the metal with units Nm^{-2} . They also suggested that impurities and point defects could override the effect of the Peierls stress.

The stress needed to propagate a dislocation, is significantly less than the stress needed for block slip. A cell block is defined as a contiguous group of cells in which the same set of glide systems operate. For a dislocation to propagate, only a small fraction of the bonds within the crystal are being broken compared to breaking all the bonds across the middle plane simultaneously as in block slip [65]. Plastic flow will

then occur in the direction of the Burgers vector, **b**. The Burgers vector dictating the magnitude and direction of a lattice distortion within a crystalline lattice [65].

2.1.2 Deformation Twinning

A different mechanism for plastic deformation is twinning. This can occur in HCP metal crystals, BCC or BCT metal crystals. A cause of twinning in a material can be if the slip planes within crystals are not aligned to the stress axis in a favourable way. Twin formation can occur when a shock loading event happens due to the high strain rate, or during a process such as annealing or via growth twinning caused by deformation of a larger substitution ion. The twin is a mirror image of the original lattice arrangement. This occurs as the new lattice arrangement is then more thermodynamically placed for slip to occur [83]. Figure 7 shows a depiction of a twin forming with the reflection occurring around the twin plane.

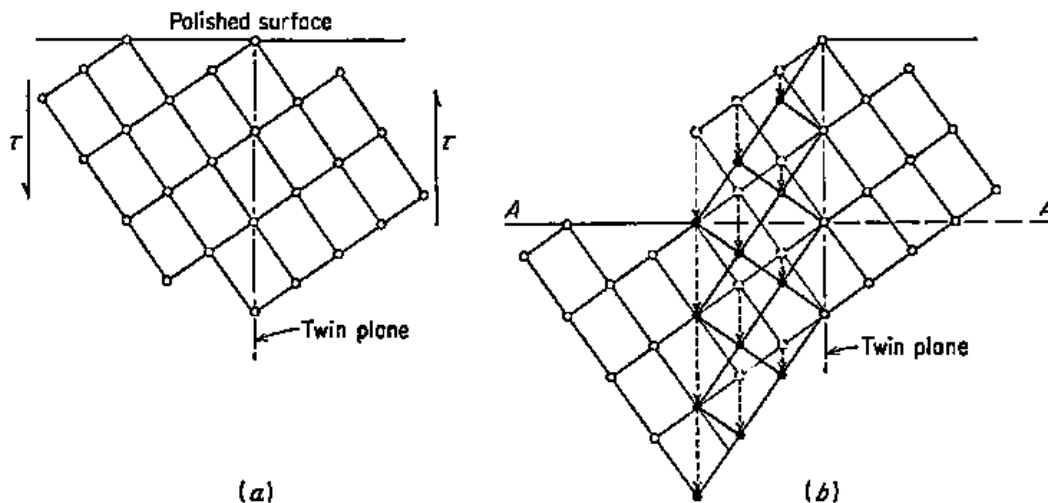


Figure 7: General diagram of twin formation within a lattice structure [113]. (a) represents the original structure, while indicating where the twin plane is and (b) represents the formation of the twin around that plane (as a direct reflection of the original twin plane).

Twinning is a highly favoured mechanism during shock loading, especially in low stacking fault FCC metals [84]. Metals, which would not usually twin under quasi-static loading will twin under shock loading conditions as the strain rate is higher, giving more energy into the system. Deformation twinning is also seen in BCC and HCP metals and is usually caused by the dislocation mobility [85]. If the shear stress required for dislocation motion/formation is greater than that for twin formation, i.e. a higher Peierl's stress, then twinning will be favoured. In situations where slip is no longer possible due to dislocation build up or in materials with a low number of slip systems, twinning will become the favourable method of deformation, as it provides a new plane for slip to occur [85].

Twinning is dependent upon six main factors: 1) pressure, 2) crystallographic orientation, 3) SFE, 4) existing substructure, 5) grain size and 6) pulse duration. Sufficient pressure and pulse duration are required for the formation of twins, to allow for a long enough time window for them to form, approximately 1 ns, and enough pressure to occur, which varies according to the type of metal [86]. Champion and Rohde discovered that during a 10 GPa shot on an austenitic i.e. FCC steel, no twins were found for a pulse duration of 0.065 μs , whereas twins were found for a pulse duration of 2 μs , showing that they had enough time to form [87].

The crystallographic orientation also has an effect, DeAngelis and Cohen, found that twinning occurred in copper single crystals at 14 GPa when the shock wave was travelling along the (100) axis but at 20 GPa when travelling along the (111) axis [88]. Pure copper has a SFE of 78 mJ m^{-2} , which would make it unlikely to twin at shock

pressures under 20 GPa but in this scenario the orientation makes it more favourable in the (100) axis [89].

If there are already enough dislocations within the substructure of the material, then twinning is less likely, as twinning involves creating another slip system. If there are many dislocations already then dislocation generation and motion will actively compete against twinning. HCP metals are more likely to twin than BCC or FCC systems as they have less slip systems available for deformation. HCP metals have 3-6 slip systems depending on the metal compared to 12 in an FCC system or 48 in a BCC system, which is also dependent on the type of metal [65]. If the grain size of a metal is larger, which can be achieved through annealing, then twinning is more likely to occur also. Rahman *et al.* investigated the effect of grain size on twin initiation stress in a twinning induced plasticity (TWIP) steel. They confirmed the Hall-Petch relationship, which is defined as smaller grain size leading to yield strengthening of a material and also found that these materials were less likely to twin [90]–[92]. This relationship is based upon dislocation mechanisms of plastic deformation, where grain boundaries prohibit the movement of dislocations.

Lee stated that suppression of mechanical twinning via grain refinement is similar to that of ϵ martensite formation via grain refinement, therefore the mechanical twinning is slowed by the increase in SFE near grain boundaries where atoms of carbon are segregated by the increased stress of dislocations [93]. In further work, Kang *et al.* carried out work on a TWIP steel and stated that decreasing grain size accelerated dislocation accumulation at the same strain, changing the slip mode from planar to non-planar and delaying mechanical twinning [94].

Back stress is a long-range type of stress caused by the pileup of geometrically necessary dislocations, which are necessary to accommodate for plastic bending in a crystalline material [95]. A high level of back stress narrows the stacking fault width, facilitating dislocation cross slip, reducing the interactions between partial dislocations which are required for mechanical twinning.

Twinning tends to favour high strain rates and lower temperatures more so than slip by dislocation, due to less energy being available for dislocation motion [96]. However, in a shock event, peak shock temperatures can be higher. In the case of stainless steel 21-6-9, the predicted temperature rise from ANSYS® Autodyn was 57 °C and 107 °C for a 6 GPa impact and 16 GPa impact respectively, well below cementite (an iron carbide phase) formation temperature of 725 °C or the higher stainless steel heat treating temperature of 1040 °C [64], [97].

Stainless steel 21-6-9 has a low SFE, which has been measured around 41 mJ m⁻² [98]. Twinning is the main mechanism for deformation in FCC materials with a SFE approximately between 18-45 mJ m⁻² [99]–[101]. In steels with a SFE higher than 18 mJ m⁻² a transformation from the austenitic γ - phase would initially lead to twinning and then α' -martensite. Whereas in materials with a SFE lower than 18 mJ m⁻² the transformation would occur from γ -austenite to ϵ -martensite and then α' -martensite [102].

Park *et al.* reported that an austenitic steel with a SFE below 20 mJ m⁻² tends to transform into ϵ - or α' - martensite, whereas twinning is dominant between 20-50 mJ

m^{-2} [103]. In austenitic stainless steels early stages of deformation are usually dominated by slip, then stacking faults are formed before twinning dominates the deformation [104]. It has been estimated that annealing twins were found in 75% of the grains from the manufacturing process and account for 45% of the areas sampled [1]. This involved hot working at $\sim 1090^\circ\text{C}$ and solution annealing at $\sim 1063^\circ\text{C}$. Annealing of steel is used to reduce hardness, lower internal stresses and increase ductility.

When Brusso *et al.* carried out experiments they found that there was a large amount of strengthening which occurred with increasing shock pressure up to 20 GPa to large amounts of twin formation. It was also noted that dislocation density saturated faster with shock than other low SFE materials [105]. Grains and annealing twins are shown in Figure 8.

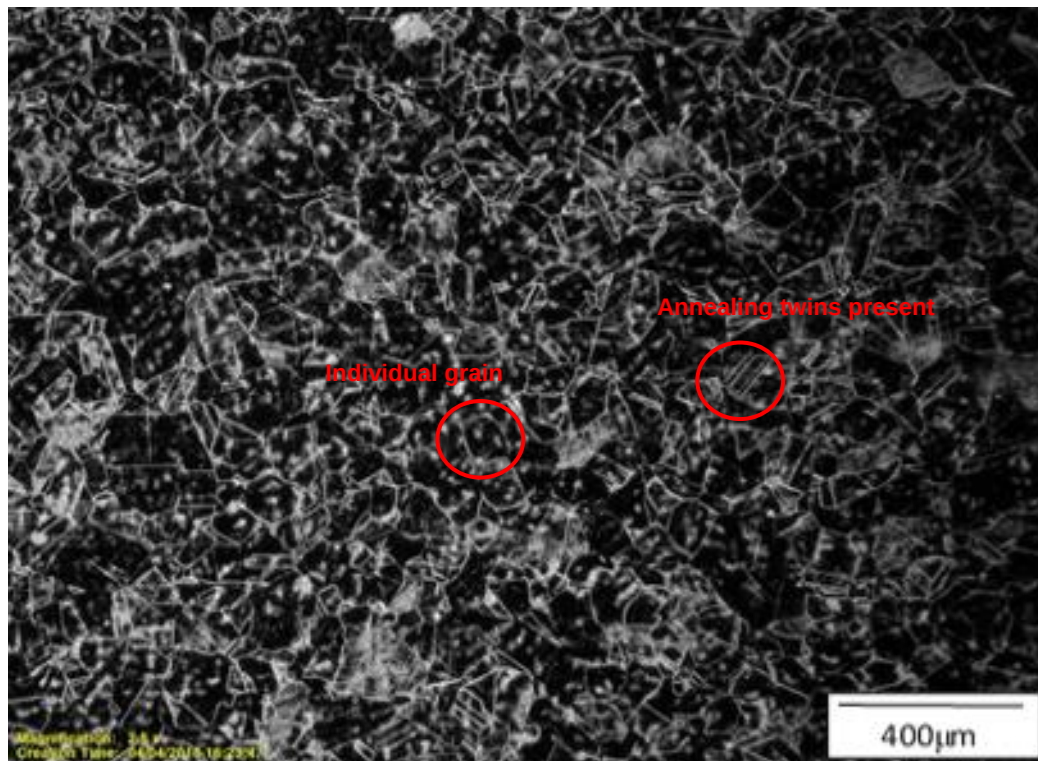


Figure 8: Representative control sample of stainless steel 21-6-9 using dark field imaging to highlight the twins present. Dark field imaging involves the tilting the beam until it diffracts the light, scattering light off the sample creating a high contrast image, as opposed to bright field imaging where the light is attenuated on the sample from the incident beam.

2.1.3 Slip Planes

Slip occurs on crystal planes, which possess the highest degree of atomic packing. The direction of slip within a plane is determined by the direction of the highest degree of atomic line density which is the number of atoms divided by the unit length of the direction vector [83]. The definition of slip is plastic deformation produced by the motion of dislocations. Plastic deformation is the permanent deformation that results from slip in response to applied shear stress [65]. The effects of this slip resulting in permanent plastic deformation is shown in Figure 9.

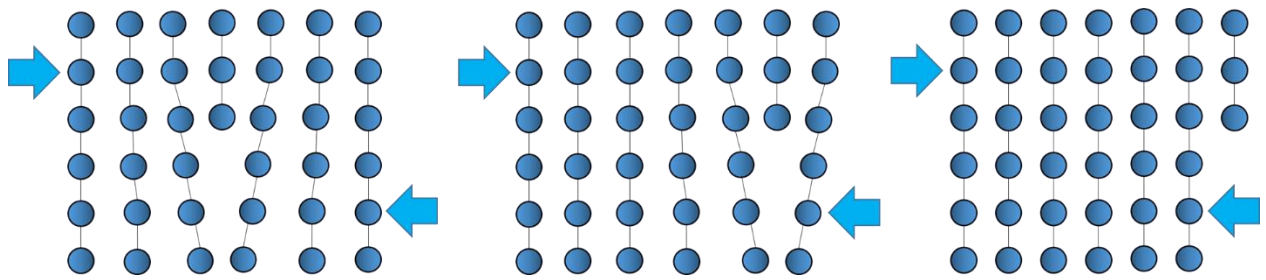


Figure 9: Example of slip occurring due to a partial dislocation within the lattice

FCC materials i.e. copper and stainless steel have 12 slip systems. Their slip systems are of type $\{111\}$ and their direction is of type $\langle 110 \rangle$. There are four octahedral planes (111) , $(1\bar{1}1)$, $(11\bar{1})$ and $(\bar{1}11)$, six $\langle 110 \rangle$ directions, each one common to two octahedral planes giving 12 slip systems overall [106]. An image of what the (111) plane looks like in an FCC crystal is shown in Figure 10. Compared to BCC crystals, which can contain up to 48 slip systems, FCC crystals are much less likely to deform through slip and more likely through twinning.

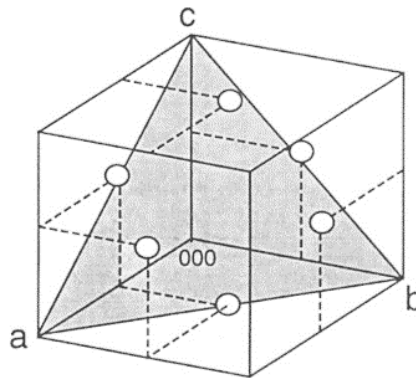


Figure 10: The (111) plane in the FCC system is shown shaded [106]

2.1.4 Plastic Flow in Metals

After a metal has reached its elastic limit, which is the limit where the material will return back to its original size and shape, any further stress, which is applied to it will

strain the metal in a plastic manner, where the strain is no longer directly proportional to the stress and the deformation becomes permanent [83]. Upon removal of the stress, the elastic strain recovers but the plastic strain does not as the lattice has been deformed by the breaking of a number of atomic bonds caused by the movement of dislocations [107].

Figure 11 refers to a typical stress strain curve of a metal showing the yield strength or elastic limit after the linear (proportional) stress and strain within the elastic zone. The plastic zone then consists of a period of strain hardening where the strain increases at a greater rate than the stress, then finally necking and fracture of the material from C to D where the material begins to and then eventually fails. The overall strain along the bottom is the change in length of the material.

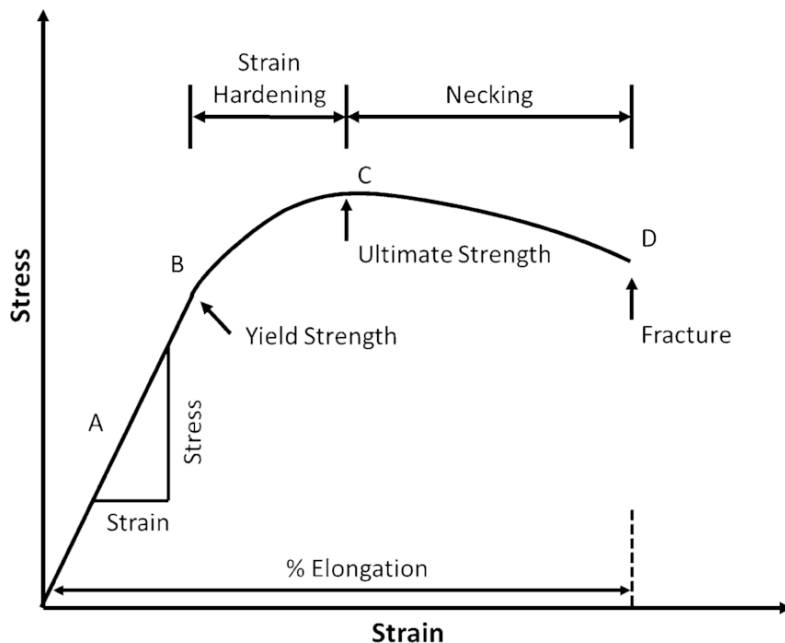


Figure 11: A typical stress-strain curve [108]

Metals in general are known to have high elastic modulus values for example copper (117 GPa), stainless steel (~200 GPa) and niobium (103 GPa) [109]. The amount

which they yield plastically before fracture, determines whether they are brittle or not [65]. A brittle material fractures a short time after plastic deformation begins to occur. Plastic deformation can occur via twinning or slip in metals [83].

In FCC metals, the effect of twinning to the flow stress is inversely dependent upon the greater the spacing between twins within a grain because dislocations are prevented from spreading by the twin/matrix interface this is given in Figure 12 [110], [111].

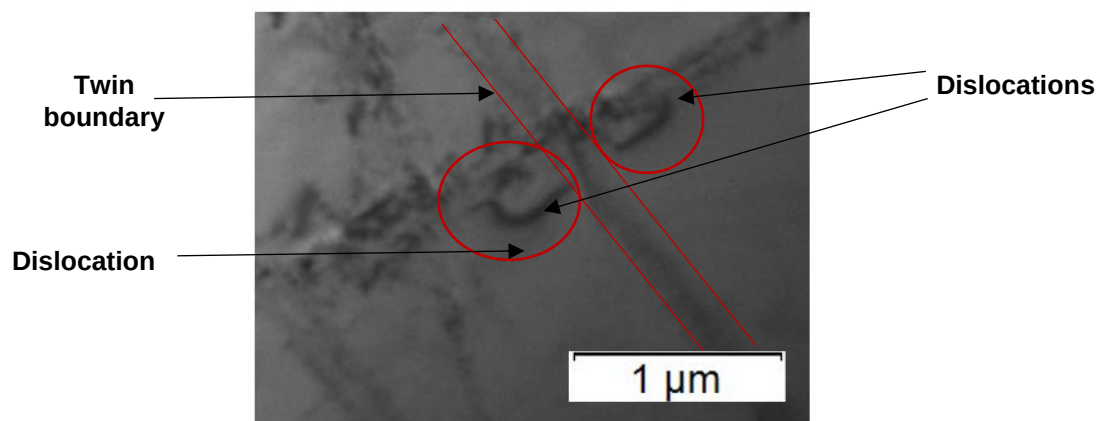


Figure 12: Twin inhibiting dislocation from spreading in 12 GPa shocked and soft recovered stainless steel 21-6-9. Two dislocations are seen either side of the twin, marked in red circles. The twin boundary is given by two red lines. This image was captured on a TEM.

The 'pinning' (inhibition of further movement) of dislocations occurs at interfaces where rapid deformation twinning has occurred. This is assumed to inhibit further development of the microstructure, fixing the yield surface and preventing further plastic flow from occurring [13].

2.1.5 Strength and Toughness

The strength of a material is its ability to withstand an applied load without failure or plastic deformation [65]. Dislocation movement produces additional dislocations and when dislocations run into one another it impedes the movement of further dislocations causing strengthening within the material i.e. the strength of the material is related to the shear stress needed to propagate dislocations throughout the matrix [77], [111].

Toughness is the ability of a material to absorb energy and plastically deform without fracture [65]. High toughness results from a good combination of strength and ductility; hence high strength and high ductility will result in high toughness. On a stress strain curve the toughness is represented by the integral of the curve, where the resulting value is material toughness and has units of energy per volume [112].

Toughness is affected by strain rate or loading rate, temperature and the notch effect, where if a notch is present a stress concentration occurs and the material will be more likely to fail as it cannot stand the simultaneous elastic and plastic deformation, this is carried out under laboratory testing conditions as the Charpy impact test [112]. If the temperature is lowered, the toughness and ductility decrease as the material becomes more brittle due to less energy because crystal defects become less mobile [112].

2.1.6 Hardness

Hardness is defined as the resistance to localised plastic deformation induced by mechanical indentation or abrasion [113]. Hardness is empirical in nature and therefore not a basic property of a material but encompasses yield strength,

toughness, ductility and wear resistance [114], [115]. The hardness value varies depending on the load size, as microhardness testing of less than 1 kg is used for variations in surface hardness of a material whereas if a hardness test is carried out above 1 kg a bulk hardness value can be obtained [115]².

2.1.7 Phase Transformations

To define a phase transformation a definition of a phase must be given; where a phase is a region of space where all physical properties of a material are uniform [65]. A phase transformation occurs when these physical properties change for example in steel this could be from ferrite to cementite. The phase is formed from the elements present in the material. Phase transformations can occur during heating, cooling or shock events [65]. As previously discussed within stainless steels, martensite can form from austenite through quenching or shock events provided that the SFE is below 20 mJ m⁻².

There have been numerous examples of phase transitions under shock loading [20], [45], [116]–[119]. Duvall and Graham investigated phase transitions under shock-wave loading [120]. Phase transformations are likely to occur where there is twin formation or dislocation formation due to the defects introduced into the material by the shock wave [120]. The pressure for phase transformation to occur is different for each material, as well as other factors such as temperature, grain size and defects already present in the material [120]. Having described the fundamentals of crystalline

² Therefore it is important to note the load size during testing.

structures, a brief description of the project materials will be given in Section 2.2 and further expanded upon in the results and discussion Chapters 4, 5 and 6.

2.2 Project Materials

The following metals were used in this project: stainless steel 21-6-9 and copper. Oxygen-Free High-Conductivity (OFHC) copper is the first material to be explored because it is a well characterised material within the literature available on its microstructure post shock, for both standard and recovered samples [50], [62], [121]–[125]. Therefore, it can be used for comparative purposes whilst setting up the recovery process [54], [126]–[129]. Additionally, copper has a face-centred cubic (FCC) structure as does stainless steel 21-6-9, which contains 21% chromium, 6% nickel and 9% manganese, showing similarities during deformation, such as twinning [63], [128], [130].

Sencer *et al.* studied similarities between copper and stainless steel 316L, which is an austenitic stainless steel similar to stainless steel 21-6-9 [128]. Though no twinning was shown at 6 GPa for copper, it was for stainless steel 316L [128]. Dislocation formation was shown for both copper and stainless steel 316L; with dislocation cell formation in the copper. Stainless steel 316L also showed evidence of planar slip [128]. Copper has a higher SFE of 78 mJ m^{-2} making deformation twinning less likely to occur at lower pressures of less than 10 GPa than the stainless steel 316L [122], [131].

Stainless steel 21-6-9 is an austenitic stainless steel with a FCC structure; part of the 200 series of stainless steels, using the properties of chromium and nickel to keep the

microstructure stable [1]. Chromium is added to steel to increase resistance to oxidation, providing the steel with corrosion resistance due to the formation of a self-repairing passive layer of chromium oxide on the surface of the stainless steel [73]. Stainless steels usually have a minimum of 10.5% chromium [73]. Manganese is added to improve hot working properties, increasing strength, toughness and hardness [73]. Manganese is an austenite-forming element and has been used as a nickel substitute in the American Iron and Steel Institute (AISI) 200 and 300 series of austenitic stainless steels, where they are all FCC in crystal structure [73]. Nickel is added to high chromium stainless steels to form austenite, which is responsible for high toughness and high strength. It also improves the resistance to oxidation and corrosion [73].

From experiments which Hills carried out, ϵ -martensite (HCP crystal structure) formation occurred at pressures above 30 GPa [101]. However, work carried out by Furnish *et al.* determined that, having carried out experimental work between 10-50 GPa, that there was no clear sign of a phase change as the Hugoniot (the Equation of State (EoS) of a material – a description of its high strain behaviour) was primarily linear [20], [132]. If there had been any indication of a phase change then there would have been another linear Hugoniot at a different gradient highlighting a change in the microstructure.

The full chemical composition of this particular stainless steel 21-6-9 is shown in Table 1.

Table 1: Table showing full chemical composition of stainless steel 21-6-9

Element	Chemical Composition, %
Aluminium	<0.01
Carbon	0.022
Cobalt	0.06
Chromium	19.79
Copper	0.27
Iron	Balance
Manganese	8.86
Molybdenum	0.26
Nitrogen	0.31
Niobium	0.03
Nickel	6.44
Phosphorous	0.024
Sulfur	0.001
Silicon	0.26
Tin	0.009
Titanium	<0.01
Vanadium	0.04
Tungsten	<0.05

The analytical techniques utilised within this thesis were: optical microscopy, X-ray diffraction (XRD), Transmission Electron Microscopy (TEM), hardness testing and compression testing. Computer modelling was carried out in order to validate the recovery and to further develop the design of the recovery targets (detailed in Chapter 6).

2.3 Shock

The experiments detailed in this thesis are dependent upon the fundamentals of shock wave propagation, seeking to explain shock effects on metals, by providing a background on material strength evolution and the causes behind different types of deformation.

2.3.1 Shock waves

A shock wave is a wave, which travels faster than the speed of sound in the medium which it is travelling through [2]. As well as detonation of explosives, shock waves can also be caused by uniaxial strain and inertial confinement, with no lateral flow of materials, allowing high levels of stress to build up [2]. When this hydrostatic component reaches a high enough stress level, it exceeds the dynamic flow stress, which is the flow stress at the strain rate at the wave front, and can be assumed to have no resistance to shear. The shock wave provides a discontinuity in the material properties of the medium, such as energy, density, pressure and temperature [2], [133]. The interaction between the shock wave and a material is highly complex due to reverberations and directional changes with the wave after it has interacted with the material [2].

Shock waves can be either elastic or plastic. An ideal elastic wave is a mechanical disturbance that propagates through a material causing oscillations of the particles inside the material about their equilibrium positions but with no other change [134].

When the stress of a ductile material exceeds its elastic limit, plastic deformation then occurs; in both quasi-static and dynamic deformation. If a pulse enters a material, which exceeds the elastic limit, then both elastic and plastic deformation will occur.

To fully understand the nature of material and shockwave interaction, a fully one-dimensional shock is required, where the shock wave travels only in one dimension. This allows an analysis of the material under known conditions. Figure 13 shows a uniaxial strain curve for an elastic perfectly plastic material i.e. a metal, where there is no work hardening, and a loading and unloading curve of a material under uniaxial strain. This is where the strain in the x-plane does not equal zero ($\epsilon_x \neq 0$) and the strain in the y and z plane does equal zero ($\epsilon_y = \epsilon_z = 0$) and the overall stress within the x, y and z planes does not equal zero ($\sigma_x \neq \sigma_y = \sigma_z \neq 0$).

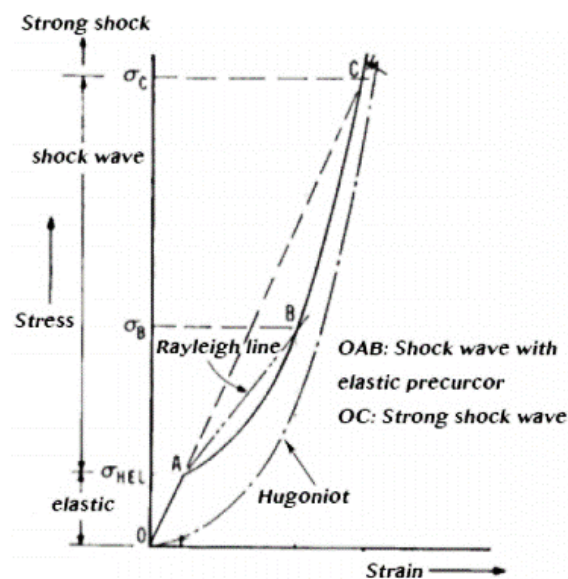


Figure 13: The theoretical Hugoniot stress-strain curve of a material when it is shock loaded. Where O-A is the elastic precursor, as A is the Hugoniot Elastic Limit (HEL) [4]

The yield point for uniaxial strain is known as the Hugoniot Elastic Limit or HEL, which is the maximum stress for elastic wave propagation in plate geometries, where the

thickness of the plate is negligible compared to other dimensions. The Rayleigh line is the line that the shock wave travels on while the curve is the loci of all of the Rayleigh lines. It is a line as it represents the discontinuity of the jump state.

2.3.2 Rankine-Hugoniot Relationships

The calculations of shock wave parameters are underpinned by the Rankine-Hugoniot equations [3][84].

It is assumed that [2], [133]:

- A shock is a discontinuous surface with no apparent thickness
- The shear modulus of the material is taken to be zero (the material has zero strength), so it responds to the wave as a fluid not a solid, which occurs at high strain rates
- Body forces and gravity are assumed to be negligible
- There is no elastoplastic behaviour (neither plastic not elastic deformation)
- The material does maintain its original phase

There are five key parameters, which are used within a shock loading event. These are shock velocity, U_s , particle velocity, U_p , density, ρ , pressure P and internal energy E . The particle velocity is the velocity of the individual elements, which propagate the shock wave through the medium with a velocity U_s . The shock velocity is the velocity at which the induced shock wave travels through the medium, therefore particle velocity is always slower than that of the shock velocity [2].

The Rankine-Hugoniot relations give a description of how a material behaves under shock using these five parameters. These relations are supported by the conservation equations; of mass, momentum and energy.

The derivation of the conservation equations can be pictured with a piston penetrating a cylinder with a set cross-sectional area, as in Figure 14, where the piston is initially at rest with pressure P_0 , density ρ_0 , and a particle velocity of u_p . Once the cylinder has begun to move, after a certain length of time, t_1 , the compressed region in front of the piston has a distance to $U_s t_1$, where U_s is the velocity of propagation of the disturbance in front of the piston. In shock wave propagation this is known as the shock velocity. The distance that the piston has moved within time t_1 is denoted by $U_p t_1$.

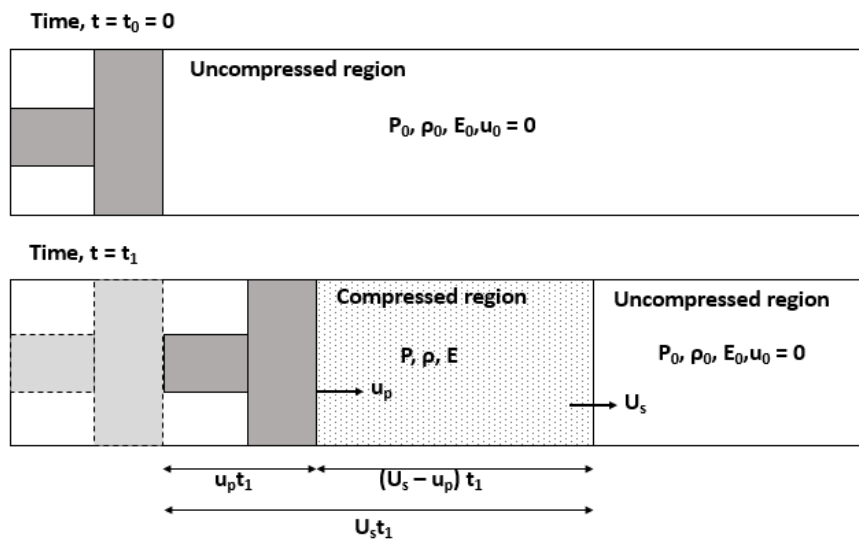


Figure 14: Idealised piston moving in a cylinder with compressible fluid, analagous to a shock wave travelling through a material (Adapted from Meyers [2])

These relations, which will be described in more detail, provide the linear Hugoniot EoS of the form:

$$U_s = S \cdot u_p + C_0 \quad \text{Equation 3}$$

U_s = Shock velocity (ms^{-1})

S = Empirical value

u_p = Particle velocity (ms^{-1})

C_0 = Sound velocity in a material at zero pressure (ms^{-1}) $\approx C_B$ = Bulk sound velocity (ms^{-1})

Where:

$$C_B = (C_l^2 - 4/3 C_s^2)^{1/2} \quad \text{Equation 4}$$

C_l = Longitudinal Sound Speed (ms^{-1})

C_s = Latitudinal (shear) Sound Speed (ms^{-1})

C_B can be obtained experimentally by measuring the longitudinal sound speed in a material and the shear or latitudinal sound speed in a material.

Two shock parameters are needed to define the shock response of a material, as the others can be obtained by derivation of the Rankine-Hugoniot relationships. These relationships involve the conservation of mass, the conservation of momentum and the conservation of energy. These conservation equations are used as the system is closed.

Conservation of Mass:

The conservation of mass describes the mass going into a process equalling the mass going out of the process. Within the reference frame of the laboratory, before the shock front enters the target, it is travelling at a velocity of $U_s - U_0$. After entering the material, the material itself is compressed by the amount $U_s - U_p$, which leads to an increase in density to ρ from ρ_0 , as the volume is decreased and the relation $\rho = m/v$ applies [135]. In this case, the mass moving toward the front is the same as the mass moving away from the front.

$$U_s \rho_0 = (U_s - U_p) \rho \quad \text{Equation 5}$$

U_s = Shock velocity
 U_p = Particle velocity
 ρ_0 = Initial density
 ρ = Density after shock

Conservation of Momentum:

The conservation of momentum means that the difference in momentum must be equal to the impulse ($F \cdot dt$) per unit of the cross-sectional area. Momentum is defined as the mass of an object multiplied by the velocity and the impulse given by a force multiplied by the change in time. The impulse is then equated to the change in momentum and the initial particle velocity is assumed to be 0, giving the equation below. When the $U_s - U_p$ state is known, the hydrodynamic pressure can henceforth be derived.

$$P - P_0 = \rho_0 U_s u_p$$

Equation 6

U_s = Shock velocity
 U_p = Particle velocity
 ρ_0 = Initial density
 ρ = Post-shock density
 P_0 = Initial pressure
 P = Post shock pressure

Where $\rho_0 U_s$ is also referred to as Z , the shock impedance.

Conservation of Energy:

The conservation of energy is derived by setting up an equation where the work done by P minus the work done by P_0 is equal to the difference in the total energy (kinetic and internal) between the two sides of the shock front.

$$E_1 - E_0 = \frac{1}{2} U_p^2$$

Equation 7

E_1 = Shock front energy
 E_0 = Initial energy
 U_p = Particle velocity

By using these Rankine-Hugoniot relations, the Hugoniot relationship, which provides the locus of available jump states i.e. the relationship between states either side of the shock wave: before and after, can be calculated. By carrying out experiments in different conditions the materials EoS can be experimentally obtained. This is primarily represented in two different planes, with the U_s - U_p relationship and the P - V Hugoniot equations. Figure 15 shows the EoS in the shock velocity and particle velocity plane in a quartz ceramic. The change in gradient indicates a phase change in the material.

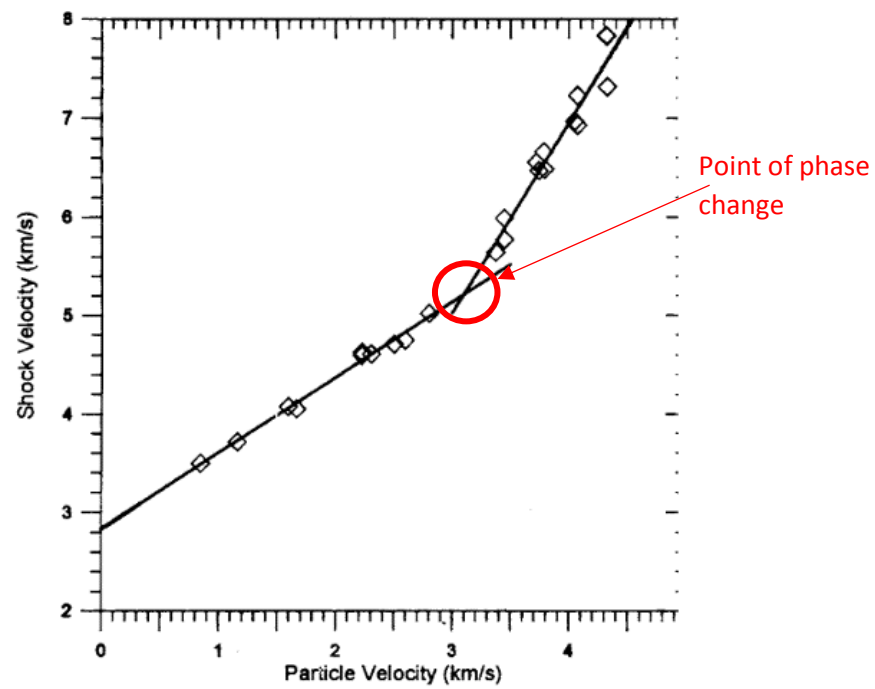


Figure 15: EoS in the U_s - U_p plane for a quartz ceramic [224]

Figure 16 is the Hugoniot presented in the P - V plane. The shock loading travels up the Rayleigh line and unloads on the relief line (isentropic). The Hugoniot is the locus of all states reachable by a shock transition but is not the loading path [136]. The Rayleigh line is where the loading occurs. This represents a linear relationship between P and V [135].

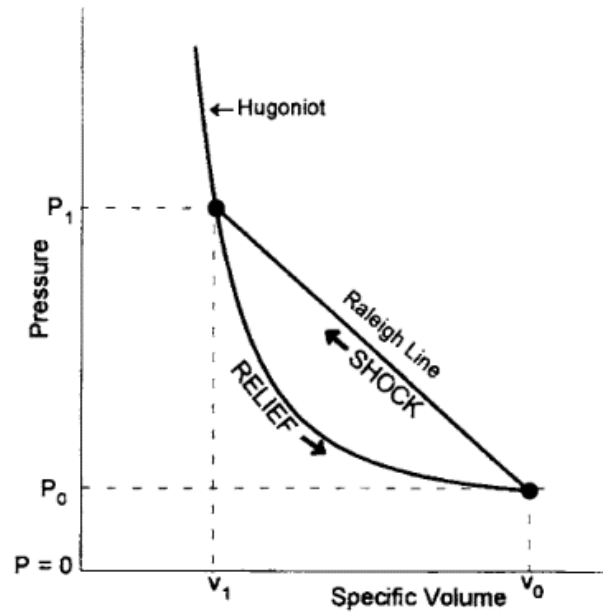


Figure 16: Shock loading and unloading EoS in the P-V Plane [224]

The isentrope of a material is incredibly close to its Hugoniot up to 20 GPa, so when solving problems, the Hugoniot is considered to be the unloading pathway [2]. The area confined by the Hugoniot and Rayleigh line is the waste heat i.e. the energy lost during the cycle.

2.3.3 Differences between Quasi-Static and Dynamic Deformation

There is a difference between static or quasi-static and dynamic deformation. In quasi-static deformation, there is always a situation of static equilibrium. Therefore any forces which act upon it sum to zero [137].

Conversely, during dynamic deformation, a shock wave with an established velocity travels through a medium. This dynamic deformation occurs when the deformation is imparted from one object to another. This creates an uneven amount of stress, as one

part of the body is stressed, while the other part of the body is yet to receive the stress, as the stress travels through the body, at a certain velocity this causes a dynamic deformation, carried as a wave [2].

Therefore, the main difference between quasi-static and dynamic deformation is that dynamic deformation includes wave propagation whereas quasi-static deformation is considered as a sequence of different equilibrium states [2]. Quasi-static deformation is governed by the equations of mechanics of materials where summed forces are equal to zero and summed moments are equal to zero [2]. This project focusses on dynamic deformation.

2.3.4 Yield Stress Versus Strain Rate

Material properties are strain rate dependent [2]. The graph in Figure 17 shows the strain rate dependence of copper (FCC) on its yield stress. The relationship initially appears to be linear within the quasi-static region up to approximately 10^3 s^{-1} . However, when entering dynamic strain rates above that of 10^3 s^{-1} , there is an exponential increase in the yield stress versus the strain rate. This helps to explain why there are therefore significant changes in the way certain materials deform in dynamic conditions, such as certain phase changes or high levels of twinning, which are not otherwise observed in quasi-static conditions.

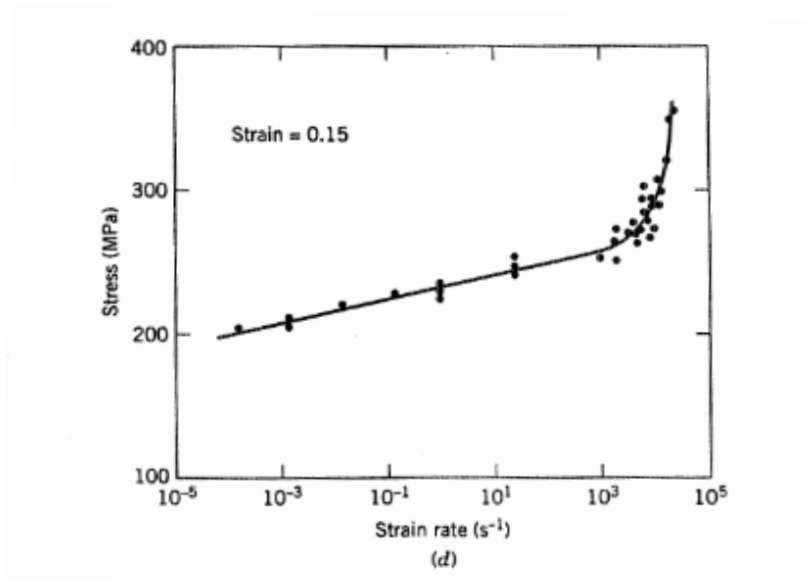


Figure 17: Graph showing changes in stress versus the strain rate [2]

2.3.5 Mie-Grüneisan Equation of State

The Mie-Grüneisan Equation of State is an EoS that relates the pressure and volume of a solid at a given temperature [2]. It is used to determine the pressure in a shock compressed solid. The bonding and phonon frequencies are affected by the shock stress and linked to thermal expansion and heat capacity [2]. This equation relates the internal structural and crystallographic shock loading changes [2].

$$P - P_{0k} = (\gamma/V) (E - E_{0k}) \quad \text{Equation 8}$$

P = Pressure

V= Volume

E= Energy

γ = Grüneisen constant (where $\gamma = 1$ corresponds to frequencies being inversely proportional to changes in volume)

2.3.6 Development and Use of Gas Guns

Gas guns are a way of introducing a shock wave into a material. Gas guns were first employed experimentally during the 1940s and gained prominence in the 1950s and 1960s [138]. A research group lead by Workman at the New Mexico Institute of Mining and Technology (NMIMT) collaborated with the United States Army Ballistic research laboratory to increase the launch velocity of powder guns, post World War II [139]. The first light gas gun was used in 1948 and launched models at velocities of more than 2.5 km s^{-1} [139]. They experimented with the pump piston mass, solid propellant mass, initial light gas charge pressure, projectile release pressure and projectile mass. They also discovered that changes in chamber volume, pump tube length, pump tube diameter, convergence angle of the taper section, launch tube diameter and launch tube length all affected the launch velocity [139].

Light gas gun facilities were further developed at NASA Ames to look into both space and missile research, which grew exponentially toward the end of the 1950s [11], [139], [140]. By the 1960s, there were several dozen operations in Western Europe, Canada and the US. The US navy investigated armour for satellite protection in the 1950s, which increased the research in increasing gas gun velocities as well as studies in the impact mechanics on the shielding materials themselves [139]. There were increases in the velocity capabilities of light gas guns during the 1950s and 1960s and from the 1970s research focussed on both increasing velocity as well as the understanding of the shock response of materials.

During this time various experiments were carried out on the explosive loading of metals by authors such as Meyers, Gupta and Murr and Yust and Harris [141], [142].

Murr, Gupta and Meyers carried out explosive loading of rapidly solidified superalloy powders to investigate hardness and microstructure of the materials [142]. Yust and Harris investigated the prevalence of dislocations and twins in explosively compacted alumina [141]. Due to the 'densification' caused in these experiments, difficulties were faced when trying to determine why certain microstructural processes had occurred.

Overall, these experiments indicated a depth change in properties based on a triangular loading pulse. This highlighted the fact that hardness measurements, grain size/ shape and dislocation density changed markedly with distance from the impact interface. Therefore, a more controlled experiment is required to obtain a sample, which has been loaded to a well-defined and desired state, for analysis with lower penetrating depths in metals such as XRD. Recovery using gas guns can provide what is known as a square-topped pulse, allowing for this well-defined loading to occur.

There are different methods of propulsion used within these gas guns, but in the case of the light-gas single stage and two-stage guns, light gases such as helium and air are used. The pressure from these gases is used to drive a projectile such as a ball bearing or sabot down the barrel of the gun to impact a target [2], [138], [139]. Figure 18 shows a 50 mm bore, 6 m long single stage gas gun, which was used in this project.



Figure 18: Image of a 50 mm bore, 6 m long single-stage gas gun

The impedance matching technique can be used to obtain the particle velocity, U_p and pressure, P , of the target [2]. This technique matches the flyer plate shock state to the shock state of the target at a given impact velocity. This can be done graphically on a pressure particle velocity graph by inverting the curve of the flyer plate and offsetting it by the value of the impact velocity, U_s . This curve intersects onto the curve of the EoS of the target material. Where the two curves intersect the pressure, P , and particle velocity, U_p can be obtained.

2.3.7 Spall Fracture

Spallation failure involves the initiation and development of voids caused by tensile releases within a material [2], [143]. Using the gas-driven plate impact technique, when a compressive shock wave propagates through a material, it reflects off the rear

surface of that material and returns towards the impact side as a rarefaction wave [2], [12]. An interaction then occurs with a reflected wave coming from the impact side of the sample caused by the compression of the flyer plate forming a region of high tensile stress at the spall plane. Then incipient spall will occur, where voids are nucleated due to the tensile stress [144]. This is shown in Figure 19. However, the stress is not high enough for more voids to form and coalesce causing full spallation.

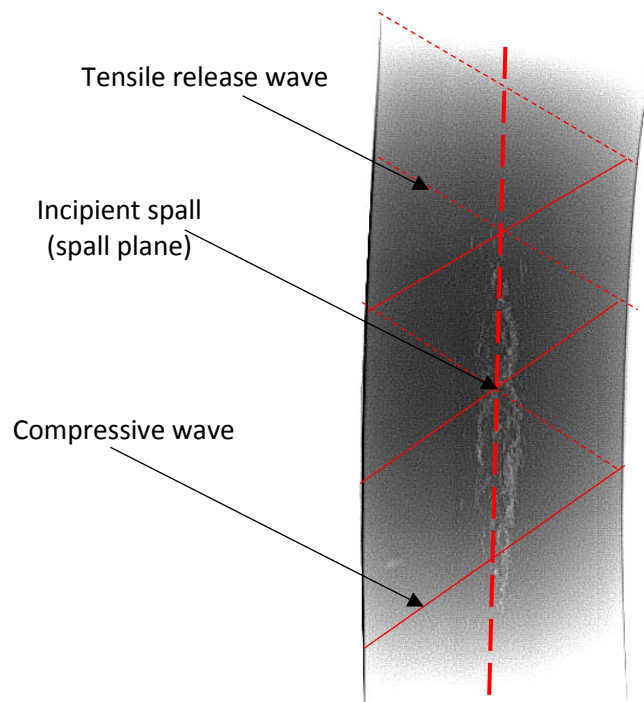


Figure 19: Spall plane within stainless steel 21-6-9. Image produced from micro CT scan. Red lines indicate overlap of tensile release waves and compressive waves causing incipient spall to open up in the spall plane.

2.3.8 Nucleation of Spall in a Metal

Spall fracture is a type of material failure. The process involved can nucleate more than a million cracks or microscopic voids per cubic centimetre within a sample [145]. The nucleation rates are dependent upon the levels of stress and temperature, where if the stress is increased the nucleation will be increased. Temperature can affect the

phase of a metal, hence it could lead to increased or decreased nucleation. The nature of spall fracture means that the stress levels reached during spall fracture are significantly higher than the stress, which would produce fracture under static loading.

Work carried out by Brown *et al.* involved looking at the microstructural effects on damage nucleation in shock-loaded polycrystalline copper [146]. Spall was induced via laser driven plate impacts, at a low stress <6 GPa [144]. Laser pulses can be used to drive thin flyer plates (in the order of microns) at targets, due to the energy of the laser these impacts can reach velocities of more than 13 km s⁻¹ [147].

Thissell *et al.* looked at nucleation and growth of voids in spall processes in shock loaded tantalum [148]. The loading conditions were varied to study the damage evolution from incipient to full spallation [148]. Overall they found that the VISAR trace did not reflect the damage evolution in the centre of the target due to the thickness of the target [148]. There was pronounced linking of ranges of voids where there was a higher level of tensile hydrostatic stress caused by a smaller level of porosity [148]. Overall the research showed that voids can initiate and propagate in a similar manner to that of dislocations with regards to them being a line defect and their dependence is related to the release pathway.

Electron backscatter diffraction (EBSD) was used to obtain statistics on grain boundary misorientation within the spall plane and all grain boundaries, which contained damage. They found that overall grain boundary angles of both 25° and 50° were favourable for having damage regardless of processing condition, this was exacerbated when the sample was plastically pre-strained using to its advantage the

Bauschinger effect [149] [150] [151]. When plastic deformation occurs in one direction and then occurs in the opposite direction, the internal stress distribution of the material changes, also changing the material properties. Strain hardening occurs, increasing the yield strength in the direction of applied force, while decreasing the yield strength in the opposite direction. The drop in yield strength is defined as the Bauschinger effect [151].

2.4 Computer Modelling and Recovery

Computer modelling, when carried out with parameters, which can accurately reflect those of experimental scenarios, can provide an important insight into real-world scenarios. When the model computes strains, stresses, velocities and other parameters related to shock wave propagation, it is called a hydrocode, due to the hydrodynamic behaviour of the material [152]. Various factors are required for an accurate model including appropriate computational power, accurate information on the material as well as the selection of the correct model type [7], [60].

Computer models are used to imitate experimental scenarios using information that has been gathered. Within a hydrodynamic context, computer models such as ANSYS® Autodyn can be used to simulate impacts involving high strain rates. Hydrocodes are used for modelling fluid flow at all speeds, as well as solids which behave hydrodynamically when impacted at higher pressure [7]. Hydrocodes are underpinned by the conservation of mass, momentum and energy, as well as an EoS depicting the behaviour of the material being modelled and a constitutive model

relating strain, strain rate effect, damage and internal energy, providing a description of the effect of deformation i.e. changes in shape or strength [7].

Computer modelling is necessary to model the recovery process which is being developed within this thesis, due to the complexities and cost of the target manufacture. It has the benefit of being cheaper, as multiple models can be run at the same time. One can model the recovery process by creating flyer plate impacts with single disc targets into more complex forms by adding concentric rings and spall plates around the targets to capture latitudinal and longitudinal shocks. However, the computer model is only as accurate as the data, which is put into it, which is why experimental work is necessary to define input parameters for the model.

Computers have a finite memory allocation and can only represent a continuous media by forming discrete pieces or cells. In hydrocodes and other computer models, this involves the definition of a 'mesh' to approximate the shape of the article being modelled and is divided into individual divisions or 'cells'. This is represented in Figure 20a and 20b.

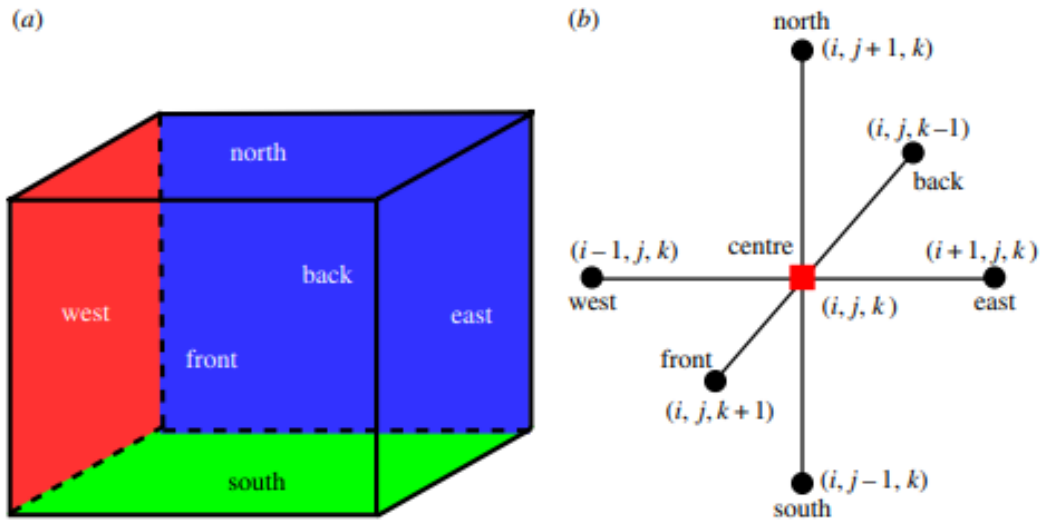


Figure 20: 3D Computational cell representation using vectors [153].

The accuracy of the computer models depends upon the size of the mesh [7]. A finer mesh will produce more accurate results, however, this can vastly increase computational processing time [152]. Therefore, a balance must be struck between accuracy of results at key points of the model and the run time, as increasing the fineness of the mesh does not always improve the accuracy of the model.

Within the shock physics literature, computer modelling allows for the creation of highly detailed targets and a simulation of their impact. For this project in particular, the development of highly detailed recovery targets is possible in order to verify the models using the experiments as well as developing further iterations of future recovery models and/or modelling other materials of interest.

Lagrangian hydrocodes are defined as being performed on a fixed grid system, whereas Eulerian modelling is contained within a grid and objects can travel freely across the grid. Eulerian modelling provides a spatial description whereas Lagrangian

provides a material description [152]. In Lagrangian modelling the computational grid is embedded and deformed with the material; this is conceptually more simple and efficient to solve than Eulerian programming as there is no advection term, which describes the mass flow across element boundaries [154]. Also, as Lagrangian methods track the flow of individual masses, history dependent constitutive models are easy to implement.

Eulerian methods are useful where material domains become heavily distorted or different materials are mixed together. The material flows through the grid as opposed to with the grid, this makes boundaries difficult to define and there are also issues with changing density in each element dependent upon the material passing through it [154].

Boundaries in Lagrangian programming are easier to keep track of, whereas boundaries in Eulerian coding can be difficult to keep track of depending upon mesh size. For example if two different materials intersect between grid boundaries, then the material which takes up the larger space will be the material which takes up that particular grid space; leading ultimately to uncertain boundary edges [152].

Lagrangian models can incur error, where a large grid distortion occurs i.e. during high velocity impacts, as the impact boundaries can distort to the point where a fixed grid inverts on itself and creates a negative mass [152]. This is corrected by replacing the zone where extreme distortion and compression has occurred with a new undistorted grid; within hydrodynamic modelling computer programs including ANSYS® Autodyn the removal of the grid is called erosion and can be set to a certain

value [155]. Mass, momentum and energy are then conserved by revaluating the old grid's properties and transferring them to the new grid. If this did not happen, then as the distortion becomes more complex, the timestep becomes increasingly smaller until it approaches zero, whereby the model would fail [154].

Hagelberg *et al.* carried out simulations of impacts into sandstone using smooth particle hydrodynamics and the discrete element method [156]. The experimental samples were subjected to shock loads ranging from 2.0 GPa to 9.8 GPa and were either 1 μ s to 2 μ s in duration and were carried out on both wet and dry samples of Berea sandstone. The computational models had qualitative agreement with the experimental work for both wet and dry samples. Simulated damage was the same as the experimental recovered samples, this was achieved by carrying out grain scale simulations with a wide range of geophysical parameters with large domains to help develop continuum models [156]. However further development would be required to reflect the heterogeneity of damage evolution, requiring a larger computing domain and longer processing times. This highlights the complexity in crystalline materials, accounting factors such as differences in grain size and defects within the structure. To model this complexity, more detailed material data is required. This can be obtained using different analytical techniques.

Smooth particle hydrodynamics (SPH) [157], which is a similar method to Lagrangian FEA involves the interpolation between particles where the fluid information is known. The function has to be differentiable and continuous [157]. Interpolation is carried out by having an associated kernel of data attached to the particle, which is a symmetrical function resembling a Gaussian in shape. This method can be used to also carry out

FEA using an appropriate interpolation technique. The technique has been used to model blast, hypervelocity impacts and other hydrodynamic events. The limitations would appear to be with fragmentation and providing enough information to accurately predict it in a high velocity event [158].

As well as this, SPH could not initially describe complex material properties such as elasticity and strength. However, developments in SPH and linking it to FEA by Attaway *et al.* and Johnson and Gordon have led to improvements within the field, allowing for more advanced computational analysis of materials [158]–[160].

SPH can also be poor at defining boundaries and the density can vary dramatically during impact events [158]. However, this still means that they can still be relevant in certain astrophysics areas such as planet and star formation due to the simplicity of SPH. It also does not suffer from the same Lagrangian problems from large deformation as the particles are not connected so do not need to be eroded and replaced in areas of large deformation, commonly used in planetary science [158].

Finite difference method and the finite element method can also be used [7]. The former uses difference equations to replace the spatial derivatives in the differential equations. Unlike differential equations, which are taken over an infinite space difference, equations are taken between a series of numbers so that it can be solved numerically i.e. as in code. It is a simple method but limited by complex geometries due to having a structures grid arranged in rows and columns. Higher accuracy is obtained with smaller cell size but this can significantly increase computational times [7].

The Finite Element Method differs from the Finite Difference Method as it separates the modelling space into elements as opposed to discrete points [36]. The elements can be curved or rectangular and don't need to be arranged into a structured grid. Usually polynomial interpolation functions are used to represent the variation of variables over a singular element [152]. Robertson *et al.* looked at numerical simulation of impact and fast transient phenomena using ANSYS® Autodyn 2D and 3D. Various models can be accessed such as Johnson-Cook, Zerilli –Armstrong and Steinberg-Guinan, which include thermal softening strain rate hardening and strain [161]. Robertson confirmed that in scenarios where the boundary is important Lagrangian is usually the preferred method as a finer mesh would be required in an Eulerian model vastly increasing computational time.

Banerjee compared the Johnson-Cook, Steinberg-Guinan, Zerilli-Armstrong, Mechanical Threshold Stress and Preston-Tonks-Wallace models to look at the errors in tension, compression, high and low strain rates and high and low temperatures, displayed in Figure 21 [161]. The Johnson-Cook model is empirical [161]. However, the strain rate dependence was found to be too low at high temperatures. The Steinberg-Guinan model is semi-empirical and is widely used in high strain rate scenarios (those above 10^3 s^{-1}) alongside the Johnson-Cook model. The Zerilli-Armstrong model is based on simplified dislocation mechanics. The Mechanical Threshold Stress model included the flow stress due to intrinsic barriers to thermally activated dislocation motion, dislocation-dislocation interactions and includes strain hardening [161]. The Preston-Tonks-Wallace model is used for extreme strain rates (those in the order of 10^{11} s^{-1}) and temperatures up to melt [161].

Condition	Average MA Error (%)				
	JC	SCGL	ZA	MTS	PTW
All Tests	36	64	33	23	17
Tension Tests	25	20	19	14	18
Compression Tests	45	126	50	35	10
High Strain Rate ($>100 \text{ s}^{-1}$)	29	22	20	15	18
Low Strain Rate ($<100 \text{ s}^{-1}$)	45	219	76	49	5
High Temperature ($>800 \text{ K}$)	43	90	40	27	16
Low Temperature ($<800 \text{ K}$)	20	20	17	15	14

Figure 21: Comparison between Johnson-Cook (JC), Steinberg-Guinan (SG), Zerilli-Armstrong (ZA), Mechanical Threshold Stress (MTS) and Preston-Tonks-Wallace (PTW) models [161].

In more recent years, authors including Vignjevic et al., Bourne and Gray have carried out modelling for recovery targets using both Lagrangian and Eulerian modelling [41], [60], [61]. Part of this is due to developments in processor size meaning that more intricate models can be run at a fraction of the time than they used to when computers were first being developed.

The most up to date recovery targets have been modelled by Bourne and Gray and Vignjevic *et al.* [41], [61]. Bourne and Gray used Eulerian modelling to look at momentum trapping recovery, as used in this project. Conversely, Vignjevic *et al.* used Lagrangian modelling to look at momentum trapping recovery, which is known to be more accurate at boundaries, which works well when observing the way spall plates and rings interact with the recovery section of the target [41], [61].

Bourne and Gray evaluated recovery models using an Eulerian approach [41], [54]. They modelled a 3 mm Cu flyer at 500 m s^{-1} with a pulse duration of $1 \mu\text{s}$ and a 10 GPa

stress level. Five gauges were placed along recovery axis in total. The set-up is given in Figure 22.

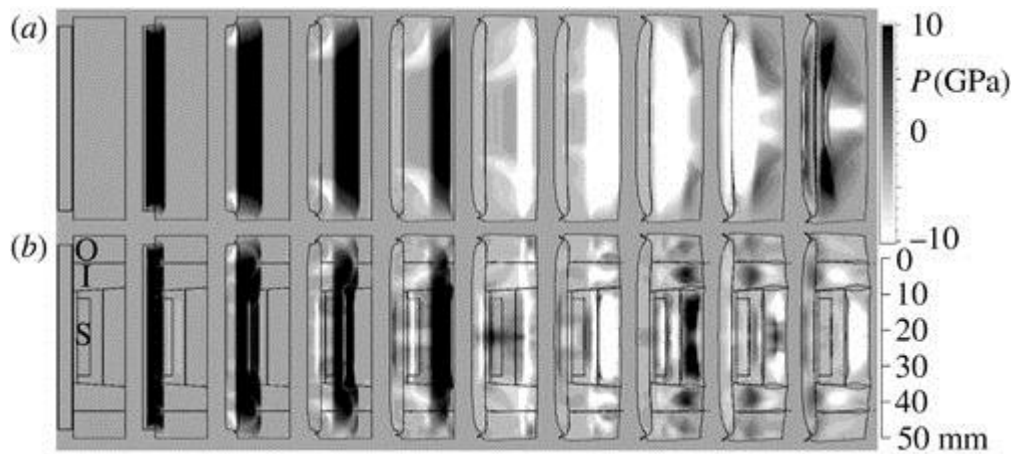


Figure 22: Impact of copper flyer onto copper target at 500 m s^{-1} [41]. (a) Indicates a copper flyer impact while (b) shows the recovery target impact, taken from the LANL design [54], [55], [162]. O and I are the outer target rings while S is the recovery sample.

Vignjevic et al. carried out Lagrangian simulations on a shock recovery plate impact experiment [60], [61]. This method was decided due to its ability to accurately track history variables such as plastic deformation and to accurately look at boundary interfaces. The new design overall resulted in a decrease of residual velocity (by 38%) and the radial displacement (by 27%), this design is shown in Figure 23.

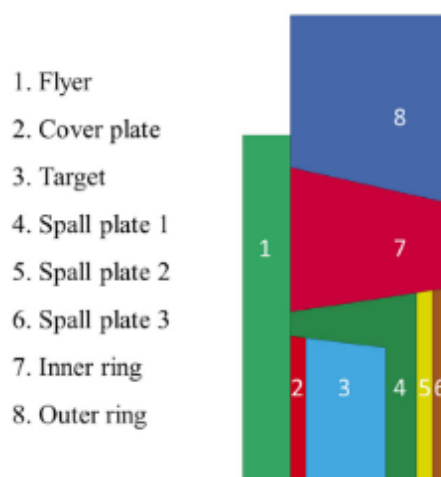


Figure 23: New design of target where spall plate 1 is twice as thick as spall plates 5 and 6 (not to scale).

In 2005 Bourne and Gray devised a computational design for recovery experiments for ductile metals, which differ in failure mode from brittle materials [41]. They built upon previous target design by Blumenthal *et al.*, Gray and Llorca *et al.* [57]–[59], [163] They classed these target designs, which were known to keep residual plastic strain on the recovery target low, as a baseline design.

Bourne and Gray wished to recreate the idealised single loading and unloading, square-topped pulse [41]. Due to the incorporation of the lateral release and rear spall plate, tensile interactions do not enter the centre recovery portion of the target. This means that the pulse duration is solely dependent upon the flyer thickness. Gray *et al.* (1989) determined that two concentric rings around the central portion of the target was an optimum number for trapping lateral momentum. However, as later discussed, Vignevic *et al.* modelled different target designs to come up with an optimal design where the recovery sample would not be impinged by the inner ring [61]. Originally there were issues with the inner guard ring preventing the release of the recovery and spall discs, which was eventually rectified by creating a tapered guard ring. The limitations were with the machining of the targets due to the need for interference and slip-fit joints [61].

Both Jones and Gray *et al.* looked at the significance of the dynamic unloading process of radial release in shock-loaded samples on the mechanical behaviour post-shock [54] [164]. The design consisted of a series of concentric rings around the central recovery portions of the target, which captured lateral release waves and a spall element at the rear to capture longitudinal release waves; a target design that has continued through to present work. This built on earlier work by Stevens & Jones

(1972) who studied this effect by using 2D Lagrangian finite-element modelling [165]. These experiments and simulations showed that by using concentric, lateral momentum trapping rings, uniaxial loading dominates radial release. Figure 24 shows their experimental set-up.

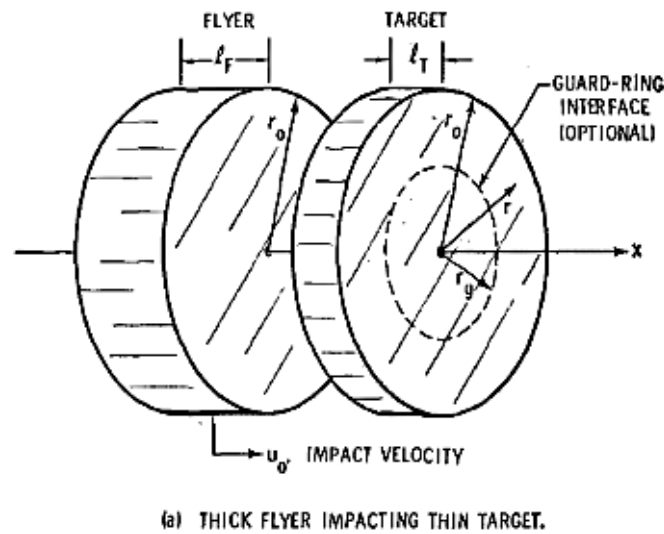


Figure 24: Experimental set up by Stevens and Jones [165]

Post-shock, targets must be decelerated to prevent re-impact, so that it is only the single shock pulse that is affecting the target. The temperature must also be lowered by quenching so that the microstructure evolves purely from the shock pulse. Smith (1958), Dieter (1961), Zukas (1966) and Gray (1993) met these two requirements by plunging the sample into a water bath immediately after the release of the shock pulse (temperature not specified); both quenching and decelerating the recovery sample [140], [166], [167]. The method has shown to be reproducible by several authors [54],

[58], [59], [163], however is not appropriate for the recovery of brittle materials which would crack / fail under the resultant thermal shock.

In order to impact and successfully recover brittle materials, the previously discussed 'star flyer' method has been used. On this front, Raiser et al. (1989), carried out a series of experiments on soft recovery for ceramics (Al_2O_3) in order to analyse resultant micro-cracking [168]. A star flyer was used and the ceramic backed with a steel plate, to mitigate the magnitude of the release waves.

Drawbacks to the numerical models developed by Bourne and Gray (2005) include limited experimental data set usage (e.g. limited experimental validation) [41]. There was only one set of data used, which was from Millet *et al.* (1997) from copper impact tests [169]. Furthermore, the simulation included modelling the copper with infinite tensile strength and not allowing spall to occur. The joints, which were also modelled, represented a worst-case scenario approach as the targets were designed with an interference fit, where the individual pieces of the target are held together by friction, whereas the modelled targets included interfaces with a 100 μm separation. Further, contact and friction were not modelled as it would add complexity to the simulation. The Murnaghan EoS was used with an elastic perfectly plastic strength model [3]. However, despite limitations to the model, the simulations indicated that less reloading occurred within the target if there were more rings to capture latitudinal release and multi-layered spall plates to capture longitudinal release.

Further modelling of recovery targets using Lagrangian analysis has been carried out by Vignjevic et al. (2015) in order to develop an efficient recovery target which has

only been affected by the initial shock front [60]. Lagrangian FEA was used as it allows history variables for plastic deformation to be tracked and for the treatment of contact interactions. Consequently, this work was designed to improve upon Stojkovic (2003), Bourne and Gray (2005) and d'Almeida et al.'s (2009) work, which used Eulerian models [170], [171]. The recovery target design used was the baseline model with two concentric rings and four spall plates. The authors employed a 10 μm thick gap between components to account for machining; suggested to be more realistic than Bourne and Gray's suggested tolerances of 100 μm (modelled in previous work). The test set up used involved a flyer plate of 25 mm diameter with 3 mm thickness and an initial velocity of 300 m s^{-1} . The target material considered was a H04 tempered Oxygen-Free, High Conductivity Copper (OFHC copper).

The Lagrangian analysis revealed that because of the low thickness and number of spall plates, multiple short duration pulses propagated between the two plates and release waves originating from the rear of the flyer plates and the rear of the last spall plate superposed. The release wave from the final spall plate transmitted back to the recovery specimen before the spall plates had separated. Further to this, uniaxial strain did not occur within the sample laterally either because the velocity of the inner ring was higher than the recovery specimen, causing impingement and pressure concentration around the sample.

Analysis based upon data gathered by Furnish, Gray and Bingert (2011) on annealed copper spall recovery samples, with three outer rings and one spall plate found the same issues as in the previous target recovery design [172]. In addition, the specimen had a high residual velocity of 23.6 m s^{-1} , which may have caused deformation upon

impact into the recovery material. This would seem unlikely as it is a comparatively low velocity to impact into the recovery material.

Using the data gathered from the previous two models, Vignjevic *et al.* developed an improved recovery target design using a Lagrangian model; one which removed the issue of the specimen contact with the inner ring [61]. Several iterations were required to do this until a final configuration was proposed. This configuration decreased the number of spall plates from four to three in a 2-1-1 mm configuration as this lengthened the interaction of the release wave with the 2-mm thick spall plate, hence preventing reloading of the specimen.

The Lagrangian finite element and finite difference method will be used, as provided by ANSYS® Autodyn as it is able to model complex target geometries well, while also including the most up to date material properties, the exact model e.g. Steinberg-Guinan etc can be selected upon experimental requirement [173].

In conclusion, by analysing the material deformation properties of these FCC and BCC materials and using computer modelling to predict and understand their behaviour, the combination of experimental and modelling work will allow for both the development of the recovery technique as well as the understanding of their respective material behaviours.

3 MATERIALS AND METHODS

There are three main disciplines within the context of this project, shock loading of materials, material analysis and computer modelling. Each of these topics will be explored in detail, as well as addressing the materials themselves in the project, copper and stainless steel 21-6-9.

3.1 Shock Loading

Shock loading of a material can be achieved in numerous ways [2]. Shock loading of a material can lead to dynamic deformation of that material, where deformation in the material is caused by the shock wave [137]. Dynamic deformation can occur in terms of plastic wave deformation using the Taylor test, the Hopkinson bar test or the expanding ring test, as well as the gas driven flyer plate technique [2]. In this project, the gas-driven plate impact technique is used to achieve shock loading of the material. High strain rates are required to understand material behaviour, as materials behave differently at high strain rates, where there is a shock front, compared to low strain rates or quasi-static loading [2]

In order to access strain rates higher than 10^4 s^{-1} in the laboratory, launchers of varying configurations are used, these range from electromagnetic systems to multi-stage launchers and laser shock peening [2], [38], [174]. However, for strain-rates up-to 10^6 s^{-1} single-stage gas guns using the flyer-plate impact technique provide a ready, accessible and repeatable means to achieve this effect. An example of a flyer plate attached to a polyoxymethylene (POM) sabot is shown in Figure 25.



Figure 25: 10 mm copper flyer plate attached to POM sabot

The configuration for a typical single-stage gas-gun is shown in Figure 26 and Figure 27. All gas guns operate using gas to propel the target down the barrel. Therefore, a high pressure chamber containing the driving gas is needed. This gas is usually air or helium. Helium provides a higher projectile velocity compared to air because of its faster sound speed caused by its lower molecular weight [2].

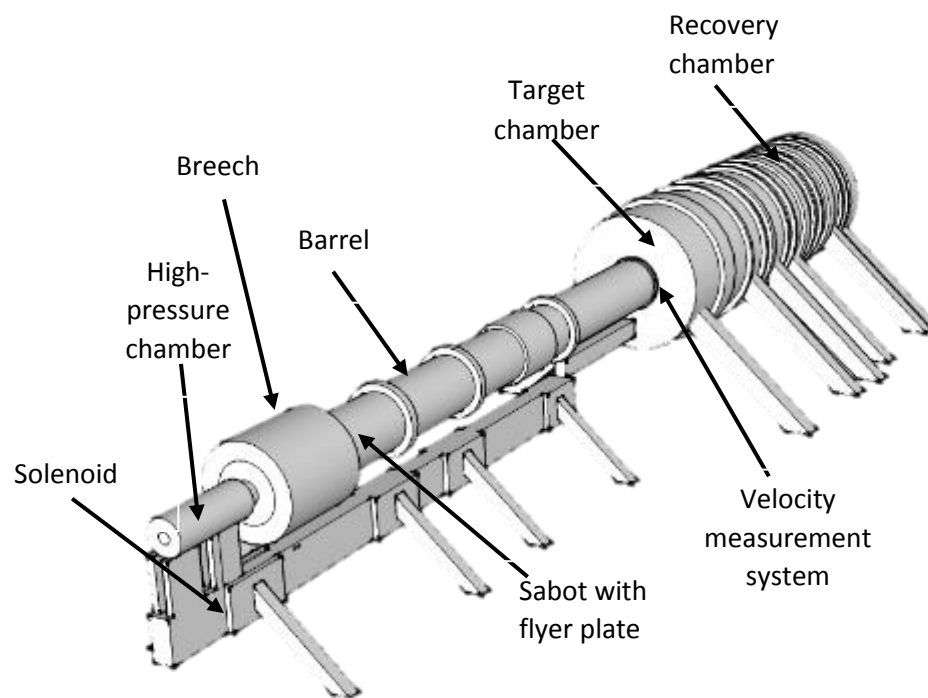


Figure 26: Configuration of the 50 mm bore 6 m long single-stage gas gun, used for the majority of shots carried out, both recovered and unrecovered. (Drawn using Google Sketch-Up®.)

The next stage is a fast release mechanism, which can either be a set of bursting diaphragms or a solenoid. This allows the gas to be released once the desired pressure has been obtained. The bursting diaphragms, that are housed within the breech of the gun, can be configured to burst at a specific pressure. The flyer plate attached to the sabot is placed within the breech, which then passes through the barrel, where it's velocity is measured using light gates. This system consists of a series of lasers split into two channels, which behave as light gates, allowing the velocity to be calculated from the distance between the gates, as the system measures

the light gate timings. Hence, velocity is equal to the distance divided by the time ($v=x/t$).

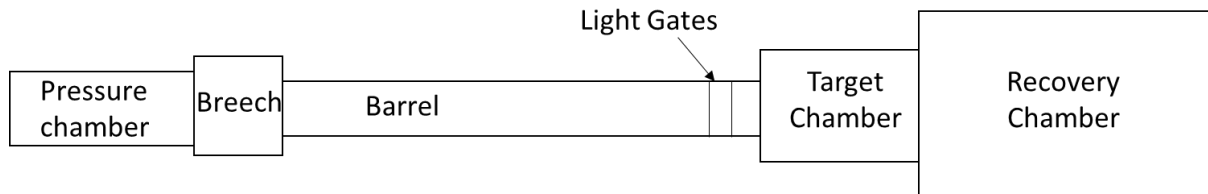


Figure 27: Side profile of single-stage gas gun (not to scale)

The flyer plate then passes through the barrel extension which the target is affixed to. Finally, once the target has been impacted, there is a recovery chamber, which is designed to allow soft capture of the target by impeding its trajectory post impact due to several meters of cotton bundles, which allow the target to be retrieved.

Via a process of inertial confinement, these gas-guns can be used to allow evolution of shock waves in loaded targets using plate-impact experiments. When a target is shock loaded one dimensionally, the lateral confinement of the target means that radial flow cannot occur in the target centre. Consequently, a one dimensional state of strain, though not stress, is established [2]. By monitoring the passage and evolution of this shock wave, it is possible to clarify different materials' responses to high strain rates.

3.2 Target Design and Modification

3.2.1 Plate-impact experiments

For this project, the selected method of shock wave production is via the plate impact method. This is where the flyer plate impacts the target in a planar manner, where the plate impact is confined to one direction, in this case, parallel to the target, where shock wave propagation in the target is primarily in one dimension, excluding any lateral reloading [2], [138]. However, the shock wave does spread outwards from the point of impact so the actual one-dimensional zone is limited to a central portion of the target. Shock wave propagation through a single plate target i.e. with no recovery is shown in an x-t diagram in Figure 28.

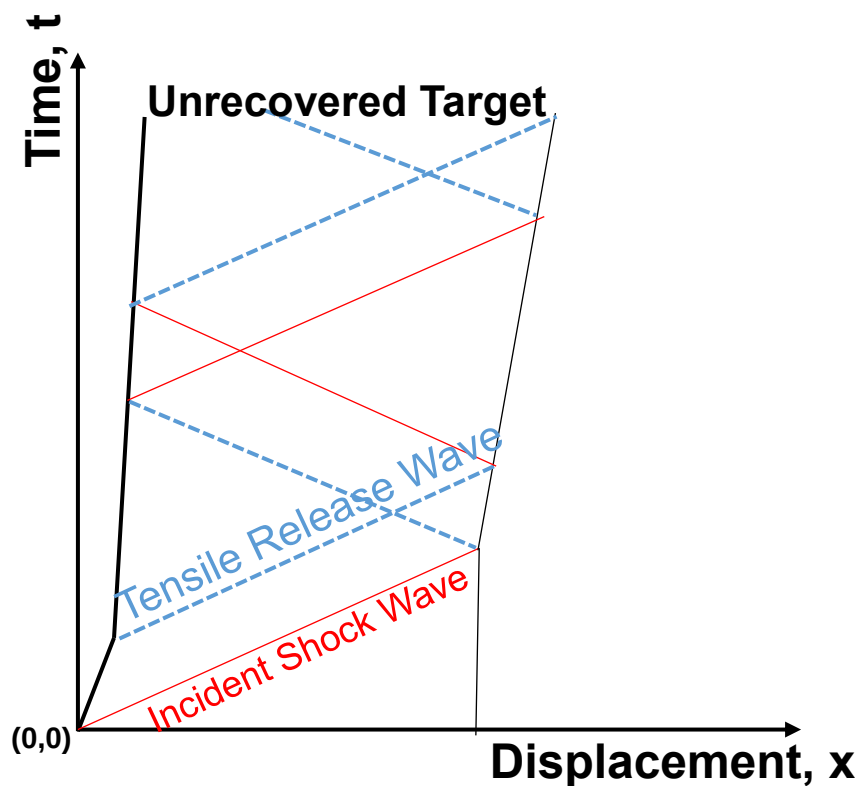


Figure 28: X-t shock diagram for an unrecovered sample

As the flyer plate impacts the target, a shock wave, beginning from the point of impact, travels through both the flyer plate and the target as a compressive wave; this is indicated in the diagram by the two solid lines travelling outwards from the point (0,0) [2]. When these compressive waves hit the boundary of the flyer plate and target respectively, they reflect back into the material. This will happen for the majority of the shock wave, as there is a free surface after the target.

As there is a free surface, the shock wave will reflect back into the target as a tensile release wave. Where these two waves overlap, spall can occur. Further to this, the appropriate target design can be made using multiple spall plates and latitudinal rings, to prevent spall from occurring in the specific part of a recovery target, to allow for full uniaxial recovery to occur [2], [41].

3.2.2 Shock Recovery Technique

Momentum trapping of shock waves to inhibit reflections into the target can be achieved by incorporating guard rings around the target being recovered and spall plates, reflections into the target are reduced, as the rings and spall plates separate because of momentum. This is shown in Figure 29, where in a) there is a single target plate with no recovery and b) a recovery target with one spall plate backing it and one concentric guard ring around the recovery target.

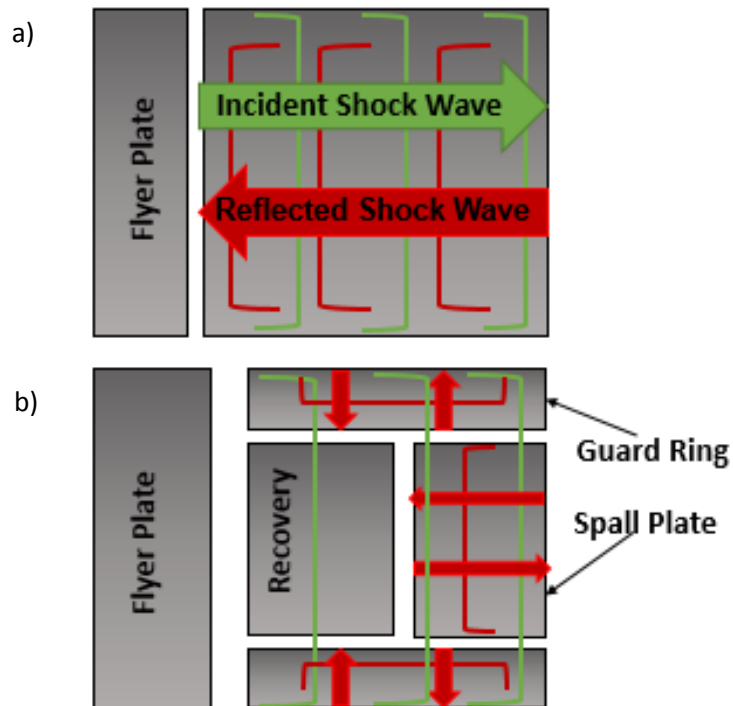


Figure 29: Simplistic diagram showing effects of guard rings and spall plates on recovery. a) Standard target b) Recovery using one concentric guard ring and one spall plate.

With the standard target in Figure 29a, the shock wave propagates through the target until it reaches a boundary, where it will reflect both laterally and longitudinally into the target reloading it repeatedly until the shock wave dissipates. In the recovered sample (Figure 29b), the shock wave will travel through the entire target, as the pieces will have an interference fit, where each joint is held together by friction, where it will also reflect at the boundaries and reflect back into the target. However, before this is able

to happen, the guard ring and spall plate separate from the recovery target mitigating the reflections that can reverberate back into it.

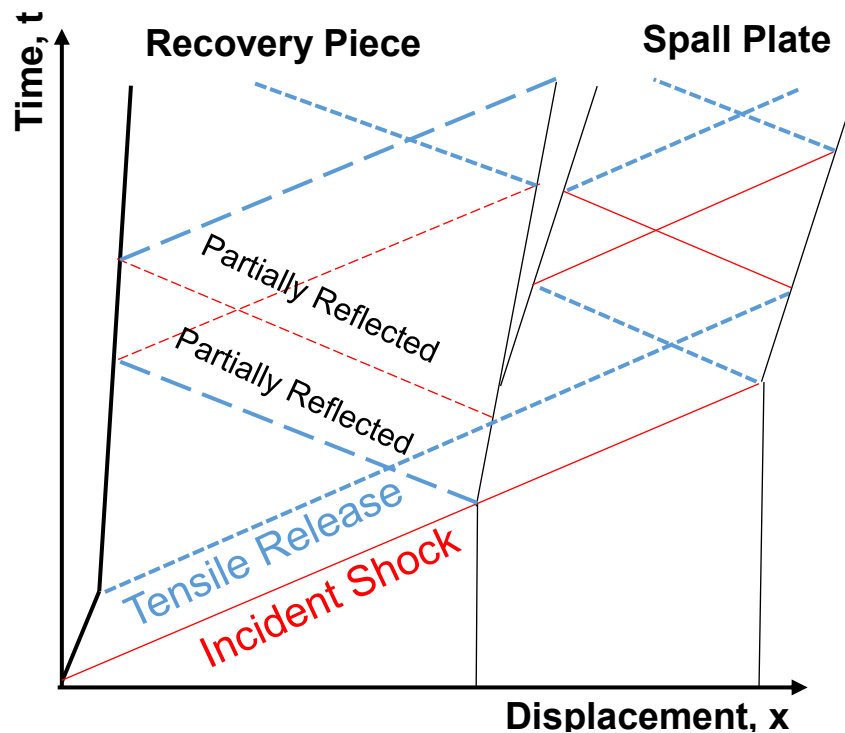


Figure 30: X-t diagram for a recovered sample with a spall plate to the rear of the target

Figure 30 is an x-t diagram showing a recovery target that has been simplified to one spall plate to the rear of the target, assuming an interference fit. Now, when the shock wave reaches the rear side of the recovery target, some of it will be transmitted to the spall plate and some will be reflected back into the material. This helps to reduce the amount of reflections within the target as the spall plate separates, preventing the shock wave from re-entering the target.

Using this information, experimentally, the standard target consisted of a 5 mm deep stainless steel 21-6-9 plate set into a PMMA surround using Loctite® Hysol 9461 epoxy adhesive to secure it. This is shown schematically in Figure 31a. The recovery target, Figure 31b, consisted of a 5 mm thick 21-6-9 sample with a 1 mm thick cover plate and 5 mm thick spall plate in front and four 1 mm spall plates on the rear. These were surrounded by three concentric rings, which had an interference fit inside one another of 70 mm, 35 mm and 23 mm outside diameter, to prevent 2D release waves from affecting the sample. There was a taper on the inner concentric ring which allowed for smooth release of the central portion of the target preventing impingement of the recovery sample, as suggested by Vignjevic *et al* [60], [61].

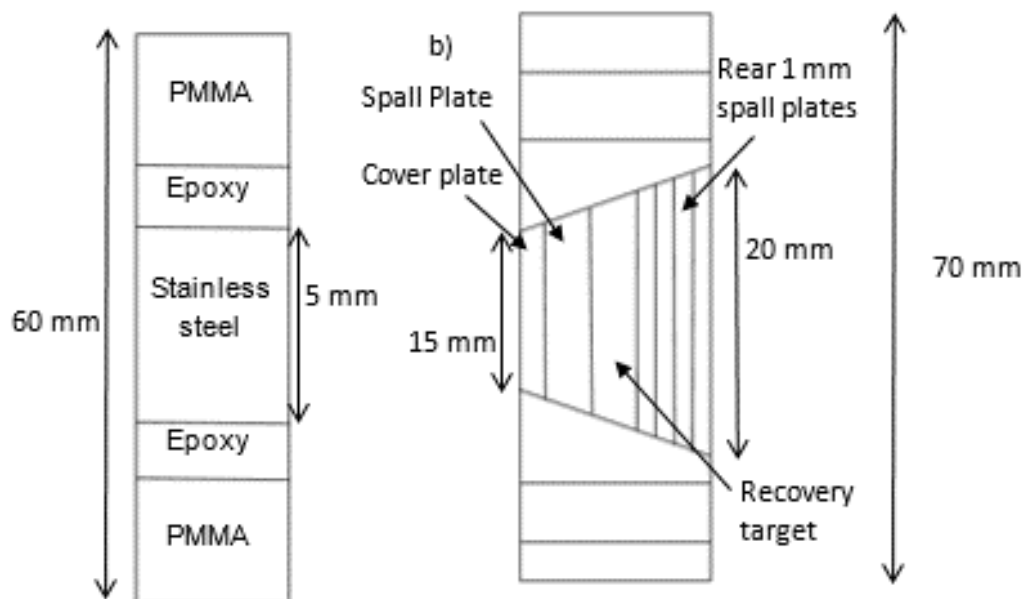


Figure 31: a) Standard target. b) Recovered target made entirely of stainless steel 21-6-9. (Figure not to scale). Impact occurs from the left hand side of each target.

As well as having a shock recovery target, there also needs to be a recovery assembly inside the target chamber. Figure 32 shows the recovery assembly. On the left side of the diagram is the barrel extension, which is also used to hold the target in place and the recovery assembly is to the right of this. The aims of the recovery assembly are to

reduce the velocity of the target and to dissipate its energy, so that it is gently decelerated to a halt.

The first part of the assembly is a stainless steel stripper plate, which is used to help separate the guard rings away from the recovery target as well as having the ability to absorb a large amount of energy due to the inverse taper on the rear side. Next within the assembly is a series of aluminium meshes, which help to absorb the impact, in a similar way to a spring. Finally, there is a bag of high absorbency pads in a sealable plastic bag filled with water to help quench the sample as it comes through. The entire assembly is contained within a large steel barrel, which is able to freely move through the target chamber of the gun once impacted. More detailed images of each component are shown in Figure 33.

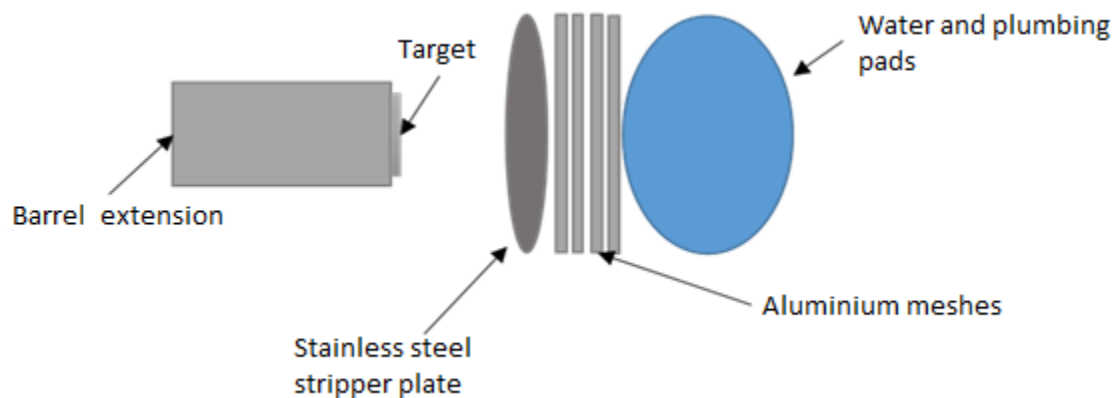


Figure 32: Diagram showing recovery method in the target chamber of the gas gun

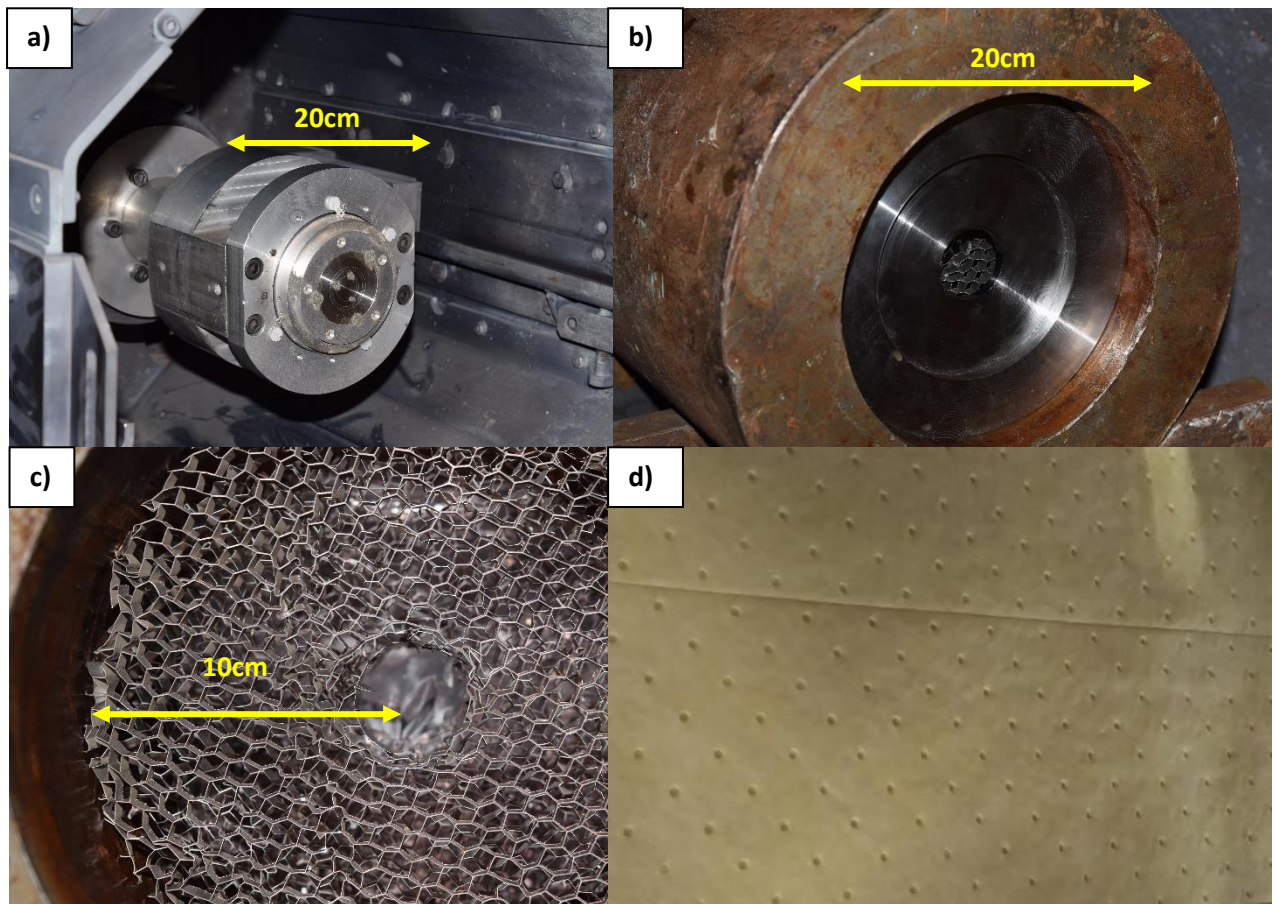


Figure 33: Recovery set up in gun: a) Barrel extension with recovery target mounted to the end. b) Steel barrel containing stripper plate c) Aluminium mesh discs set behind stripper plate d) Plumbing pads, which are wet placed within zip top bags to help quench/decelerate the recovery target.

There is an upper limit both theoretically and experimentally to recovering a sample. Experimentally, the recovery is limited by how much the target can be decelerated and the energy absorbed without reimpacting onto spall rings and plates. Also, the steel barrel which captures the recovery target weighs approximately 15 kg. Therefore, if the shock pressure were to be higher than 18 GPa, it risks damaging the recovery chamber inside of the gun.

As the shock velocity increases, the one-dimensional zone decreases and there is less time for the guard rings and spall plates to separate before the reflecting waves

re-enter the recovery piece. This is due to the higher wave speed and increased compression of the sample due to the increased impact pressure. Therefore, the target size would need to be larger to account for this but the bore size of gas guns is finite, therefore there is an upper limit at which shock recovery can occur.

Further to this, the flyer plate thickness has a distinct effect on the recovery of the target and the way that the spall plates behave due to the shock reflections. To achieve a full soft recovery, a flyer plate thickness of 2 mm to 3 mm is ideal, as the shock wave reflection indicates on the x-t diagram in Figure 34 that the tensile release waves overlap and would cause spall where the rear four flyer plates are. Though, because the flyer plates are present, the tensile releases instead cause them to separate, reducing reflections back into the recovery target, allowing for a predominantly 1D recovery.

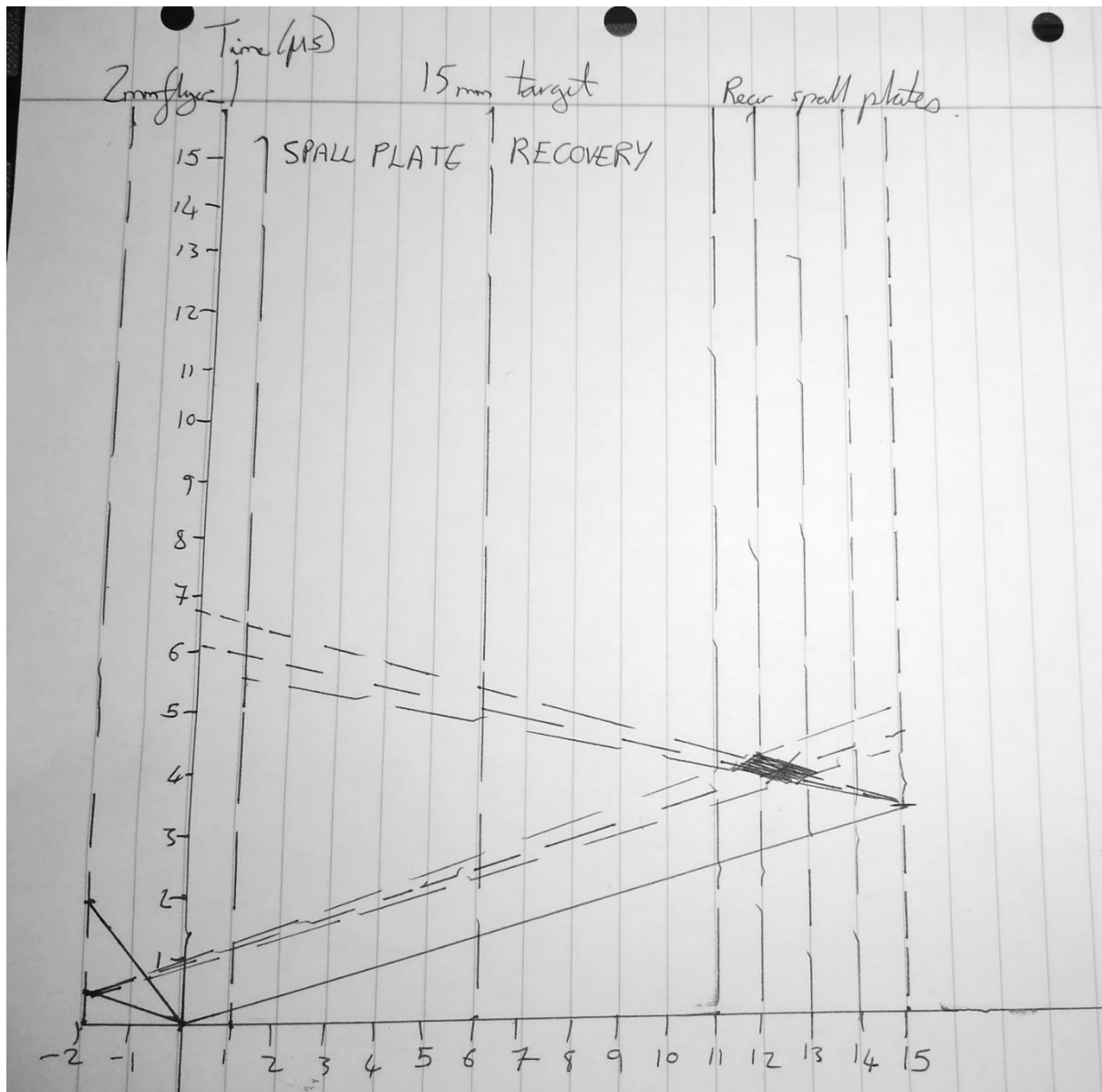


Figure 34: Simplified x-t diagram of 2 mm flyer plate impact with full recovery target

For the experiments in this project, excluding the flyer plate control example of 2 mm, a 10 mm flyer plate was used. The tensile release waves therefore overlap, as shown in Figure 35, where the front 5 mm spall plate is, creating a different loading history to that of the 2 mm flyer plate. Though it is not fully 1D, there are some 2D releases reverberating through the target, some of the releases are mitigated, creating a partial recovery.

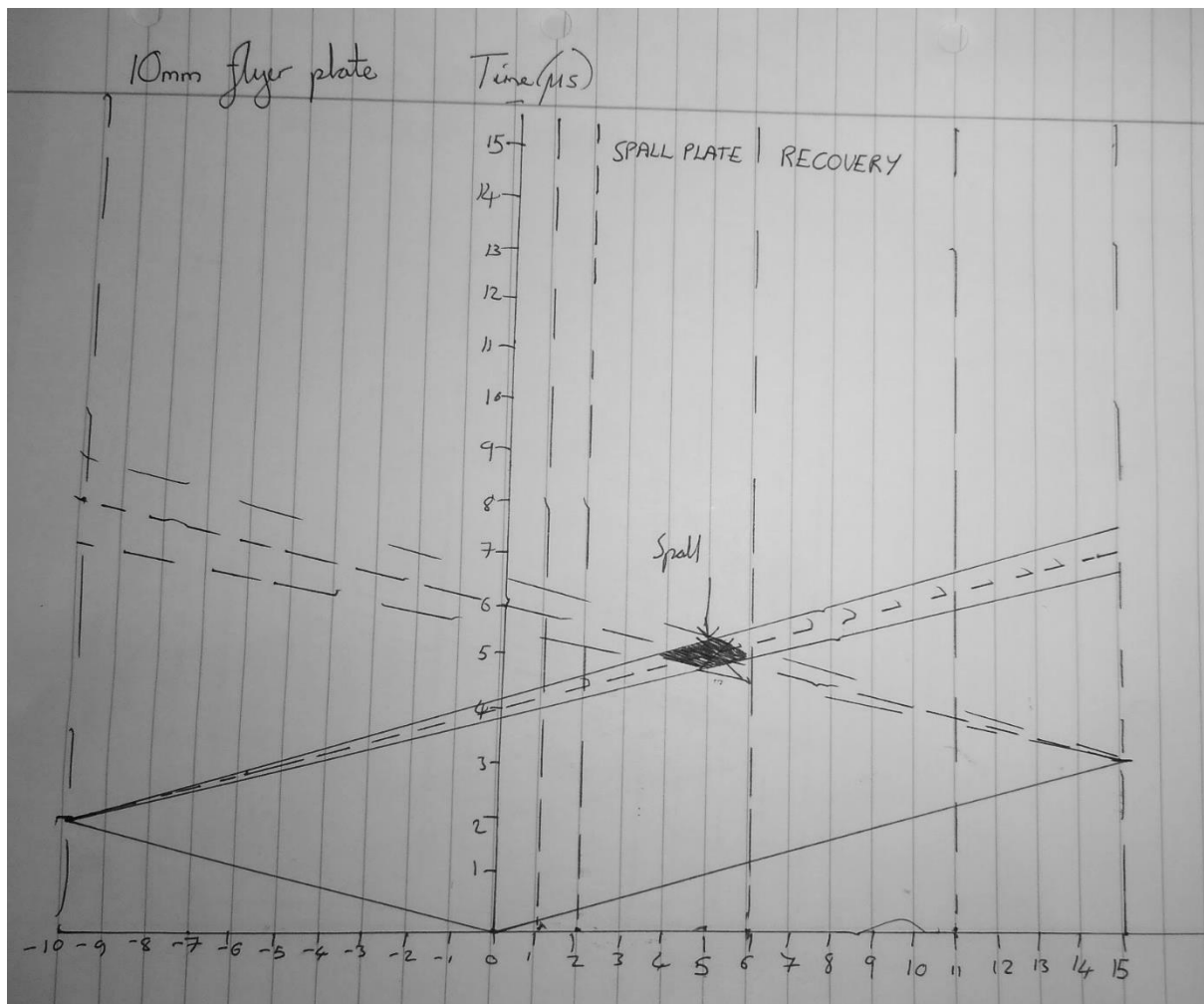


Figure 35: Simplified x-t diagram of 10 mm flyer plate impact with full recovery target

To further investigate how the rear four spall plates behave in this target, these were modified in thickness and in number from four 1 mm targets to two 2 mm targets, as well as one 4 mm target. This was to investigate how these affected the partial recovery. By having the 2 mm flyer plate comparison, analytical quantification in the effects on recovery, from a standard shot, to a partial recovery and finally a full recovery shot can be made. This reflects Stevens and Jones' research, where they investigated the diameter to thickness ratio of targets to see the effects on the tensile stress amplitude [165]. Further to this, Gray et al. found that a diameter to thickness

ratio greater than 7 to 1 appeared to reduce releases in the central 1D portion of the sample [165].

A series of 23 experiments were carried out on the stainless steel. This involved a range of recovered and non-recovered shots from ~6 GPa - 18 GPa. This range was selected as ~6 GPa can be reliably obtained from the bursting discs (2.0 mm- 1.0 mm) used in the gun and 18 GPa is the highest pressure that the gun can achieve using the recovery system inside the recovery chamber without causing any damage.

3.3 Analysis Techniques

3.3.1 Hardness

Hardness gives an indication of shock hardening due to mechanisms such as dislocation pile up and twinning [175]. Used in conjunction with Transmission Electron Microscopy (TEM) and yield strength from compression testing, hardness can give comparisons between recovered and unrecovered materials.

Indentation hardness testing was first widely used in the beginning of the 1900s by Johan August Brinell, who popularised the use of the Brinell test, which in the standard method uses a spherical indenter [176]. The Vickers hardness test was developed in 1921 by Robert L. Smith and George E. Sandland at Vickers Ltd as an alternative to the Brinell method [177]. The Vickers test has a larger hardness scale than that of the Brinell and can be used for all metals. The Vickers test also scales, as the calculation is irrespective of the indenter size, which is a diamond tipped indenter with an included angle of 136° [177]. Vickers testing can be used for microhardness testing, which is

hardness testing carried out with a force of less than 1 kg. This also mitigates strain hardening effects from the indenter itself. Hence it was decided that Vickers microhardness testing would be the most appropriate.

To enable hardness testing to begin each sample was sectioned in half (semi circle with radius 7.5 ± 0.5 mm). The full cross-section of the sample was then mounted in Bakelite using a mounting press, then ground down using a series of sandpapers from 240 grit to 600 grit. A 3 μm diamond polish was then used (Struers® DiaDuo-2, 3 μm) until a mirror finish was achieved.

A diamond tipped pyramid indenter is used to give a hardness value based on the load size and measurements between opposite corners of the pyramid indent. Within this project microhardness testing was selected as it can give an insight into variations across the sample, rather than just one overall value that macro hardness testing gives. All samples were tested at 300 g using an Indentec HWDM-7 microhardness tester. This is indicated in Figure 36.

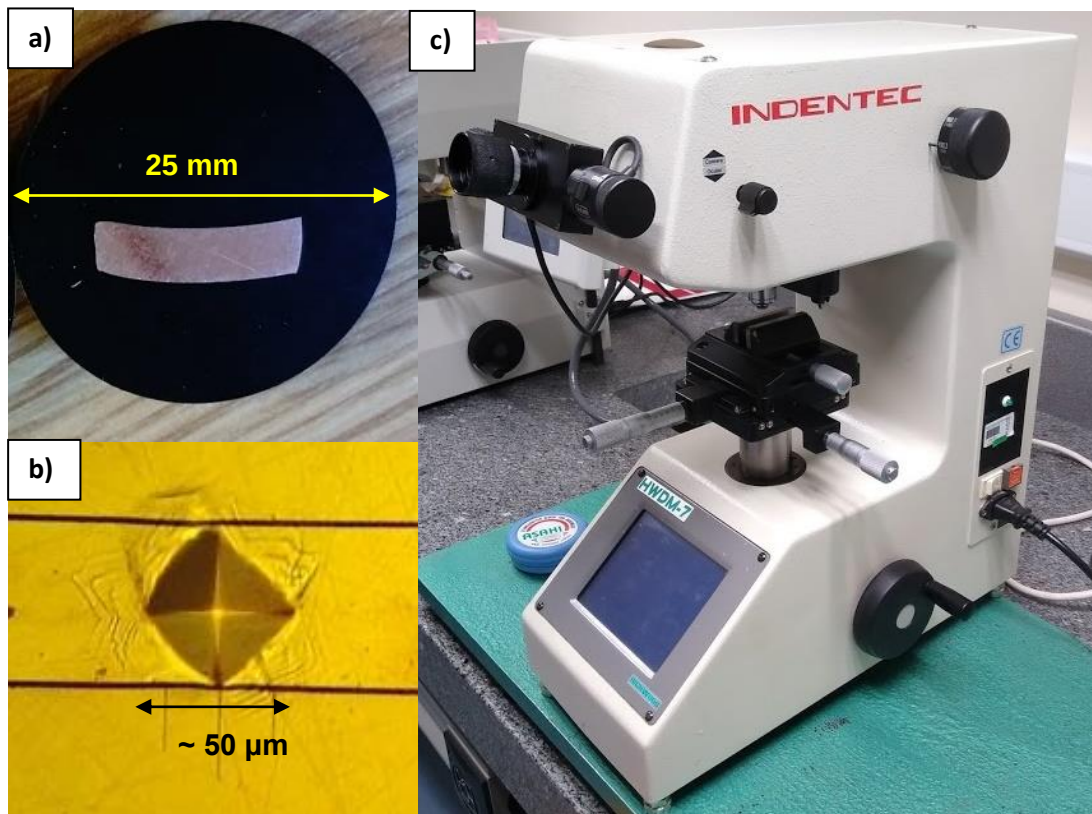


Figure 36: a) Stainless steel cross-section mounted in Bakelite. b) Image of pyramid shaped indent from microhardness tester on copper. c) Indentec HWDM-7 micro hardness tester.

3.3.2 Optical Microscopy

In 1590 three Dutch spectacle makers Hans Jansen, Zacharias Jansen and Han Lippershey invented the compound microscope [178]. It consisted of an eyepiece and objective lens. In 1663 Robert Hooke published a folio volume titled *Micrographia*, providing a wide range of microscopic views of familiar objects [179]. Van Leeuwenhoek improved microscopic resolution up to $\sim 0.7 \mu\text{m}$, which also helped to create the study of microbiology in 1674 [180]. These single lensed microscopes were used until the mid 19th century. The main drawback was chromatic aberration, which meant that different wavelengths of light did not come to the same focus [180]. This was corrected in 1733 where Chester Moor Hall, discovered that the combination of a

convex crown glass lens and a concave flint glass lens corrected chromatic aberration inside of telescopes [181].

The microscope used for this project was the Olympus BX53M, which is a compound light microscope. All light microscopes work in a similar manner. One is detailed in Figure 37.



Figure 37: Labelled compound light microscope (©OnFocus Laboratories)

The main features of the compound light microscope are an illuminator, which provides a light source at the base of the microscope to reflect off the object being imaged [182]. There is then a condenser, which is a two lens system, which collects and

concentrates the light, directing it to the Iris diaphragm, which regulates the amount of light entering the system [182]. The mechanical stage is used to hold the item being imaged, as well as fine and course adjustment to get the image in focus [182]. The nose piece allows the degree of magnification to be changed and finally, the ocular lens or eyepiece further magnifies the image and allows it to be seen [182].

The samples were prepared in the same way as the hardness testing, however, a finer polish was used on the sample, a 3 μm diamond polish. An Olympus BX53M microscope was then used to obtain images with the Olympus Stream image analysis software. Grain size and twinning density can be acquired from optical microscopy. The samples were all taken at the same magnification (20x) to allow for comparative analysis.

Twinning density provides information how many twins per grain that there are in each sample. This was achieved through optical microscopy using an OLYMPUS® BX53M microscope in conjunction with the OLYMPUS® stream software and by counting individual grains and then individual twins by hand, hence providing a result in twins per grain as a comparative proportion of the sample.

3.3.3 Transmission Electron Microscope (TEM)

Transmission electron microscopy (TEM) was first developed in 1928 at the Technical University of Berlin by Ruska *et al.* and imaging was achieved in 1931, based on the principles of the cathode ray [183]. In the same year Reinhold Rudenberg, the

scientific director of Siemens, patented the electrostatic lens microscope [183]. Since then, refinements have been made but the principles remain the same.

TEM has similarities to light microscopy. However, instead of utilising photons to produce an image, electrons are used instead. They have a shorter wavelength than photons, which allows for a greater resolution. There are three main aspects to a TEM. A simplistic diagram showing the main features of a TEM is shown in Figure 38. Firstly, there is an electron gun, which produced an electron beam by accelerating the electrons toward an anode. Secondly, a series of lenses, including the objective lens are used to focus the electrons toward the movable specimen stage. Finally, there is an image recording system, which can either be a fluorescent screen or charged coupled device, linked to a computer (CCD) [184].

TEM has similarities to Scanning Electron Microscope (SEM) but it has significantly higher resolution of 0.2 nm compared to SEM which has a maximum resolution of 10 nm [185]. TEM can provide a plethora of information including phase information, dislocation type and density, twinning and stacking faults.

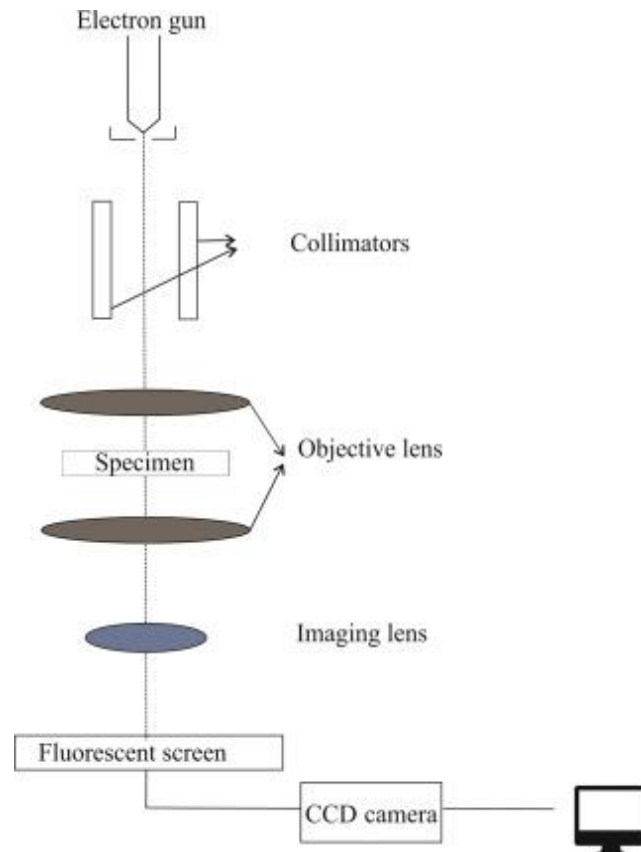


Figure 38: Diagram of a Transmission Electron Microscope [225]

For the stainless steel 21-6-9 targets the TEM allows for the austenitic structure to be observed with any changes in dislocation density or twinning, which can be counted by hand or using imaging software, such as Image J [186].

In order for imaging to occur the sample must be thin with the maximum size being 250-500 nm. Otherwise electrons are unable to transmit through the sample, making image formation impossible.

Sample preparation begins by sectioning, for the samples in this project, a cross section was taken from the centre of the sample, this was achieved using abrasive wet cutting with a silicon carbide wheel, which cools the blade down to prevent the microstructure of the sample being changed due to heat evolution. A bar was initially cut using a silicon carbide cutting disc, which is suitable for soft to medium metals, from the centre of each recovery sample to provide an insight into the cross-section of the sample.

The next stage, grinds the sample down from 500 μm to 300 μm thickness using a grinding wheel with 600 grit paper [187]. The next step was to punch out the discs used within the TEM using a punch measuring 3.05 mm. Approximately five discs were obtained for each section. From there the specimens were further polished down using a 1200 grit piece of sandpaper to 100 μm thick. The final stage involved twin jet electropolishing the samples using 80% phosphoric acid and 20% deionised water at 13.4°C with 2 V. An alarm indicates when the perforation of the sample has completed and therefore the polishing of the sample is complete. An image of this perforation is shown in Figure 39.

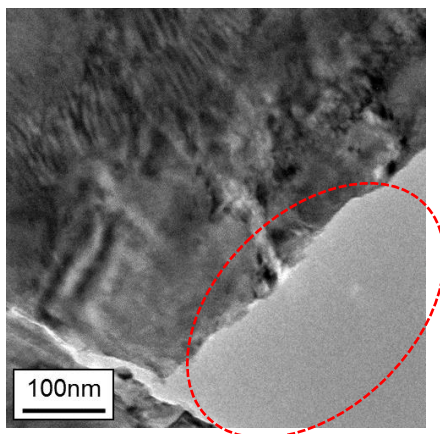


Figure 39: TEM image showing central perforation of the sample. The red circle indicates the edge of the perforation.

Once the sample had been polished, it was washed immediately in ethanol then methanol and dried in between sheets of filter paper. The sample should be shiny and is tested in the TEM to confirm that appropriate imaging can be obtained. The images obtained were from the central cross-section of the sample, indicated in Figure 40.

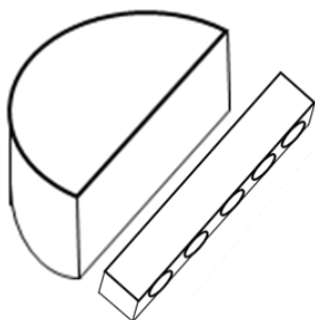


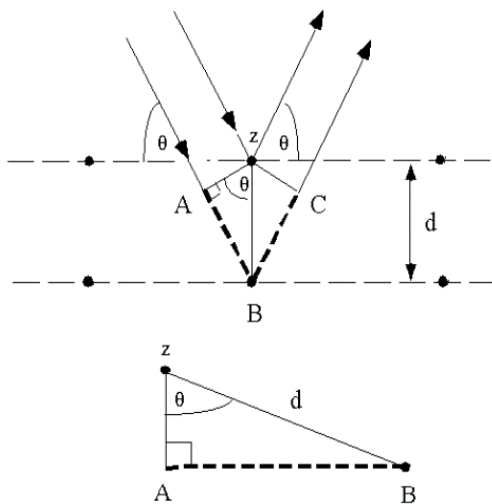
Figure 40: Sample taken from centre of recovery target cross-sectionally as shown. 5 samples were taken from each cross-section, which measured 3 mm diameter compared to the ~5 mm height of the overall sample.

3.3.4 X-ray Diffraction (XRD)

X-ray diffraction, usually shortened to XRD is an analysis technique, which uses diffracted photons produced by an X-ray beam, which then undergo constructive interference, to obtain crystallographic information about a material. Bragg's Law is the underpinning theory behind X-ray diffraction relating the wavelength of the X-ray with its diffraction angle within the crystallographic lattice via its spacing. Bragg's Law is given in Equation 8 [188].

Bragg's Law derivation:

$$n\lambda = AB + BC$$



where AB and $BC = d\sin\theta$

therefore $n\lambda = d\sin\theta + d\sin\theta$

leading to:

$$n\lambda = 2d\sin\theta$$

However, as n is the order of diffraction and is taken to equal 1, Bragg's Law is:

$$\lambda = 2d\sin\theta$$

Equation 9

λ = Wavelength of X-ray (m)

d = Interplanar distance of Miller Planes (m)

θ = Angle of diffraction ($^{\circ}$)

The X-ray beam interacts with the atoms within the crystal, causing the X-ray to diffract at discrete angles [65]. A detector then collects the intensity of radiation diffracting back from each of these angles (2θ). From the information gathered the average spacing between atoms can be obtained as well as the crystallographic orientation and measurement of the size, shape and stress of crystalline regions. The angle where X-rays are detected provides information on the element, spacing and orientation allowing a match of the material to be found through computer databases, such as TOPAS [65].

Each peak at a certain angle represents a certain orientation of an elemental crystal lattice. As each type of lattice has a different spacing, different elements and base structures can be identified, such as the crystallographic orientation and strains within the sample. Problems can arise where different crystal lattices have peaks at similar angles but software can be used to try and match the peaks, and/or resolve intensities to try to identify the correct compound.

The narrower the peak, the lower the dislocation density. As the samples, excluding the control sample, have been shocked the width of the peak is wider and can also be used to determine the dislocation density using the Rietveld method, which was not

used in this project but would be a good opportunity for future work [189]. The XRD spectra baseline also increases in intensity with increasing angle, this is due to the XRD being used on a metal, causing fluorescence, and can be corrected for using the TOPAS software [190].

The XRD was carried out using a CuK α source of 8.04 keV. Ideally Co would have been used with a steel sample as Fe fluoresces with a Cu source, causing the background to slope due to the production of polychromatic as opposed to monochromatic radiation. However, this wasn't available and therefore was not able to be utilised.

3.3.5 Compression Testing

Compression testing after impact can provide an important analysis into the residual strength of a material by comparing the impacted sample to a non-impacted compression sample [191]. Quasistatic compression testing was carried out by Lucideon Ltd at a rate of 0.01524 cm min⁻¹. Typical quasistatic tests are carried out at around strain rates of 0.01 s⁻¹ [192].

Two compression samples were extracted from each shocked sample of size 2 mm by 5 mm, using wire Electrical Discharge Machining (EDM). This technique was used as tolerances of ± 0.005 mm could be achieved, as well as a no contact, no force process, therefore not adding in stress to the samples as traditional machining would. One sample was taken as close to the centre of the sample as possible indicated in

Figure 41 'A', as some samples had already been sectioned for other analysis. Sample A is within the central zone of the sample, whereas Sample B is closer to the outside of the sample so some differences may be observed dependent on the success of the recovery, excluding that of the control sample. These differences in standard versus partially recovered samples can also be shown in the hardness testing.

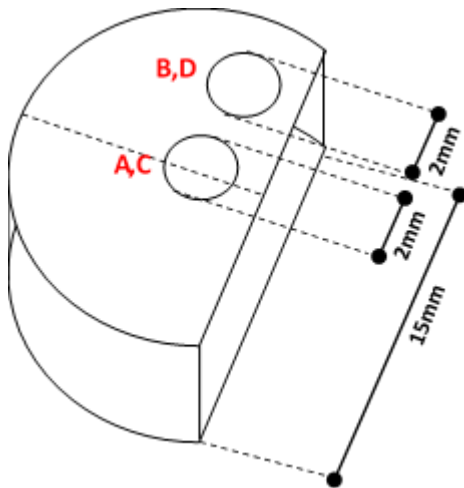


Figure 41: Schematic showing the location where the compression samples were taken from. Samples were 2 mm diameter and approximately 3 mm deep.

The tests were carried out in a universal servo hydraulic test frame. They were compressed between two carbide platens at a rate of $0.001524 \text{ cm s}^{-1}$. The lower platen was articulated to allow for even loading of the specimen to account for any error in the parallelism. The displacement of the platens was measured using three extensometers and averaged to calculate the strain.

3.4 Shock diagnostics of Material Properties

3.4.1 Temperature Measurements

When a shock front travels through a material, the material is compressed and there is an associated temperature rise [2]. This is due to the energy created from the material deforming inelastically and the increase in entropy as shown in Equation 9:

$$PV = nRT \qquad \text{Equation 10}$$

P = Pressure (Pa)

V = Volume (m³)

n = Number of moles

R = The gas constant (J mol⁻¹ K⁻¹)

T = Temperature (K)

There are two ways to measure the temperature evolved during a shock event. One of them is by observing the radiation emitted and the other is by embedding thermistors (thermocouples) which can be used to record temperature [193]. Especially when using a recovery technique, it is difficult to get an understanding of the temperature within the target sample that is being recovered because if a new component is introduced to the recovery target, which is not impedance matched then shock reflection becomes an issue. Temperature gauges have a certain time lag and techniques such as optical pyrometry and near infrared radiation would not be able to measure the peak temperature inside the target, only on the surface. Therefore, for the scope of this project, estimated temperatures were obtained from computer modelling data on ANSYS® Autodyn [64]. Further to this, no temperature induced traces were detected in the post experimental analysis of the metal.

3.4.2 Sound Speed Measurements

The sound speed is an important factor when determining shock properties of a material. The bulk sound speed is linked to the EoS of a metal, as it is the approximate value of the sound speed of the material under zero pressure (see *Equation 4*). The bulk sound speed can be calculated by measuring both the longitudinal and shear wave speed within the material.

The Panametrics 500 PR as in Figure 42a was used as a preamplifier for the signal to then be sent to a TDS 5104 oscilloscope as shown in Figure 42a. For the longitudinal sound speed measurements, a V102 1 MHz transducer was used and for the shear sound speed a V152 1 MHz transducer was used, which are shown in Figure 42b. Treacle was used as a conductive medium to enable a tight bond between the metal and transducer, reducing any air gaps and making the trace clearer on the oscilloscope. The time interval was then measured, by measuring the time difference between each 'packet' on the trace.



Figure 42: a) Preamplifier and b) transducer images (not to scale)

3.4.3 Het-V Measurements

Laser interferometry was first developed in the 1960s by Barker and Hollenbach, who described the first laser interferometer [2]. VISAR (Velocity Interferometer System for Any Reflector) is a velocity measurement system based on laser interferometry, developed in the 1970s by Barker and Hollenbach for use within shock research [194]. Since 2003 PDV (photon doppler velocimetry) also known as Het-V (heterodyne

velocimetry) has been used in shock physics research also [49]. Het-V estimates the velocity of a reflector that is moving by observing the Doppler shift of the light scattered from its surface [195]. The light source used is an infrared laser.

As the surface of the target moves towards the laser, the reflected light has a different frequency to the incident light. This difference is given in Equation 10.

$$f'/f = (c + 2u_p)/c \quad \text{Equation 11}$$

f' = Reflected frequency (Hz)

f = Initial frequency (Hz)

c = Speed of light (ms^{-1})

u_p = Particle velocity (ms^{-1})

In order to observe the shift in frequency, the reflected light is split and one of the split beams is given a known delay. These two beams are then recombined creating an interference pattern, which can then be used to obtain particle velocity [49], [196].

The surface finish of the target affects how much light is collected and therefore how accurate the measurement is, ideally in angled Het-V a diffuse surface is required to effectively fan the reflected light so that it can return to the detector [195], [197], [198]. In a study carried out by Zellner *et al.* seven different surface finishes: fly-cut, painted, sand blasted, sanded (240 grit), saw cut, surface ground and waterjet cut were compared against a control sample to look into the reflected power measurement compared to the incident angle of the laser [195]. They determined that when a surface had more texture i.e. sandblasted, painted or as is (usually machining marks are present) there was a greater retention of reflected power, which is useful when trying to maximize reflected signal measurements for Het-V at non-incident angles [195].

It was necessary to introduce a 15° angle of incidence to the recovery target, so that when shocked, the recovery pieces could separate without being impacted by the structure holding the Het-V fibre, this is discussed in more detail in Results I: Copper. The machining marks on the stainless steel targets were used to fan out the reflected light by turning them perpendicular to the incident light. The Het-V set-up is shown in Figure 43.

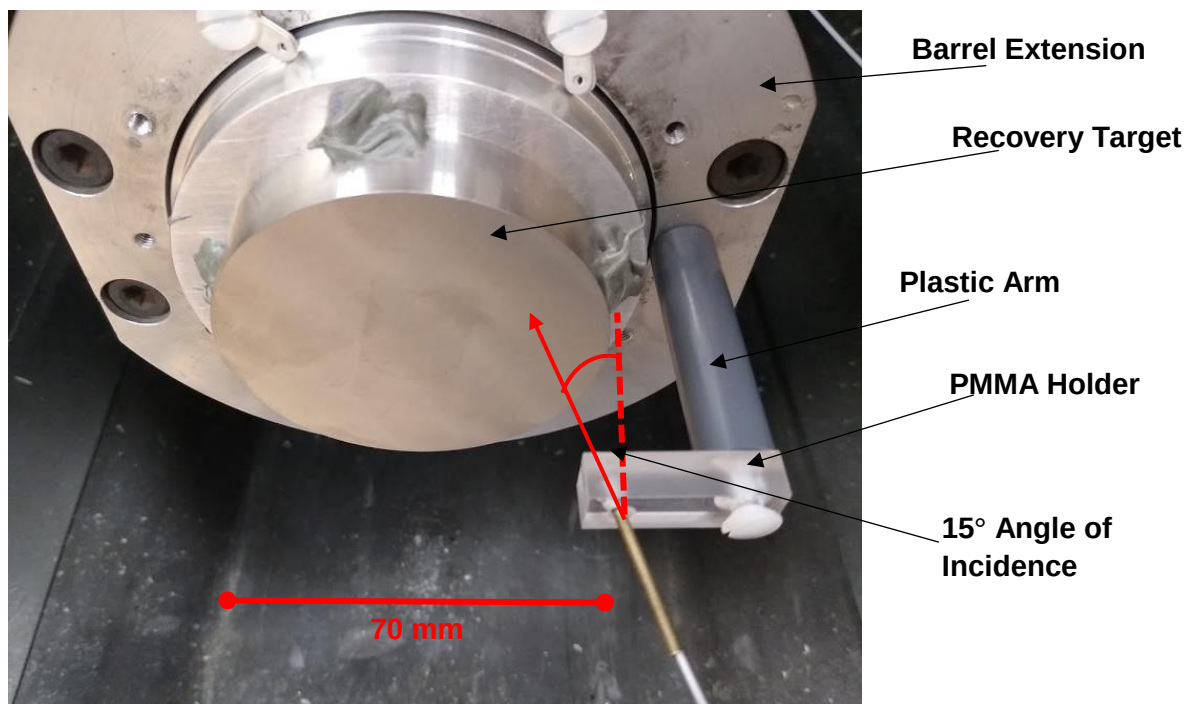


Figure 43: Recovery target set up with Het-V fibre set at a 15 degree angle in PMMA

3.4.4 Gauges

Manganin gauges work by measuring the applied shock pressure using their electrical resistance and thermal resistance [199]. They are most valued as a high-pressure sensor as their electrical resistance is a linear function of applied pressure, whilst their thermal resistance allows them to measure the shock waves without being affected by any temperature increases [199]. The gauges can then be used to

plot the shock velocity versus the particle velocity within the sample using the impedance matching technique (see Section 4.1) [2], [133].

3.5 Computer Modelling

Firstly, the verification of the project is needed, this will be confirmed by carrying out single plate impacts, then adding spall plates and rings around the target until the full recovery target is reached. Experimental work will be carried out to verify these models before developing the target further and creating a new full recovery target.

Once the full recovery target has been modelled, the number and thickness of spall plates behind the recovery target will be varied to provide an insight into the best recovery design.

Due to the effects of shock temperature on the structural evolution of the metal, it is important to be able to get an estimate of the temperature through both modelling and experimentally using gauges. Problems with gauges arise when the detection time is significantly slower than the time of the peak shock temperature, therefore only an estimate can be obtained. Taylor *et al.* carried out research into thin film metal thermistors with microsecond time responses as the time resolution is limited by the conduction of heat from the sample to the gauge [193]. Research carried out by Rosenberg and Partom found contradictory results to that of Bloomquist and Sheffield looking at the temperature rise in 2 GPa shocked PMMA [200], [201]. There are also issues affecting temperature estimation within modelling, as the model is only as accurate as the data provided for it.

This can be achieved by implementing gauges instead of spall plates to see what is occurring within the target itself, as well as using the Het-V diagnostic to measure the particle velocity within a target, which can then be directly compared across to the computer models used.

Thus far, modelling has been carried out with a single plate impact, a simple recovery involving one spall plate and one guard ring, a more complex recovery including two spall plates and two rings. From there the current recovery target has been modelled and the thickness of the spall plates have begun to be changed as due to the distances involved, longitudinal release can cause more changes to the microstructure with spall etc than that of latitudinal release where the diameter of the ring is usually significantly higher than that of the recovery target thickness.

The understanding behind what goes on with plastic strain, shock and particle velocity as well as pressure inside of a target during a recovery experiment can start to verify the recovery technique. Initially a copper plate was modelled 5.0 mm thick with a 2.5 mm copper flyer plate. Impacts were carried out at three different pressures: 7 GPa, 10 GPa and 14 GPa, which had also been carried out experimentally to be able to verify theoretical results.

Mesh size is an important factor to consider when carrying out computer modelling as that can affect the results of the model if the mesh size is too small or too large. In order to combat this, a parametric study was carried out where models were run with a mesh size of 0.10, 0.05, 0.03. They were then compared using the results from the

gauge data. This test was carried out on a simplistic model, which had one spall plate and one ring surrounding recovery.

A balance must be struck between getting a fine enough mesh size to accurately display the data at each individual point but not so fine that the model has an excessively long run time. Further to this, during dynamic deformation, as the mesh must be replaced when large amounts of deformation occur during impact so as to avoid issues such as negative mass, if the mesh size is too fine, this may cause inaccuracy problems. The computer specifications used in this project are Intel Core i7-3770 at 3.4 GHz with 16 GB of RAM with the 64 bit version of Windows 7.

4 RESULTS I: COPPER

4.1 Hugoniot of Copper

Copper is a well-characterised material mechanically and under dynamic loading, with a well-defined Hugoniot [54], [126]–[129]. The Hugoniot of copper is shown in Figure 44 with data from LASL [17]. As previously discussed, the Hugoniot provides information on the shock response of a material, as it represents its EoS under shock conditions.

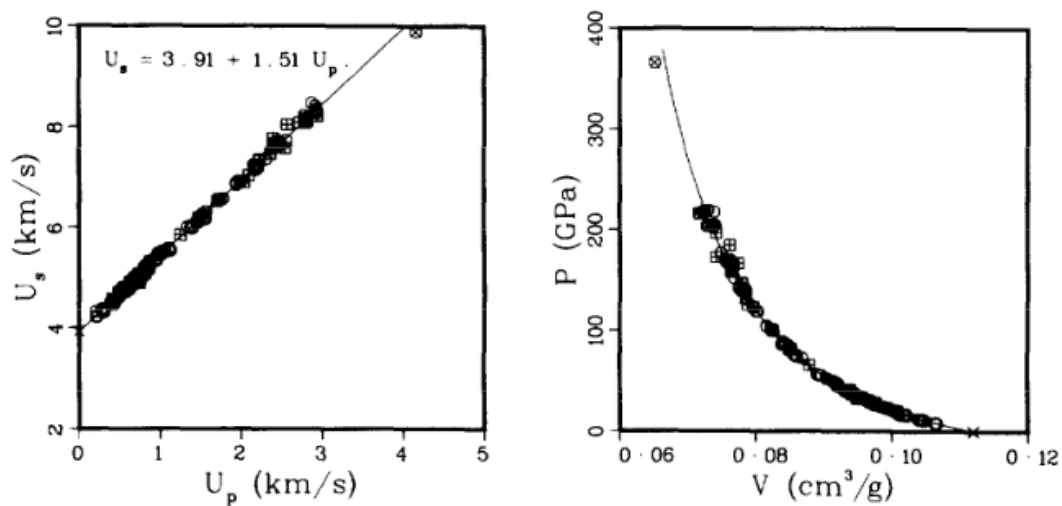


Figure 44: Hugoniot of polycrystalline OFHC copper in both the U_s - U_p plane and the P - V plane [17]

Initially, three experimental shots to determine the Hugoniot copper were completed, for experimental familiarisation and confirmation that the shots carried out lay upon the already well characterised Hugoniot from LASL shock data [17].

The experimental work on OFHC copper included shots incorporating longitudinal manganin gauges. Manganin gauges work by measuring the applied shock pressure using their electrical resistance and thermal resistance [199]. They are most used as a high-pressure sensor as their electrical resistance is a linear function of applied pressure, whilst their thermal resistance allows them to measure the shock waves without being affected by any temperature increases [46], [86], [130], [202]. The gauges can then be used to plot the shock velocity versus the particle velocity within the sample using the impedance matching technique. Figure 45 shows an image of a manganin gauge.

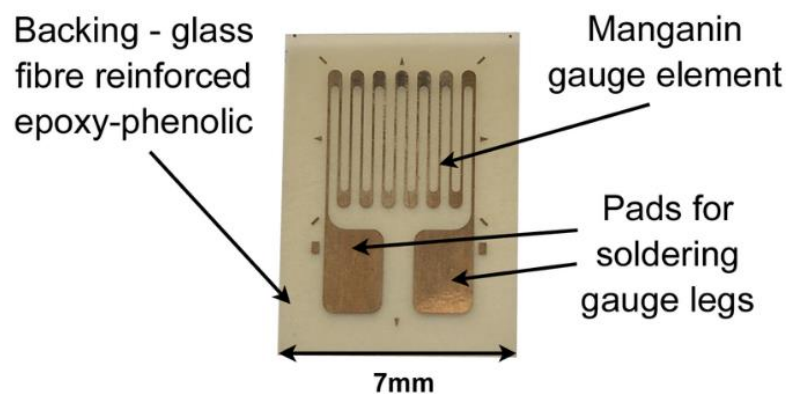


Figure 45: Labelled image of a manganin gauge

Two of these validation shots are shown in Figure 46 and corroborate pre-existing Los Alamos Shock Laboratory (LASL) data on OFHC copper [17]. The resulting straight-line graph of the shock velocity versus the particle velocity is the Hugoniot of copper, which is its EoS.

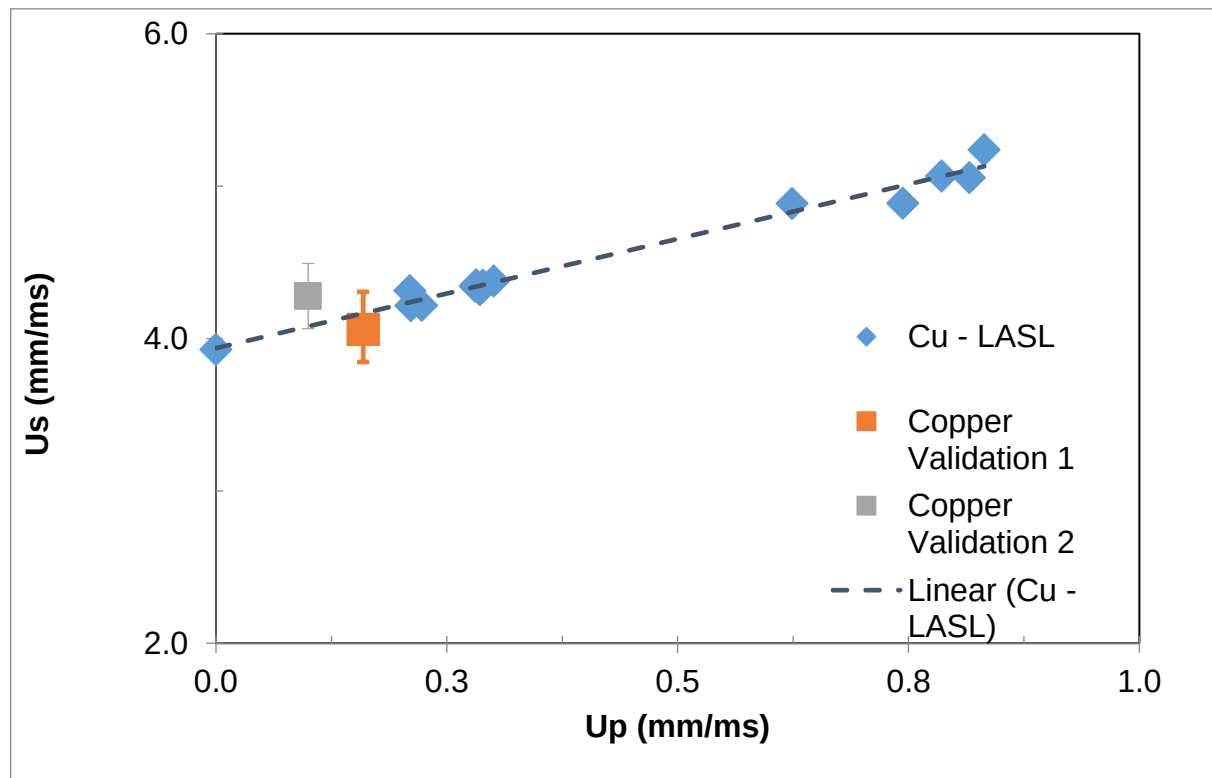


Figure 46: Experimental validation of the Hugoniot of Copper [17]. Los Alamos catalogued shock data from a wide range of materials at a different range of velocities to obtain the EoS of those materials. This data can be used as a comparison for experiments conducted by other laboratories.

4.2 Spall Targets

Previous research has focussed upon pulse duration and strain rate effects on incipient spall fracture in copper [14]. Johnson *et al.* looked at real time VISAR measurements of the spall fracture of copper [14]. Copper targets were 4 mm thick and the flyers were chosen to produce pulse durations of between 0.3 – 1.2 μ s with

release strain rates ranging from 0.2 to $0.8 \mu\text{s}^{-1}$ at the spall plane using impactors that were 1 mm , 2 mm , 2.25 mm , and 3 mm with an impact stress of 6 GPa [14]. Overall, they found that the pulse duration and tensile strain rate had the principal effects on void formation within the sample [14]. Void formation occurs usually when there is either fracture or interface decohesion (separating of interfaces) of a second phase particle [203], [204]. Void growth is driven by plastic flow and depends upon the stress state and microstructure of the material [203].

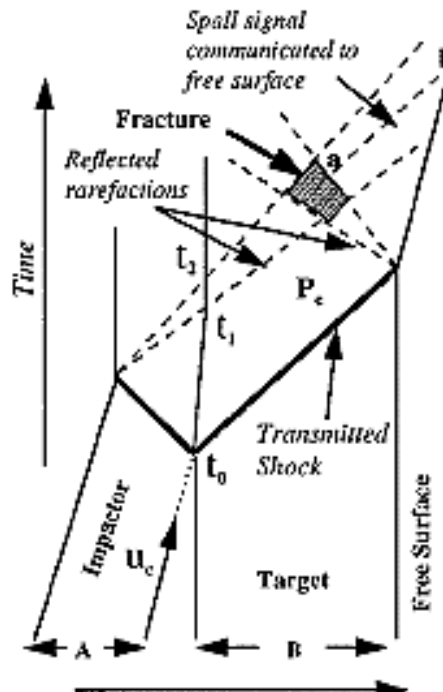


Figure 47: x-t diagram of Johnson et al.'s experiment [14]

Figure 47 indicates that when the target is approximately twice the thickness of the impactor, the rarefaction waves meet in the middle of the target causing spallation at the centre of the target [14]. The material to the left of the spall plane or fracture area has been brought to rest and the material to the right is moving with a particle velocity of $U_c/2$ (where U_c is the impact speed) [14]. Once the material has reached an upper

tensile limit, defects no longer control the rate of fracture and the material pulls apart at a comparatively instantaneous rate [14]. Conversely, Stevens and Jones confirmed that where the flyer plate is thicker than the target, the tensile releases from the flyer plate will not reach the target before separation occurs [165].

Experimentally, spall targets were manufactured, whereby a central disc was surrounded by two guard rings. These guard rings help to mitigate lateral release waves, allowing the tensile releases to only occur within the longitudinal plane. This was similar in nature to the experiments carried out by Escobedo *et al.* whereby a 4 mm thick copper spall target with three lateral momentum trapping guard rings surrounding a 15 or 20 mm thick disc was impacted by a 1.4 mm copper flyer or a 2.0 mm quartz flyer [205]. Photon Doppler velocimetry was used to calculate the spall strength of the copper targets, which was between 1.21 and 1.70 GPa with the resultant damage volume having values between 0.27 to 3.56. They concluded that the predicted spall damage relied not only on the free surface velocity, which is the velocity occurring at the target boundaries but also the measurement of the decompression and recompression rates. Henceforth the damage fields were caused by the energy supplied from the shock impulse and time spent dissipating.

Figure 48 shows the copper targets that were used in this project. The copper target was 5 mm thick with a copper flyer plate thickness of 2 mm to allow for spall in the centre of the sample. This resultant spallation was found to be ductile in nature shown in Figure 49.

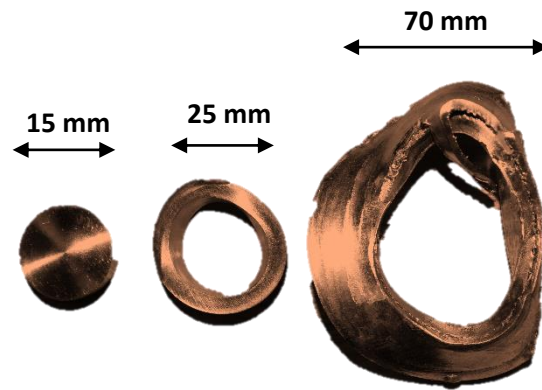


Figure 48: Copper target with central portion and two guard rings measurements indicated are those prior to impact

In Figure 49 microvoid coalescence of ductile fracture cusps can be seen on the spall face. Ductile fracture is expected on FCC metal crystals at ambient temperature.

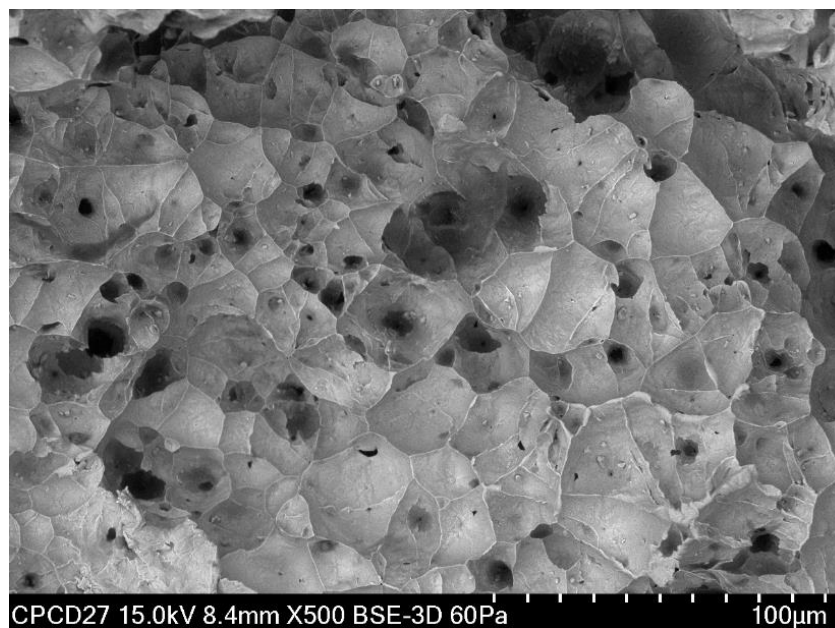


Figure 49: Spall face from 7 GPa lateral recovery spall target

4.3 Copper Recovery

Copper was also used as the primary material to develop the recovery system inside the gas gun and the recovery target itself.

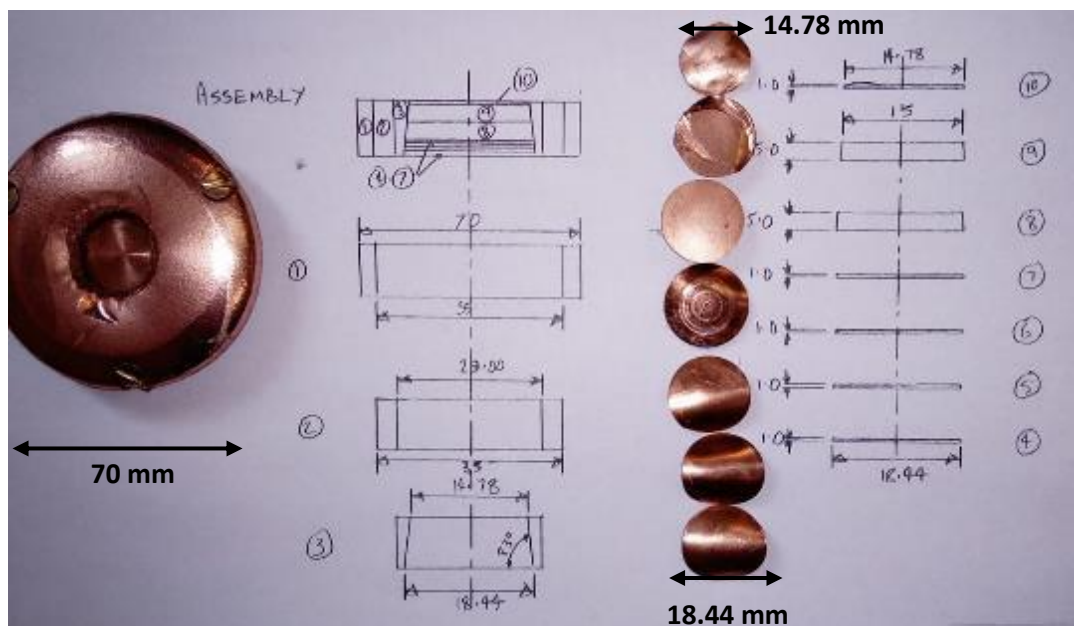


Figure 50: A full recovery target post shock. Spall plates have taken the majority of the reloading, whereas the recovery sample (8) remains unaffected

The first experimental series demonstrated that the current method was not optimal due to the target showing reimpact (hence reshocking) because of the recovery method that was originally in place. This is shown in Figure 51. The technique was modified and improved by adding absorption pads to the water (quench) bag. This aided the experimental technique by absorbing energy and encouraging separation of the spall plates and recovery piece.



Figure 51: Spall plates having reimpacted the recovery target

4.3.1 Fit of Plates

The fit of plates is vital in recovery targets. If there is not an interference fit between plates and rings, spallation can occur within the target. Gray investigated this phenomenon in shock loaded copper [54]. All targets were 10 mm thick with a pulse duration of 1 μ s. The residual strain ϵ_{res} ranged from 1.5% up to 26% [54]. The changes in residual strain resulted in a change in deformation characteristics. OFHC copper with $\epsilon_{res} = 1.5\%$ showed dislocation cell characteristics, whereas the samples with an $\epsilon_{res} = 26\%$ showed primarily deformation twins, which have been disputed to not appear until a pressure of 15 GPa is achieved [54]. The reload yield strength also increases by 40% [54]. Strains of less than 2% occurred when the tolerances of the target were slip fit with a 0.0254 mm clearance [162]. Whereas plastic strains of 26% were obtained when there was no cover plate to shield the recovery target from the impact and the tolerances were 0.05 mm or higher [54].

The example in Figure 50 shows the recovery target and Figure 51 shows the 5 mm thick spall plate. Spall is seen to occur within the recovery portion of the target as well as the spall plate. However, these targets did not have an interference or slip fit. There

was a gap of approximately 0.05 mm, between the central disc portion of the target and the guard rings, which would have created a significant air boundary as shown in Gray's work, causing free surfaces at each plate boundary before the shock front had a chance to travel to the rear and sides of the target, creating significant tensile releases [54]. As the central plates were not held in laterally, there would also have been separation longitudinally earlier than expected, enhancing this effect.

Figures 52 and 53 show the consequences of having a significant air boundary. Void formation and propagation is present in the recovery portion of the target and more dramatically in the 5 mm spall plate where there is almost completion of full spallation. Microhardness testing (at a load of 300 g) confirms the differences in hardness across each sample, showing the strain affects where void formation is not visible but has clearly initiated.

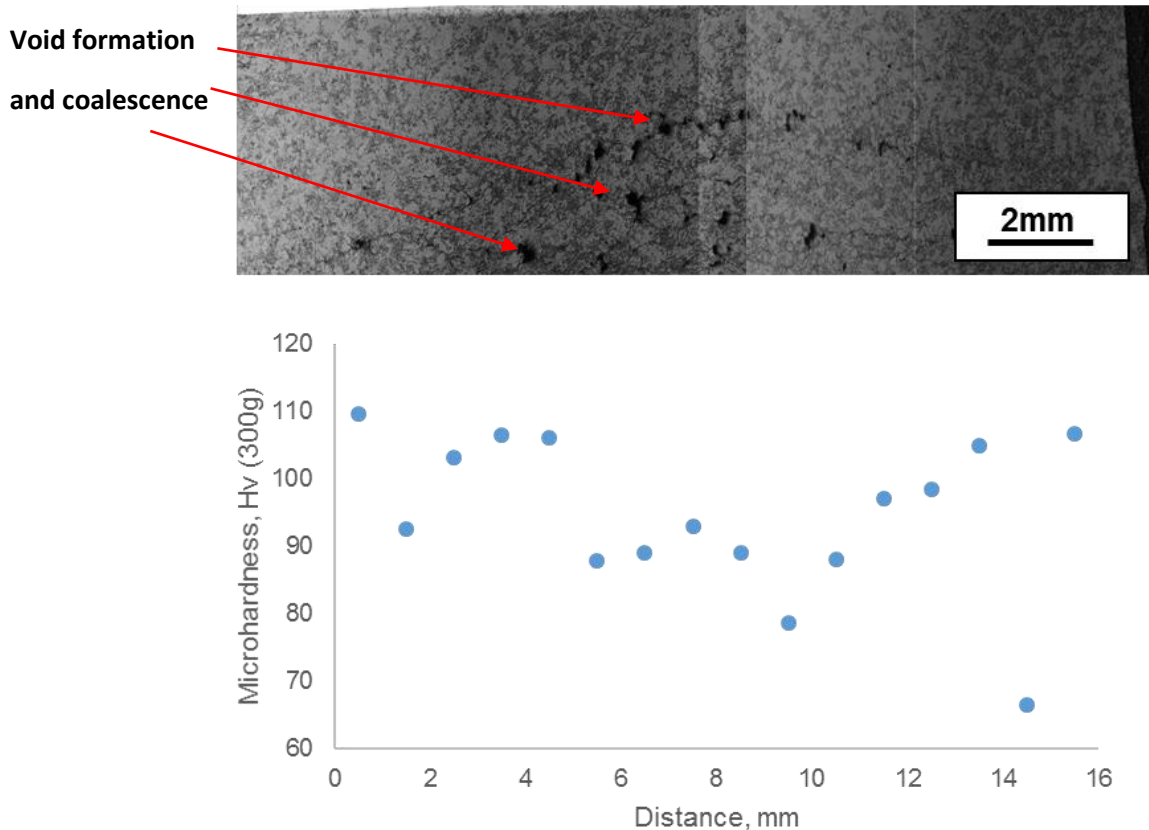


Figure 52: 6.3 GPa recovery portion of target, with significant air boundaries in the full target.

In Figure 52, evidence of void formation and hence spall can be seen in the micrograph where small voids can be seen, with some beginning to coalesce into longer cracks. The evidence of spall is also confirmed with the variations in hardness which have a range of 45 ± 5 Hv. Where the copper is less hard, voids have formed and therefore when the indenter has contacted the target these voids have collapsed creating a larger indent and leading to a lower hardness result. These lower values appear especially prevalent in the centre of the target between 5 mm and 11 mm across, which would be expected from the overlap of the tensile releases.

Figure 53 show similar hardness figures to those of Figure 52 though there is a visible spall plane rather than a lower amount of individual voids and some coalescence. Multiple cracks can be seen suggesting tensile overlaps in different parts of the target. The hardness testing once again reflects a large variation, this time of 30 ± 5 Hv, reflecting the differences where more voids or cracks are present.

The ideal tolerances as quoted by Bourne and Gray and Vignjevic et al. should be 0.01 mm, which was subsequently carried forward to the work on stainless steel 21-6-9.

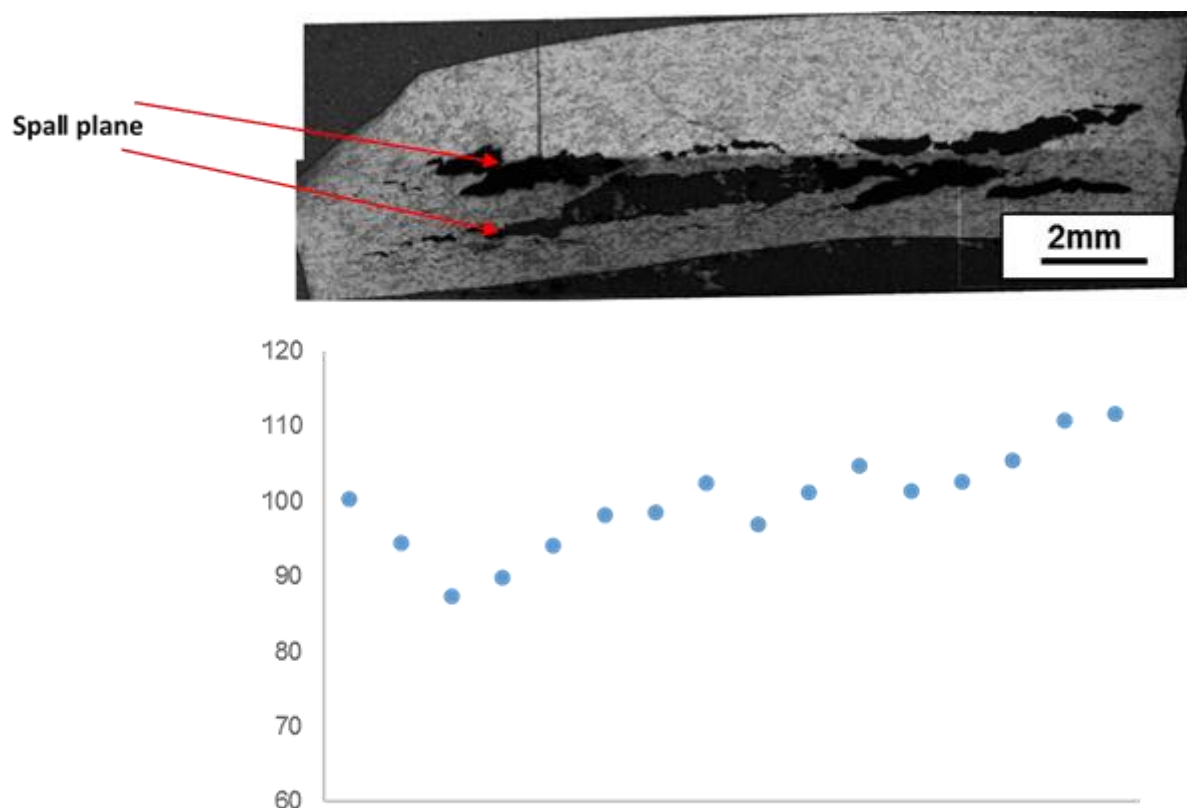


Figure 53: 6.3 GPa 5 mm spall plate portion of target, with significant air boundaries in the full target. 16

4.4 Analysis: TEM

Below a certain pressure threshold, copper can be seen to form dislocation cells under TEM, this was shown experimentally in Figure 54 with a recovery sample impacted at 7 GPa. This concurs with previous research where bulk specimens showed that a pressure of 14.5 GPa was required for deformation twinning to become dominant over dislocation cell formation [54]. Further to this, looking at the research from Gray *et al.* it would suggest that the plastic strain is approximately 2% or less [54].

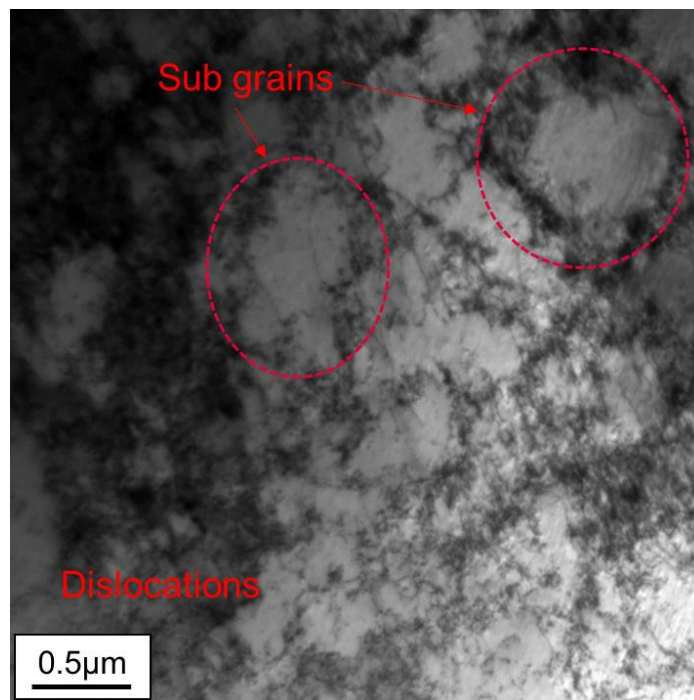


Figure 54: 7 GPa Cu Cross-section showing dislocation cell (subgrain) formation

4.5 Conclusions

Overall, the copper experiments allowed for development of experimental understanding and refinement, as well as providing insight into the effects of flyer plate thickness and henceforth pulse duration on target deformation. Computer modelling also allows for further understanding of these effects in Chapter 6.

The knowledge gained on correct interference fit of all of the components within the target, as well as correct alignment for recovery to successfully occur was then used to inform the experimental work carried out on the main focus material stainless steel 21-6-9. Further to this an increased number of analytical techniques were employed to analyse the novel contribution of data given from the stainless steel including plastic strain data, hardness testing, yield strength, twinning density and dislocation density.

5 RESULTS II: STAINLESS STEEL 21-6-9

5.1 Introduction

Overall, 28 experiments were carried out on stainless steel 21-6-9. Of these experiments, 11 were standard, where there was a single plate target, with no form of recovery system in the gas gun (see Section 3.2.2, Figure 31a). These standard shots were used as a baseline to compare the effectiveness of both partial and full recovery. One shot was a full recovery experiment (Section 3.2.2, Figure 34), which was used to compare against the partial recovery system's effectiveness. Finally, 16 of these shots were partially recovered shots where there was some reflection inside of the target as discussed in Section 3.2.2 (Figure 35).

5.2 Characterisation of Stainless Steel 21-6-9

Characterisation of stainless steel 21-6-9 was achieved through measurements of the density, hardness, dislocation density, twinning density from optical microscopy, CAI testing and XRD. These values are included as control measures in each section to compare against the different experiment types.

5.2.1 Density of Stainless Steel 21-6-9

Archimedeian immersion was used to measure the density of the received stainless steel 21-6-9 [206]. Archimedeian immersion is where the loss of mass of an object when suspended in a fluid with a known density, is equal to the mass of the displaced fluid. Therefore, the volume and then density can be calculated using Equation 12 [206]:

$$\rho_{solid} = \rho_{liquid} (m_{air} / (m_{air} - m_{liquid}))$$

Equation 12

ρ_{solid} = Density in Solid (kg m^{-3})
 ρ_{liquid} = Density in Liquid (kg m^{-3})
 m_{air} = Mass in Air (kg)
 m_{liquid} = Mass in Liquid (kg)

Three measurements were taken and then averaged to give a value of $7.81 \pm 0.01 \text{ g cm}^{-3}$. This is in agreement with the literature value of $7.818 \pm 0.022 \text{ g cm}^{-3}$ [1].

5.3 Shock Experiments Undertaken

A combination of analysis techniques were used to determine the microstructural changes in stainless steel 21-6-9. Each technique on its own provides data that represents partial analysis of the post experimental sample. When all of these different pieces of analysis are combined, a more holistic representation of the deformation characteristics of the steel can be obtained, for both a standard and partial or fully recovered sample as shown in Figure 55. Where the standard samples have been fired and collected from the cotton bundles at the back of the gun, and the partially recovered samples and fully recovered sample consist of multiple spall plates and rings and have passed through the capture system.

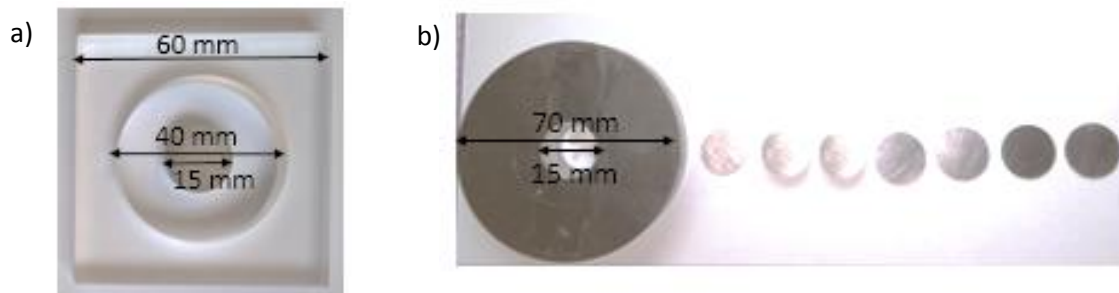


Figure 55: a) Standard target and b) Recovery target with two guard rings and six spall plates

The comparison between standard, partially recovered and fully recovered samples, allows a control study, to demonstrate the necessity for recovery itself, as well as giving a worst case scenario from deformation of the standard sample. Development of the recovery target was also explored by changing spall plate thickness at the back of the target to see whether improvement could be made on the partial recovery target design. This involves reducing the shocks within the target through the number and orientation of spall plates. Table 2 provides an overview of each stainless steel 21-6-9 experiment with the velocity, shock pressure and analysis undertaken on each target; standard, partially recovered or fully recovered.

Table 2: Overview of stainless steel shots undertaken with notes on the velocity, recovery and analysis carried out on each.

No.	Recovery	Velocity (ms⁻¹)	Shock Pressure (GPa)	Analysis carried out
CG0	Control	-	-	XRD, TEM, Optical, Hardness, CAI
CG1	No	370	7.0	XRD, TEM, Optical, Hardness, CAI
CG2	Yes	400	7.6	XRD, TEM, Optical, Hardness, CAI
CG3	Yes	600	12.0	XRD, TEM, Optical, Hardness, CAI
CG4	Yes	370	7.0	XRD, TEM, Optical, Hardness, CAI
CG5	Yes	685	13.6	XRD, Optical, Hardness, CAI
CG6	Yes	524	10.0	XRD, Optical, Hardness, CAI
CG7	No	321	6.0	XRD, Optical, Hardness, CAI
CG8	No	718	14.4	XRD, Optical, Hardness, CAI
CG9	No	631	12.4	XRD, Optical, Hardness, CAI
CG10	Yes	700	13.9	XRD, Hardness, CAI
CG11	No	318	5.9	XRD, Hardness, CAI
CG12	No	397	7.5	XRD, Hardness, CAI

CG13	No	446	8.5	XRD, Hardness, CAI
CG14	No	~600	12.0	XRD, Hardness, CAI
CG15	No	~700	14.0	XRD, Hardness
CG16a	No	730	16.0	XRD, Hardness
CG17	No	725	16.0	XRD, Optical, Hardness
CG18	Yes	725	16.0	XRD, Optical, Hardness
CG19	2x2 mm	760	15.3	XRD, Optical, Hardness
CG20	1x4 mm	769	15.0	XRD, Optical, Hardness
CG21	Yes	761	15.0	XRD, Optical, Hardness
CG22	2x2 mm	370	7.0	XRD, Optical, Hardness
CG23	1x4 mm	370	7.0	XRD Optical, Hardness
CG24	Yes	332	6.2	XRD, Hardness, Optical
CG26	Yes	538	10.4	XRD, Hardness, Optical
CG27	No	650	12.8	XRD, Hardness, Optical
CG29	Yes	552	10.7	XRD, Hardness, Optical
CG30	Full	370	7.0	XRD, Hardness, Optical

5.3.1 Heterodyne Velocimetry

Heterodyne velocimetry (Het-V) data provides an accurate U_p or particle velocity value to be obtained from a shot rather than using the impedance matching technique and calculating the value of the data obtained from the light gates. The particle velocity values obtained from the Het-V, which was carried out during experiment CG17, matched with the calculated values from the impedance matching technique, this provides confidence in the calculated shock pressure values, which are given for each experiment.

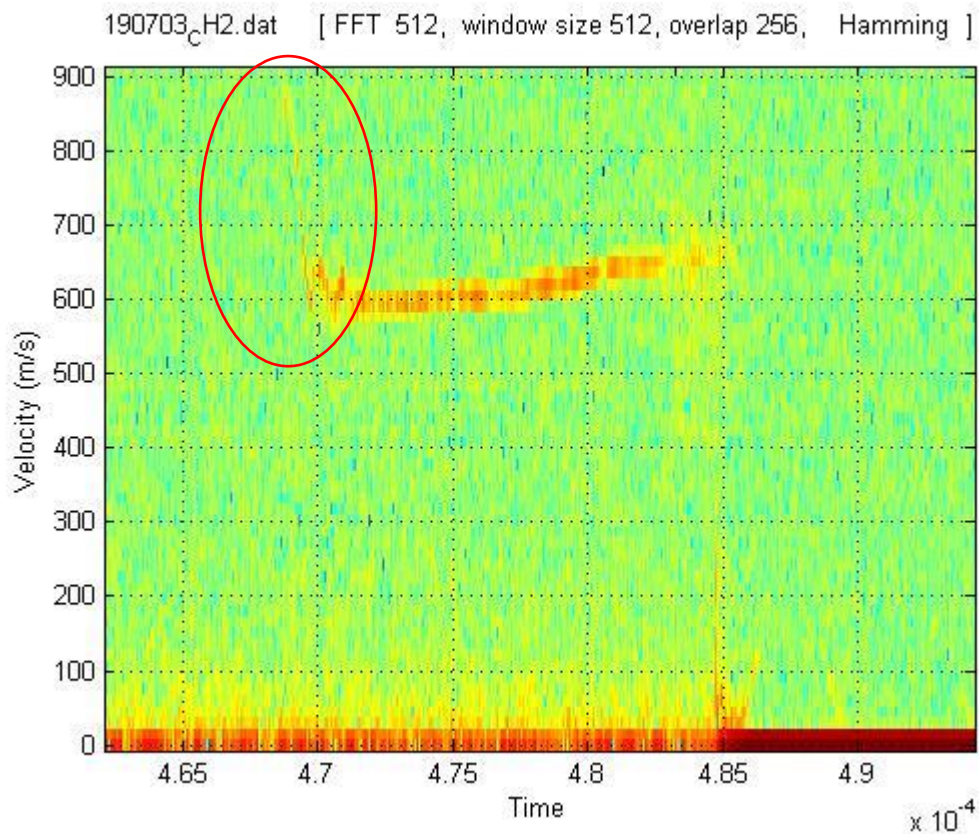


Figure 56: Het-V trace from 16 GPa standard shot. The tensile release wave can be faintly seen in the highlighted circle in red

5.4 Statistical Methods and Error Propagation

Statistical methods and error propagation have been used to give an estimate of the error for each technique. The main two methods used in this project are ANOVA and standard deviation of the sample leading to calculation of the standard error.

5.4.1 Analysis of Variance (ANOVA)

In order to calculate errors for twinning density Minitab® was used with its Analysis of Variance for Designed Experiments (ANOVA). This means that the variance difference

between each pressure for the standard and shock recovered samples as a comparison to see if there is a true difference.

One way ANOVA assumes that the data is independent and normally distributed, with the two groups having different means but the same variance. In the case of the twinning density, the two groups are 'Partially Recovered Samples' and 'Standard Samples'. Or, by comparing the three different full recovery designs are '4x1' mm, '2x2 mm' and '1x4 mm'.

Where the significance level is $\alpha = 0.05$, if the p-value is equal to or smaller than α then there is a high probability of statistical significance, whereas if the p-value is larger than α the differences between the means are unlikely to be of statistical significance.

The results for the recovered versus the standard samples are given in Table 5. Equal Variances were assumed for the analysis. The P-value is 0.038 showing that there is a statistical significance between standard samples and partially recovered samples in twinning density.

Table 3: ANOVA of recovery samples versus no recovery samples

Null hypothesis	All means are equal
Alternative Hypothesis	Not all means are equal
Significance Level	$\alpha = 0.05$

Factor Information		
Factor	Levels	Values
Sample	2	Standard, Recovery

Analysis of Variance					
Source	DF	Adj SS	Adj MS	F-Value	P-Value
Sample	1	250.2	250.22	9.28	0.038
Error	4	107.8	26.95		
Total	5	358			

Model Summary			
S	R-Sq	R-Sq(adj)	R-sq (pred)
5.19181	69.89%	62.36%	32.24%

Means				
Sample	N	Mean	StDev	95% CI
No Recovery	3	24.84	5.88	16.52, 33.17
Recovery	3	11.93	4.39	3.60, 20.25

The results for the modified recovery samples are given in Table 5. The P-value for the modified targets is 0.767. As this is greater than $\alpha = 0.05$, no statistical significance is suggested, which implies that the effect on partial recovery for the modification of the targets is negligible for twinning density. Therefore, no difference was noted in the different rear spall plate configurations for twinning density, which can be an indicator for recovery success.

Table 4: ANOVA comparing modified recovery targets

Null hypothesis	All means are equal
Alternative Hypothesis	Not all means are equal
Significance Level	$\alpha = 0.05$

Factor Information		
Factor	Levels	Values
Sample	3	1x4, 2x2, 4x1

Analysis of Variance					
Source	DF	Adj SS	Adj MS	F-Value	P-Value
Sample	2	34.91	17.45	0.29	0.767
Error	3	180.77	60.26		
Total	5	215.68			

Model Summary			
S	R-Sq	R-Sq(adj)	R-sq (pred)
7.76261	16.18%	0.00%	0.00%

Means				
Sample	N	Mean	StDev	95% CI
1x4	2	6.75	7.01	-10.72, 24.22
2x2	2	5.25	5.04	-12.22, 22.72
4x1	2	10.95	10.31	-6.52, 28.42

Where: DF= The total degrees of freedom
 Adj SS = Adjusted sums of squares
 Adj MS = Adjusted mean squares
 F-Value = The test statistic
 P-Value = To determine whether each main effect and the interaction effect is statistically significant

The total degrees of freedom are the amount of information in the data. The analysis uses the information to estimate the values of unknown population parameters. The adjusted sums of squares are measures of variation for different components of the model. The order of the predictors in the model does not affect the calculation of the

adjusted sum of squares. The adjusted mean squares measures how much variation a term or model explains, assuming that all other terms are in the model, regardless of the order they were entered. Unlike the adjusted sums of squares, the adjusted mean squares consider the degrees of freedom. The F-value is used to calculate the P-value, which is the probability that measures the evidence against the null hypothesis.

5.4.2 Error Propagation

Error propagation is vital when processing data, as multiple errors including human, instrumental, environmental and procedural all add up to produce some level of uncertainty in a measurement or reading. An appropriate statistical method must also be used when processing data to ensure the most accurate error estimate.

The error for the dislocation density obtained from XRD was calculated using standard deviation of the sample then calculating the standard error [207]. Sample standard deviation was used as the samples analysed would be representative of a larger population i.e. a greater number of experiments would need to be carried out to create a large enough data set for population standard deviation to be used. The sample standard error is given in Equation 11 where:

$$s_x = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}} \quad \text{Equation 12}$$

Where: s_x = Standard deviation of sample
 n = Number in the sample taken
 x_i = An individual value
 $\bar{x}$ = The mean of the population

The standard error is then given as the standard deviation divided by the square root of the population size.

5.5 Analysis of Stainless Steel 21-6-9

5.5.1 X-ray Diffraction: Dislocation Density

Dislocation density utilises X-ray diffraction to ascertain the amount of dislocations within a given area or volume of a sample [208]. Typically, area is used rather than volume, as it can be difficult to ascertain the exact sample thickness. This is because the thickness of the sample is in the order of 10^{-6} so the errors in measurement would be large and the thickness varies over the whole sample. Additionally, there is a presumed homogeneity of dislocations throughout the sample, which is not true, especially post shock, as changes in grain size, dislocation number and propagation can cause changes in strength and hardness properties, which may be heterogeneous due to shock reverberations [165]. As previously discussed, dislocations are one of two types, edge or screw, but are usually a combination of both. An example of a dislocation in stainless steel 21-6-9 is shown in Figure 57.

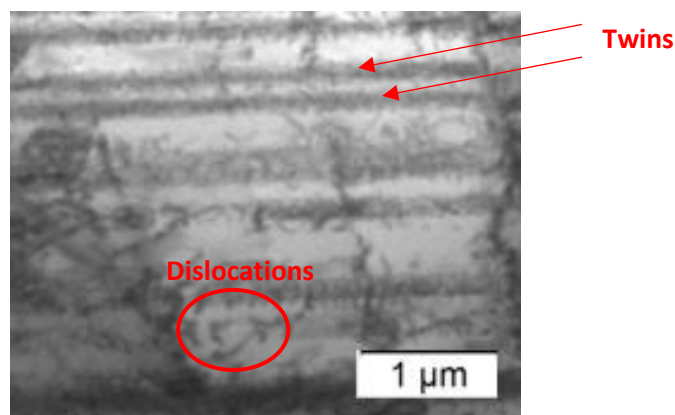


Figure 57: TEM image of stainless steel 21-6-9. Twins are highlighted as well as a dislocation.

X-ray diffraction does have some challenging aspects, including a small spot size of between 0.4 mm fine focus to 2.0 mm broad focus [209]. Regarding phase changes in the metal, there is also a 2% detection limit, meaning that there must be more than 2% of an impurity to show in the pattern. In this scenario, the impurity would be a different phase i.e. ϵ - or α' - martensite. If there is another phase or material incorporated into the sample, peak overlap may occur and penetration depth is limited to approximately 10-20 μm , which only indicates a small amount of the sample depth ($\sim 0.4\%$) [210].

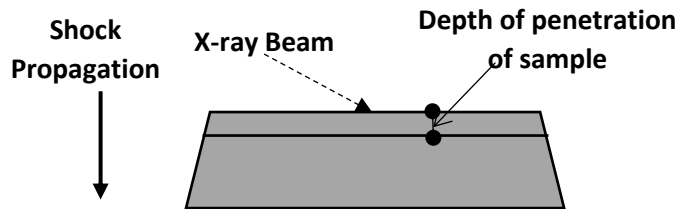


Figure 58: Image showing set up of sample during XRD. Depth of penetration of sample is not to scale

Dislocation density was calculated using the Scherrer Equation (Equation 12, which relates the size of grains in a solid to the broadening of a peak in a diffraction pattern [211]):

$$B(2\theta) = K\lambda / (\beta \cos\theta) \quad (2) \quad \text{Equation 13}$$

Where: $K = 0.94$ (for FWHM spherical crystals with cubic symmetry) [211]

B = mean size of grain (could be smaller than or equal to actual grain size)

λ = X-ray wavelength (m)

β = line broadening at full width half maximum (FWHM)

θ = Bragg angle ($^\circ$)

$$\text{Dislocation density} = 1/B^2 \text{ [211]}$$

Equation 14

The results for this are shown in full in Appendix B.

The shock pressure was calculated using the Rankine-Hugoniot relations (Equations 3-7) [137].

$$C_B = (C_l^2 - 4/3 C_s^2)^{1/2} \quad \text{Bulk sound speed}$$

$$U_s = S \cdot U_p \cdot C_0 \quad \text{Hugoniot linear equation}$$

$$U_s \rho_0 = (U_s - U_p) \rho \quad \text{Conservation of mass}$$

$$P - P_0 = \rho_0 U_s U_p \quad \text{Conservation of momentum}$$

C_l = Longitudinal sound speed (ms^{-1})
 C_s = Latitudinal (shear) sound speed (ms^{-1})
 C_B = Bulk sound speed (ms^{-1})
 U_s = Shock velocity (ms^{-1})
 S = Material constant (~ 1.49 for stainless steel and copper [212])
 U_p = Particle velocity (ms^{-1})
 C_0 = Sound velocity in a material at ambient pressure $\approx C_B$ = Bulk sound velocity (ms^{-1})
 P_0 = Initial pressure (Pa)
 P = Final pressure (Pa)
 ρ_0 = Initial density
 ρ = Final density

The values for C_l , C_s and C_B were obtained through sound speed measurements. The values for this are given in Table 5.

Table 5: Measured longitudinal and shear sound speed using ultrasound testing in copper and stainless steel 21-6-9. The bulk sound velocity has been calculated from the two values using Equation 4.

Material	C_l (ms^{-1})	C_s (ms^{-1})	C_B (ms^{-1})	Density (g cm^{-3})
Copper	4674	2242	3891	8.90
SS 21-6-9	5754	3140	4467	7.81

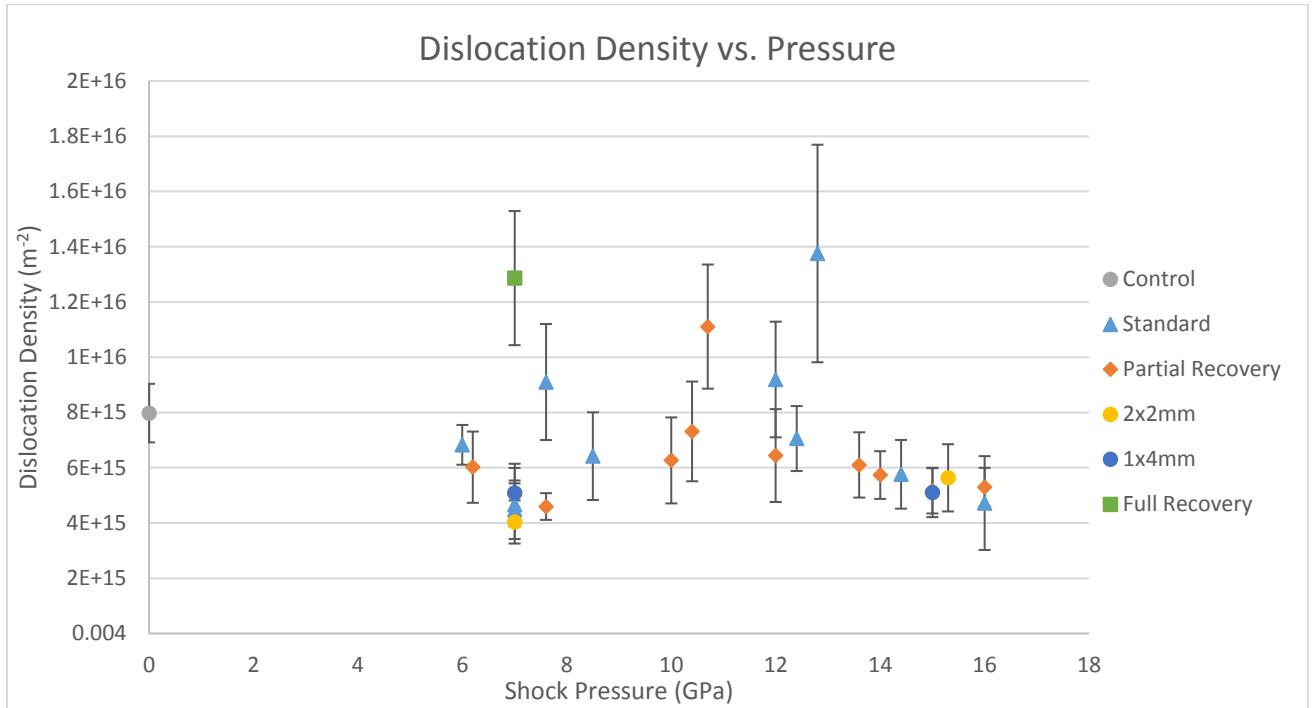


Figure 59: Dislocation density from XRD data obtained using the Scherrer Equation, where shock recovery refers to the process involving a multi-ringed and plated target with a soft capture system placed in the target chamber. The error given is the standard error calculated from the standard deviation of the sample shown in Appendix B.

The graph for dislocation density is given in Figure 59. The dislocation density increases with increasing pressure until 12 GPa, where it peaks at $1.37 \times 10^{16} \text{ m}^{-2}$ for standard samples and at $1.11 \times 10^{16} \text{ m}^{-2}$ for samples which have been partially recovered. It then decreases above 12 GPa for both standard and partially recovered samples. The variation of the recovered samples is lower than that of the standard ones.

It is noted that the standard samples in Figure 59, while deviating from the partially recovered samples between 8 GPa – 12 GPa follow the same trend as the partially recovered samples above 12 GPa. Reasons for this could be that XRD is measuring boundaries within the sample, through the detection of different orientations, which may be a grain boundary, dislocation or twin. Therefore, if different mechanisms

become more prevalent at certain pressures the dislocation density calculated is going to change.

The full recovery sample has a dislocation density of 1.29×10^{16} . This is higher than the partial or the standard recovery which have values of 5.09×10^{19} . Overall the variation of the values is large. If there is an increase in twinning density, dislocation propagation will be reduced. If grain size were to increase due to an annealing affect then the dislocation density would also decrease. There is also a large error in the measurements, which suggests that obtaining the dislocation density through the Scherrer equation is not an optimal method, in depth TEM or SEM data would be required for this. This is why multiple techniques include twinning density are being included to determine what the main mechanism or mechanisms are.

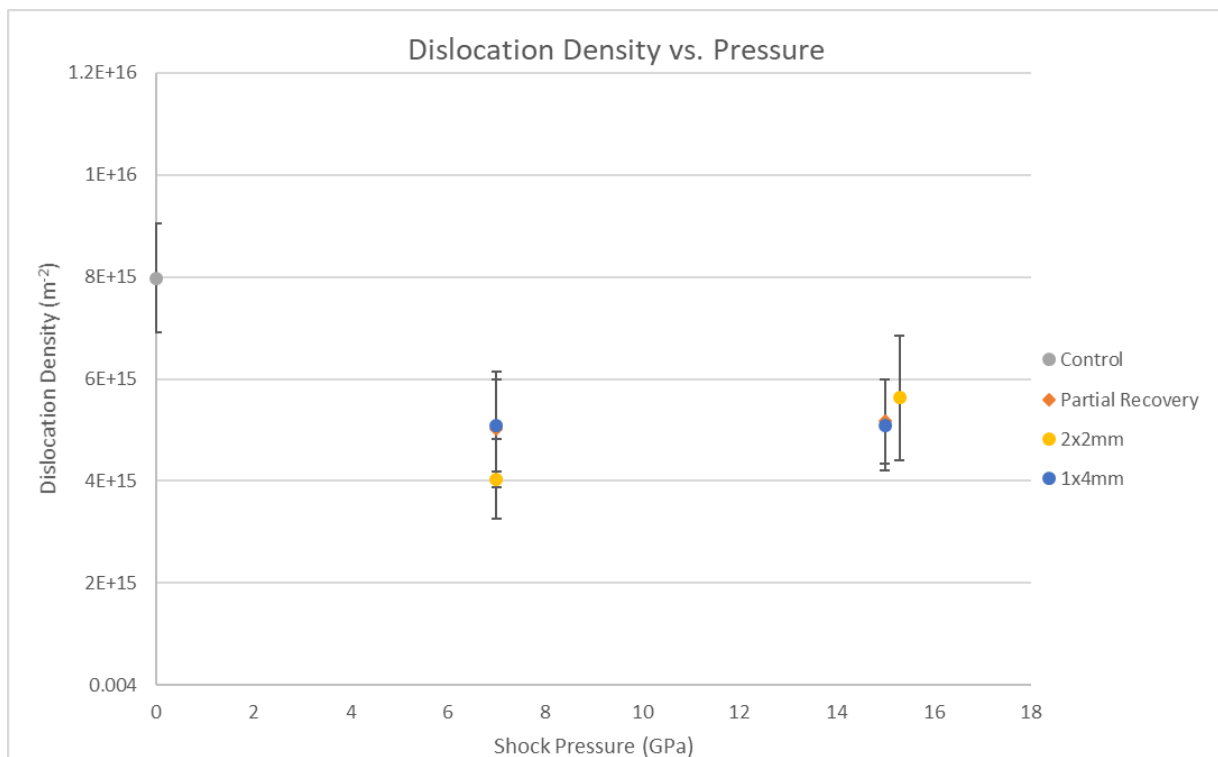


Figure 60: Dislocation density versus pressure for the modified samples with 2x2 mm spall plates and 1x4 mm spall plates compared to the original shock recovery design of 4x1 mm plates.

Figure 60 shows the respective dislocation densities for each modified recovery sample. The original recovery sample having four 1 mm spall plates, and the two modified recovery samples having two 2 mm spall plates and one 4 mm spall plate respectively. At 7 GPa the dislocation density is similar at $5 \times 10^{15} \text{ m}^{-2}$ for both the four 1 mm spall plates and the one 4mm spall plate, whereas the dislocation density is lower for the two 2mm spall plates. However, at a pressure of 15 GPa the two 2 mm spall plates have a higher dislocation density than the one 4 mm spall plate and four 1 mm spall plates.

The errors are large, so dislocation density obtained from the Scherrer equation may not be a good indicator of recovery without having other data available. This confirms the limitations noted by Zhang *et al.* where use of XRD is acknowledged to be limited by low sensitivity, noise interference and a small sample area [213]. Zhang *et al.* used Electron Backscatter Diffraction (EBSD) to carry out more in depth analysis, which would be considered for future work here [213]. Banerjee *et al.* also used TEM and EBSD in conjunction with XRD to verify their findings on strain rate dependent deformation behaviour [214].

5.5.2 Optical Microscopy: Grain Size

Dislocation density values are obtained as the reciprocal of the average grain size. Therefore if the XRD dislocation density values are flipped a graph of the average grain size would be produced. These include the same disadvantages as previously discussed, which include the detection of boundaries, regardless of whether it is a dislocation, grain boundary or twin, so for comparison the average grain size from

optical microscopy samples has been included, which was calculated from manual grain counting. This is shown in Figure 61.

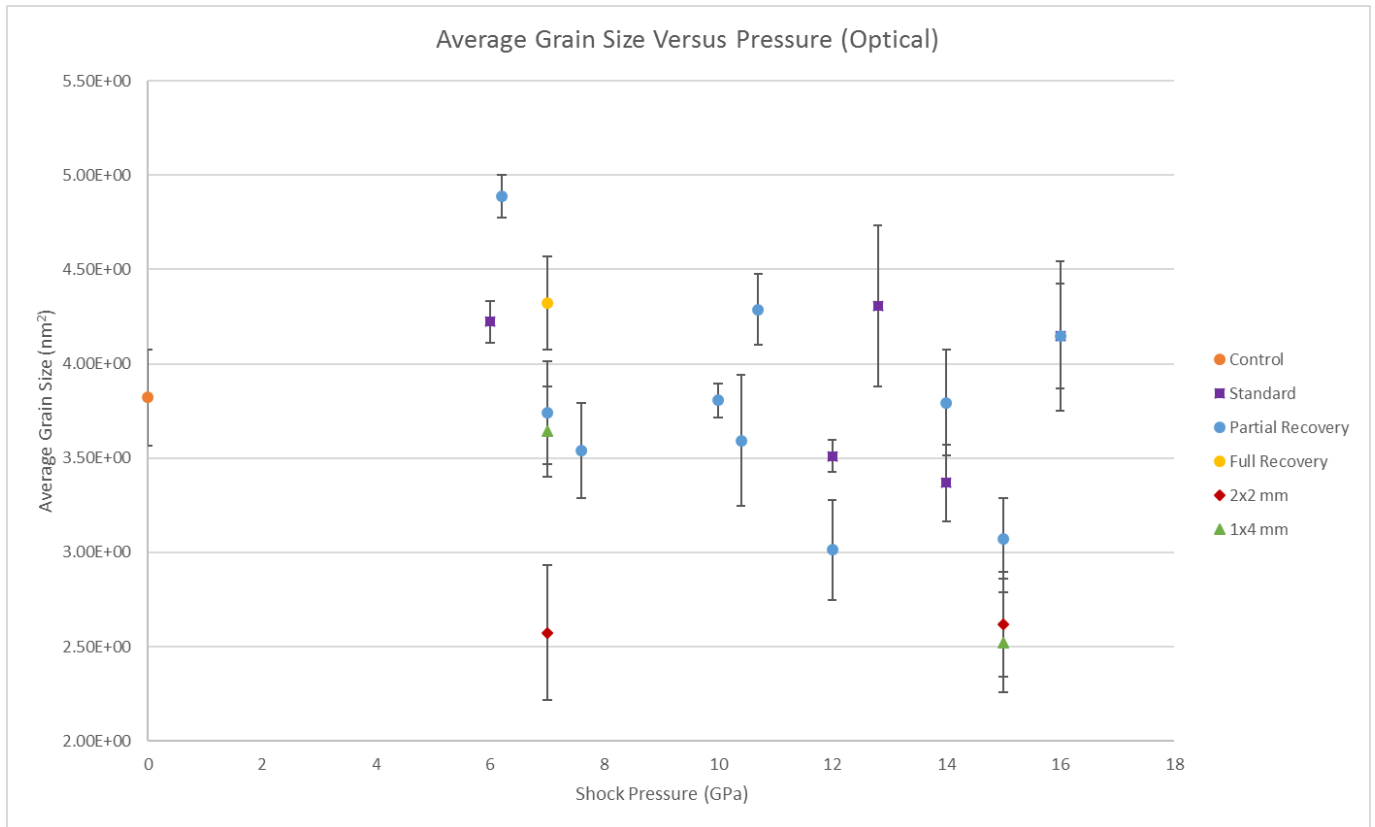


Figure 61: Average grain size versus shock pressure using manual counting technique

For the optical samples in Figure 61, for the partially recovered samples, the average grain size varies between values of 3.00 ± 0.27 nm to 5.00 ± 0.04 nm. The standard samples show varying grain sizes between 3.40 ± 0.25 nm to 4.30 ± 0.14 nm. The fully recovered sample has a grain size of 3.60 ± 0.25 nm.

A general trend in the standard samples of a decrease of 1.00 ± 0.50 nm can be seen, this could be due to dislocation propagation causing subgrains and saturation, where twinning and break up of subgrains occurs at high levels of strain [215]. The increased

twinning and decreased grain size lead to an increase within the yield stress (strength) in the compression testing (Section 5.5) explaining why the overall yield stress is higher in the standard samples versus the recovered samples. However, further information is required from the other analysis techniques to make any definitive statements. Overall, all of the samples have grain sizes which are in the order of nm and changes in the grain size can be reflected in the variation of reverberations of the sample.

The full recovery sample has a grain size which falls within the error of the control sample (3.60 ± 0.25 nm versus 3.80 ± 0.30 nm), suggesting that recovery has been successful. However, the values for the partial recovery vary even between 6 GPa and 7 GPa, suggesting different success rates of recovery causing changes in grain size. It would be expected to have a wider variation of grain size towards the outside of the sample due to the greater number of reflections occurring and changing the microstructure, as well as being outside of the 1D zone. Perhaps in the randomised selection of images for grain counting more images were obtained from the outside of the sample. However, some variation would be expected, as the grain size could be affected from initial machining of the target as well as the shock itself. Overall, there is no immediate correlation of any of the recovery types.

5.5.3 X-ray Diffraction: Phase Changes

XRD can also be used to investigate phase changes within a material. As previously discussed, phase changes can cause changes to the material properties, which could lead to changes in strength, hardness and twinning. Previous authors have disputed the threshold where it begins, as some authors including Hills discovered an unknown

phase from around 5 GPa where dislocation bands crossed but most authors believe the change happens above 15 GPa [101]. XRD is unlikely to detect any phase change below that of approximately 2% of the overall composition of the material, with this research only one of the partially recovered samples gives any indication of a phase change below that of 15 GPa (see Figures 62-65).

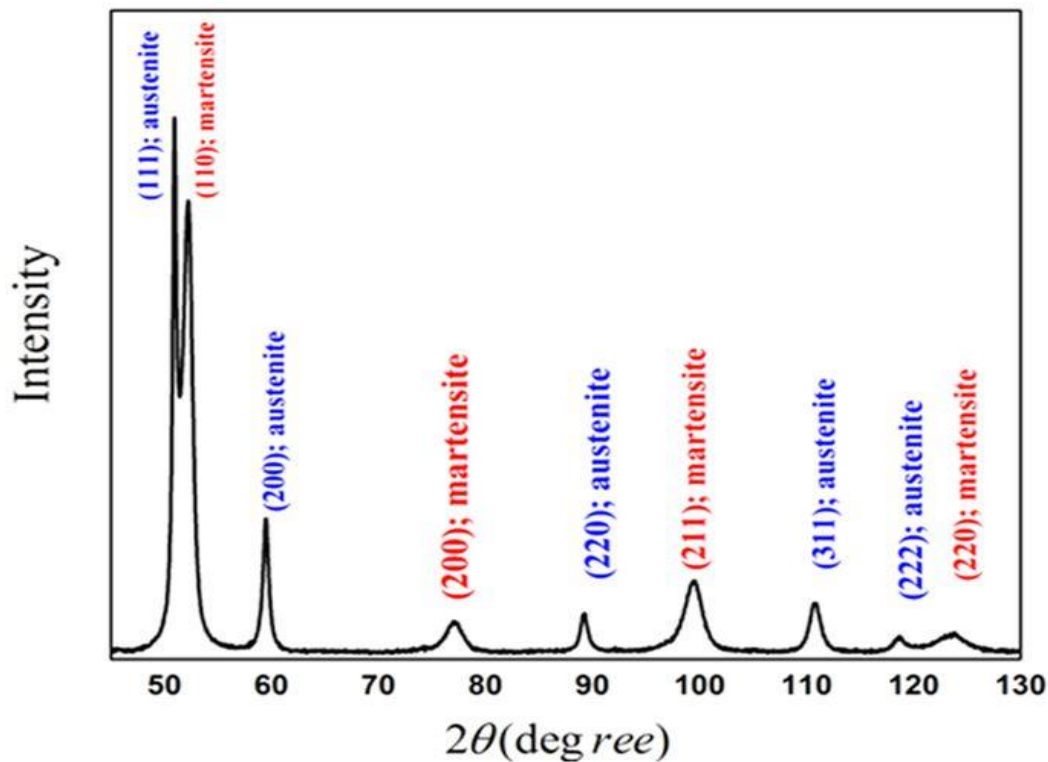


Figure 62: XRD trace of austenite and martensite to compare against XRD traces obtained of stainless steel 21-6-9 from experimental work [226]

The martensitic phase change (α') would be observed primarily at (110) and as can be seen in Figures 63 and 64, there is no indication of a phase change at this point. In Figure 65 there is a peak which could be identified as the (110) α' peak, however none of the other samples which underwent X-ray diffraction contained this peak (see Appendix B). An explanation of this is that in the other samples martensite was present but in quantities lower than 2% or it could be the Cu $k\beta$ peak for (111) γ -austenite.

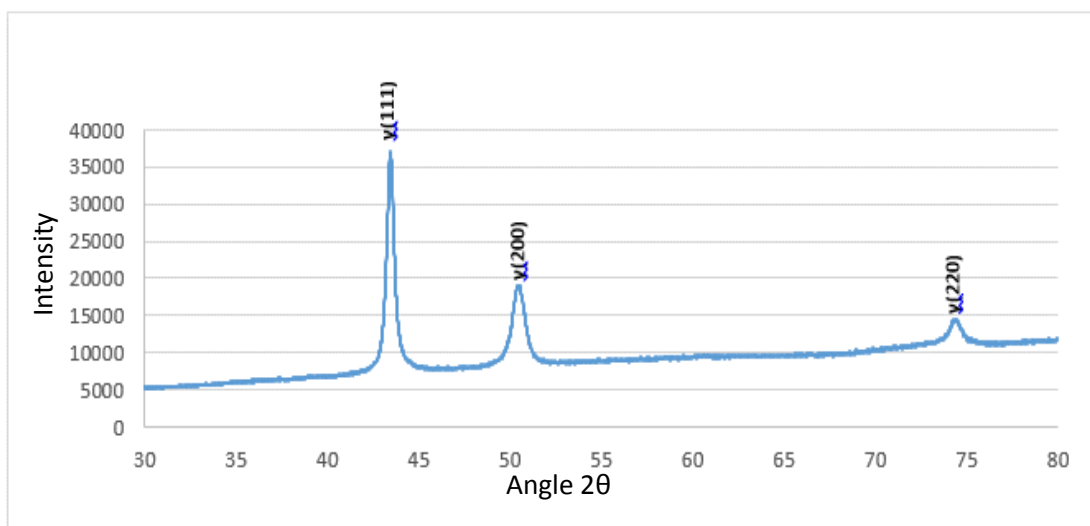


Figure 63: XRD trace of control stainless steel 21-6-9 sample. The sample shows the (111), (200) and (220) peaks for the γ -austenite phase of stainless steel 21-6-9.

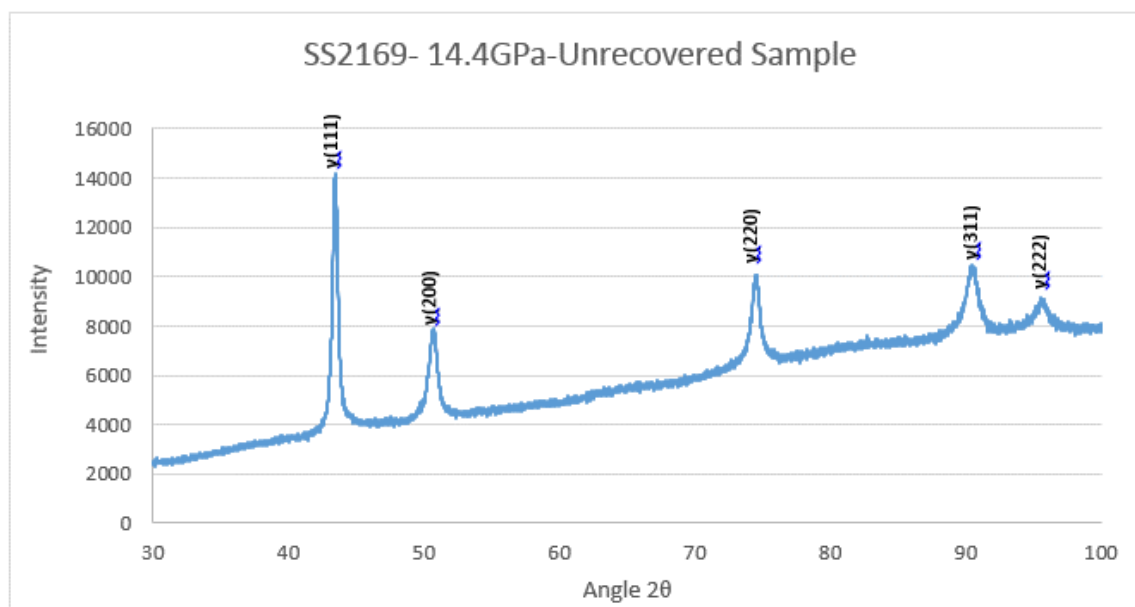


Figure 64: XRD trace of a standard sample with a shock pressure of 14.4 GPa. The (111), (200), (220), (311) and (222) peaks for the γ -austenite phase are indicated.

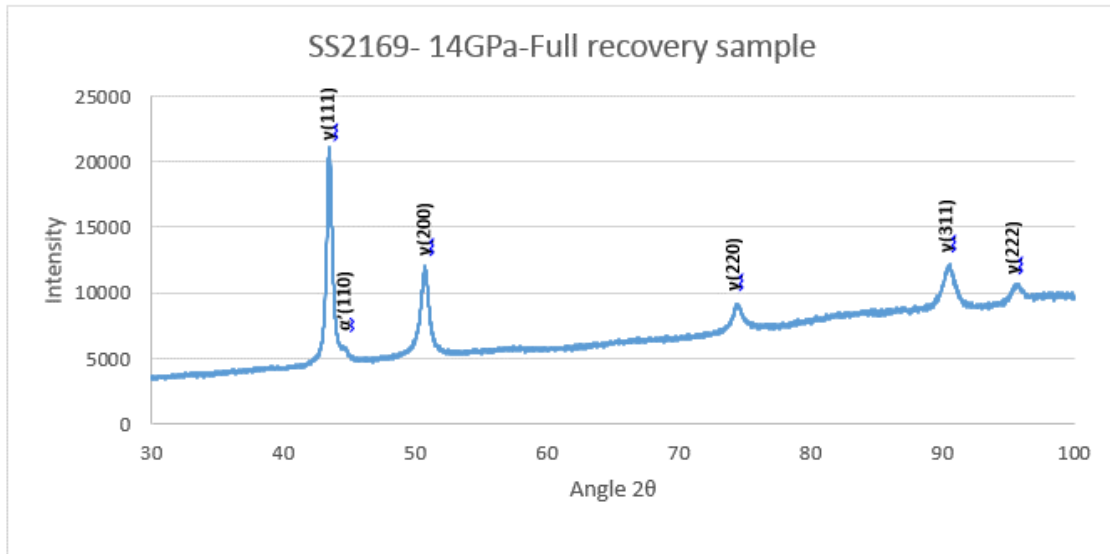


Figure 65: XRD trace of a partially recovered sample with a shock pressure of 14 GPa, where the (111), (200), (220), (311) and (222) peaks are shown for the γ -austenite phase. There is a slight peak at approximately 45° , which suggests the presence of the α' -martensitic phase of stainless steel 21-6-9 or the Cu $k\beta$ peak for γ -austenite.

5.5.4 Optical Microscopy: Twinning Density

One of the main deformation mechanisms of stainless steel 21-6-9 is that of twinning. Twinning density can give an indication of how many twins per grain there are in each sample. This can be achieved through optical microscopy by counting individual grains and then individual twins, hence providing a result in twins per grain as a comparative

proportion. An example of how these twins look in stainless steel 21-6-9 is given in Figure 66.

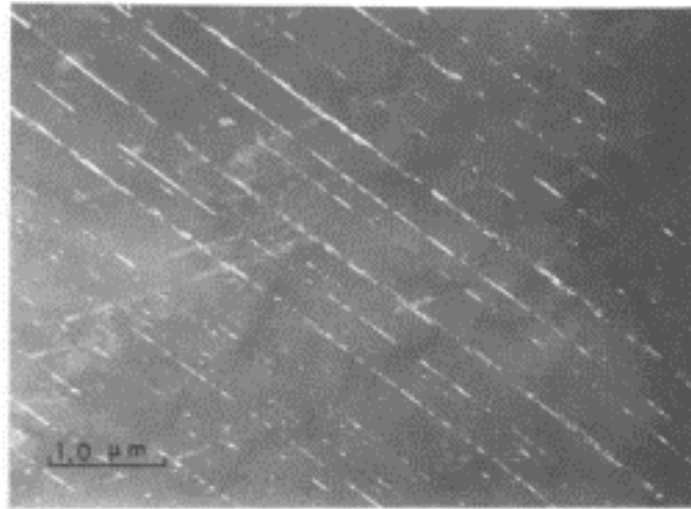
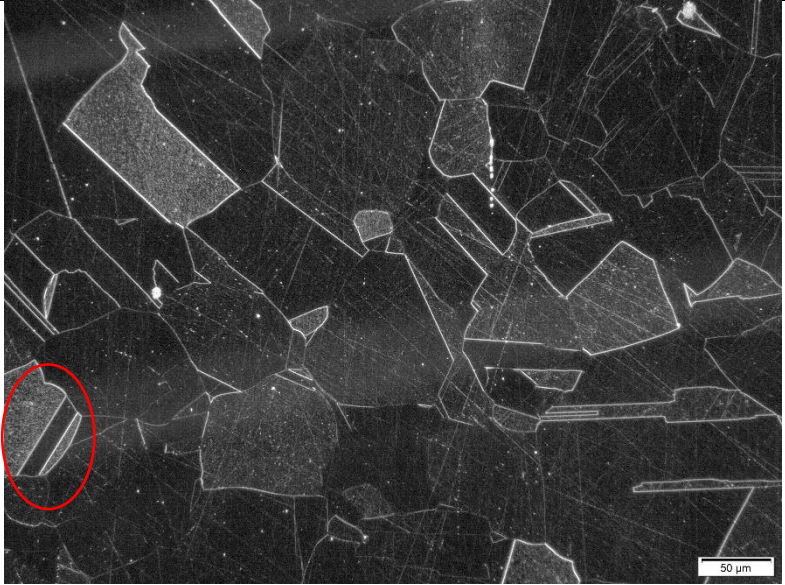


Figure 66: A dark field transmission electron micrograph showing segmented twins in stainless steel 21-6-9 shock deformed to a pressure of 7.5 GPa in the $[110]$ zone axis $[11]$.

In austenitic steels, twins display as a series of lines, which may be mistaken for stacking faults [216]. To mitigate human error and make the sampling selection as unbiased as possible, micrographs of each sample were taken as a grid with between 18- 27 sample areas. A ‘true’ random number generator, which uses atmospheric noise to generate numbers was used to select 5 random sample areas from each individual sample [217]. These five areas were then counted and tabulated.

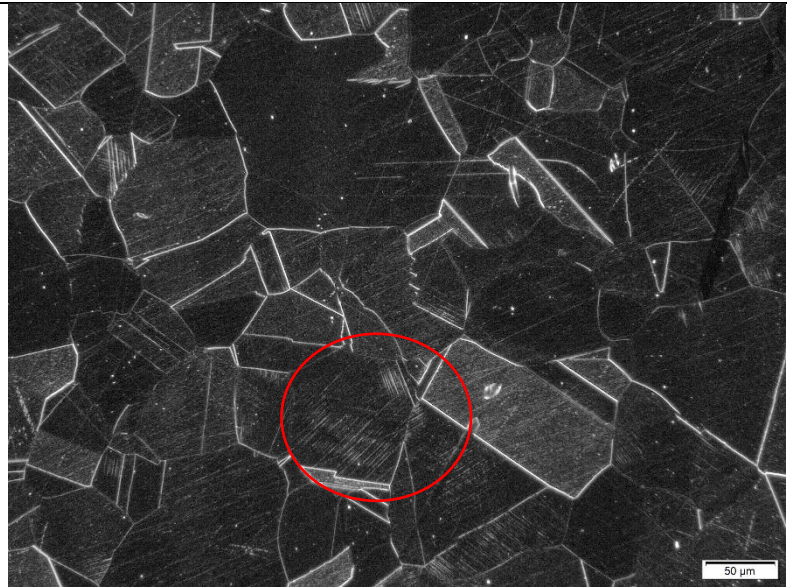
Table 6 gives a representation of each experiment type: standard, partial recovery, full recovery and the modified partial recovery of 2x2 mm spall plates and the 1x4 mm spall respectively at 7 GPa. A comprehensive list of representative micrographs from all experiments taken can be found in Appendix B.

Table 6: An optical image showing a sample area of a selection of targets. The red circle indicate the presence of twinning.

Control- Equiaxed grains with some annealing twinning present, example shown with a red circle	
Standard 7.0 GPa- Changes in grain size and shape with presence of deformation twinning compared to control, example shown with a red circle	

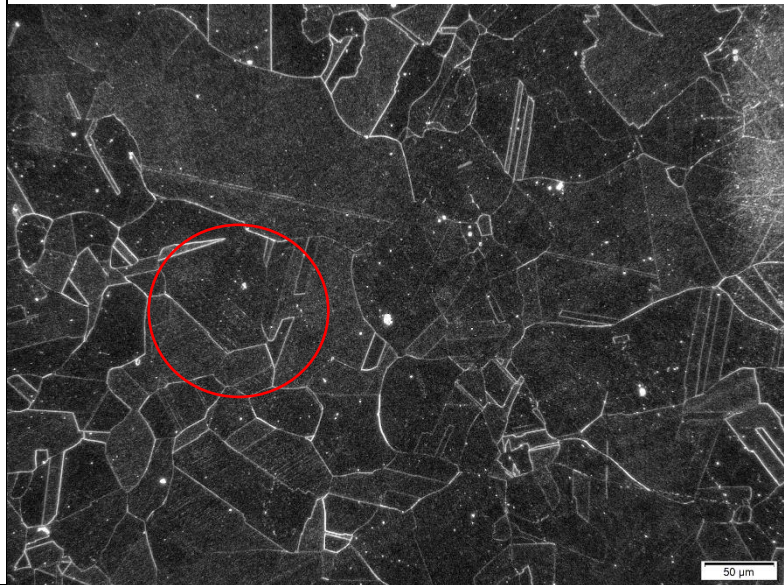
Partial Recovery 7.0

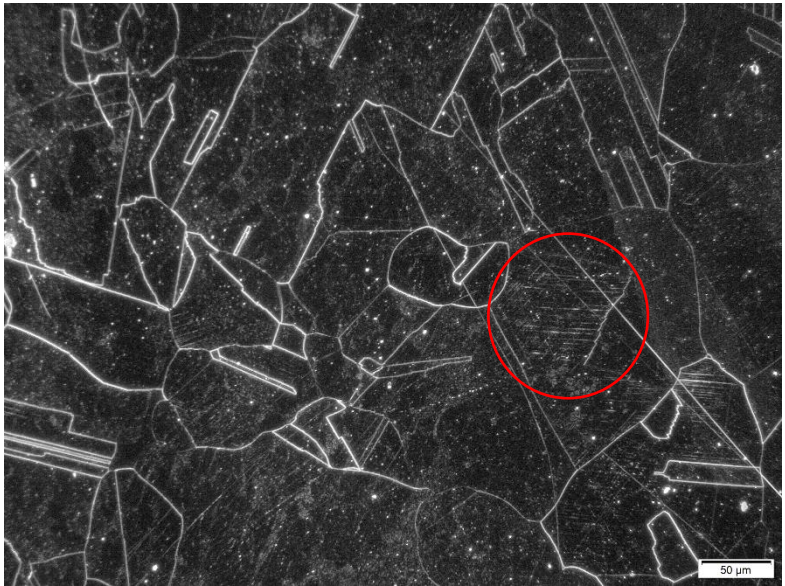
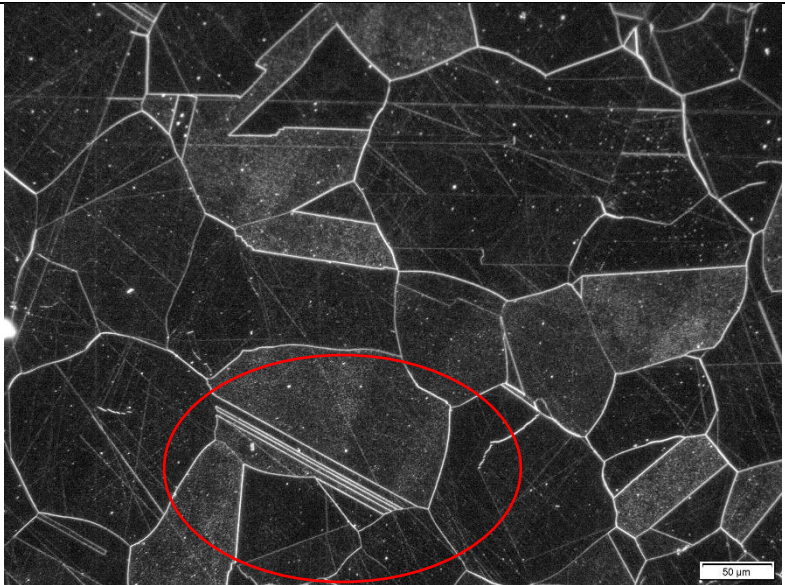
GPa- The majority of grains are equiaxed with increased twinning compared to the control sample, example of deformation twinning shown with red circle



Partial Recovery (2x2

mm) 7.0 GPa- An increase in twinning (deformation) compared to the control sample with the majority of grains equiaxial. Example of deformation twinning shown with red circle.



Partial Recovery (1x4 mm) 7.0 GPa- An increased number of twins compared to the control sample with some variation in grain size. Example of deformation twinning shown within one grain given by red circle	
Full Recovery 7.0 GPa- Equiaxed grains with a similar number of twins as the control sample. Annealing twins shown with red circle.	

After the five counts were completed these were then averaged and the proportion of twins per grain was plotted for each sample. This plot is shown in Figure 67.

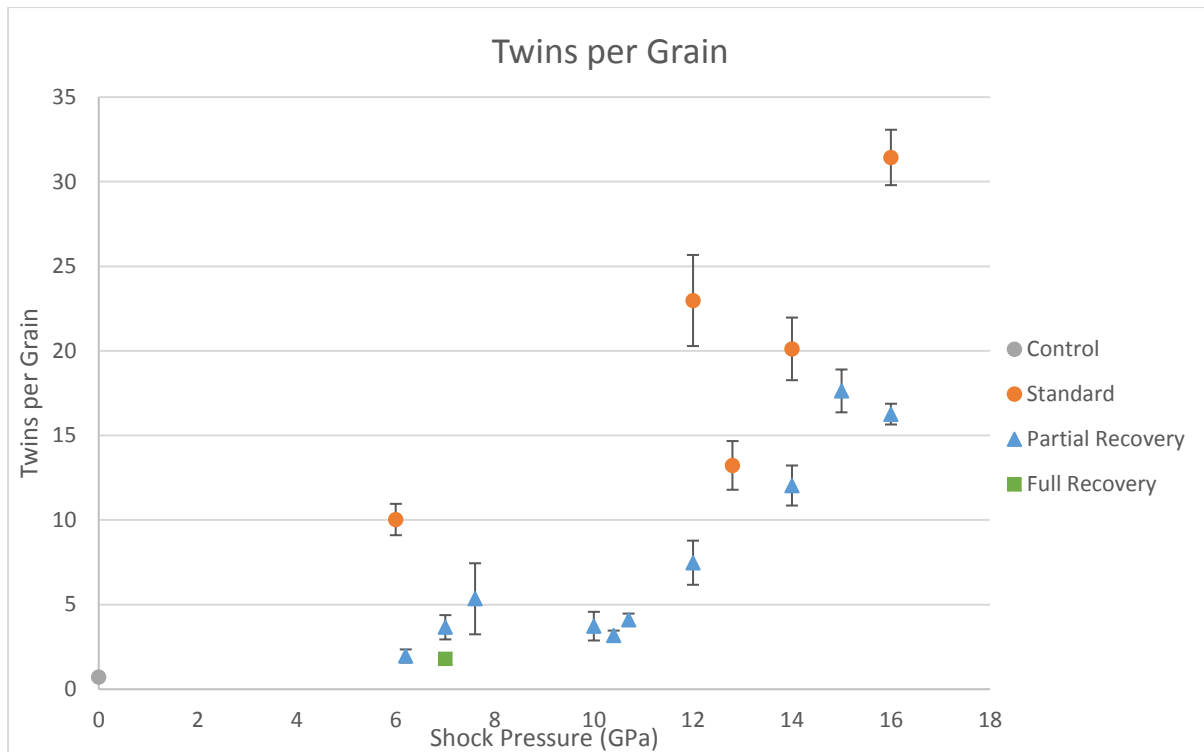


Figure 67: Graph showing the proportion of twins per grain for a series of shock recovered samples from 7 GPa - 13.6 GPa. The error calculated is the standard error of the standard deviation of the sample.

Figure 67 shows that as the shock pressure increases, the number of twins per grain also increases. Twinning would be expected as the main form of material deformation due to the low (42 mJ m^{-2}) SFE of the stainless steel.

As expected the standard recovery shows an increased number of twins per grain compared to the partial recovery system. At 12 GPa and 16 GPa the largest differences can be seen between the two at approximately 15 twins per grain. Where twinning is one of the main types of deformation, it would be expected that more reverberations would lead to increased twinning. At pressures above 20 GPa it would be expected that the standard samples would begin to saturate with twins and/or dislocations inhibiting further twin initiation and propagation, as well as formation of α' -martensite [101].

The full recovery target in Figure 67 at 7 GPa has a twinning density of 1.80 twins per grain compared to the control sample 0.72 twins per grain. As the full recovery target shows 1D deformation it would be expected to have lower twinning density than both the standard and partial recovery, which it does, confirming the success of the recovery.

At 7 GPa the partial recovery shows a twinning density of 3.66 twins per grain whereas the standard recovery shows a twinning density of 10.03 twins per grain at 6 GPa. From this data it can be inferred that twinning density is a good indicator of recovery as well as providing key mechanical deformation information on the stainless steel itself.

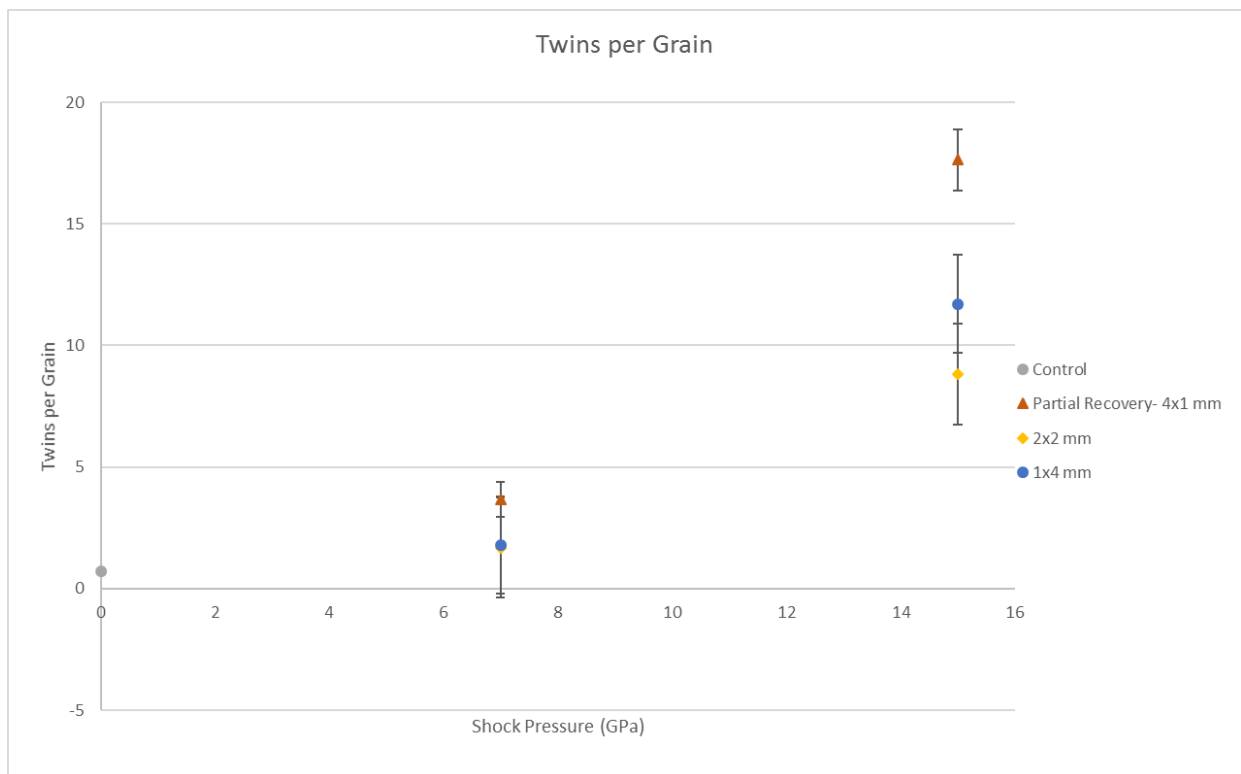


Figure 68: Twins per Grain for modified samples with the rear spall plates: 4x1 mm, 2x2 mm and 1x4 mm.

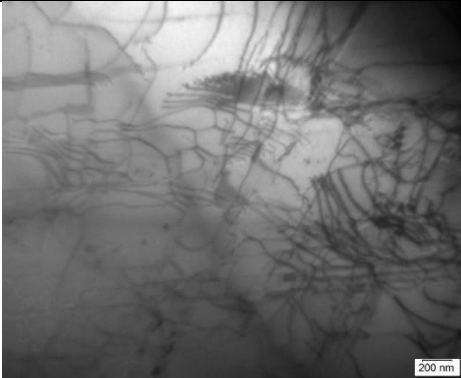
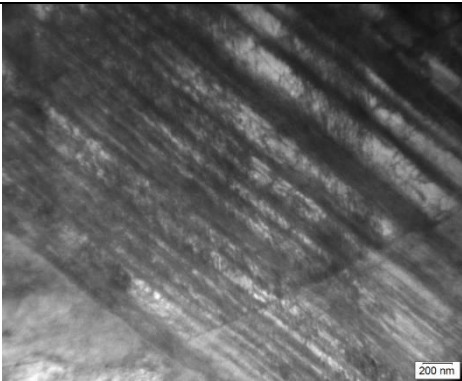
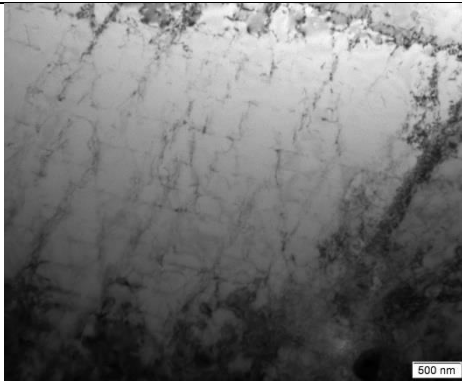
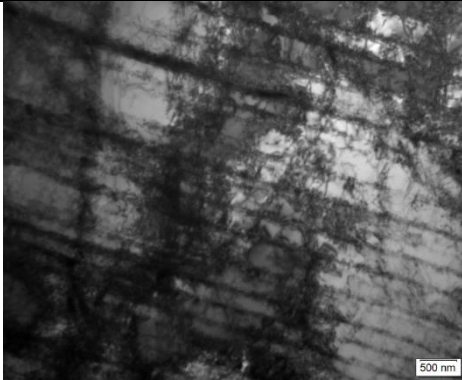
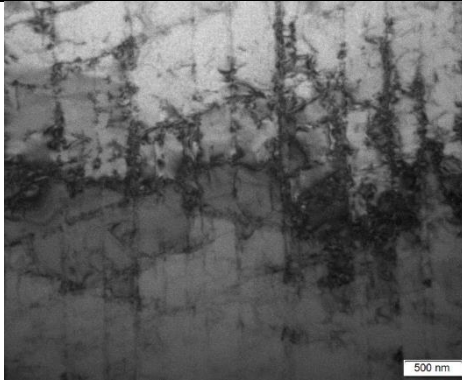
Figure 68 is the twinning density for the modified partial recovery samples. The twinning density is lower for the two modified samples. This difference is more pronounced at 15 GPa than at 7 GPa with a difference of 9 twins per grain between the original partial recovery and the 2x2 mm rear plate recovery. The lower twinning density is because, as explained in Section 3.2.2 Figure 34, in the partial recovery targets, the tensile waves overlap in the rear four spall plates. By modifying these plates to be thicker, more time is given for the plates to separate before the shock wave reflects back into the rest of the target. The 2x2 mm rear plate configuration is the most effective, as the plates are thicker than the 4x1 mm plates of the original target allowing twice the time for separation whilst also having less mass than the one 4 mm plate allowing for a higher velocity of separation due to the momentum.

Although twinning density and grain size, show changes within the microstructure, in order to see the effects on plasticity, yield strength and hardness both compression testing and hardness testing are required to give an overview to these effects. These are given in Section 5.5.6 and Section 5.5.7 respectively. Compression testing allows for information on the plasticity or plastic strain to be obtained, while hardness gives an overview of the overall effect of mechanisms such as twinning, shock hardening and dislocation density, which can give a good indication of the success and comparative effects of recovery.

5.5.5 Dislocation Density: TEM

A limited number of experiments were able to be analysed using TEM. To obtain the dislocation density, these images were analysed using a contrasting technique in Image J and then analysing the subsequent pixel proportions while taking into account the area analysed [186]. Restrictions on accuracy of obtaining dislocation density through TEM have been discussed by many different researchers and there are authors attempting to come up with novel ways of more accurate estimations [218]. However, the main restriction without including the difficulties in counting individual dislocations in dense tangles, is the small sample size of 3 mm diameter and an image area size within microns or nanometers. It is difficult to suggest that it is representative of the whole sample, especially depth-wise with the TEM foils being in the order of microns thick as opposed to millimetres or centimeters. To deal with this issue, several foils were punched from across the sample, providing a slightly larger area to analyse. A representative sample from each TEM image is shown in Table 7.

Table 7: Representative TEM image of each stainless steel sample

 CG0- Dislocations inside the control sample	
 CG1- Large number of twins present, dislocations visible behind	 CG2- Dislocations present with a deformation band on the RHS
 CG3- A series of twins present in the stainless steel with dislocations visible	 CG4- Deformation bands present formed from dislocation pile-ups

In Table 7, differences in the deformation characteristics are visible in each image. The control sample, CG0, shows some dislocations present though they are widely spaced and not tangled or piled up. Both standard recovery samples, CG1 and CG3,

show large numbers of twinning and dislocation tangles compared to the partially recovered samples, which do show an increased number of dislocations and some deformation bands formed from dislocations but no large numbers of twinning.

In Figure 69 a graph has been plotted for the dislocation density obtained for each sample from the TEM. It can be seen that as the shock pressure increases, the dislocation density also increases in what initially appears to be a linear fashion and then appears to level off. This would be expected as dislocations begin to saturate there is not room for dislocation nucleation and propagation. Table 7 CG1 show the 12 GPa sample which qualitatively shows a much denser number of dislocations than in any of the other images, highlighting the previous point.

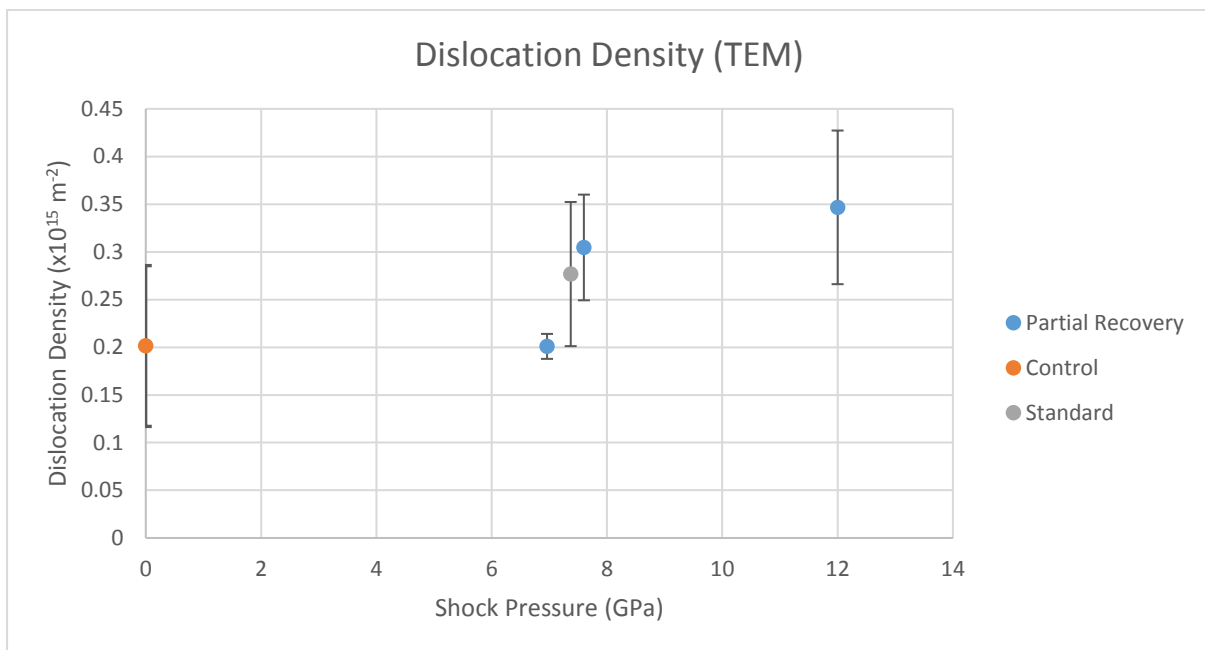


Figure 69: Dislocation Density obtained from TEM images using Image J

This dislocation data is directly contradictory of the dislocation density obtained from XRD, suggesting that the XRD values have indeed been affected by factors other than dislocations, the TEM data giving a more accurate depiction.

5.5.6 Compression Testing

The samples, which were selected for compression testing, are shown in Table 8. Set A1 were the recovery samples, where the compression sample was taken from the centre. Set B1 were the compression samples taken from the edge of the recovery sample. Set A2 and B2 are taken from the centre and outside of the standard samples respectively.

Table 8: Table for compression testing samples, where the A1 and B1 CAI samples are taken from the centre and outside of the partial recovery target respectively. A2 and B2 are the CAI samples taken from the centre and outside of the standard target.

Set A1- Partial Recovery (Centre)	Set B1-Partial Recovery (Edge)	Set A2-Standard (Centre)	Set B2-Standard (Edge)
CG4A- 7.0 GPa	CG4B- 7.0 GPa	CG11A- 6.0 GPa	CG11B- 6.0 GPa
CG2A- 7.6 GPa	CG2B- 7.6 GPa	CG7A- 6.0 GPa	CG12B- 7.0 GPa
CG6A- 10.0 GPa	CG6B- 10.0 GPa	CG1A- 7.0 GPa	CG13B- 8.5 GPa
CG3A- 12.0 GPa	CG3B- 12.0 GPa	CG12A-7.0 GPa	CG9B- 12.0 GPa
CG5A- 13.6 GPa	CG5B- 13.6 GPa	CG13A- 8.5 GPa	CG14B- 12.0 GPa
CG10A- 14.0 GPa	CG10B 14.0 GPa	CG9A- 12.0 GPa	CG8B- 14.0 GPa
		CG14A- 12.0 GPa	
		CG8A- 14.0 GPa	

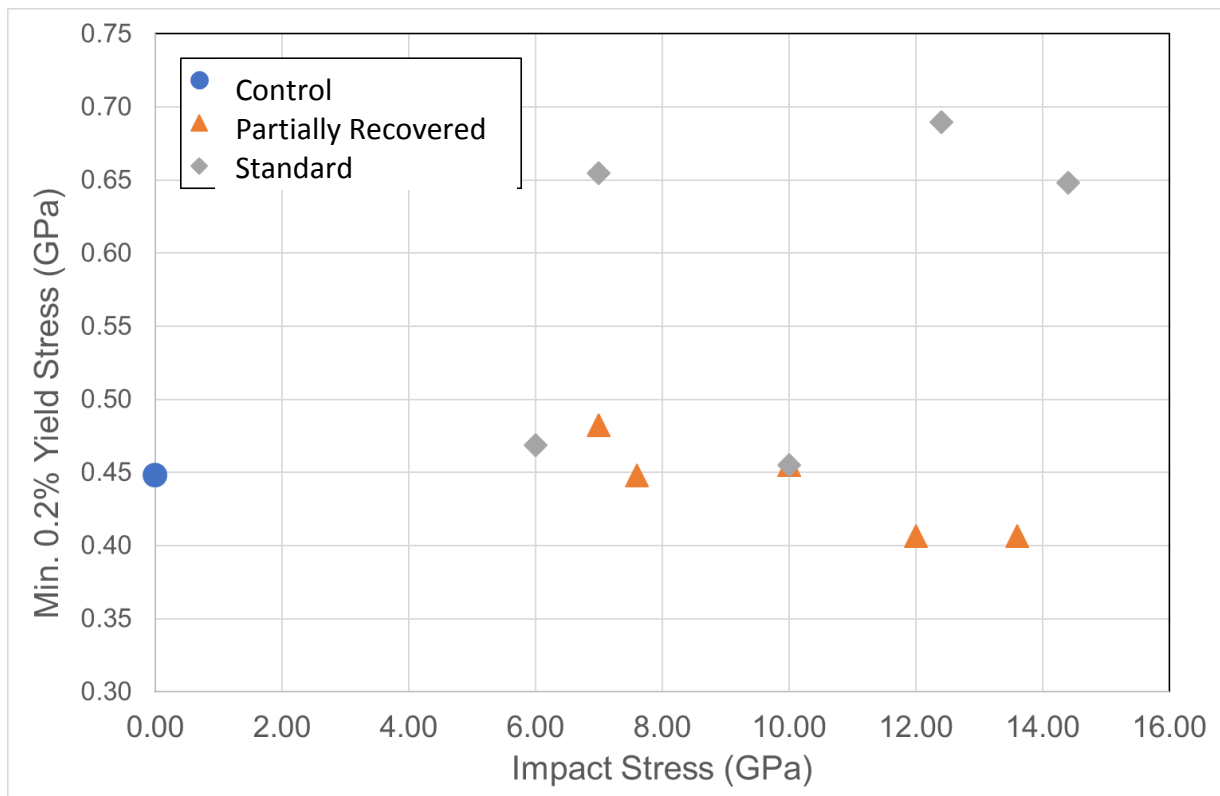


Figure 70: Yield stress (offset by 0.2%) versus the shock pressure (impact stress) for standard and recovered samples ranging from 6 GPa to 14.4 GPa. An offset of 0.2% is used where the transition from elastic to plastic behaviour is not easily detected. The offset yield point is the stress at which 0.2% plastic deformation occurs. These samples were an average of all of the results (edge and centre).

The initial results of compression testing are given in Figure 70, where the offset yield stress (a good indicator when the yield point is not clear) has been plotted against the shock pressure (impact stress). These results indicate a general trend that the yield stress is higher for the standard samples as opposed to the recovered samples. The recovered samples decrease overall by approximately 0.7 GPa of the yield stress from low shock pressure to high shock pressure, whereas the standard samples vary significantly more but generally increasing from 0.46 GPa to around 0.65 GPa.

The yield stress would be expected to be higher for the standard samples as they have undergone shock strengthening due to the build-up and saturation of dislocations, shown in the hardness data (Section 5.6), where saturation is shown to occur at

around 12 GPa at 300 Hv in Figure 76. As twinning also occurs, an increased number of twins would lead to a higher yield stress, as confirmed from the increased twinning density in the standard samples. Whereas the recovered shocked samples showed a consistent average micro hardness of around 220-240 Hv (Section 5.6 Figure 76)

In order to compare the partially recovered and standard samples to the control sample, it is necessary to displace the strain (along the x-axis) to get the true plastic strain through shock and release by [55], [191], [219], [220]:

$$\epsilon_{res} = 4/3 \ln (V/V_0) \quad \text{Equation 15}$$

Where: V = shocked volume
 V_0 = ambient specific volume
 ϵ_{res} = plastic strain

substituting in $(V/V_0) = (U_s - U_p)/U_s$

Where: U_p = Particle velocity
 U_s = Particle velocity
 ϵ_{res} = plastic strain

Hence:

$$\epsilon_{res} = 4/3 \ln ((U_s - U_p)/U_s) \quad \text{Equation 16}$$

As an example for a shot with a pressure of 7 GPa, the values for U_s and U_p would be $4.22 \text{ mm } \mu\text{s}^{-1}$ and $0.19 \text{ mm } \mu\text{s}^{-1}$ respectively. Therefore:

$$4/3 \ln ((4.22-0.19)/4.22) = - 0.061$$

An example of the Excel spreadsheet used to plot the offset graphs is given in Table 9, which shows the calculation for the true strain and then the strain offset. This then translates through to Figure 72 where each pressure gives a different offset of 0.061, 0.094 and 0.109 respectively.

Table 9: Example of Excel spreadsheet for offset calculation given in Equation 17

CG0A			CG4A				CG3A			
True Stress	Stress MPa	True Strain	True Stress	Stress MPa	True Strain	Strain Offset	True Stress	Stress MPa	True Strain	Strain Offset
psi		in/in	psi		in/in		psi		in/in	
-125.62	- 0.86612	0	18.52	0.13	0	0.061	11.06	0.08	0	0.094
-64.22	- 0.44278	0	39.53	0.27	0	0.061	49.47	0.34	0	0.094
-112.65	- 0.77669	0	28.73	0.20	0	0.061	47.27	0.33	0	0.094
-17.42	- 0.12011	0	57.61	0.40	0	0.061	47.10	0.32	0	0.094
-39.47	- 0.27214	0	72.15	0.50	0	0.061	52.05	0.36	0	0.094

Results overall indicate differences in the post shock hardening (explained in Figure 71) between the shock recovered samples with the momentum trapping targets and recovery rig in the back of the gun and the standard samples, which were taken without the recovery system. An example of shock versus work hardening interpretation is shown in Figure 71. The shock hardening is indicated at the yield point as indicated in the graph, where if the yield point is higher than the control sample, shock hardening

has occurred. Whereas, work hardening is observed from the gradient after the yield point of the graph. If the gradient is steeper than the control sample then there is a greater degree of work hardening but if the gradient is shallower, then there is a lower degree of work hardening.

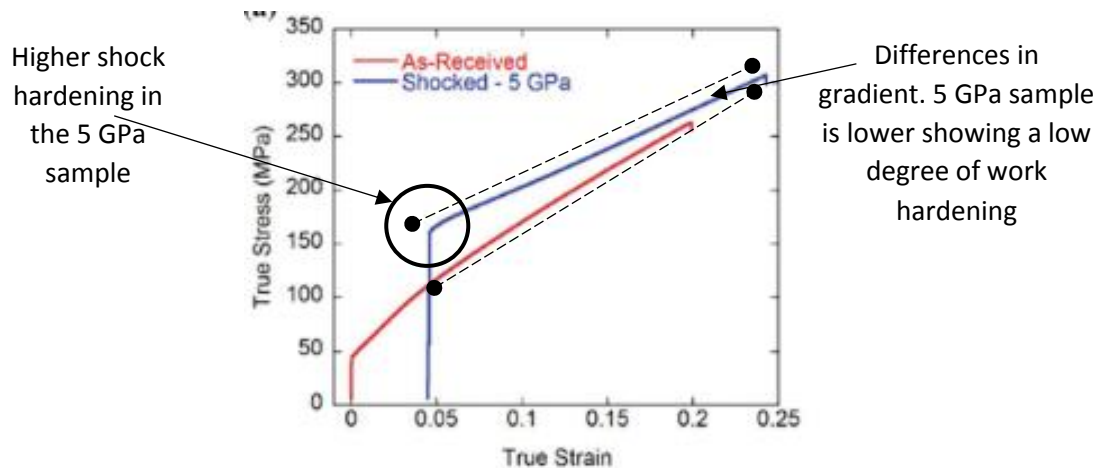


Figure 71: Example of a high degree of shock hardening and a low degree of work hardening from Millett et al. [191]. The yield point for the blue trace is higher than that of the red trace, indicating a higher degree of shock hardening. Whereas the gradient of the blue line past its yield point is lower than that of the as received sample showing a lower degree of work hardening.

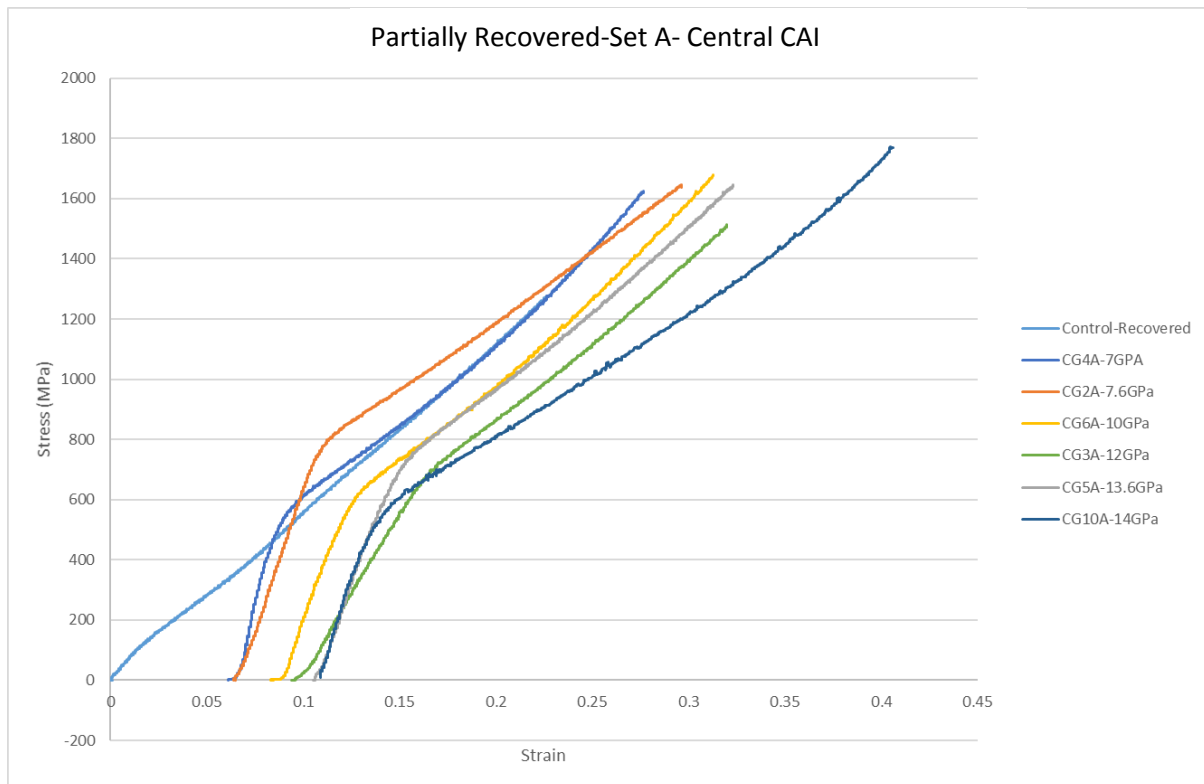


Figure 72: Partial recovery central set A compression testing results

In Figure 72 it can be seen that the work hardening for the recovered set A samples is approximately the same for all of the samples. Shock hardening is present in the 7.6 GPa and 7 GPa shocked samples, however, no other samples show shock hardening. The 7.6 GPa and 14 GPa samples show a lower degree of work hardening, suggesting that shock hardening was predominant in the deformation of the 7.6 GPa sample.

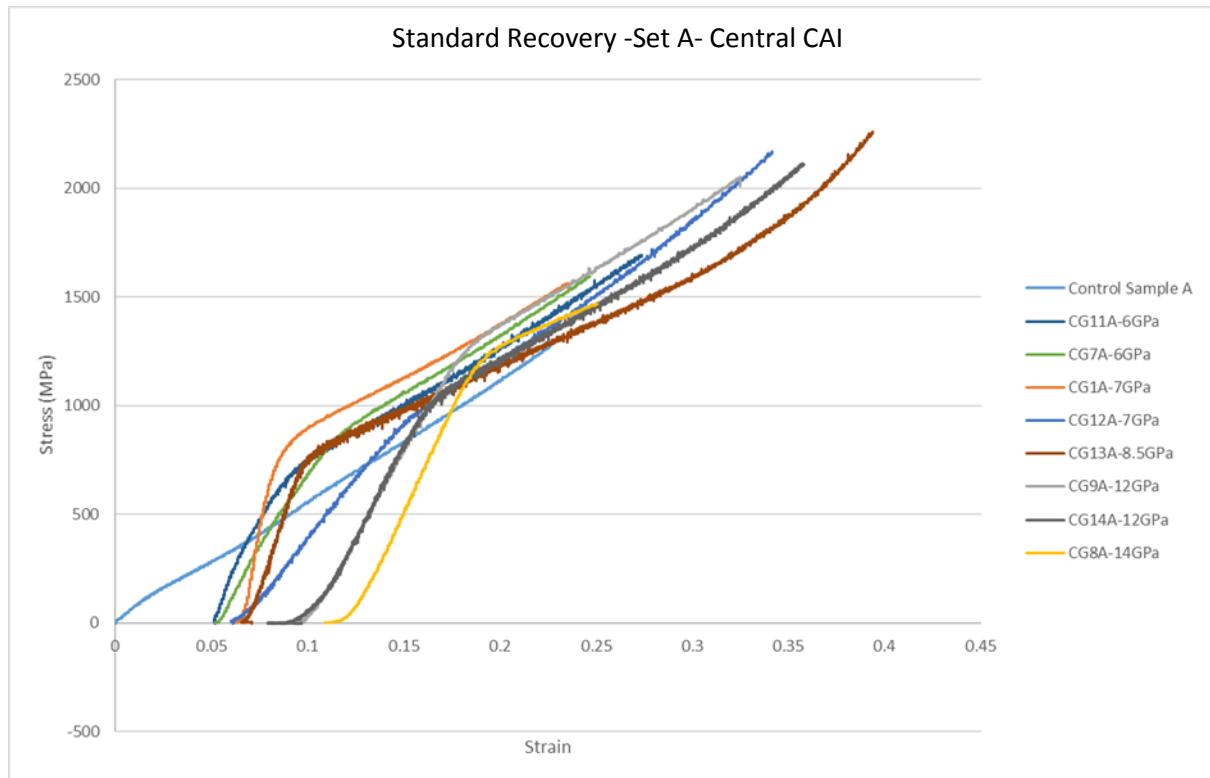


Figure 73: Partial recovery outer set B compression testing results

Figure 73 shows shock hardening in all of the samples, 7-14 GPa, however there is no work hardening. Figure 73 also shows no shock hardening in any of the standard samples but does show some work hardening in the 14 GPa sample, suggesting dislocation build up.

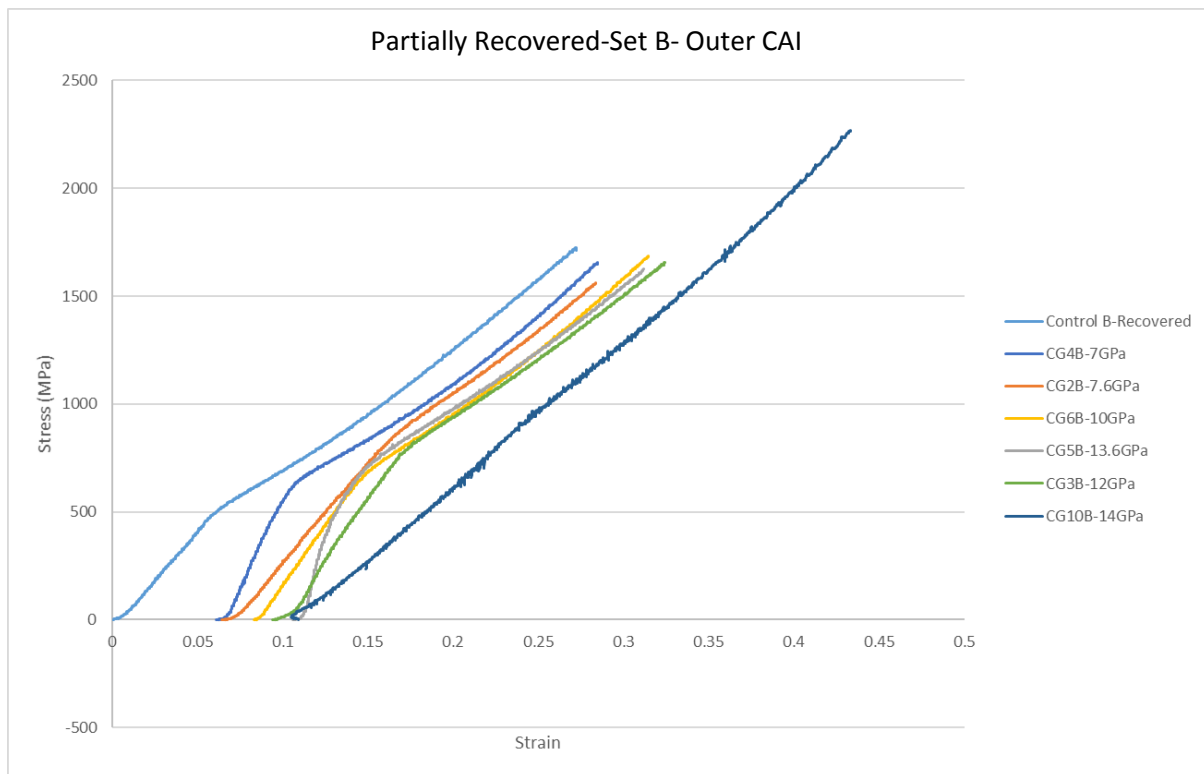


Figure 74: Compression testing of partially recovered samples set B

Figure 74 displays no shock hardening in the recovered samples and also no work hardening, therefore proving that recovery has been successfully carried out. If there were a significant level of reverberations inside the sample, there would be at least some shock or work hardening. The yield strength for the control sample is at 500 MPa and gradually increases with increasing pressure to 600 MPa for the 7 GPa shock recovered sample, 800 MPa for the 12 GPa sample and finally 900 MPa for the 14 GPa sample. There is no work hardening of the samples, suggesting similar amounts of dislocations in the control sample and shock recovered samples. The trace for CG10B does not show a clear elastic rise, which could indicate a different sampling rate or that the faces of the sample were not parallel.

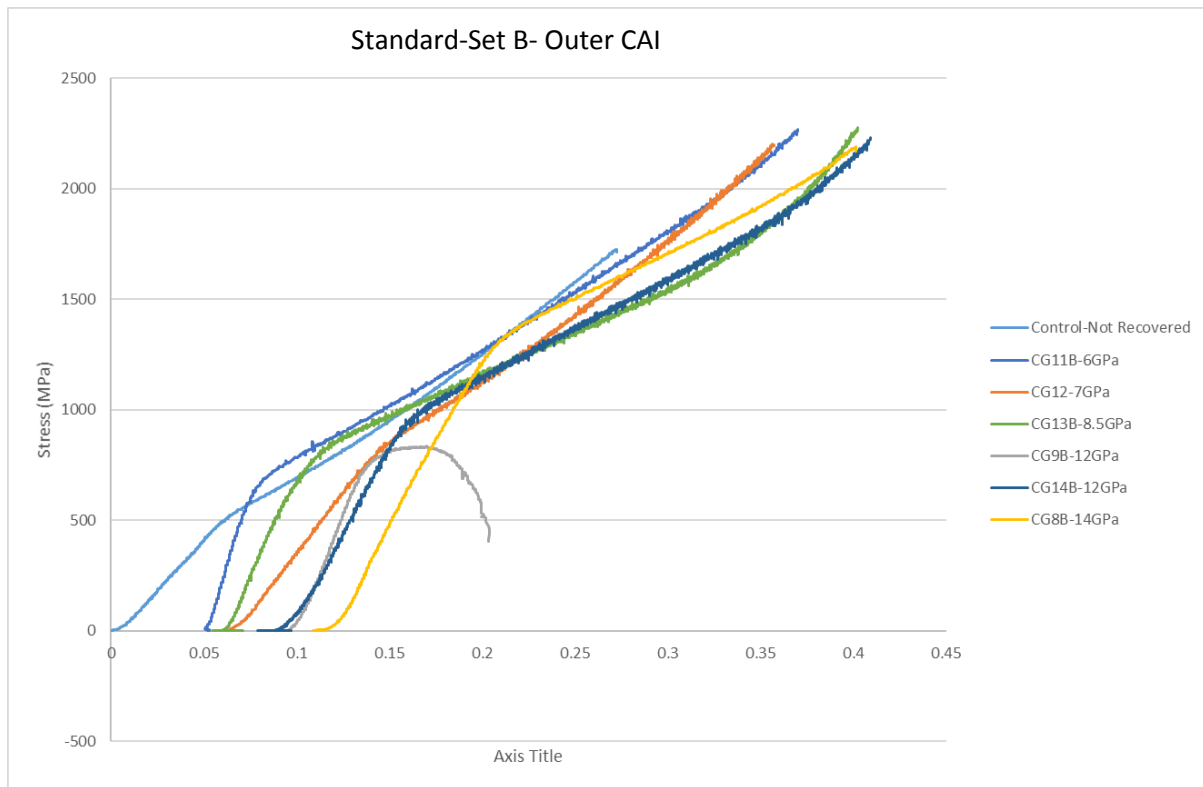


Figure 75: Compression testing of standard samples set B

In Figure 75 sample CG9B (12 GPa) underwent failure during the compression test, as can be seen from the gradual decrease in stress but increase in strain before failure occurs. As these samples were not recovered, it is likely that voids have formed due to tensile release within the sample causing the early failure compared to that of the other samples.

In both standard sets A and B (Figures 73 and 75) the work hardening is slightly higher above 8.5 GPa, however this is limited and would suggest that there may be some shock formed dislocations impeding newer dislocations post shock and preventing work hardening [221].

Overall, it is interesting to note that in all but one (7 GPa outer sample) of the standard recovery samples showed shock hardening, which is reflected in the data obtained from both the qualitative analysis of the TEM images, showing large amounts of dislocation tangles, deformation bands and twinning and the quantitative dislocation density figures calculated using Image J. Further to this, tensile releases were confirmed visually during microscopy in the centre of the sample, which would create areas of high stress and strain, leading to shock hardening.

Whereas, in the partially recovered samples, only two samples showed shock hardening but with a lesser degree of work hardening. The 7.6 GPa sample also showed a higher twinning density than that of the 10 GPa experiments and a higher dislocation density from the XRD data, perhaps suggesting that the partial recovery was not fully successful, especially as the other partial recovery data did not show shock hardening, including the outer sample from the 7.6 GPa experiment. Overall, partial recovery was successful in reducing reverberations, which is also reflected in the lower yield strength of the metal compared to standard recovery.

In order to verify these conclusions on work and shock hardening from the compression testing, further information is required on the hardness of the material. Hardness testing can provide comparative information for each of the samples, to show any increases or decreases in the hardness of the material.

5.5.7 Hardness testing

Microhardness testing was carried out on each stainless steel sample. This was done in a 3x7 array in 2 mm increments so that individual changes across the sample can

be observed. These values were then averaged to give one overall hardness value of the sample for comparison with the other samples. Once these values were obtained they were plotted as below average microhardness versus shock pressure. The hardness testing was carried out with a 300 g mass. The Vickers test scales proportionally, so values obtained would be equal using a weight between 100 g and 1000 g, provided the corners of the pyramid can be read accurately by the user. Table 10 provides all of the average hardness values from the array, for each type of experiment, while Figure 76 shows the same data plotted as a scatter graph.

Table 10: Hardness testing data, showing each type of recovery and the average hardness value obtained from a microhardness array across the sample

Code	Shock Pressure	Average Hardness	Notes
CG0	0.0	183.7	Control
CG2	7.6	219.7	Partial Recovery
CG3	12.0	244.1	
CG4	7.0	220.8	
CG5	13.6	236.5	
CG6	10.0	208.9	
CG18	16.0	241.0	
CG21	15.0	216.8	
CG24	6.2	215.3	
CG25	10.4	314.3	
CG26	10.4	256.3	
CG29	10.7	267.3	
CG7	6.0	245.4	Standard
CG8	14.4	304.9	
CG9	12.4	304.6	
CG17	16.0	301.4	
CG27	12.8	314.0	
CG19	15.0	224.8	2x2mm
CG22	7.0	189.7	1x4mm
CG20	15.0	228.3	
CG23	7.0	184.1	Full Recovery
CG30A	7.0	185.3	

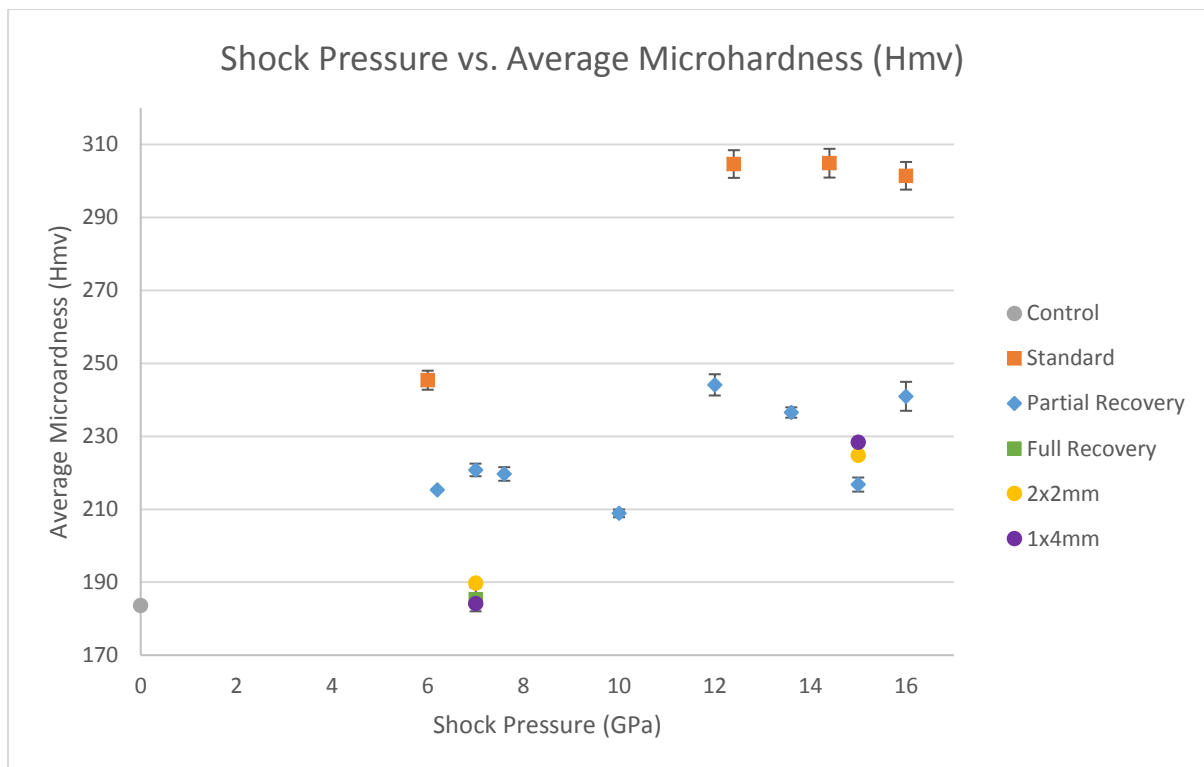


Figure 76: Graph showing average microhardness of each shocked sample against its respective shock pressure

The results in Figure 76 indicate that as the shock pressure increases so does the average microhardness of the sample. There is also a difference between the standard and partially recovered samples. The standard samples have a microhardness, which is between 300 Hv at approximately 7 GPa and 60 Hmv at 14 GPa.

The modified samples with spall plates of 2x2 mm and 1x4 mm are of interest compared to the 4x1 mm recovery sample used in the majority of experiments. On the graph the 1x4 mm plate recovery target shows a lower hardness than the current target (4 x 1 mm) used at 7 GPa pressure and an average hardness that matches the recovery at 15 GPa. Experimentally, due to the similarity in mass and momentum, the

recovery target reimpacted with the 1x4 mm spall plate, which would lead to a higher hardness.

The 2x2 mm sample shows a consistent lower hardness at both 7 GPa and 15 GPa compared to that of the current recovery target. As the plates had a different mass to the recovery target, they did not appear to reimpact. Further to this, as the plates were thicker the momentum trapping could have been more successful as there was more time for the plates to separate before the shock wave transmitted back into the recovery target. Also, there would have been less shock reflections because there were half the number of spall plates, decreasing the number of dislocations and therefore decreasing the overall hardness. Therefore it can be inferred that larger numbers of reflections lead to increased dislocations causing increased hardness of the material.

The full recovery sample has a value of 185.3 ± 3.3 Hmv compared to that of the control sample which has a microhardness value of 183.7 ± 1.7 Hmv. This confirms that the full recovery was successful with just a 1D shock wave as it suggests that less microstructural changes have occurred due to less reverberations. It also provides a comparison for the partial recovery, which has a value of 220.8 ± 1.73 Hmv and the standard target that has a 245.4 ± 2.6 Hmv. Also, due to the volume of measurements across each sample, the hardness testing has a much lower error than the other analysis techniques, giving an accurate representation of the sample as a whole, linking into the twinning density, which shows an increase with the samples reflective of the increases in hardness. The increased yield strength with increasing shock pressure in the standard samples is reflected in the hardness results also.

Overall, from the hardness testing it can be seen that the partial recovery has a higher hardness from the full recovery, which confirms that the theoretical x-t diagrams are correct in terms of the reverberations. The full recovery sample is within the same error of hardness as the control sample, which is reflected in having a low number of twins per grain and a grain size consistent with the control sample. Although CAI testing was unable to be carried out, it would be expected that there would be little to no shock or work hardening within the sample.

Higgins *et al.* found hardness to be a good indicator for different experiments carried out on copper [175]. They found increases in hardness with increasing shock pressure and with different treatments on the copper as well as different recovery techniques. They found that this increase in hardness originated from increased dislocation density in the copper caused by dislocation cells, which form when it is shocked. Similarly, the increase in hardness in this case can be seen to be caused by increased shock hardening in the standard samples, where many reverberations will have occurred.

Further to this, with increasing shock pressure, increased twinning also occurred. The dislocation density data obtained from the XRD was not shown to have a trend in increasing or decreasing. However, the dislocation density data obtained from the TEM images shows an increase in dislocation density, which would lead to increased hardening. The predicted temperature rise from the computer models would be too low causing an annealing effect in the metal, therefore grain size would not be expected to change by a large amount, which is reflected in the XRD data not showing

a large change in dislocation density, as it is the inverse of the grain size as shown in Equation 13.

Hardness increases with dislocation density. As more dislocations form and propagate through the material, this causes the overall hardness to increase. Further to this, twinning can also increase hardness of the material. However, although hardness testing can show useful insights into changes in strength and hardness of the material, the cause of the hardness cannot be obtained. Therefore, by combining the information obtained on dislocation density from XRD, the plasticity data from the compression testing and the twinning density from optical microscopy a much bigger picture can be built up of what deformation is occurring in the material. The discussion between these relationships are going to be developed further in Chapter 6: Results III: Computer Modelling.

5.6 Summary

Overall, a series of 21 flyer-plate impact experiments were carried out on stainless steel 21-6-9. The recovery technique was validated by comparing the analytical results from the shock recovered momentum trapping samples, as well as more qualitative analysis, whereby spall was present in the standard samples, which would be expected from tensile release waves, compared to the recovery samples, which did not show evidence of spall.

Further to this, in compression testing, the standard samples showed evidence of both shock and work hardening, whereas the recovery samples showed neither shock nor

work hardening. This is also evidenced in the hardness testing results, where the recovered samples only show a range of 20 HmV but the standard samples show a range of 60 HmV, three times as much.

Twinning density indicates an increase from 4 twins per grain in the 7 GPa recovered sample to 18 twins per grain in the 18 GPa recovered sample. In the standard samples, at 6 GPa there is 10 twins per grain up to 32 twins per grain at 16 GPa. This evidences that twinning is the predominant form of deformation in stainless steel 21-6-9.

In Chapter 6 the results from the stainless steel 21-6-9 are compared with predictions from the computer models for each scenario, to see the effectiveness of comparing a standard recovery with multiple reverberations versus a partial recovery with some reflections and finally a fully one-dimensional recovered shot. With regards to the computer models, the main indicator used for the effectiveness of the recovery is effective plastic strain. In these results, in Section 5.4.5 direct comparisons can be made from the stress strain curves produced from CAI testing. However, due to the large error margins caused by a small sample size of 2 mm diameter with a depth of 5 mm other techniques were required to measure the material strength evolution of the steel. By combining all of these techniques together, a complex and reliable picture of the microstructural evolution of the stainless steel can be obtained for use in material response prediction of the material under shock between pressures of 6 GPa up to 16 GPa.

6 RESULTS III: COMPUTER MODELLING

6.1 Introduction

Computer modelling can provide information on complex structural behaviour. Initially, experimental validation of the computer models was required to verify their accuracy, this has been carried out in a range of hydrodynamic applications including Maudlin *et al.* who validated their computer models using the Taylor impact test and Panov *et al.* who wanted a failure model to accurately reflect failure in tensile aluminium alloys [222], [223]. Panov *et al.* found that an accurate failure model, combined with the mechanical threshold stress model and an accurate EoS reflected empirical data of dynamic tensile failure [223]. Validating computer models is necessary to develop ever more accurate descriptions of material strength for high performance applications across multiple industries [222].

ANSYS® Autodyn was selected to run these computer models as it runs hydrocode models, allowing prediction of the behaviours of the shocked metals including stress, plastic strain and temperature. This can be predicted using infinitesimally small gauges which can be placed anywhere in the model, avoiding the restrictions of experimental gauges, which can affect recovery as well as the difficulty in placing the gauge within the target. This research involved having several target iterations from a single standard plate, all the way up to a full recovery target. Modifications of the rear four 1 mm spall plates were then conducted with two 2 mm spall plates and one 4 mm spall plate. This was to investigate the effects of changing the interfaces on the tensile release wave from the recovery target, to look at the changes in material deformation.

The 2D Lagrangian model was selected as it is a simplistic model with a comparatively lower run time than that of a 3D model, as there are less nodes. The symmetry of the modelling was axial as opposed to planar as axial symmetry repeats around the 2D object allowing for the circular shape of the targets to be constructed, whereas planar extends the model to infinity in the z-axis, which would not depict the experiment accurately.

Lagrangian modelling was chosen over Eulerian as it can be more efficient with regards to boundary definitions, especially where there are interests in the properties at the interface or boundaries of a model, as the grid is plotted onto the object rather than plotting the object onto the mesh. Eulerian would be preferential for explosions due to the fluid flow over the mesh. However, as the shock impact experiments with metals are much more confined the Eulerian method would be unnecessary.

6.2 Fidelity Study of Mesh Size

A fidelity study was carried out on the mesh size of the targets. As previously discussed, the mesh size can affect the data, making the model inaccurate if the resolution is too low but having longer run times with a finer, high resolution mesh.

The models used were a first approximation of a recovery target consisting of a flyer plate impacting a target, which had one guard ring around the outside and one plate backing it. This was to simplify the output readings, in order to gain an idea of how different mesh sizes would work with the recovery technique. The velocity was fixed at 370 ms^{-1} , as experimental data is available at 370 ms^{-1} (7 GPa) for each type of experiment. Figure 77 shows the placement of the gauges within the model. Two and

four are placed in the centre and on the edge of the recovery target, eight and six are placed on the spall plate and 15 and 13 are on the recovery ring.

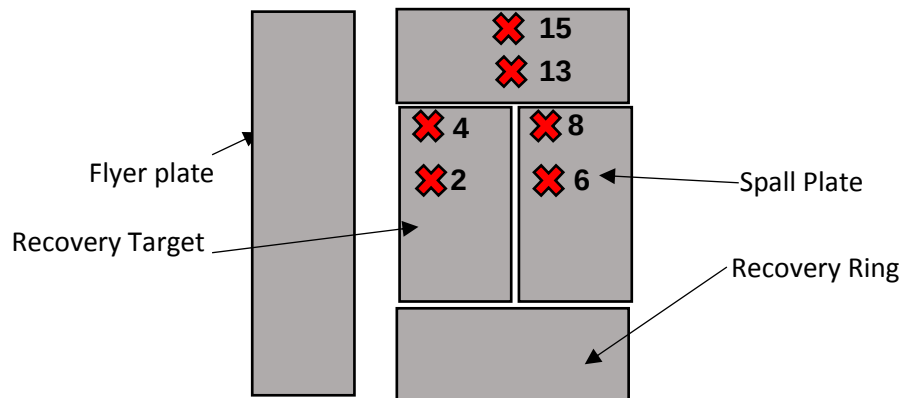


Figure 77: Flyer plate impacting target with one ring and one spall plate. Full diagram shown. Red crosses indicate gauge placement.

Overall the run times for each mesh size were: 2h 38m for the 0.1 mm mesh, 9h 9m for the 0.05 mm mesh and 31h 57m for the 0.03 mm mesh, see Table 11. These were respectively at 10,100 cycles, 20,200 cycles and 30,300 cycles, as a higher number of cycles is needed for a higher resolution mesh to counter any effects for the erosion and replacement of the mesh itself [152]. Further to this, more cells in the mesh mean more calculations and a smaller time step required [152]. These run times will vary depending upon the computer specifications.

Table 11: Mesh size in Lagrangian model versus the computational run time

Mesh Size	Computational Run Time
0.1mm	2h38m
0.05mm	9h9m
0.03mm	31h57m

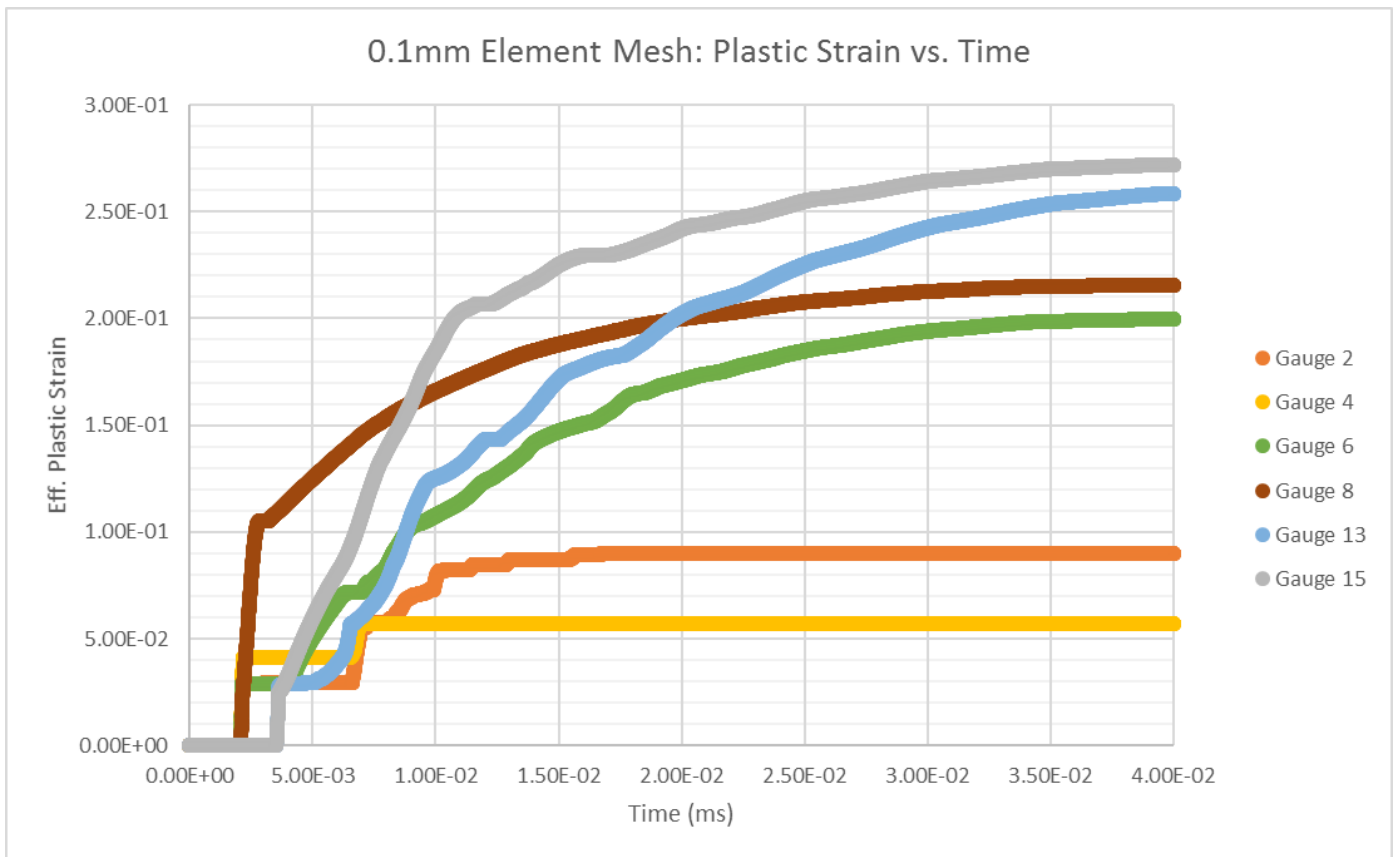


Figure 78: Graph from lowest resolution mesh (0.1mm) displaying plastic strain. Gauge locations are shown in Figure 76.

The graphs shown in Figures 78-80, from this fidelity testing would indicate that the models all have very similar profiles. However, gauge four on the lowest resolution mesh (0.1 mm) has a completely different profile to gauge four on the medium (0.05 mm) and highest resolution (0.03 mm) meshes. Gauge four is nearest to a boundary, hence in the lowest resolution mesh, the value from a particular node will be averaged. Therefore, the node may have a lower value for the overall area it covers, rather than

a higher value as it is at the boundary. The boundary may be under a higher rate of strain, due to impingement of guard rings or reflections occurring at the boundary.

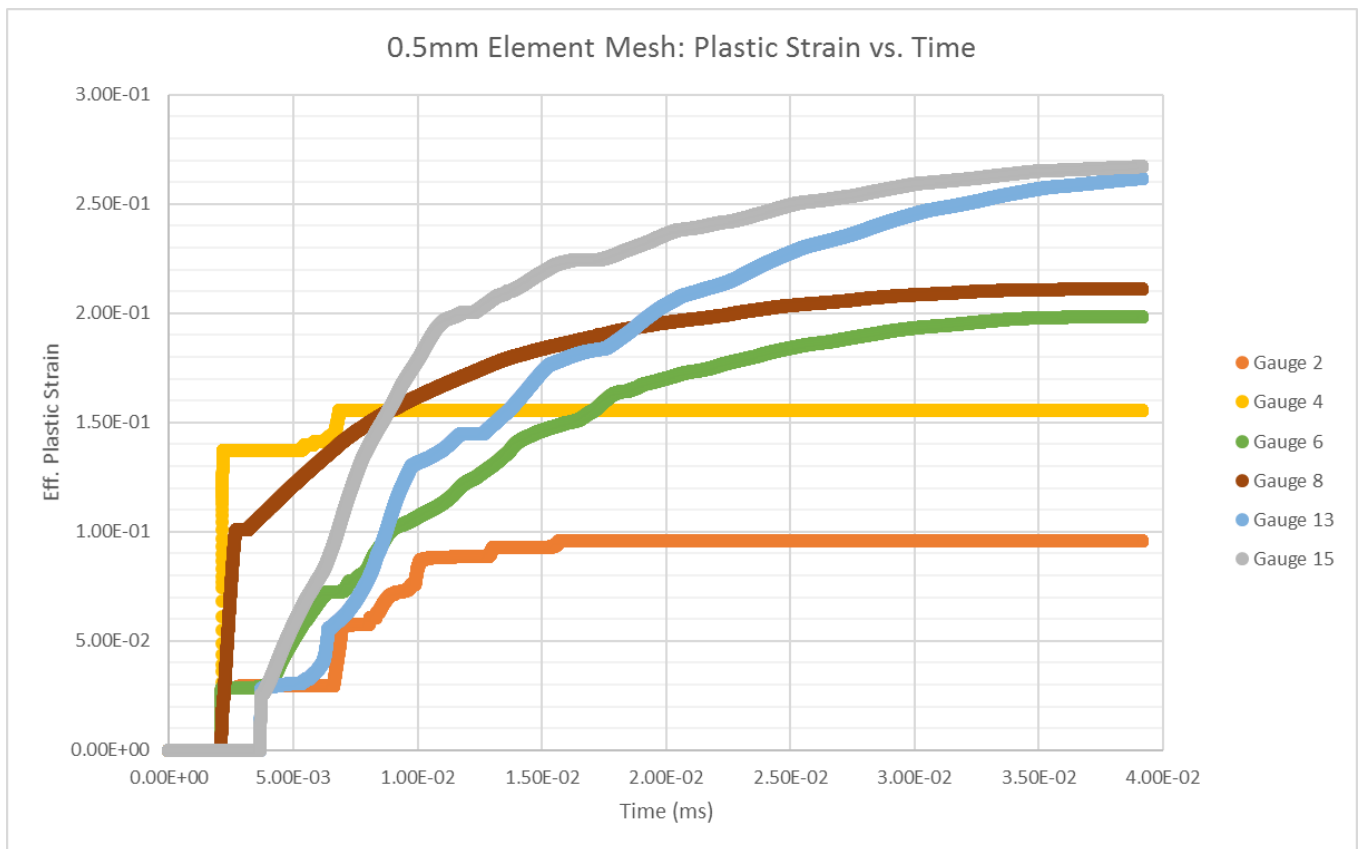


Figure 79: Graph from medium resolution mesh (0.05 mm), showing plastic strain vs. time

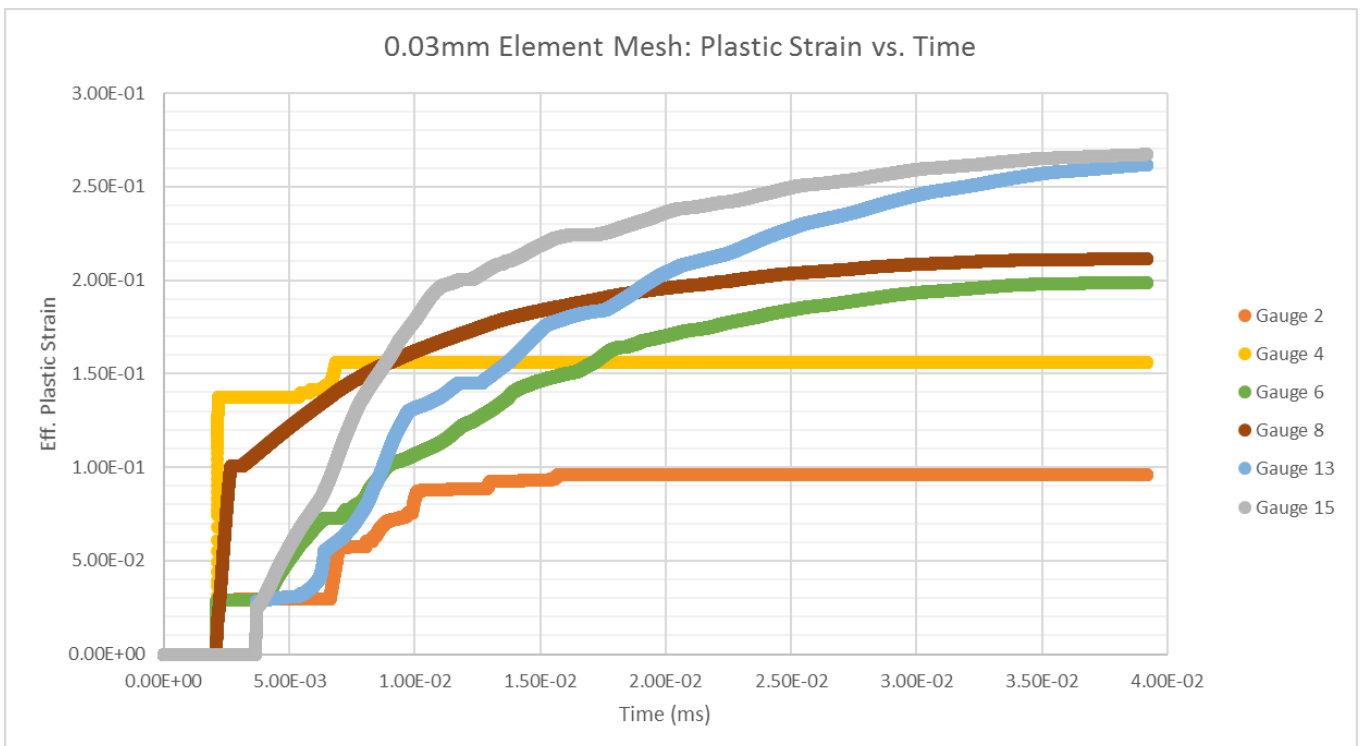


Figure 80: Graph from high resolution mesh (0.03 mm), showing plastic strain vs. time

The 0.05 mm mesh was the most suitable as it has been balanced between accuracy and run time (i.e. between a finer mesh but having a longer run time). The accuracy was obtained by reading the gauge values. The main difference is that the plastic strain in the 0.1 mm size mesh differs in gauge four, compared to the 0.05 mm and 0.03 mm mesh size as gauge four is near a boundary so where the resolution is lower, the strain may have not been accurately calculated. The 0.05 mm mesh allows for an appropriate separation between each component of the complex target, so that the desired spacing of 0.1 mm can be achieved, which is within the tolerances of the manufacture of the experimental samples and based upon the literature from Vignjevic et al. [60].

6.3 Validation of Models

To validate the computer models with the empirical data, it was necessary to create a simple single plate impact experiment, to highlight the reason needed for an appropriate recovery method. Then, this plate impact was developed into a simple recovery experiment consisting of one guard ring and one spall plate. A model was then created to match the experimental design of the target in order to be able to compare and validate the computational methods with the experimental ones.

A model was also created to reflect the set-up of the standard experiments for comparative purposes. In all of these examples, the Steinberg-Guinan model for stainless steel 21-6-9 was used within ANSYS® Autodyn with data gathered from LLNL experiments [64], [105], [152]. An erosion factor of 2 was used and an impact velocity of 500 ms^{-1} to simplify any forthcoming calculations. A velocity of 500 ms^{-1}

was selected as it is the midpoint for all of the experiments carried out between $\sim 300 \text{ ms}^{-1}$ and 700 ms^{-1} , as well as being used for the x-t diagrams, which show the anticipated spall plane/overlap of tensile releases. The erosion factor was selected due to the high strain rate, as it has previously been used in similar ballistic calculations, where it was determined that an erosion factor higher than 1.0 matched empirical data [155]. A factor of two means that when the geometric strain reaches 200% the cell gets deleted and then replaced with a new cell with 0 deformation.

There was also a need to determine whether the thickness of and number of spall plates was appropriate. The longitudinal momentum traps were selected as, due to the shorter length, have significantly more impact on the target than the latitudinal targets, as the shock will take longer to travel back from them. Further to this, Vignjevic's work helped to reduce the impingement problem of the latitudinal targets, which would be the primary concern [60].

In the case of the longitudinal spall plates, they needed to be numerous enough to help trap the momentum but thick enough that they have time to separate before the shock wave has time to reflect back into the recovery target. To simplify the experiment, the rear four 1 mm thick spall plates were selected and two iterations were created; one where they were replaced with two 2 mm thick spall plates and the other where they were replaced with one 4 mm thick spall plate. This was to see if the plastic strain, one of the indicators of how well a target has recovered, could be reduced any further. Having less spall plates could also potentially mitigate any tolerances within manufacture that could add up with a high multitude of smaller parts. It is also more efficient to have the targets manufactured.

Each set up is given below, with its respective plastic strain graph. Figure 81 shows the single plate impact, where gauges are placed near the shock face (two and four) and to the rear face (six and eight) and finally in the centre and edge of the target (13 and 15).

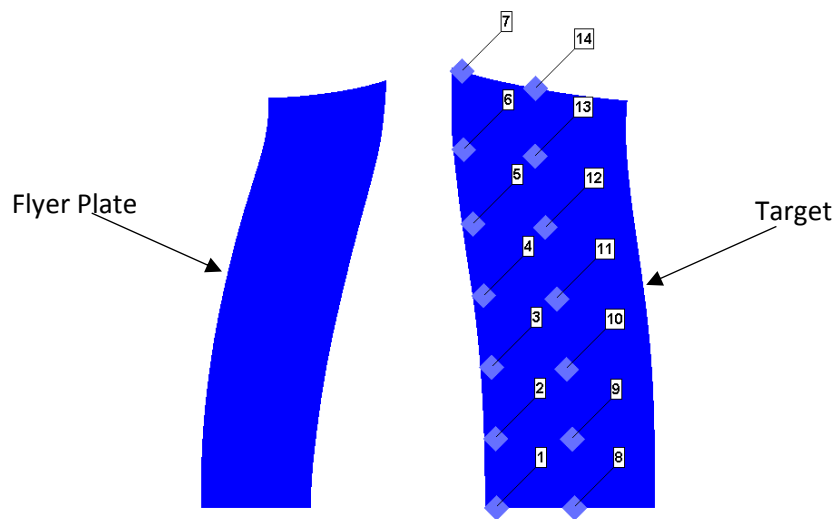


Figure 81: Single plate impact modelled using ANSYS® Autodyn. Gauges are shown in the diagram.

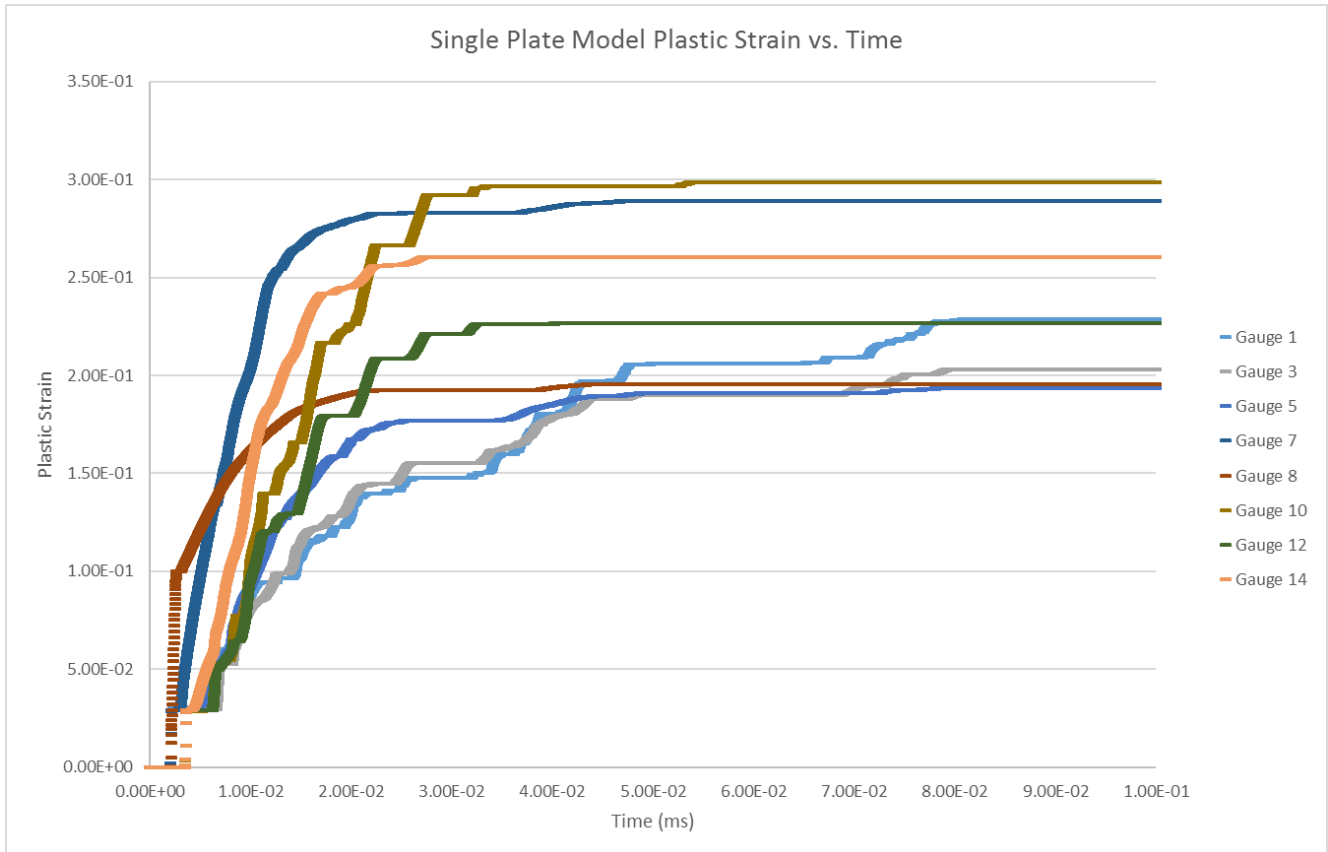


Figure 82: Plastic strain produced from a single plate impact. Gauges 1-14 are placed throughout the target as indicated in Figure 80. The lowest plastic strain is in Gauges 3, 5 and 8 (within the 1D zone) and the highest in Gauge 7, 10 and 14 (edge of target and centre with tensile releases).

As can be seen in Figure 81, fourteen gauges were placed within the single plate, this allowed for an overview of what occurred within the plate at different times. Within this scenario plastic strain is used as an indicator for recovery, which is useful for understanding the chosen recovery target. Individually, it is useful to look at where high and low strain areas are expected to be within the model, which will be compared directly using compression testing in the case of the experimental recovered and standard models.

Figure 82, shows that there are high areas of plastic strain in the model particularly at Gauges 5, 10 and 14. This is because they are located halfway through the model, therefore tensile releases will reflect off the target surfaces and meet in the centre, forming a spall plane. This is depicted in the x-t diagrams in Figure 34 and Figure 35 (pages 83 and 84).

Recovery involves mitigating these spall effects as much as possible, hence the need for momentum traps within the target, to prevent these reflections and tensile releases from causing dense areas of plastic deformation and spall.

Figure 83 displays a standard impact. The standard impact was carried out in two ways. One method was to attach a 15 mm diameter, 9 mm thick circular target to a 1 mm backing plate, which is what is shown in Figure 84. The other method consisted of a 15 mm diameter, 10 mm thick circular target mounted in a PMMA square using epoxy resin. The second method was developed as the first method caused the target to be confined, meaning that the plastic strain was higher than it would be in a standard recovery, hence the second method was used, to eliminate this confinement issue. Figure 84 displays the plastic strain in the standard impact.

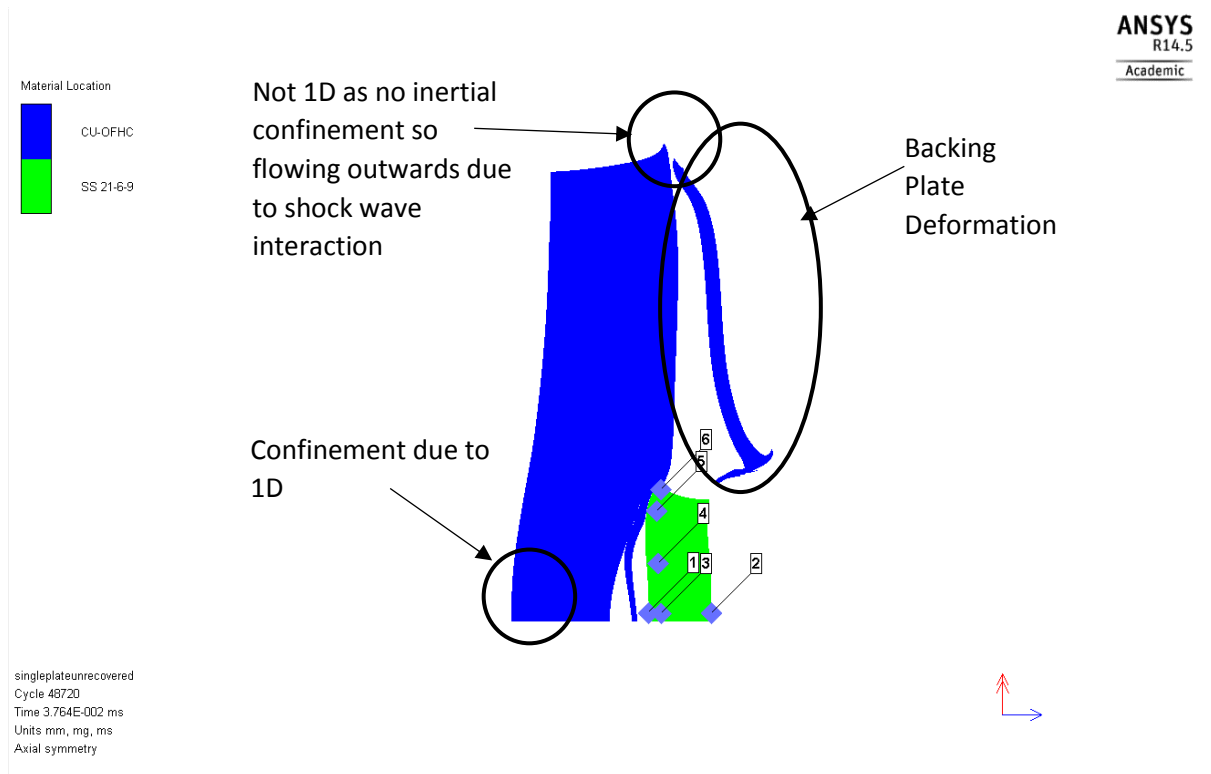


Figure 83: Standard impact, where standard piece is attached to a 1mm thick Cu flyer plate. Gauge numbers are shown in the diagram.

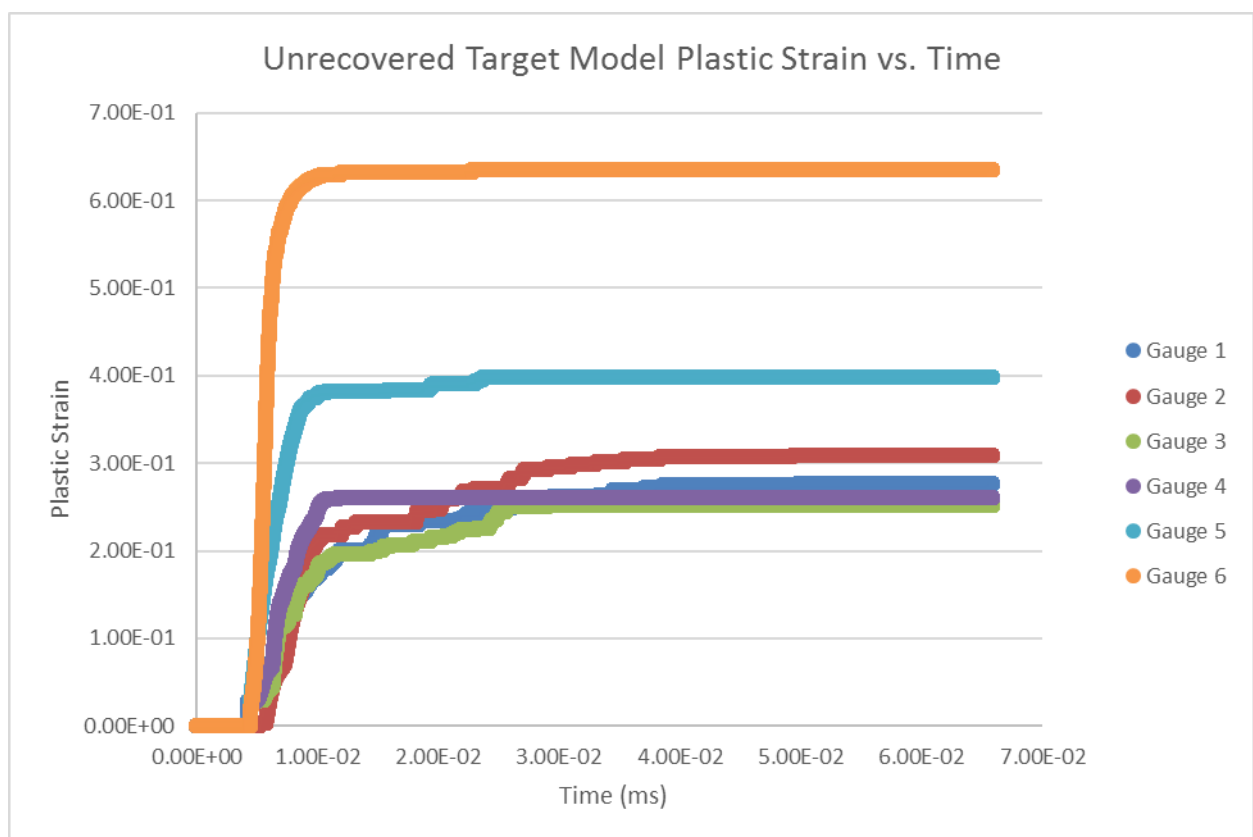


Figure 84: Plastic strain graph produced from standard target

Due to the size of the sample, six gauges were placed onto the modelled target (Figure 83). Gauge one and gauge two were the front and rear gauges and three, four, five and six were set 0.5 mm back and vertically up the target to gain insight into what was occurring laterally. Further to this, the particle velocity and displacement can also be obtained at these points to give comparisons as to how well a recovery target has worked by checking whether the target has been impinged on the outside by any guard rings.

In this model, gauge six shows the highest level of plastic strain. When Figure 83 is consulted, it can be seen that where the flyer has impacted with the flyer plate, the top of the target has also remained in contact with it. This was confirmed experimentally where some of the standard targets were found still attached to the target plate. This has naturally caused a large amount of plastic deformation, which is why the strain in this region is twice as high as the regions indicated by the other gauges.

Gauges one, two, three and four have lower plastic strain and appear to have releases from the flyer plate, where five and six are impinged by the plastic flow of the flyer and flyer plate around the edges of the target. This is likely due to the shock releases and momentum separating the two but trapping the region around the edge of the target. To mitigate this, experimentally, samples were set using epoxy resin into a PMMA disc, so that the flyer plate issue would be lessened.

Figure 85 shows the model created with one guard ring and one spall plate. Where the thickness of the recovery sample and the spall plate is 7.5 mm. The graph in Figure

86 begins to show signs of recovery and highlights the need for complex target design which includes guard rings and spall plates.

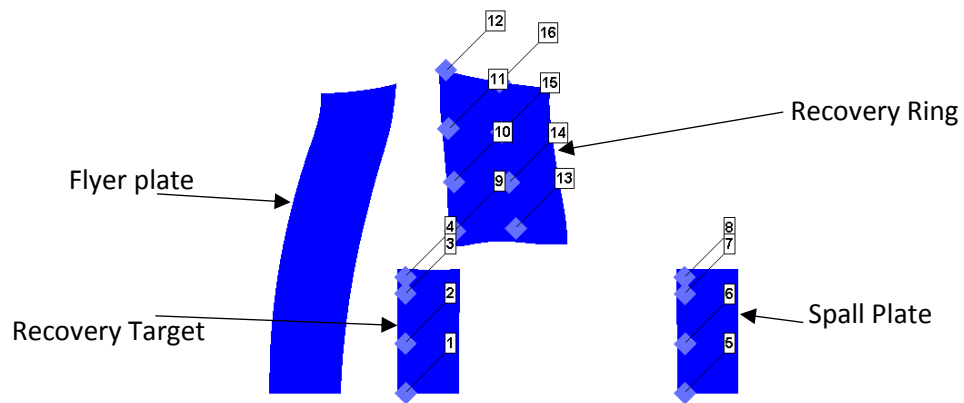


Figure 85: Impact displaying one guard ring and one spall plate this model was carried out under axial symmetry, so shows the upper half of the 2D model. Gauge numbers are shown in the diagram

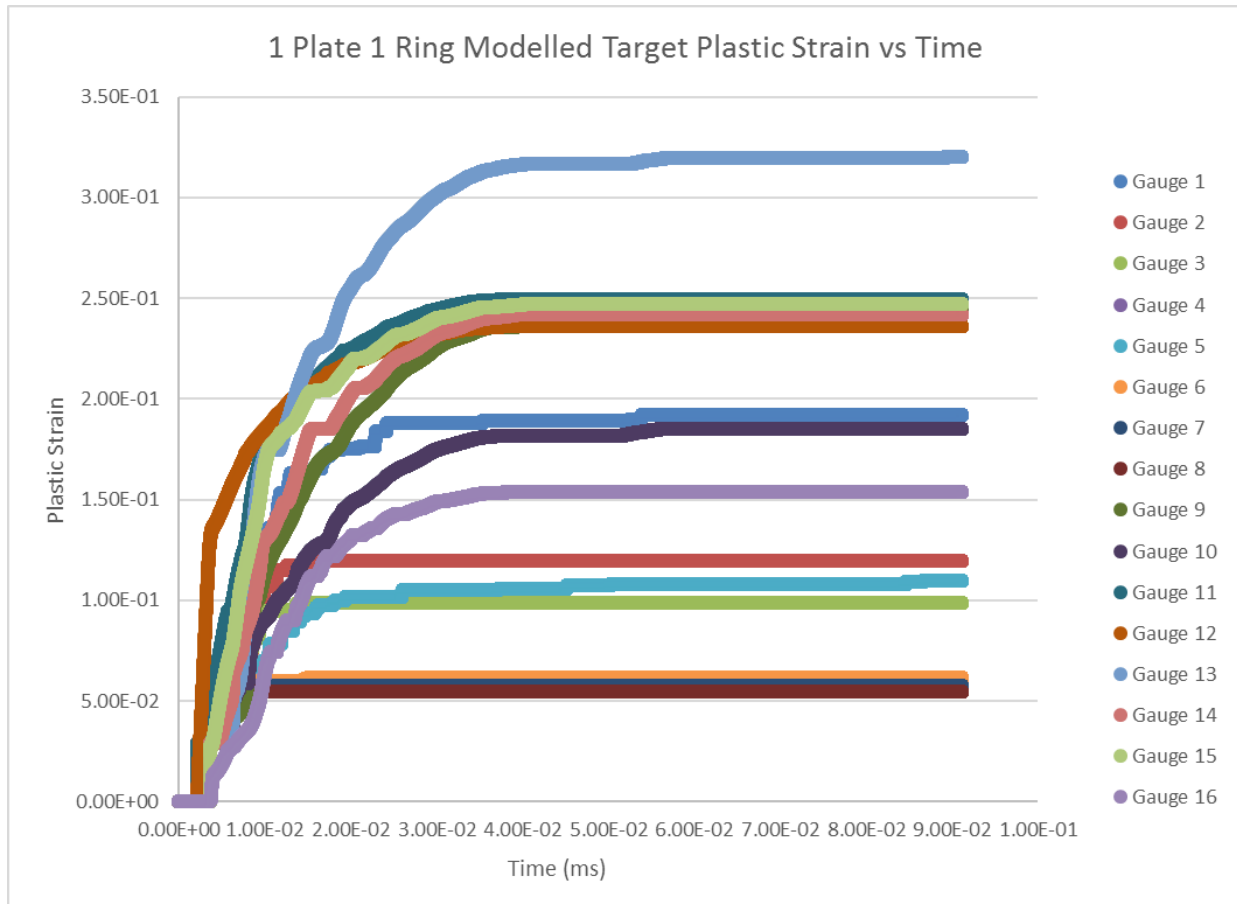


Figure 86: Graph showing plastic strain for flyer impact with one guard ring and one flyer plate

Although the highest plastic strain is around 3.5×10^{-1} like in the single plate impact. The lowest plastic strain is four times less than that of the lowest plastic strain within the single plate impact, at around 5×10^{-2} , with a significant amount of variation in-between. In this case, the recovery target is to the rear or right hand side, this can be confirmed with the lowest amounts of plastic strain within the target, as the front spall plate is more likely to remain in contact with the flyer for longer, hence more reflections and more strains occurring within the spall plate.

The highest strain values are generally within the guard plate, this is due to both latitudinal and longitudinal shock reloading. The value for gauge 16 is lower as it is on the boundary edge in the middle of the guard plate, where most strain will be central

within the guard plate due to tensile release, which is shown with the high level of strain in gauge 13. However, overall the lowest values are shown within the recovery target, showing the importance of momentum trapping within recovery.

The computer model for the initial experimental design for the full recovery stainless steel target (the 4x1 mm configuration) is shown in Figure 87. The gaps between the rings and plates were made to match the tolerance level during manufacture; a value of 0.1 mm. The recovery sample itself is given by gauges 8, 9 and 10. These have the lowest plastic strain occurring within them. Gauge 8 has a higher plastic strain, which is probably due to it being in the centre of the sample so will suffer from more reflections than gauges 9 and 10. However, the overall value is low and shows the effects of recovery upon plastic strain.

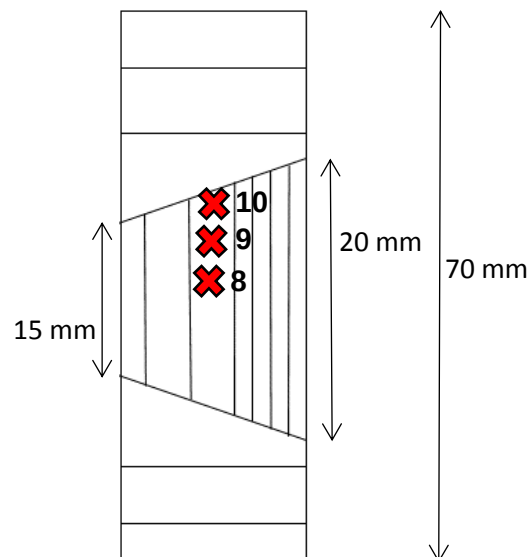


Figure 87: Full recovery (current model with four 1 mm spall plates to the rear), showing gauge locations.

The overall thickness of the target is 15 mm.

Figure 88 shows the plastic strain for the full recovery scenario, where the plastic strain varies between 0.06 and 1.11. Gauge 8 has a comparatively higher plastic strain due to boundary limitations of the model.

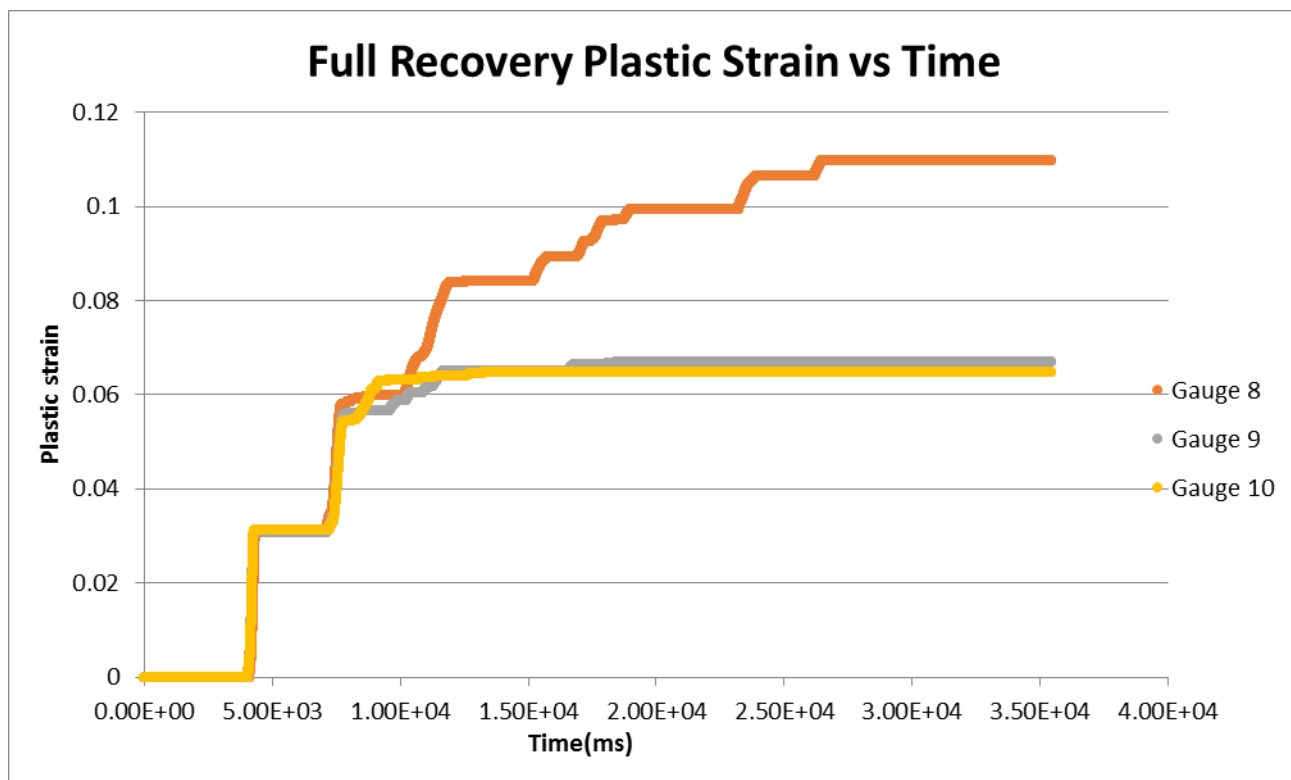


Figure 88: Plastic strain shown from modelled full recovery event

6.4 Validation of Failure Model

One of the three requirements of a computer model is that of a failure model. A failure model is a model that depicts how a material would fail empirically. To simplify matters, the initial validation models were run without a failure model. In order to correct for this, a plastic strain failure model was added and run on the full recovery stainless steel 21-6-9 target to compare differences in the plastic strain. The results are shown in Figure 89. There is minimal difference, approximately less than 5% seen in the plastic strain with the plastic strain failure model versus without. Therefore, having run

several failure models including the Johnson-Cook and stress-strain failure model, a failure model was not determined necessary within the bounds of this project, as the Steinberg-Guinan model used within the stainless steel was adequate for predictions.

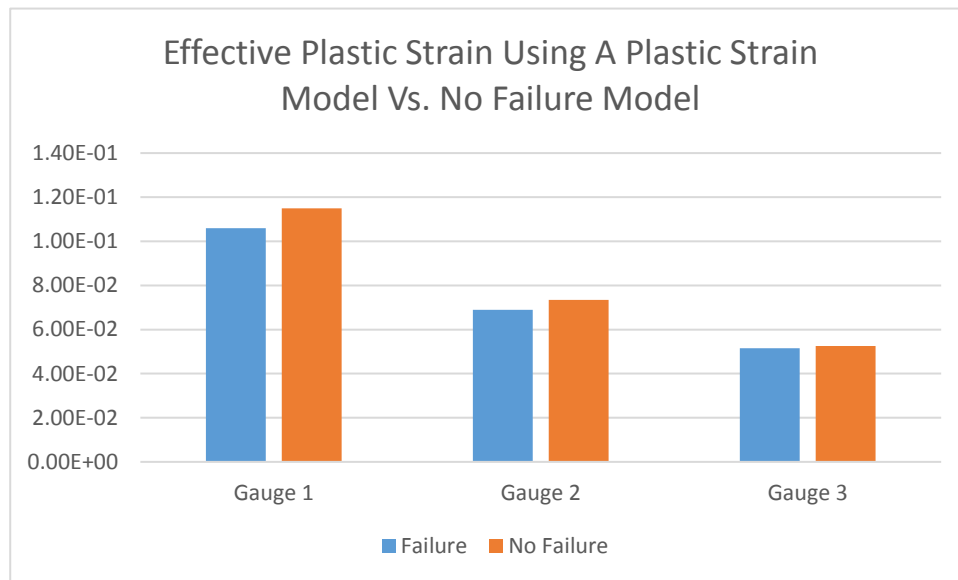


Figure 89: Effective plastic strain with and without a failure model. The failure model included is a plastic strain failure model.

6.5 Modifications to the Stainless Steel 21-6-9 Partial Recovery Target

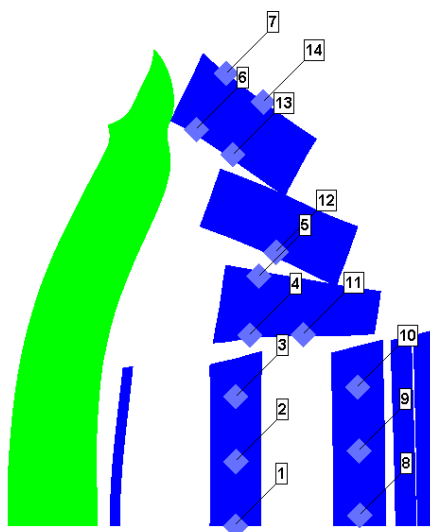
Having modelled the full recovery target used for most of the experimental work, modifications to the rear four 1 mm spall plates were carried out. These two other iterations have two 2 mm spall plates and one 4 mm spall plate. These were chosen to be modified as the smaller distance involved is the target depth of 15 mm, which is less than the diameter of the target, which is 70 mm, therefore the longitudinal reverberations are more likely to affect the recovery of the target before the plates separate than the lateral capture of the guard rings.

Material Location



SS 21-6-9

CU-OFHC



fullrecoveryspallplates2x2
Cycle 43390
Time 4.158E-002 ms
Units mm, mg, ms
Axial symmetry



Figure 90: Full recovery with modified rear spall plate with a 2x2mm configuration. Gauge numbers are shown in the diagram.

Figure 90 shows the same target configuration as the full recovery but with two 2 mm thick spall plates added to the rear (right hand side) of the target in the image. This is compared to four 1 mm targets. The comparisons found in the plastic strain will be discussed below in Figure 91.

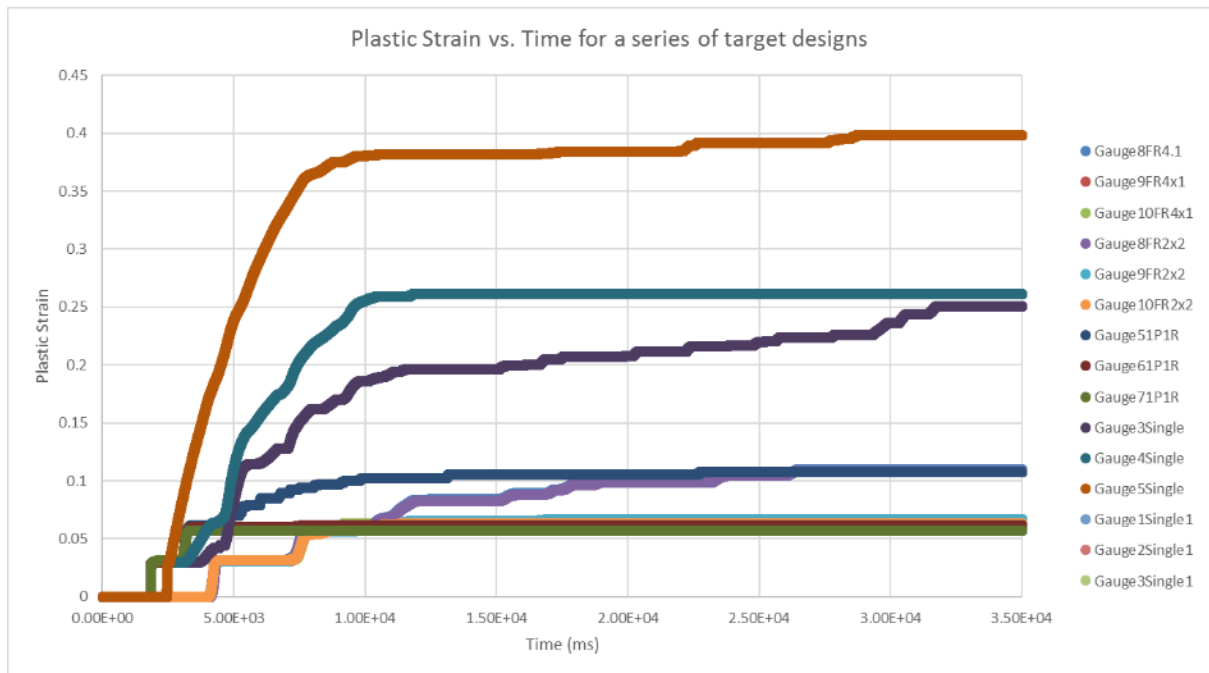


Figure 91: Overview of plastic strain within the same region for each model. The 4x1 model, the 2x2 model, the simple recovery of one plate and one ring, as well as the single plate impact.

Figure 91 provides an overview of each model set up and the comparative plastic deformation for the same or a similar region. As expected the highest strain is from the standard sample attached to the flyer plate, the next lowest is from the single plate impact. The lowest values are from the full recovery in its three different iterations, these are further dissected in Figure 92.

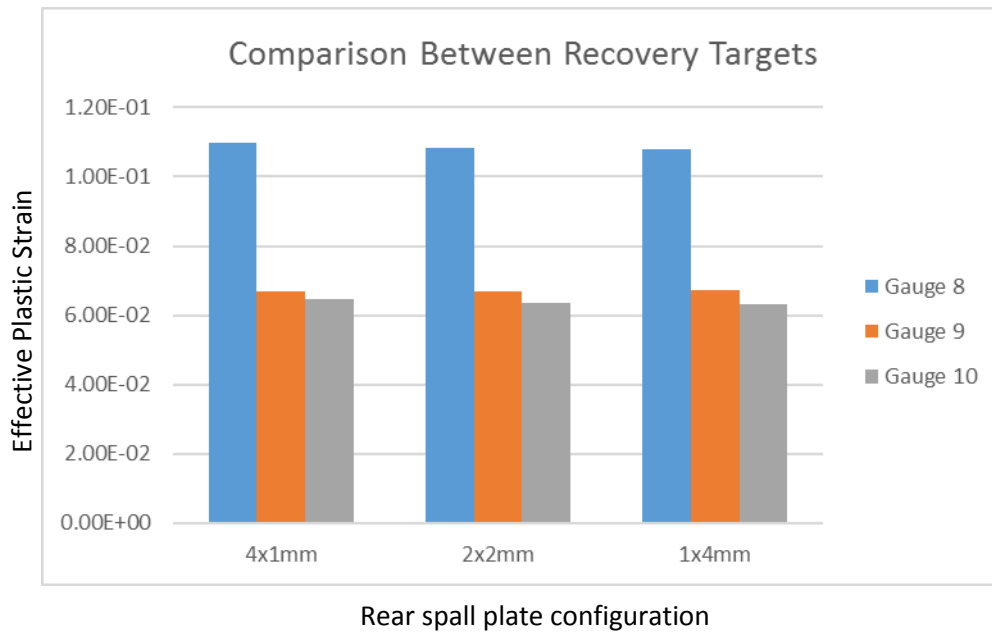


Figure 92: Comparison of effective plastic strain by varying spall plate number and thickness at back of target: 4x1mm, 2x2mm and 1x4mm

Experimentally, each target showed a difference in recovery but computationally the models only show negligible difference. This highlights an area where the model would need further parameters to reflect this. However, experimentally, it would seem promising to change the target design, which would also make the machining of the targets easier, as there would be less interfaces therefore less machining error. The thicker plates may further mitigate the shock waves, so that there is more time for the plates to separate versus the time for the shock waves to be reflected.

In a traditional recovery impact, with a 3 mm flyer plate, the rear four 1 mm spall plates are placed in the spall plane where the two tensile release waves overlap, so these spall plates separate due to the release waves, leaving the recovery target unaffected. Where partial recovery has occurred but not fully one dimensional using interface

effects, these spall plates behave in a different manner, therefore by modifying the number and thickness this can be fully investigated.

Having looked at previous research, it has been found that usually an increasing amount of spall plates and rings is, on the whole, better, however, if the spall plates are too thin, then they do not have time to separate as they trap the momentum.

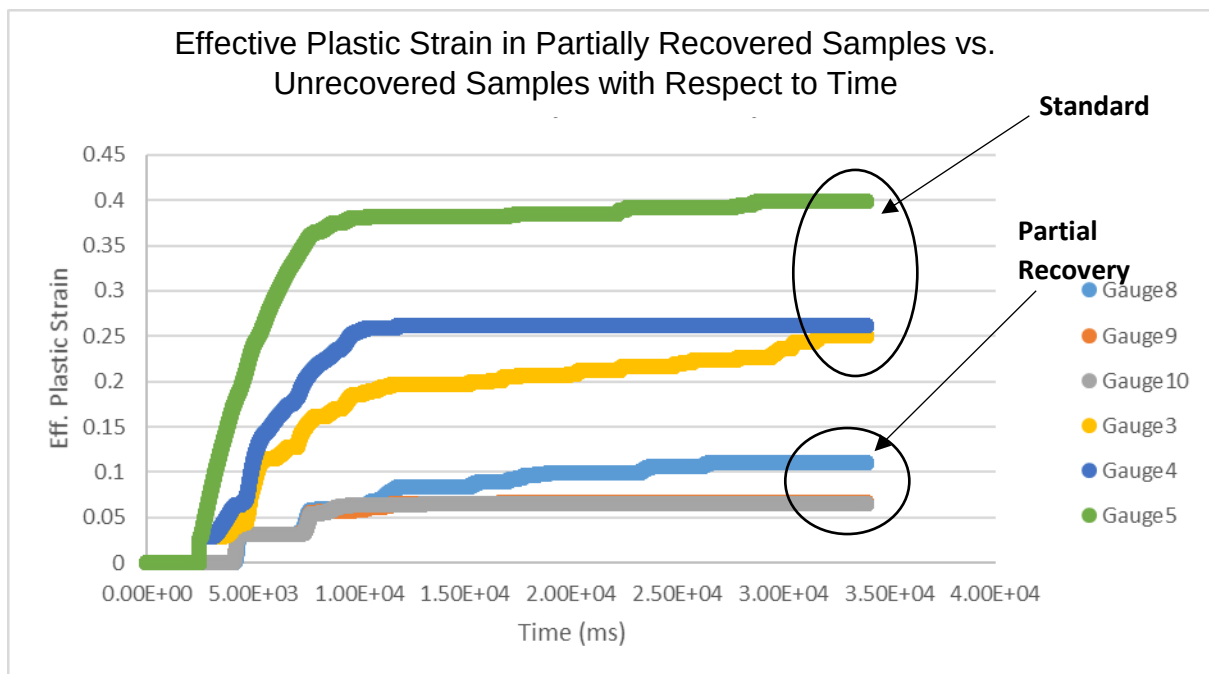


Figure 93: Graph showing comparisons between the modelled partially recovered samples versus the standard samples

Models were created to imitate the experimental work carried out with standard versus partially recovered samples. It is immediately apparent in Figure 93 that in the recovered samples (Gauges 8, 9 and 10), the plastic strain is lower than that of the standard samples (Gauges 3, 4 and 5). These gauges were selected as they are evenly spread from the centre to the outside of the recovery and standard sample.

Also within the standard samples it can be seen that there is a greater range between the lowest plastic strain compared to the highest plastic strain. These models were run with a starting velocity of 500 m s^{-1} to compare with empirical data.

However, when comparisons are drawn between the compressive sample results and the computer models, each velocity hence shock pressure will be matched in order to gain more accurate comparisons between results.

Overall, it can be seen that as spall plates are added the overall measured plastic strain is reduced, therefore on the whole, adding more plates and rings improves recovery. However, with the modified spall plate thickness experiments it can also be seen that if the spall plate is too thin it cannot trap momentum as efficiently.

6.6 Comparisons Between Partial Recovery and Full Recovery Target:

Empirical versus Computational

The full recovery target, using a 2 mm flyer plate to allow for 1D recovery is show in Figure 94.

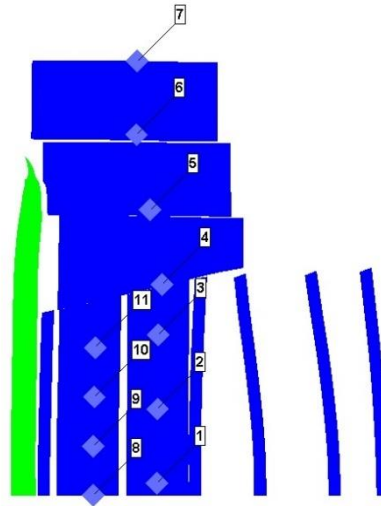


Figure 94: Full recovery with 2 mm copper flyer plate. Gauges shown in diagram.

Figure 95 shows the effective plastic strain differences between the full recovery, partial recovery and standard recovery targets. The plastic strain is lowest for the full recovery at 5.0×10^{-2} , next higher for the partial recovery which is at 1.0×10^{-1} and highest for the standard shot at 2.5×10^{-1} . This computer data is reflected well in the experimental results, which show that the lowest hardness data is for the full recovery, than the standard and partial recovery. The twinning data also confirms this observation.

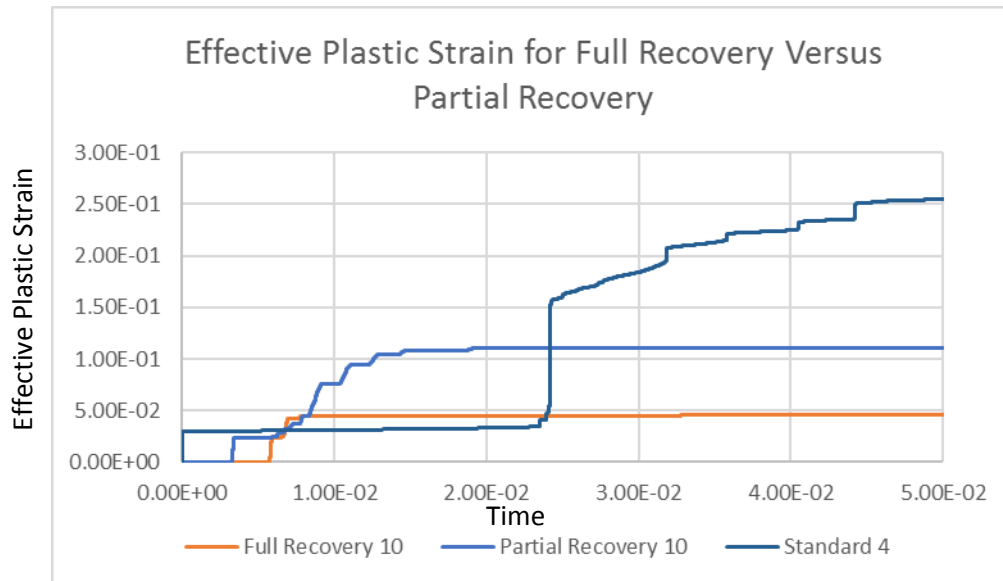


Figure 95: Effective plastic strain versus time for full recovery, partial recovery and standard recovery targets

6.7 Summary

Overall, the computer models have provided information into the plastic strain of the targets. They show that the current model that is in use for the full and partial recovery systems provides adequate momentum trapping of the shock reflections. Further development of this would involve looking at whether the model is appropriate for all materials and where those limitations lie.

It was interesting that the computer model did not show differences in effective plastic strain for the modification of the targets, where some differences were shown in grain size of the samples, twins per grain and hardness. This highlights a limitation of the models and suggests that the ability to input further parameters would make the model predictions more accurate.

ANSYS® Autodyn has been shown to provide good general predictions of recovery by using the effective plastic strain as an indicator. Further parameters, such as the ability to plot a stress-strain curve which would be comparable to compression testing would be a useful addition. The shock pressures obtained computationally were in line with the shock pressures calculated experimentally, as well as the Het-V data. It was useful to gradually model the recovery process to show improvements in strain but the limitations were shown, where more fine detail was needed, which could in future, be inputted from the data obtained in this thesis, showing the differences between partial and full recovery and hence improving the accuracy of material prediction, especially of stainless steel 21-6-9 but using a similar technique could be used to optimise the input parameters of many other FCC materials.

7 CONCLUSIONS

This project has provided a holistic understanding of the material strength evolution of stainless steel 21-6-9 under standard conditions, partial recovery conditions (including modified samples) and a full recovery target. This has been undertaken, firstly, to compare the overall effects of the recovery technique, secondly, to prove its necessity and finally cataloguing the stainless steel in different states of recovery, for comparison against the Steinberg-Guinan model based upon LLNL Hugoniot shock data for stainless steel 21-6-9 in ANSYS® Autodyn [64], [105], [152].

The first objective of the project consisted of redeveloping an operational recovery system for hydrodynamic gas-gun experiments on metals. This was achieved through experiments and modelling work on OFHC copper samples, as shown in Chapter 4. Previous alignment issues, preventing successful recovery from occurring through reimpact were corrected, and the quality of the fit of the targets (interference fit) was improved to prevent spallation occurring within the sample due to air gaps. The partial and full recovery target design iterated upon previous models and modifications, which allowed for effective release of the guard rings and spall plates without impingement, provided that alignment and capture prohibited reimpact [41], [60], [61]. Further modelling carried out on the recovery target confirmed its success in reverberation reduction.

The second objective was to characterise stainless steel 21-6-9, as it had not previously been characterised using a full or partial recovery technique. The seminal stainless steel 21-6-9 paper on dynamic material deformation was based upon standard shock experiments by Hills and Rack, which used TEM, hardness testing,

tensile testing and optical microscopy [101]. As computer modelling has improved since then, recovery techniques have become necessary to improve the understanding and validate predictions of material behaviour. This material behaviour can then be used in material selection for use in environments, which may be difficult to simulate experimentally i.e. outer space. By conducting both partial recovery experiments and a full recovery experiment, the microstructure under different loading conditions has been ascertained and can be applied to computer models to improve their accuracy, especially on stainless steel 21-6-9, which is critical to the functioning of high performance components in extreme conditions due to its corrosion resistance and stable γ -austenite phase structure [1], [130].

The overall material properties for stainless steel 21-6-9, indicated differences in recovery where standard recovery had no mitigation of reverberations leading to increased stress and strain and spallation in the steel. Partial recovery included less reverberations, leading to lower stress and strain and no spallation of the sample and full recovery was a fully 1D shock with no reverberations passing through it, hence no spall and lower hardness and twinning density.

Higgins *et al.* found hardness to be a good indicator of different recovery experiments on copper, where increases in hardness originated from increased dislocation density [175]. Similarly, hardness testing of the stainless steel displayed a clear difference of around 20-60 Hmv between partially recovered and standard samples, whilst also showing an increase in hardness up to 16 GPa. Previously, hardness testing had not been regarded as a good predictor of material behaviour, however, in correlation with the dislocation density obtained from TEM analysis and yield stress and strength data

from the compression testing, in Chapter 5 it is shown to be a reliable source. In the standard samples, the hardness stops increasing at around 12 GPa.

This project has shown that twinning density data shows increases twinning for the standard samples compared to the partial recovery and full recovery samples. The increase in twinning and dislocations leads to overall strengthening of the steel as shown in the yield strength values of the standard samples. As α' -martensite transition doesn't occur until 20 GPa, this means that the material characteristics will retain greater ductility for longer, which is useful in many industrial applications where embrittlement would lead to failure, as well as the strengthening effect caused by the twinning and dislocations.

Further to this, compression testing showed a difference of between 0.2-0.3 GPa higher yield stress in the standard samples to that of the partially recovered samples, confirming the previous hardness data and also verifying the higher rates of plastic strain show in computer models of the unrecovered samples.

XRD showed a potential phase change in α' -martensite in a partially recovered 14 GPa sample, though no other samples showed evidence of this change. It could also have been caused by the Cu $k\beta$ peak of austenite. As previously stated in Section 5.5.3, a phase change would not be expected until around 20 GPa due to the stability of the austenitic structure and certainly the standard 14.4 GPa sample shows no evidence of a phase change. Due to both experimental and theoretical limitations of the equipment and recovery method, higher pressures cannot be tested to see the phase change value for a recovered sample.

Computer modelling was undertaken from single plate impacts through to the current full recovery model. This model was then modified to increase the spall plate thickness and decrease the number of plates in a 1x4 mm plate configuration and a 2x2 mm plate configuration. Computationally there was negligible difference. However, experimentally, the 1x4 mm spall plate was too similar in mass to the 5 mm recovery disc and 5 mm spall plate. Therefore, it reimpacted on the recovery disc resulting in a higher hardness than the 2x2 mm recovery disc, which did have a lower strain rate experimentally.

The results of the modified experiments highlight the need for thick enough spall plates to trap momentum and separate but thin enough to have a higher velocity to separate from the recovery target itself. It also highlights a limitation of the models and suggests that the ability to input further parameters would make the model predictions more accurate. Overall, 2x2 mm plates are also less complex to manufacture than 4x1 mm plates, so should be considered for future recovery experiments.

In summary, the main aims of the project have been achieved. A recovery technique has been redeveloped for use in hydrodynamic gas-gun experiments, particularly on metals. This recovery technique could be successfully used in a number of different gas gun systems around the world, whilst currently being in use at Cranfield University. Stainless steel 21-6-9 has been characterised in different recovery states, with a plethora of hitherto unknown data provided, which can be inputted into predictive hydrocode modelling software for use in industries which have an interest in non-corrosive austenitic steels. The modification of targets from 4x 1 mm rear spall plates

in the partial recovery targets to 2x2 mm spall plates, also lead to successful recovery, while reducing the complexity of manufacture, allowing a reduction in the cost and lead time require per shot. Furthermore, hardness testing and twinning density obtained from optical microscopy have been shown to be good predictors of the recovery behaviour of a metal, which provides a cost and time efficient method of obtaining preliminary results compared to the more traditionally used EBSD or TEM data. All of these factors combine to provide a significant benefit to the field of shock physics and engineering by enabling a greater understanding of the materials used with both financial and temporal benefits.

8 FURTHER WORK

Further work would include looking at more detailed computational shock models, including those that allow for individual grains with variable grain sizes. This would allow for more accurate prediction of material behaviour, as currently single grain crystals and recovery modelling is carried out experimentally, to simplify the understanding of what is happening with the material. However, this needs to be iterated up to account for a full scale model of larger structures. Further to this, having the ability to input more data into ANSYS® Autodyn would allow improvement of the software and enhanced prediction of material behaviour.

ANSYS® Autodyn has been shown to provide good general predictions of recovery by using the effective plastic strain as an indicator. Further parameters, such as the ability to plot a stress-strain curve which would be comparable to compression testing would be a useful addition. The shock pressures obtained computationally were in line with the shock pressures calculated experimentally, as well as the Het-V data. It was beneficial to gradually model the recovery process to show improvements in strain but limitations were shown. This included where more fine detail was needed, which could in future, be inputted from the data obtained in this thesis, showing the differences between partial and full recovery and hence improving the accuracy of material prediction, especially of stainless steel 21-6-9. By using a similar technique the input parameters of many other FCC materials could be optimised.

Carrying out further full recovery experiments, rather than the one verification shot, would allow for comparison and understanding of the different complexities of shock loading. The comparisons between a standard environment, versus a partial recovery

scenario and a full recovery scenario allows for the accuracy of computer models to be developed especially as more in depth software is developed than can model specific granular structure including defects such as twins and dislocations initiating in the material and propagating. The use of TEM and EBSD would be crucial in this allowing for in depth material behaviour to be analysed. TEM would allow for in depth analysis of the microstructural changes going on in the metal. This would allow for each target being assessed in thin samples to note particular changes from the edge to the centre of each target. EBSD would allow for specific orientation information on twins and dislocations.

Further to this, by undertaking more experiments on metals with different types of crystallographic structure it can be seen as to whether the same or similar momentum trapping experiments would be successful on different metals. An extended version of this would be looking into ceramic or polymer based materials and looking at the shock pressure limits of the recovery.

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APPENDICES

Appendix A – Shot Record

Copper Shots

	Date	Type of Shot	Flyer type/thickness, mm	Velocity, ms ⁻¹	Calculated Pressure, GPa	Analysis Carried Out
CSL1	161124	Copper shot longitudinal gauge	Cu, 5			Confirmation of Hugoniot
CSL2	161129	Copper shot longitudinal gauge	Cu, 5			Confirmation of Hugoniot
CSL3	161205	Copper shot longitudinal gauge	Al, 5			Confirmation of Hugoniot
SS1	161206	304L SS Recovery target 5mm	Steel,	488	8.9	Not aligned

SS2	170119	405L SS Recovery target 5mm	Steel	485	8.9	Not aligned
SS3	170201	Stainless steel spall recovery shot spigot flyer	Steel	466	8.9	
SS4	170217	Stainless steel spall recovery shot flat flyer	Steel	466.42	8.9	CT
CU1	170302A	Copper spall recovery	Cu,	466.4	8.9	
CU2	170302B	Copper spall recovery	Cu,	370.0	7	
CU3	170307	Copper spall recovery with a Het-V probe	Cu,	484.5	9.3	SEM, Microscopy Hardness
CU4	170314	Copper spall recovery with a Het-V probe	Cu,	369.8	7	SEM, Microscopy, Hardness

CU5	170427	Copper spall recovery	Cu,	718.4	14.4	SEM, Microscopy, Hardness
CU6	170428	Copper spall recovery (annealed)	Cu,	679.3	13.5	SEM, Microscopy, Hardness
CU7	170531	Copper spall recovery (annealed)	Cu,	370	7	SEM, Microscopy, Hardness
CU8	170717	Full copper recovery	Cu, 10	363.1	6.8	TEM
CU9	170720	Full copper recovery	Cu, 10	439.8	8.4	
CU10	170905	Copper 5mm disc	Cu,			Microscopy, Hardness
CU11	170906	Full copper recovery (annealed)	Cu, 10	335.4	6.3	TEM
CU12	170907A	Full copper recovery (annealed)	Cu, 10	469.5	9	
CU13	170907B	Full copper recovery (annealed)	Cu, 10	335.8	6.3	
CU14	171213	Full copper recovery	Cu, 10	500	9.6	

		(annealed)				
CU15	171214A	Full copper recovery (annealed)	Cu, 10	350	6.3	
CU16	171214B	Full copper recovery (annealed)	Cu, 10	<70	1.25	

Appendix B – Stainless Steel Analysis

XRD Results and Calculations

	Peak Position, 2theta	Theta in radians	cos theta	FWHM in radians	Costheta *FWHM	k	Lambda nm	k*lambda	Grain size nm	Dislocation density	Average Grain Size	Average Dislocation Density	Standard Variation (Sample)
Control	4.35E+01	3.80E-01	9.29E-01	1.15E-02	1.07E-02	9.00E-01	1.54E-01	1.39E-01	1.30E-08	5.95E+15	1.14E-08	7.98E+15	2.37E+15
Control	5.11E+01	4.46E-01	9.02E-01	1.58E-02	1.43E-02	9.00E-01	1.54E-01	1.39E-01	9.72E-09	1.06E+16			
Control	7.48E+01	6.53E-01	7.94E-01	1.50E-02	1.19E-02	9.00E-01	1.54E-01	1.39E-01	1.16E-08	7.39E+15			
CG 1	4.35E+01	3.80E-01	9.29E-01	7.50E-03	6.97E-03	9.00E-01	1.54E-01	1.39E-01	1.99E-08	2.53E+15	1.56E-08	4.48E+15	1.61E+15
CG 1	5.07E+01	4.42E-01	9.04E-01	1.15E-02	1.04E-02	9.00E-01	1.54E-01	1.39E-01	1.33E-08	5.64E+15			
CG 1	7.45E+01	6.50E-01	7.96E-01	1.19E-02	9.45E-03	9.00E-01	1.54E-01	1.39E-01	1.47E-08	4.64E+15			
CG 1	9.04E+01	7.89E-01	7.05E-01	1.57E-02	1.11E-02	9.00E-01	1.54E-01	1.39E-01	1.25E-08	6.37E+15			
CG 1	9.56E+01	8.34E-01	6.72E-01	1.17E-02	7.86E-03	9.00E-01	1.54E-01	1.39E-01	1.76E-08	3.21E+15			
CG 2	4.35E+01	3.80E-01	9.29E-01	8.90E-03	8.27E-03	9.00E-01	1.54E-01	1.39E-01	1.68E-08	3.56E+15	1.50E-08	4.60E+15	1.08E+15
CG 2	5.05E+01	4.40E-01	9.05E-01	1.15E-02	1.04E-02	9.00E-01	1.54E-01	1.39E-01	1.33E-08	5.65E+15			

CG 2	7.44E+01	6.50E-01	7.96 E-01	1.01E-02	8.06E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.72E- 08	3.38E+15			
CG 2	9.02E+01	7.87E-01	7.06 E-01	1.47E-02	1.04E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.34E- 08	5.57E+15			
CG 2	9.55E+01	8.33E-01	6.73 E-01	1.43E-02	9.63E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.44E- 08	4.82E+15			
CG 8	4.35E+01	3.79E-01	9.29 E-01	7.85E-03	7.30E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.90E- 08	2.77E+15	1.41 E-08	5.76E +15	2.63E+ 15
CG 8	5.08E+01	4.43E-01	9.03 E-01	1.27E-02	1.15E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.20E- 08	6.89E+15			
CG 8	7.45E+01	6.50E-01	7.96 E-01	1.20E-02	9.59E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.45E- 08	4.78E+15			
CG 8	9.05E+01	7.90E-01	7.04 E-01	1.94E-02	1.36E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.02E- 08	9.67E+15			
CG 8	9.56E+01	8.34E-01	6.72 E-01	1.41E-02	9.50E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.46E- 08	4.69E+15			
CG 4	4.36E+01	3.80E-01	9.29 E-01	6.63E-03	6.16E-03	9.00 E-01	1.54E- 01	1.39 E-01	2.25E- 08	1.97E+15	1.55 E-08	5.01E +15	2.52E+ 15
CG 4	5.10E+01	4.45E-01	9.03 E-01	1.17E-02	1.06E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.31E- 08	5.80E+15			
CG 4	7.46E+01	6.51E-01	7.96 E-01	1.31E-02	1.04E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.33E- 08	5.64E+15			
CG 4	9.07E+01	7.92E-01	7.03 E-01	1.82E-02	1.28E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.09E- 08	8.46E+15			
CG 4	9.58E+01	8.36E-01	6.71 E-01	1.17E-02	7.84E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.77E- 08	3.20E+15			
CG 5	4.35E+01	3.79E-01	9.29 E-01	8.03E-03	7.46E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.86E- 08	2.89E+15	1.36 E-08	6.10E +15	2.64E+ 15
CG 5	5.08E+01	4.43E-01	9.03 E-01	1.20E-02	1.09E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.27E- 08	6.16E+15			
CG 5	7.45E+01	6.50E-01	7.96 E-01	1.36E-02	1.08E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.28E- 08	6.11E+15			
CG 5	9.06E+01	7.91E-01	7.03 E-01	1.99E-02	1.40E-02	9.00 E-01	1.54E- 01	1.39 E-01	9.91E- 09	1.02E+16			

CG 5	9.57E+01	8.35E-01	6.71 E-01	1.48E-02	9.96E-03	9.00 E-01	1.54E-01	1.39 E-01	1.39E-08	5.16E+15			
CG 6	4.35E+01	3.80E-01	9.29 E-01	7.68E-03	7.13E-03	9.00 E-01	1.54E-01	1.39 E-01	1.94E-08	2.65E+15	1.40 E-08	6.27E +15	3.48E+ 15
CG 6	5.07E+01	4.43E-01	9.04 E-01	1.50E-02	1.36E-02	9.00 E-01	1.54E-01	1.39 E-01	1.02E-08	9.57E+15			
CG 6	7.44E+01	6.49E-01	7.96 E-01	1.13E-02	9.04E-03	9.00 E-01	1.54E-01	1.39 E-01	1.53E-08	4.25E+15			
CG 6	9.04E+01	7.89E-01	7.05 E-01	2.01E-02	1.41E-02	9.00 E-01	1.54E-01	1.39 E-01	9.80E-09	1.04E+16			
CG 6	9.56E+01	8.35E-01	6.72 E-01	1.38E-02	9.26E-03	9.00 E-01	1.54E-01	1.39 E-01	1.50E-08	4.46E+15			
CG 7	4.35E+01	3.80E-01	9.29 E-01	7.85E-03	7.30E-03	9.00 E-01	1.54E-01	1.39 E-01	1.90E-08	2.77E+15	1.30 E-08	6.83E +15	2.78E+ 15
CG 7	5.04E+01	4.40E-01	9.05 E-01	1.45E-02	1.31E-02	9.00 E-01	1.54E-01	1.39 E-01	1.06E-08	8.93E+15			
CG 7	7.44E+01	6.49E-01	7.97 E-01	1.24E-02	9.87E-03	9.00 E-01	1.54E-01	1.39 E-01	1.40E-08	5.07E+15			
CG 7	9.02E+01	7.87E-01	7.06 E-01	1.85E-02	1.31E-02	9.00 E-01	1.54E-01	1.39 E-01	1.06E-08	8.87E+15			
CG 7	9.56E+01	8.34E-01	6.72 E-01	1.90E-02	1.28E-02	9.00 E-01	1.54E-01	1.39 E-01	1.08E-08	8.50E+15			
CG 3	4.34E+01	3.79E-01	9.29 E-01	9.77E-03	9.08E-03	9.00 E-01	1.54E-01	1.39 E-01	1.53E-08	4.29E+15	1.38 E-08	6.44E +15	3.76E+ 15
CG 3	5.05E+01	4.41E-01	9.04 E-01	1.52E-02	1.37E-02	9.00 E-01	1.54E-01	1.39 E-01	1.01E-08	9.81E+15			
CG 3	7.44E+01	6.49E-01	7.96 E-01	1.12E-02	8.90E-03	9.00 E-01	1.54E-01	1.39 E-01	1.56E-08	4.12E+15			
CG 3	9.04E+01	7.89E-01	7.05 E-01	2.08E-02	1.46E-02	9.00 E-01	1.54E-01	1.39 E-01	9.47E-09	1.11E+16			
CG 3	9.56E+01	8.34E-01	6.72 E-01	1.10E-02	7.39E-03	9.00 E-01	1.54E-01	1.39 E-01	1.88E-08	2.84E+15			
CG 9	4.35E+01	3.80E-01	9.29 E-01	8.03E-03	7.46E-03	9.00 E-01	1.54E-01	1.39 E-01	1.86E-08	2.89E+15	1.36 E-08	7.06E +15	4.70E+ 15

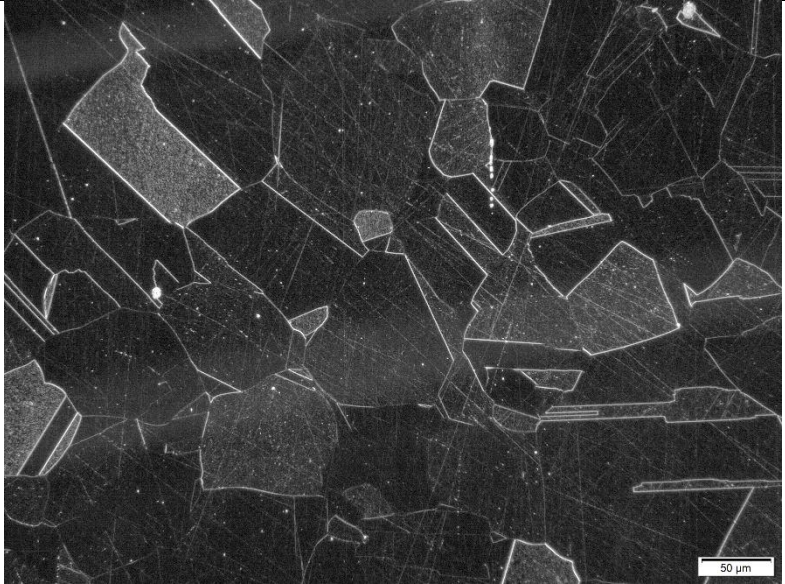
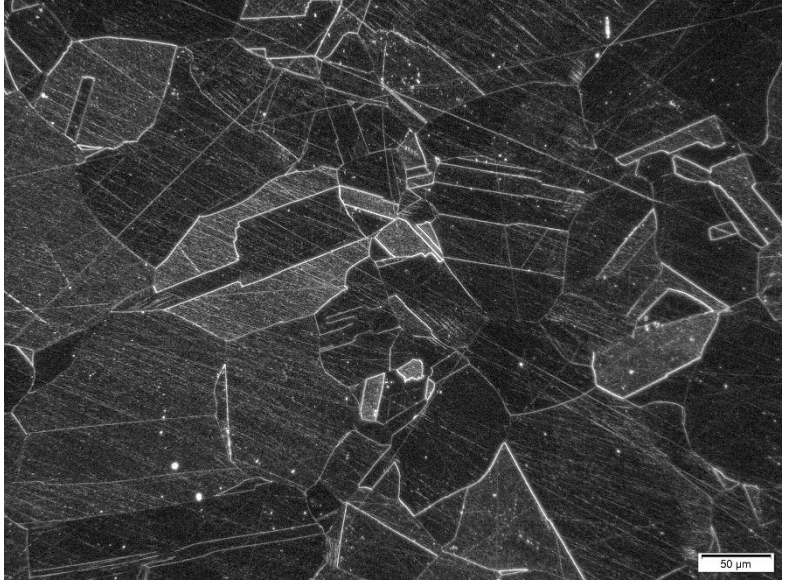
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CG 9	7.44E+01	6.49E-01	7.97 E-01	1.06E-02	8.48E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.63E- 08	3.74E+15			
CG 9	9.03E+01	7.88E-01	7.05 E-01	2.02E-02	1.43E-02	9.00 E-01	1.54E- 01	1.39 E-01	9.71E- 09	1.06E+16			
CG 9	9.56E+01	8.35E-01	6.72 E-01	1.40E-02	9.38E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.48E- 08	4.57E+15			
CG 10	4.35E+01	3.80E-01	9.29 E-01	8.03E-03	7.46E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.86E- 08	2.89E+15	1.38 E-08	5.73E +15	1.93E+ 15
CG 10	5.07E+01	4.43E-01	9.04 E-01	1.10E-02	9.94E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.40E- 08	5.13E+15			
CG 10	7.44E+01	6.50E-01	7.96 E-01	1.31E-02	1.04E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.33E- 08	5.65E+15			
CG 10	9.05E+01	7.90E-01	7.04 E-01	1.75E-02	1.23E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.13E- 08	7.85E+15			
CG 10	9.57E+01	8.35E-01	6.71 E-01	1.75E-02	1.17E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.18E- 08	7.14E+15			
CG 12	4.35E+01	3.79E-01	9.29 E-01	9.95E-03	9.24E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.50E- 08	4.44E+15	1.11 E-08	9.10E +15	3.55E+ 15
CG 12	5.04E+01	4.40E-01	9.05 E-01	1.64E-02	1.48E-02	9.00 E-01	1.54E- 01	1.39 E-01	9.34E- 09	1.15E+16			
CG 12	7.44E+01	6.49E-01	7.96 E-01	1.48E-02	1.18E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.17E- 08	7.26E+15			
CG 12	9.03E+01	7.88E-01	7.06 E-01	2.29E-02	1.61E-02	9.00 E-01	1.54E- 01	1.39 E-01	8.60E- 09	1.35E+16			
CG 12	9.56E+01	8.34E-01	6.72 E-01	1.94E-02	1.30E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.07E- 08	8.81E+15			
CG 13	4.35E+01	3.79E-01	9.29 E-01	6.11E-03	5.67E-03	9.00 E-01	1.54E- 01	1.39 E-01	2.44E- 08	1.67E+15	1.52 E-08	6.42E +15	4.68E+ 15
CG 13	5.06E+01	4.42E-01	9.04 E-01	1.62E-02	1.47E-02	9.00 E-01	1.54E- 01	1.39 E-01	9.45E- 09	1.12E+16			
CG 13	7.44E+01	6.50E-01	7.96 E-01	1.03E-02	8.20E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.69E- 08	3.50E+15			

CG 13	9.04E+01	7.89E-01	7.05 E-01	2.13E-02	1.50E-02	9.00 E-01	1.54E- 01	1.39 E-01	9.24E- 09	1.17E+16			
CG 13	9.56E+01	8.35E-01	6.72 E-01	1.31E-02	8.79E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.58E- 08	4.02E+15			
CG 14	4.34E+01	3.78E-01	9.29 E-01	9.77E-03	9.08E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.53E- 08	4.29E+15	1.11 E-08	9.19E +15	3.80E+ 15
CG 14	5.04E+01	4.40E-01	9.05 E-01	1.22E-02	1.11E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.25E- 08	6.35E+15			
CG 14	7.43E+01	6.48E-01	7.97 E-01	1.73E-02	1.38E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.01E- 08	9.87E+15			
CG 14	9.01E+01	7.86E-01	7.07 E-01	2.23E-02	1.58E-02	9.00 E-01	1.54E- 01	1.39 E-01	8.78E- 09	1.30E+16			
CG 14	9.55E+01	8.33E-01	6.73 E-01	2.30E-02	1.55E-02	9.00 E-01	1.54E- 01	1.39 E-01	8.95E- 09	1.25E+16			
CG 17	4.35E+01	3.80E-01	9.29 E-01	7.33E-03	6.81E-03	9.00 E-01	1.54E- 01	1.39 E-01	2.04E- 08	2.41E+15	1.53 E-08	4.72E +15	1.73E+ 15
CG 17	5.08E+01	4.43E-01	9.03 E-01	1.08E-02	9.77E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.42E- 08	4.97E+15			
CG 17	7.45E+01	6.50E-01	7.96 E-01	1.08E-02	8.61E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.61E- 08	3.86E+15			
CG 17	9.06E+01	7.90E-01	7.04 E-01	1.66E-02	1.17E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.19E- 08	7.08E+15			
CG 17	9.56E+01	8.34E-01	6.72 E-01	1.50E-02	1.01E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.38E- 08	5.29E+15			
CG 18	4.35E+01	3.80E-01	9.29 E-01	8.03E-03	7.46E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.86E- 08	2.89E+15	1.42 E-08	5.30E +15	1.56E+ 15
CG 18	5.08E+01	4.43E-01	9.03 E-01	1.17E-02	1.06E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.31E- 08	5.80E+15			
CG 18	7.45E+01	6.50E-01	7.96 E-01	1.43E-02	1.14E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.22E- 08	6.76E+15			
CG 18	9.05E+01	7.89E-01	7.04 E-01	1.57E-02	1.11E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.25E- 08	6.36E+15			
CG 18	9.56E+01	8.34E-01	6.72 E-01	1.41E-02	9.50E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.46E- 08	4.69E+15			

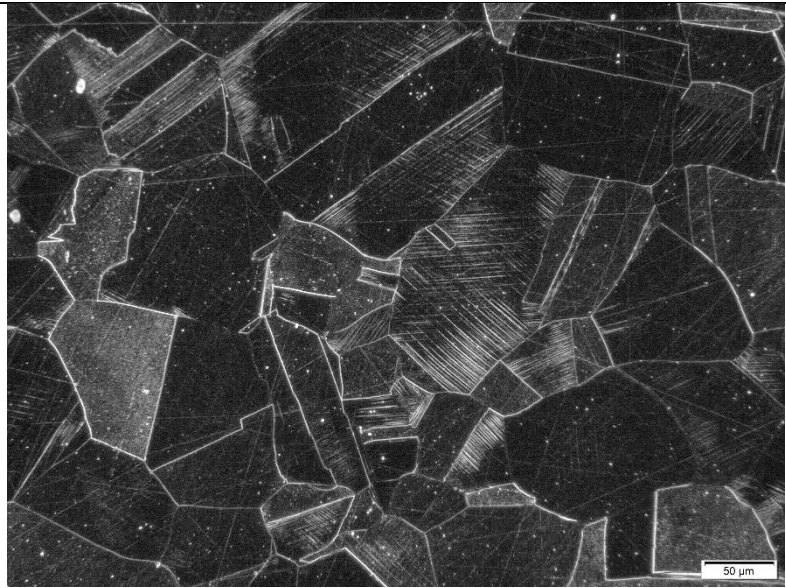
CG 19	4.36E+01	3.80E-01	9.29 E-01	8.20E-03	7.62E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.82E- 08	3.02E+15	1.42 E-08	5.63E +15	2.72E+ 15
CG 19	5.09E+01	4.44E-01	9.03 E-01	1.24E-02	1.12E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.24E- 08	6.51E+15			
CG 19	7.46E+01	6.51E-01	7.96 E-01	1.26E-02	1.00E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.39E- 08	5.20E+15			
CG 19	9.06E+01	7.90E-01	7.04 E-01	1.95E-02	1.38E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.01E- 08	9.84E+15			
CG 19	9.57E+01	8.35E-01	6.71 E-01	1.24E-02	8.32E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.67E- 08	3.60E+15			
CG 20	4.34E+01	3.79E-01	9.29 E-01	8.38E-03	7.78E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.78E- 08	3.15E+15	1.47 E-08	5.10E +15	1.98E+ 15
CG 20	5.07E+01	4.42E-01	9.04 E-01	1.20E-02	1.09E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.27E- 08	6.16E+15			
CG 20	7.44E+01	6.49E-01	7.97 E-01	1.27E-02	1.01E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.37E- 08	5.36E+15			
CG 20	9.05E+01	7.90E-01	7.04 E-01	1.73E-02	1.22E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.14E- 08	7.70E+15			
CG 20	9.56E+01	8.34E-01	6.72 E-01	1.15E-02	7.74E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.79E- 08	3.11E+15			
CG 21	4.35E+01	3.80E-01	9.29 E-01	7.50E-03	6.97E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.99E- 08	2.53E+15	1.46 E-08	5.17E +15	1.85E+ 15
CG 21	5.08E+01	4.43E-01	9.03 E-01	1.15E-02	1.04E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.33E- 08	5.63E+15			
CG 21	7.45E+01	6.50E-01	7.96 E-01	1.26E-02	1.00E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.39E- 08	5.21E+15			
CG 21	9.06E+01	7.90E-01	7.04 E-01	1.73E-02	1.22E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.14E- 08	7.69E+15			
CG 21	9.57E+01	8.35E-01	6.71 E-01	1.43E-02	9.61E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.44E- 08	4.80E+15			
CG 22	4.35E+01	3.80E-01	9.29 E-01	6.63E-03	6.16E-03	9.00 E-01	1.54E- 01	1.39 E-01	2.25E- 08	1.97E+15	1.67 E-08	4.04E +15	1.74E+ 15
CG 22	5.09E+01	4.44E-01	9.03 E-01	9.95E-03	8.98E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.54E- 08	4.20E+15			

CG 22	7.46E+01	6.51E-01	7.96 E-01	1.06E-02	8.47E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.64E- 08	3.73E+15			
CG 22	9.06E+01	7.91E-01	7.03 E-01	1.62E-02	1.14E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.21E- 08	6.78E+15			
CG 22	9.57E+01	8.35E-01	6.71 E-01	1.22E-02	8.20E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.69E- 08	3.50E+15			
CG 23	4.36E+01	3.80E-01	9.29 E-01	7.50E-03	6.97E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.99E- 08	2.53E+15	1.48 E-08	5.09E +15	2.02E+ 15
CG 23	5.08E+01	4.44E-01	9.03 E-01	1.10E-02	9.93E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.40E- 08	5.13E+15			
CG 23	7.46E+01	6.51E-01	7.96 E-01	1.24E-02	9.86E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.41E- 08	5.06E+15			
CG 23	9.05E+01	7.90E-01	7.04 E-01	1.78E-02	1.25E-02	9.00 E-01	1.54E- 01	1.39 E-01	1.11E- 08	8.16E+15			
CG 23	9.57E+01	8.35E-01	6.71 E-01	1.40E-02	9.37E-03	9.00 E-01	1.54E- 01	1.39 E-01	1.48E- 08	4.57E+15			

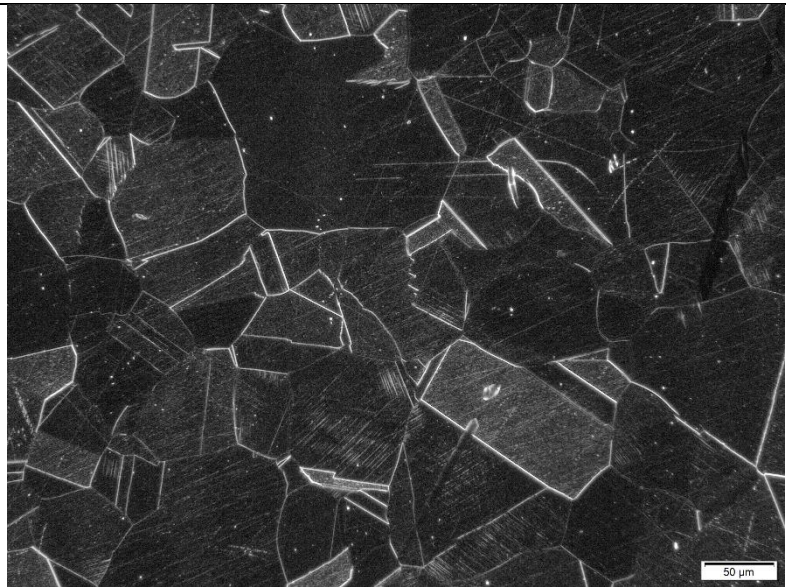
Optical Microscopy- Representative Microstructures for Twinning Counting

Sample/Pressure	Representative Microstructure (Optical)
Control	
CG2 – 7.6 GPa- Standard	

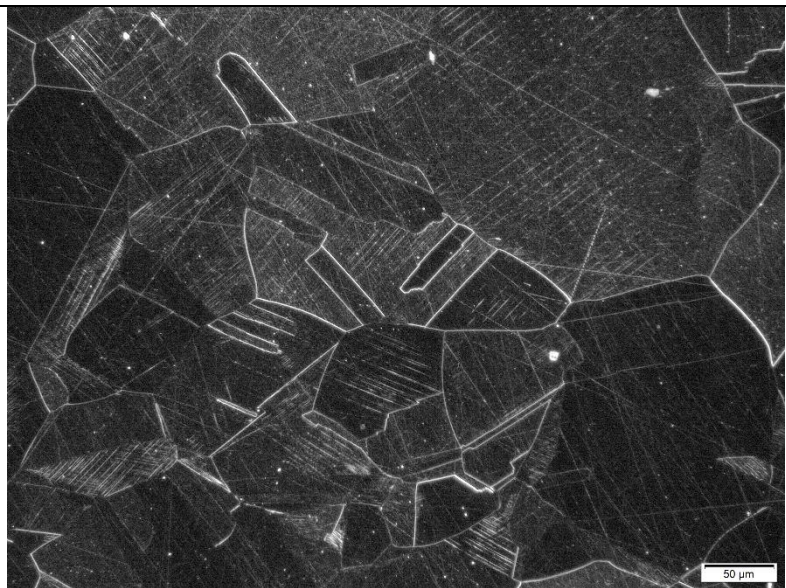
CG3 - 12.0 GPa- Partial
Recovery



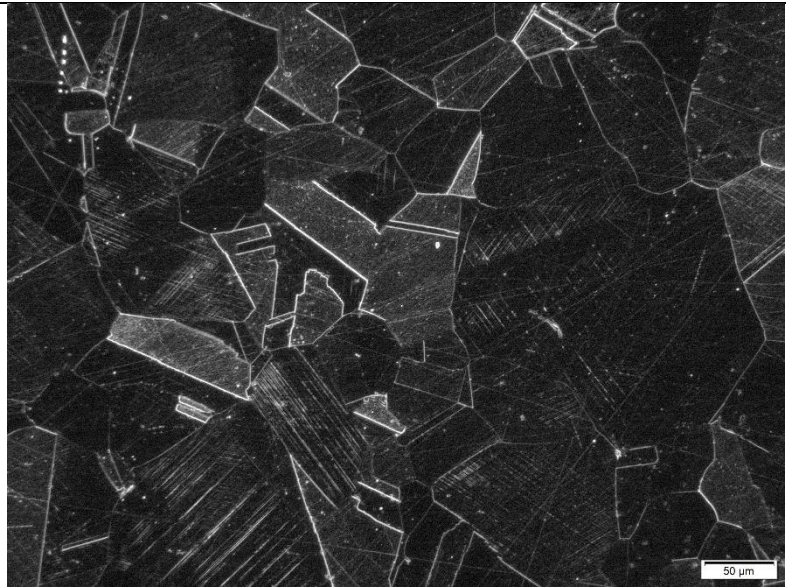
CG4 - 7.0 GPa – Partial
Recovery



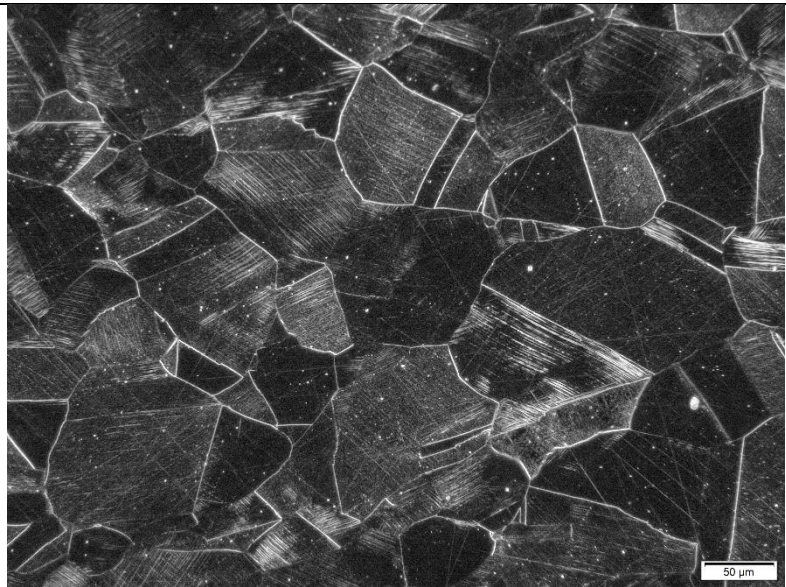
CG5 - 13.6 GPa – Partial
Recovery



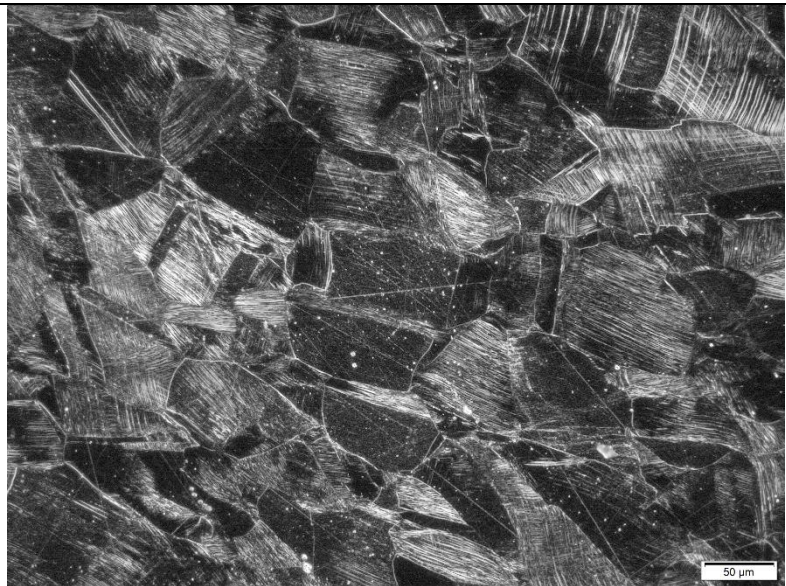
CG6 - 10.0 GPa – Partial
Recovery



CG7- 6.0 GPa – Standard



CG8 – 14.4 GPa –
Standard



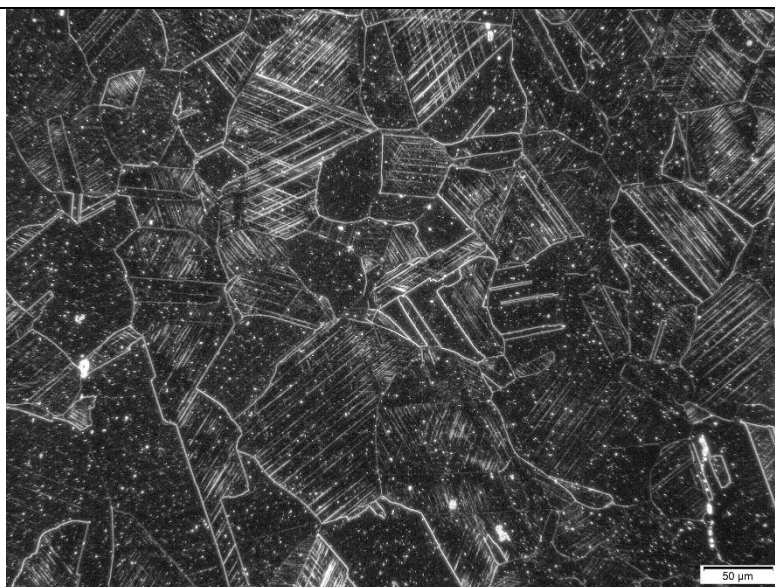
CG9 - 12.4 GPa - Standard

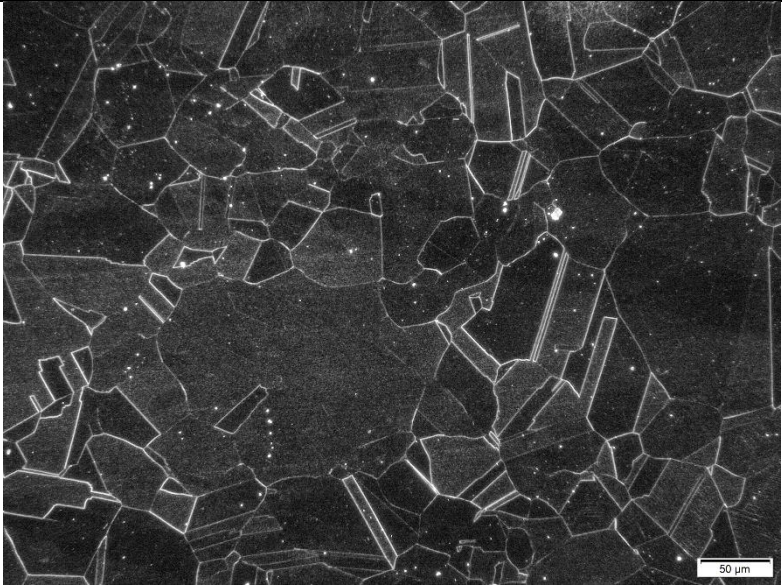
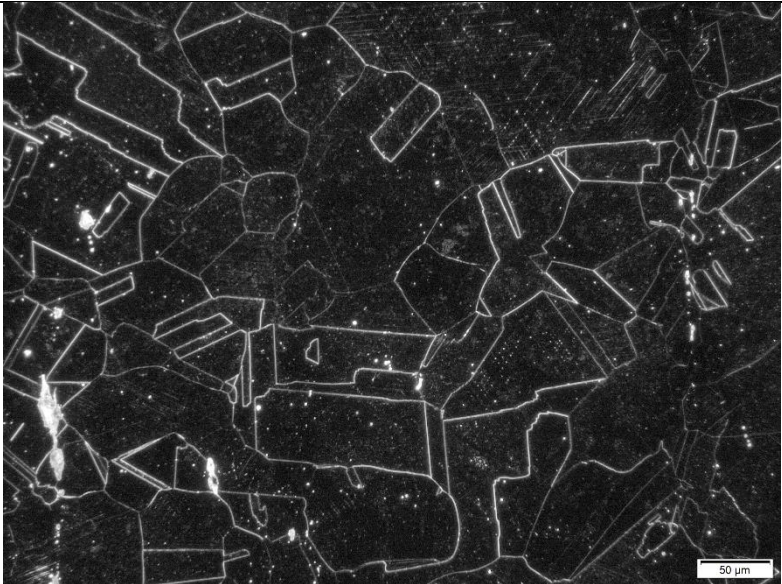


CG17- 16.0 GPa –
Standard

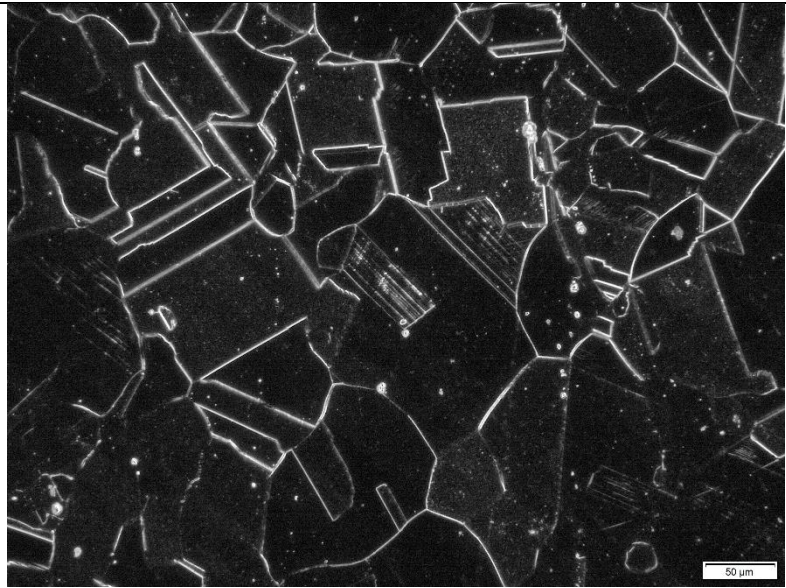


CG18- 16.0 GPa – Partial
Recovery

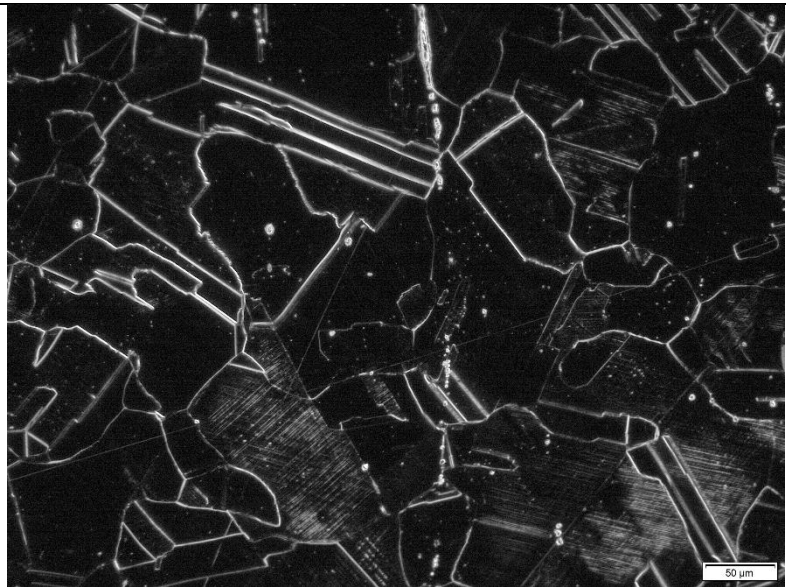


CG19- 15.3 GPa – Partial Recovery (2x2 mm)	
CG20 – 15.0 GPa – Partial Recovery (1x4 mm)	
CG21 – 15.0 GPa – Partial Recovery	
CG22 – 7.0 GPa – Partial Recovery (2x2 mm)	
CG23 – 7.0 GPa – Partial Recovery (1x4 mm)	

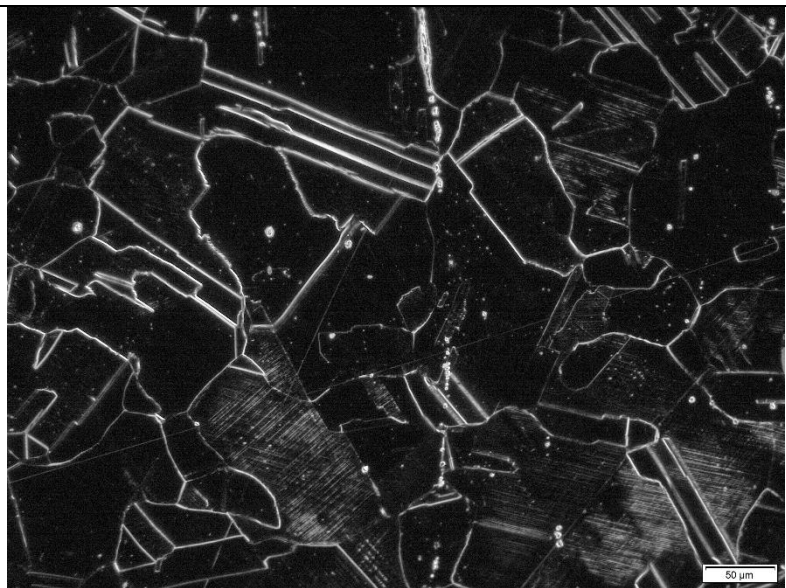
CG24 – 6.2 GPa – Partial
Recovery



CG26 – 10.4 GPa – Partial
Recovery



CG27 – 12.8 GPa –
Standard



CG29 – 10.7 GPa – Partial
Recovery



CG30 – Full Recovery – 7.0
GPa – Full Recovery

