DEFORMATION MECHANISMS OF PURE POLYCRYSTALLINE IRON SHOCKED IN EXCESS OF ONE MEGABAR PRESSURES

by
Alex Sundby
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Golden, Colorado

Date ______________

Signed: ____________________________
Alex Sundby

Signed: ____________________________
Dr. Aaron P. Stebner
Thesis Advisor

Golden, Colorado

Date ______________

Signed: ____________________________
Dr. Greg S. Jackson
Professor and Head
Department of Mechanical Engineering
ABSTRACT

High purity polycrystalline iron was shocked at pressures of 1 and 2 Mbar (100 and 200 GPa) via plasma driven compression waves created by ablating plastics using 1 nanosecond long square-shaped 70 and 150 Joule laser pulses, respectively. The experiments were performed on the Omega laser at the University of Rochester Laboratory for Laser Energetics. The iron targets were recovered post-shock. A combination of nanoindentation, microscopy, and diffraction techniques were used to characterize the properties and microstructures of the targets post-mortem as a means to understand the deformation mechanisms of iron at these extreme pressures and strain rates. The sample shocked at 1 Mbar exhibited nearly identical hardness, stiffness, and microstructure to the un-shocked material, except for a sizeable spall region where the shock broke out of the target. This result indicates that the temperature and pressure of the material as the shock released was sufficient for recovery of defect structures that may have been formed by deformation mechanisms that accommodated the shock. Thus, the post-mortem attempt to ascertain the mechanics of shock accommodation of this iron sample was inconclusive. Contrarily, the sample recovered from 2 Mbar shock showed remarkable grain refinement from ~500 µm in the unshocked state to less than 50 µm post-shock. The recrystallized grains were equiaxed and twins were not found. Instead, dramatic microbanded structures were observed to be sheared about \{3 2 2\} planes, which are ~9° misaligned from \{2 1 1\} planes. This result suggests that the Ferrite to Hexaferrum phase transformation known to occur in iron above 0.013 Mbar did not occur, but instead adiabatic heating due to shock release recrystallized the heavily deformed Ferrite that accommodated the
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CHAPTER 1: INTRODUCTION

Investigations into the strengthening effect and associated deformation mechanisms of pure iron at high pressures have astronomical, engineering, and material science impacts. Motivation for such research is further described in Section 1.1. A detailed background and review of previous high-pressure studies of iron is given in Sections 1.4.1 – 1.4.4. The introduction concludes with a review of high-pressure materials testing in Section 1.5.

1.1 Motivation

Pure iron is one of the most abundant elements on Earth, as it comprises much of the planet’s inner and outer cores. At atmospheric pressure, iron is relatively soft and malleable; pure single crystals have a tensile strength of 10 MPa. However, this metal exhibits a dramatic strengthening mechanism at high pressures, evidenced by existence in solid, non-flowing form at core pressures of 330 GPa or more [1]. With acknowledgement that tensile strength isn’t directly analogous to the ability of metals to withstand pressure at Earth’s core, there is still evidence of a dramatic strengthening mechanism present with iron at high pressures. This phenomenon makes it a potential candidate for high-pressure engineering applications such as inertial confinement fusion, pressure vessel fabrication, and heavy armoring. This strengthening is often attributed to a pressure induced phase transformation at 13 GPa whereby ferrite, the body-centered cubic (BCC) $\alpha$-phase that is stable at ambient atmospheric pressure and temperature, transforms to hexaferrum, a hexagonal closed-packed $\varepsilon$-phase. This reversible phase transformation has been well documented through high-pressure
diamond-anvil experiments [2] but less is known about how iron reacts to the extreme strain rates achieved with fast (< 1 microsecond), very high-pressure (> one Mbar) shocks. In fact, direct evidence of the phase transformation during moderate shocks in pressure regimes of 15 to 30 GPa has eluded scientists for decades, with one recent exception [3]. Thus, it is currently unknown if the martensitic phase transformation is the primary accommodation mechanism during very fast high-pressure shocks, or if another mechanism is responsible for the strength of iron in these circumstances.

To explore these mechanisms, we performed experiments in which we shocked iron samples at pressures of 1 and 2 Mbar on time scales of 1 to 100 nanoseconds to elucidate the very high-rate deformation mechanics of pure iron. These deformations are extreme, equating to shock regimes of $10^7$ to $10^{10}$ strain rates. These experiments were novel, in that the samples were not destroyed, but were instead recovered. Recovery allowed for post-mortem characterizations using nanoindentation, microscopy, and diffraction techniques. From these analyses, insights into the deformation mechanics are gained. Before describing the current work in more detail, we proceed to provide background.

1.2 The Chemical Abundance of Iron

Iron is by mass, the most abundant element on Earth. In fact, it is the main constituent of the Earth’s inner and outer cores. From a cosmic perspective, iron is abundantly created as a result of the decay of radioactive nickel-56, which is the last element produced by nuclear fusion in main sequence stars. Once the cores of these stars become nickel-rich, the internal pressure and heat within the star rise significantly, sometimes causing such enormous forces that the star collapses on itself and violently
explodes. This collapsing and exploding of massive stars is known as a supernova. Supernovae can enrich entire galaxies with nickel, which after less than a 100-day half-life, ultimately decays to the stable form of iron, iron-56 [4]. The timescales involved with these supernovae subject materials to extreme strain rates, which in turn give rise to significant deformations that have rarely been studied.

1.3 Deformation Mechanisms of Iron

Pure iron is subject to a variety of deformation mechanisms, a few of which include slip, twinning and phase transformation. For a generalized introduction to each one of these mechanisms, refer to Basic Engineering Plasticity, by David Rees [5]. The following sections outline specific studies on the phase transformation observed in iron.

1.3.1 Alpha-Epsilon Phase Transformation

Pure iron is interesting not only for its abundance and strength scaling properties, but also because it readily undergoes a pressure and temperature induced phase transformation. At room temperature and pressure, pure iron exists in a Body-Centered-Cubic (BCC), $\alpha$-phase known as “ferrite” [6]. At 13 GPa (0.13 Mbar), pure iron transforms into a Hexagonal-Close-Packed (HCP), $\varepsilon$-phase known as “hexaferrum”. Due to the high pressure and temperatures that are postulated as being the conditions within the inner and outer cores, $\varepsilon$-iron is thought to be stable or metastable in the Earth’s interior [7].

At ambient pressure, phase transformation also takes place in iron with temperature changes. As pure iron cools from a molten state, it will crystallize at approximately 1,538°C to a BCC “Delta” $\delta$-phase. With further cooling to approximately 1,394°C, the crystal structure of pure iron transforms to a Face-Centered-Cubic (FCC),
“Gamma” $\gamma$-phase known as “austenite” [6]. Finally, after cooling to approximately 912°C, pure iron reverts to the room temperature-pressure phase, $\alpha$-iron. A detailed phase diagram for pure iron is illustrated in Figure 1.1.

![Figure 1.1 - Phase diagram of Pure Iron](image)

Figure 1.1 – Phase diagram of Pure Iron. The $\alpha$-$\varepsilon$ phase transformation is shown at 13 GPa (130 kbar). [7]

It has been shown that the HCP phase can be derived from the BCC structure if loading along the [001] direction (See Figure 1.2) [2]. This compression reduces the d-spacing of the BCC [002] plane and causes the shuffling of the (110) planes in the $[1\bar{1}0]$ and $[\bar{1}10]$ directions [2]. This mechanism has been confirmed at similar pressures with shock-compressed iron via nanosecond X-ray diffraction [8]. While this proposed mechanism has been confirmed via diamond-anvil and laser shock compression at lower pressures, little is known about the mechanism at pressures exceeding 100 GPa.
A multitude of investigations have taken place which have provided indirect evidence of the $\alpha$-$\varepsilon$-$\alpha$ reversible phase transformation in iron, however, post-shot forensics had not identified microstructural signatures of this phase transformation [3].

Figure 1.2 – Mechanism of BCC-HCP phase transformation in iron. a) [110] projection of the BCC phase. b) [001] projection of HCP phase. The BCC phase is transformed to the HCP phase by compression along the [001] and shearing of the A layer along $[1\bar{1}0]$ [9].

During the efforts of Dougherty et al., they subjected circular plates of 1018 steel to shock compression tests with a flyer plate in an 80-mm gas gun. While in fabrication, the 1018 steel plates cooled to a solid state, promoting a eutectoid reaction, thus creating carbon free $\alpha$-iron grains interspersed in pearlite colonies [3]. The flyer plates created impact pressures of 14 and 16.3 GPa on the steel and then were soft recovered and characterized using electron microscopy [3].

The microscopy analysis utilized electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) to identify a rare twin mode only present after the $\alpha$-$\varepsilon$ phase transformation pressure was exceeded. Along with the most common twin in BCC metals $\{112\}<111>$, the rare twin associated with the $\alpha$-$\varepsilon$ phase
transformation was identified as \{332\}<113> twins. These rare twins were primarily found to be secondary twins because they resided within the common BCC twin \{112\}<111> (seen in Figure 1.3). An interesting aspect about the appearance of these twins is that when the entire plate was surveyed at the impact zone, the special twins only appeared near the surface of the impact. The authors concluded that at the surface of the plate, the metal almost certainly went through the reversible \(\alpha\rightarrow\epsilon\rightarrow\alpha\) phase transformation, and thus, the appearance of this rare twin in post-shot microstructures is indicative that the area was impacted with pressures sufficient enough to transform \(\alpha\)-iron to \(\epsilon\)-iron [3].

Figure 1.3 - EBSD identification of the rare twin mode found as a result of exceeding the \(\alpha\rightarrow\epsilon\) phase transformation pressure in iron. The features outlined in black are the \{332\}<113> rare twins which are secondary twins located inside white \{112\}<111> primary twins [3].
1.3.2 Plasticity at High Pressures, Long Time Scales

The \( \alpha-\varepsilon \) (BCC-HCP) phase transformation mechanism has been extensively studied with synchrotron radiation using a diamond-anvil cell [9]. In a particular experiment at the Cornell High-Energy Synchrotron Source (CHESS), polycrystalline iron was placed parallel to the loading axis and pressure on the sample was increased and then decreased. Using the diffraction patterns produced by the beam incident on the sample at various loading conditions, the experiment was able to conclude that the \( \alpha-\varepsilon \) phase transformation starts between 10.3 and 11.3 GPa [9]. At 21 GPa, the \( \alpha \)-phase was completely eliminated, only to reappear when unloading pressure to the range of 16.3 to 15.4 GPa [9]. The experiment noted that the \( \varepsilon \)-phase disappeared completely when unloading below 3 GPa [9]. While this experiment very accurately captured the \( \alpha-\varepsilon \) phase transformation with small constant pressures changes, what is not known is how this phase transformation takes place with impulse pressure in the megabar regime at nano-timescales. Even less is known about the microstructure present at these conditions.

Due to the dramatic strengthening that occurs in pure iron at high pressure, the \( \alpha-\varepsilon \) phase transformation is assumed to be a main contributor and has thus been the subject of extensive study via different methods of compressive shock loading. As opposed to past studies, this research is intended to observe the effects of laser shock loading on the microstructure observed in pure iron.

1.3.3 The Plastic Deformation of Iron at High Pressures in the Inner Core

Earth’s solid inner core is thought to be made up of a nickel-containing iron alloy [10]. Interestingly enough, this means that the core metals, which are inevitably subject
to high pressure, exhibit cubic crystal lattices (Nickel-FCC, Iron BCC) at ambient temperature and pressure (acknowledging the binary phase diagram includes other ordered intermetallic phases). Cubic lattices have a great deal of crystal symmetry and are known to withstand extreme pressures while remaining solid. The Clausius-Clapeyron effect also aids in this solid state at high pressures.

Wenk et al. have conducted diamond anvil experiments on iron powder (grain size of 0.1 – 0.5 µm) at high pressures in order to determine deformation mechanisms taking place at or around Earth’s core [10]. Their experiment utilized a synchrotron source with a goal of characterizing what deformation mechanisms lead to the anisotropy measured at the Earth’s core. During their experiment, they rotated the cell around an axis perpendicular to the diamond-anvil axis at 10° intervals, thus enabling diffraction for different lattice planes [10]. In their first run, they used a 10-µm thick, 20-µm diameter sample and captured patterns at constant pressure of 54 GPa for 10 minutes per each pattern. The second run used a sample which was 5-µm thick and 15-µm in diameter and was subjected to 220 GPa while each diffraction pattern was captured in 60 minutes [10].

The results they obtained were then compared to simulations using other HCP materials such as zinc and titanium as a baseline. The simulation takes into account the identification of several slip and twinning systems common for HCP materials and applies a finite element self-consistent approach by imposing a uniform strain path which in turn approximates the deformation in the diamond anvil cell [10]. Analysis of the synchrotron diffraction data produced a textured orientation of the various loadings in the form of inverse pole figures (IPFs). Comparing the experimental texture with the
texture produced by several simulations with differing active deformation mechanisms, the authors remark that their simulations provide good agreement with the observed texture. The analysis concludes that large plastic strain (50 – 100%) was attained and basal slip is an active deformation mechanism in $\varepsilon$-iron and possibly coalesce with other systems [10]. They arrive at this conclusion based on the activity of twinning and slip systems output by their simulation as seen in Figure 1.4.

![Figure 1.4 - Calculated activities of basal and prismatic slip in HCP iron as a function of compressive strain. The simulation uses polycrystalline plasticity assumptions with critical shear stress ratios (prismatic to basal): 2-1, 1-1, 0.5-1 and 0.1-1. The simulation shows all prismatic systems decreasing and basal systems increasing. [10]](image)

This experiment utilized small sample sizes with very small grain sizes 0.1 – 0.5 $\mu$m. The very small grain size coupled with a constant pressure delivered by a diamond anvil may not apply to experiments where iron has larger grain sizes. In fact, Bergman suggest for geophysical plausible stresses, the grain sizes at the inner core exists at a size range from 0.1 – 10 meters [11]. In addition, the constant pressure exerted by the
diamond anvil does not accurately illustrate a laser shock loading condition. Identifying deformation mechanisms at high pressure via simulation should also be confirmed using microscopy techniques.

1.3.4 **Microband Formation in Shock-loaded and Quasi-statically Deformed Metals**

Huang and Gray studied the formation of microbanding structures resulting from shock-loading high-purity aluminum (Al), copper (Cu), gold (Au) (all FCC) and niobium (Nb) (BCC) [12]. In their experiment, they used a gas gun to drive Al and Cu to 13 and 10 GPa respectively, and an explosive-driven flyer-plate technique to drive Ag and Nb to 48.6 and 37 GPa respectively. During their thorough post-shot examination of the various metals, they frequently observed pronounced microbanding structures [12]. Examples of the microbanding observed by Huang and Gray are shown in Figure 1.5 on page 12.

Double-dislocation walls approximately 0.1 – 0.4 µm apart (mostly ~0.2 µm) lying within a few degrees of crystallographic slip planes are indicative of microbands [12]. Also a characteristic of microbands, the volume within these dislocation walls has a 1-3° misorientation with respect to the surrounding matrix. This low initial misorientation is thought to increase with increasing strain. These microbands carry shear strains with them, which is evident based on the relative displacements at the intersection of several microbands or low angle grain boundaries [12]. Microbands sometimes appear to have close resemblance with coarse slip bands, because both of these band-type structures lie on slip planes. Out of all the pure metal samples Huang and Gray observed after loading, only niobium (the BCC metal) appeared to have microbands and slip bands.
coexisting together. However, although both of these features appear in the same sample, they make note that no slip bands were observed in grains with profuse microbands and vise versa [12]. The fact that this coexistence is only observed in the pure BCC metal is interesting because during ambient conditions, iron is in a BCC, $\alpha$-phase.

Based on their experimental observations, the formation of microbanding does not appear to have dependence upon crystal structure, material properties, strain level, or deformation path. In fact, they stipulate that the mechanism for microbanding may be similar in a variety of FCC and BCC metals and alloys [12]. Also, based on their analysis, thermal trapping does not appear to play a role in the substructure evolution of microbands where materials were “soft” recovered after shock loading at moderate pressures [12].

Huang and Gray proposed a mechanism for the formation of microbands. An illustration of this mechanism is shown in Figure 1.6 on page 13. A key assumption of their model is that microbands can only form when a slip-band like feature has already been induced [12]. Also mentioned in their paper, Huang and Gray discuss the work done by Jackson [13] in which he proposed that microband formation is likely the result of an unstable slip on a latent slip plane via a cross-slip mechanism from an incident glide plane [12].

1.4 High Pressure Materials Testing

Over the past several decades, the desire to study materials at higher strain rates has driven experimentation methods to new heights. Figure 1.7 on page 14 shows several compression testing methods commonly used in materials
characterization. Starting at the lower end of the strain rate spectrum with a range of $10^{-6}$ to $10^{-2}$, one can visualize a typical quasi-static testing machine. A normal quasi-static mechanical load frame can put tensile bars in tension or compression during testing and delivers a very slow, controlled strain rate. In the $10^{-2}$ to $10^{2}$ range of strain rate, one might expect a high-rate quasi-static mechanical load frame. This machine uses the same methodology as the normal mechanical load frame, but delivers an impact that is capable of exhibiting strain rates experienced during a high-speed car crash. Increasing strain rate further, Kolsky bar, gas-gun and flyer plate experiments reside in the $10^{2}$ to $10^{6}$ strain rate range. One might envision the strain rates experienced with bullets piercing through armor as being analogous to these types of experiments.

![Figure 1.5 – TEM micrographs of microbands observed in a) pure Ag shock loaded to 48.6 GPa, b) pure Cu shock loaded to 10 GPa, c) pure Al shock loaded to 13 GPa, and d) pure Nb shock loaded to 37 GPa. [12].](image)

Figure 1.5 – TEM micrographs of microbands observed in a) pure Ag shock loaded to 48.6 GPa, b) pure Cu shock loaded to 10 GPa, c) pure Al shock loaded to 13 GPa, and d) pure Nb shock loaded to 37 GPa. [12].
Figure 1.6 – Huang and Gray illustration of proposed microband formation model. a) coarse slip bands on primary slip planes (dislocations are random) b) the same coarse slip band creates polarized dislocations c) double-wall features are formed after the polarized dislocations are annihilated in the central part of the band structure d) secondary dislocations become activated between these double walls because of internal stresses from primary dislocation arrays e) formation of microband structure with stable dislocation arrays [12].

Laser compression currently serves as the highest strain rate bound of manmade compression experiments. Extreme transfers of energy, which can take place in a nanosecond, delivers strain rates that could be present during a nuclear explosion or
Laser compression currently serves as the highest strain rate bound of manmade compression experiments. Extreme transfers of energy, which can take place in a nanosecond, delivers strain rates that could be present during a nuclear explosion or the onset of a supernova. Transmitting strain rates in the $10^6$ to $10^{10}$ regimes, laser compression is currently one of the most feasible ways to simulate pressures present in the inner core of Earth or during a supernova event. In an effort to explore and fully characterize the deformation mechanics and associated high pressure strengthening mechanisms in iron, the experiment outlined in this report utilized laser compression as the means to simulate extreme pressure and strain rate.
CHAPTER 2: METHODS

From manufacturing, to post-shot analysis, the iron samples used in this research succumb to several rigorous routines. To start, a brief description of the manufacturing process of the samples and the experimental setup is described in Sections 2.1 and 2.2 respectively. The post-shot samples then endure a rigorous sample preparation procedure as outlined in Section 2.3. Analysis techniques then follow, where a description of the nanoindentation, microscopy investigation and two-surface analysis are detailed in Sections 2.4, 2.5, and 2.6 respectively.

2.1 Manufacturing of Samples

General Atomics fabricated the iron samples used in this experiment from four-nines (99.99%) high-purity iron plate stock that was purchased from Goodfellow. Multimode sinusoid patterns were coined onto the surface of the plate. Cylindrical samples 4 mm in diameter and 1 mm in height were then cut from the plate, through the coined patterns. The purpose of this microcoining was to have a macroscopic measure of plastic deformation as a result of the laser shocks, as has been described elsewhere [14], [15]. In the present work, the goal is to characterize the microstructure and associated deformation mechanism response to laser-shock loading, therefore the coined patterns are not further discussed.

2.2 Experimental Setup

The iron samples were shot on the Omega Laser at the Laboratory for Laser Energetics (LLE) at the University of Rochester (Rochester, New York). The Omega laser is one of the most powerful and highest energy laser systems in the world. On
Omega, there are 60 laser beams that when focused, are capable of delivering 30,000 Joules in a nanosecond [16]. Current research on Omega centers on high energy density research (HEDM) and internal confinement fusion.

The experiment utilized a Lawrence Livermore National Laboratory (LLNL), General Atomics (GA) and CalTech built experimental platform called the “ride-along” recovery tube. This universal recovery tube was designed specifically for studying the strength of materials at pressures greater than 1 Mbar [17]. The term “ride-along” refers to being able to perform experiments on one of Omega’s unused laser beams while not interfering with the primary experiment [17]. A picture of the recovery tube can be seen in Figure 2.1. The recovery tube was filled with aerogel which acted as a catcher for post-shot target recovery [17]. To ensure adequate recovery, the aerogel had to be the right density so that it was not hard enough to break the sample upon impact, but it also did not allow the sample to fly through the material easily [17]. In front of the aerogel was a stainless steel retaining ring that held the target stack in place. Without the retaining ring, the entire target stack could have lodged into the aerogel catcher upon laser impact. Near the end of the recovery tube was a stainless steel adaptor that connected the tube to the aluminum Nova Mount. This Nova Mount enabled the recovery tube to attach to Omega.

The main goal of this research was to study the effect of high pressure and high strain rate on pure iron. However, direct contact between the laser beam and metal would turn the metal into plasma, which is a highly undesirable result because one cannot ascertain usable strength property information from plasma. In order to deliver a compressive shockwave without melting or exploding the iron targets, a Richtmyer-
Meshkov (RM) target stack was used. The objective of using the target stack is to remove as much thermal energy as possible while simultaneously driving a pure compression wave through the targets, thus allowing for target recovery.

Figure 2.1 – Illustration of cross-section of the universal ride-along recovery tube used in laser compression experiments at Omega. This recovery tube enabled the experiment to “ride-along” on an unused beam next to a primary experiment without interference. The target stack is placed on the laser entrance side of the tube, which is then held in place by a stainless steel retaining ring.

The Richtmyer-Meshkov instability is a phenomenon that occurs when a less dense medium is accelerated into a heavier medium via shock wave passage [18]. In this experiment, an LLNL built RM target stack was used as a means to drive a compression wave from a less dense plastic ablator (CH) to a higher density metal (Iron). A close-up illustration of the target stack can be seen in Figure 2.2. One can see from Figure 2.2 that the laser entered the stack from right to left through the opening of a stainless steel mounting washer and passed through an aluminum flashing. The laser then continued through an ablator (CH) and heat shield (BrCH) before finally hitting the surface of the target. Each target stack used the same
components in the same order, but the ablators, heat shields, and laser energies were specifically designed using Hyades hydrodynamic simulations.

Figure 2.2 – Illustration of cross section of the target stack, which was mounted in front of the recovery tube. The laser enters the stack from the right, passing through the opening a mounting washer, and then penetrating the aluminum flashing in front of the stack. When the laser strikes the ablator, a square compression shockwave is sent through a heat shield, which removes a significant amount of thermal energy, and then subsequently hits the iron target. Hyades, a 1D hydrodynamic code, is used to design the target stack parameters [15].

Due to the nature of being a ride-along experiment on an unused beam, there was no in-situ VISAR attached to the target stack; therefore the experiment relied heavily on simulations for design parameters. VISAR stands for Velocity Interferometer System for Any Reflector and is used to document a complete beam velocity history vs. time as a sample surface is impacted [19]. Used in this experiment, was a 1D radiation hydrodynamic simulation code, which simulates how dense plasmas are driven by intense energies; this code was developed by John Larson [20]. Hyades used laser energy along with heat shield and ablator thicknesses from past laser shots, as input parameters, which was then tuned to match VISAR velocity, pressure and temperature
profiles at each component in the target stack [17]. The final step in calibrating the Hyades simulation was to match the simulation with VISAR velocity profiles and target exit times near the laser exit surface of the sample, which was again measured from past experiments. Once the Hyades simulation was fully calibrated, it calculated pressure, velocity and temperature profiles at each target stack component as a function of laser energy [17]. Peak pressure as a result of the applied pressure wave was then also determined from Hyades.

Hyades simulations were then used to optimize the design of the ablator and heat shield components in the target stack with reference to the attenuating characteristics of each material. Due to the relatively unknown pressure levels at which various deformation mechanisms materialize, the experiment used design parameters that allowed focus on a range of pressures for characterization purposes.

For analysis of low, mid, and high range pressure levels, laser shot energies of 70, 100, and 150 J respectively were requested. The laser beam used for the entire experiment for all laser shots had a 350-nanometer wavelength, a one-nanosecond pulse, and an 800-µm diameter circular spot geometry. Concluding the experiment, a naming convention for the samples was developed such that Fe-Unshot represented the baseline iron sample which was stamped, but not shot, Fe-2 corresponded with the 70 J requested energy, Fe-3 with 100 J requested energy, and Fe-4 with 150 J requested energy.

2.3 Post-Shot Sample Preparation

In order to perform a post-shot investigation on the iron samples utilizing microscopy techniques, the samples needed to go through a rigorous polishing
procedure. This procedure involved sectioning the as-delivered samples, mounting them, and taking them through coarse and fine polishing steps. A table summary of the coarse and fine polishing steps used in polishing the shot iron samples can be found at the end of the Fine Polishing subsection.

2.3.1 Sectioning

The process of sectioning the samples was extremely tedious and was taken with great caution. With respect to the overall size of the sample, the shot area was very small (~800 µm diameter). Therefore, large sectioning disks were not appropriate for this application. To preserve as much material as possible with a higher level of precision cutting, the post-shot samples were sectioned using a 11-1180 Low Speed Saw manufactured by Buehler LTD. A CBN Metal Bonded wafering blade, with dimensions 4” x 0.012” x ½” (manufactured by Allied High Tech Products Inc. - Part #60-20071) was used in conjunction with the low speed saw to section the samples. The RPM setting on the saw was set to 3 or 4 (out of 10) during the sectioning of all samples.

The orientation of the sample with respect to the cut was extremely important. In order to accurately observe a cross-section of the triple-sinusoid pattern stamped on the sample surface with microscopy; the experiment needed to cut directly perpendicular to the coining pattern stamped on the sample surface. Lining up the sample holder on the saw so that the blade would cut perpendicular to the pattern was not easy, therefore the utilization of an optical microscope, a light source (for viewing the pattern) and tweezers allowed for higher precision when lining up the sample for cutting.
The samples were cut intentionally thick (i.e. off-center) to ensure a sufficient amount of material was available so that polishing steps would not remove or overshoot the shot epicenter. Figure 2.3 shows an approximate representation of how the samples were cut. After sectioning, the larger half of the sample was mounted and the smaller half was put away as a backup sample if needed. Concluding the sectioning process, the samples were cleaned of oil using warm soapy water.

Figure 2.3 – Approximate sectioning lines for target Fe-4 (red). All samples were cut intentionally thick; mounting the thick side to ensure polishing wouldn’t remove material in the laser impact epicenter. The laser impact and stamped surface are evident on the post-shot sample.

2.3.2 Mounting

The next step in the sample preparation was to mount the sectioned samples in a non-conductive material that can also be used as a leverage point for hand polishing.
For this process, a LECO PR-32 Mounting Press with a Bakelite medium was utilized. Bakelite is a non-conductive thermoset resin that is commonly used for mounting applications.

First, any burrs or tailings from the sectioning process was either cut or ground off so that the sample could sit flush on a level surface. The sample was then arranged cross-section down on a level surface, and a plastic/metallic sample-clip was stretched so that the clip jaws surrounded the sample. The sample clips acted as a safeguard to ensure the sample remained upright during the mounting process.

The clipped sample was then set on top of the mounting press, sample side down. Without proceeding any further, one had to check that the sample and clamped sample clip were both flush with the mounting press head. If there was any space between the sample cross-section surface and the ram head, there was a high likelihood that melted resin could fill the space between the ram head and sample, therefore trapping the sample in a large thickness of Bakelite. Due to the nature of the size associated with the samples, this is an undesired event that could result in accidently polishing through the sample shot area.

When the clipped sample is properly arranged on the ram head, the user then retracted the ram head into the press and added approximately $30 \text{ cm}^3$ of bulk Bakelite into the cavity. If the Bakelite was added too quickly, the sample will move on the ram head and will therefore not be centered after mounting.

A crosshead was then attached to the mounting press and screwed down. The user drove up the ram and then the pressed the “start” button. The mounting press then heated up the Bakelite while it simultaneously applied upward pressure with the
hydraulic ram. After the Bakelite was completely melted and compressed, the press then went through an automatic cooling cycle. From start to finish, the process took approximately 15-16 minutes per sample and the final product was a puck-shaped mounted sample with a 1.25" diameter and 1" height (seen in Figure 2.4). These dimensions are ideal for the analysis because the electron microscopy laboratory at the Colorado School of Mines had sample holders that are specific for these dimensions.

![Figure 2.4](image)

Figure 2.4 – A cross-section of a sample mounted in Bakelite. The black edge on the corner of the silver sample cross-section is a remnant of the mounting procedure and is polished away before continuing with the polishing procedure.

Inevitably, there was some small layer of Bakelite between the sample cross-section surface and the bottom of the mount. Before starting the grinding stage, this layer was removed.
2.3.3 Coarse Grinding

The grinding stage was an essential part of the sample preparation that needed to be taken with caution. As before with sectioning, the grinding step was sensitive to material size, and the shot areas of interest for the samples were very small and could be polished through with relative ease. The object of the grinding steps was to methodically use coarse sandpaper to remove material and “grind-down” to a finer polish. Unless otherwise noted, the following steps were all performed on the LECO DS-20 Wet Sanding unit.

Before beginning the grinding process, the person performing the grinding needed to take note of the direction of grinding with respect to the orientation of the sample. Completing a “grinding” grit size step was somewhat subjective; therefore being able to take into account orientation of scratches in the sample became important as an indicator of step completion. After each satisfactory grinding step, one rotated the sample 90 degrees with respect to the direction of grinding from the previous step. Utilizing this method and an optical microscope, one could easily see if the scratches embedded in the material from the previous step had been ground away, which was the indication that it was appropriate to start the next grit size.

Technique was important when holding the sample during coarse grinding. Unlike fine polishing steps where motion was applied to the sample via electric motor, the coarse grinding process utilized human muscle power to drive the sample across sandpaper. This downward force applied by the user was difficult to keep constant and the intention was not to apply so much force that deep scratches pervaded the sample surface. Keeping this in mind, extra consideration was given during the larger grit sizes.
Using the thumb on one side of the sample with index and middle finger on the other side, one gently pushed the sample in one direction along the sandpaper roll. The idea was to add as little force as possible to keep the sample surface in contact with the sandpaper. With the addition of water between the sandpaper and sample, one essentially glided the puck across the sandpaper. After one “stroke” across the roll of sandpaper, the sample was lifted up and the same motion completed (i.e. perform one motion only on the sandpaper). This ensured even distribution and direction of scratches. The quality of the polish was largely dependent on utilizing the proper handling technique.

The first grit size on the wet sanding unit was 240 (50 μm particle size). Since this was the first and largest grit size, it was easy to see the scratches embedded in the material. Once uniform scratches were observed (after 5-7 motions depending on down force applied), the sample was turned 90 degrees and was started on the next grit size. The second grit size was 320 (29.5 μm particle size). To observe for completeness, one took the sample to an optical microscope and made sure the scratches from the previous step had almost entirely disappeared (ground away). This second step took approximately 10 forward one-way motions. The third grit size on the wet sanding unit was 400 (18.3 μm particle size) and it took approximately 15-20 forward one-way motions to erase the scratches from the previous step. The fourth and final grit size on the sanding unit was 600 grit (10.6 μm particle size). This took approximately 20-25 forward one-way grinding motions to erase the scratches from the previous step.

The final coarse grinding step was performed on an 800 grit (7.8 μm particle size) sandpaper PSA disk. The adhesive disk was not placed on a wheel; rather it was laid
on a flat surface. Before grinding on this disk, the disk had water applied to it, acting like a lubricant so the sample did not stick and overly embed into the sandpaper. The technique here was to use a front and back motion on the sandpaper, again being careful not to add too much downward force. After several full motions front and back on the sandpaper, it was advised to change the orientation of the user’s grip on the sample so that a stochastic scratch pattern could be achieved. In addition to changing the user’s grip, one also utilized the entire grinding disk by changing starting locations to begin motions. This step was considered complete after about two minutes on the sandpaper, or until a uniform scratch size across the sample was observed via optical microscope.

### 2.3.4 Fine Polishing

The fine polishing steps were the most crucial steps for obtaining a satisfactory polish that was suitable for high quality microscopy. Handling pressure, time, and technique all play an important role in achieving a good polish during these steps. The polishing procedure now transitioned from using sandpaper, which used hands as the driving force, to polishing wheels, which used electric motors to provide movement.

In the fine polishing steps a premium diamond suspension slurry (heavy concentration) manufactured by LECO was used as the polishing medium. This suspension was sprayed via squirt bottle onto a cloth with varying levels of nap (i.e. length of fibers on polishing cloth). The polishing cloth had an adhesive side that was carefully stuck onto the top of a metal polishing wheel. Before continuing, the user had to ensure that there were no air pockets between the cloth and the wheel. This could have yielded an uneven surface resulting in a poor polishing step. For the fine polishing
evolution, a VP-160 Variable Speed Polishing Unit manufactured by LECO with an 8” polishing wheel was used.

The VP-160 has an adjustable RPM setting from 0 – 600 RPM. Usually there is a trade-off between longer polishing times and a higher RPM setting on the wheel. With longer polishing time, pitting has been observed in soft materials like that of pure iron. From the preliminary polishing tests, it was concluded that the samples could utilize a high RPM setting without significant pitting. For all polishing steps on the VP-160 unit, the RPM ranged from 400-450 RPM.

The addition of the polishing wheel added more nuances to achieving the proper technique. First, the relatively high RPM setting created a significant lateral force to the mounted Bakelite puck. Without the proper hold on the puck, it was very difficult to maintain contact with the polishing cloth and suspended media. A low center of gravity approach was applied. To do this, the thumb and middle finger were placed low (close to bottom) on both sides of the puck and the index finger was placed on top. Using this grip, one was easily able to apply enough force to resist the intense motion from the high RPM setting and increase an even exposure to the polishing media. In order to get a more homogenous and even movement of diamond media across the sample, the sample was moved in circles, counter to the motion, around various radii of the wheel. In addition to this movement, the sample grip was rotated every few minutes.

The first fine polishing step was using the 6-µm diamond slurry on an 8” Lecloth polishing pad by LECO. Enough of this solution was applied to the wheel to ensure that the polishing pad remained wet at all times. Every few minutes, more 6-µm diamond slurry was added to the polishing cloth while in motion. To enable the diamond slurry to
last longer without more slurry application, periodically a red Diamond Compound Extender (by LECO) was added to the wheel (for both 6-µm and 1-µm sized media). During the initial testing of the polishing procedure, the tests showed that the 6-µm diamond step was crucial for observing pitting in the final polish. Using a trial and error method, it was found that with a small application of downward force, scratches could be successfully removed from the 800-grit step in approximately 20 – 30 minutes using 6-µm diamond slurry. If the sample exceeded 30 minutes on the 6-µm step, large pitting was observed that could not be removed using 1-µm diamond slurry.

The last step on the polishing wheel was using the 1-µm diamond slurry on an Imperial polishing cloth by LECO. The Imperial pad had a much finer nap, which was ideal for application of very small polishing medias. Much like the 6-µm step, the Imperial cloth was saturated with 1-µm diamonds and an additional application of 1-µm slurry or diamond compound extender was added every few minutes (or when the polishing cloth felt dry). Polishing time was important on the 1-µm step because removing the 6-µm sized scratches was essential to continue. It was found that if there was a small increase of downward pressure than was applied in the 6-µm step, the 6-µm scratches could be removed in approximately 45-60 minutes. It is possible to achieve this step quicker with the addition of more downward force on the wheel, but in doing this, there is also a higher risk of embedding material in the sample. Ultimately, the polishing step was considered done when there were no more remnant scratches from the 6-µm step.
A comprehensive summary table of all polishing steps is shown in Table 2.1. Following the completion of the polishing steps in Table 2.1, a satisfactory polish was obtained for EBSD study on the electron microscope.

### 2.3.5 VibroMet

The VibroMet is a vibratory polisher that provides the finishing touch on the polishing procedure. Aside from electro polishing, this is the preferred last step by many microscopists if they intend to perform analyses that require high quality finishes. To start, the user attached an adhesive polishing cloth with a fine nap to the circular polishing area. After that, a generous amount of 0.05-µm suspended colloidal silica was applied to the surface of the wheel. The Bakelite mounted sample was then secured

<table>
<thead>
<tr>
<th>Grinding Type</th>
<th>Grit/Particle Size</th>
<th>Polishing Medium</th>
<th>Polishing Apparatus</th>
<th>Time</th>
<th>Downward Force Applied</th>
<th>Notes/Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coarse</td>
<td>240/50 micron</td>
<td>Sandpaper</td>
<td>LECO DS-20 Wet Sanding Unit</td>
<td>N/A</td>
<td>Little as possible</td>
<td></td>
</tr>
<tr>
<td></td>
<td>320/29.5 micron</td>
<td>Sandpaper</td>
<td>LECO DS-20 Wet Sanding Unit</td>
<td>N/A</td>
<td>Little as possible</td>
<td>~5 - 7 one way forward motions, or until homogenous scratches are observed</td>
</tr>
<tr>
<td></td>
<td>400/18.3 micron</td>
<td>Sandpaper</td>
<td>LECO DS-20 Wet Sanding Unit</td>
<td>N/A</td>
<td>Little as possible</td>
<td>~10 one way forward motions</td>
</tr>
<tr>
<td></td>
<td>600/10.6 micron</td>
<td>Sandpaper</td>
<td>LECO DS-20 Wet Sanding Unit</td>
<td>N/A</td>
<td>Little as possible</td>
<td>~15 - 20 one way forward motions</td>
</tr>
<tr>
<td></td>
<td>800/7.8 micron</td>
<td>Sandpaper</td>
<td>8” LECO PSA Disk on level surface with water</td>
<td>2 minutes</td>
<td>Little as possible</td>
<td>~20 - 25 one way forward motions</td>
</tr>
</tbody>
</table>

| Fine          | 6 micron           | Diamonds in Suspension | LECO Locotch Pad (with PSA) on LECO VP-160 Variable Speed Polisher | 20 - 30 minutes | Some downward force | Wheel @ 400 RPM, changing grip and location on Polishing pad frequently |
|               | 1 micron           | Diamonds in Suspension | LECO Imperial Polishing Cloth (with PSA) on LECO VP-160 Variable Speed Polisher | 45 - 60 minutes | More downward force than 1 micron step | Wheel @ 400 RPM, changing grip and location on Polishing cloth frequently |
|               | 0.05 micron        | Colloidal Silica     | VibroMet 2 - Vibratory Polisher by Buehler | 75 - 90 minutes | Weight of sample holder only, no additional weights | Amplitude = 60%, clockwise cycle |

Table 2.1 – Polishing procedure used for high-purity (99.99%) iron samples. This polishing procedure uses 3 different polishing medias and the majority of the procedure involves hand polishing.
down in a sample holder via setscrew. Before continuing one needed to observe that all of the weight of the mounted sample was on the surface of the puck (i.e. not the holder), which allowed for even distribution of force across the sample. The VibroMet adjusts vibrational speed via percent amplitude. The user selected the desired operating amplitude, then while the machine was running, the sample was gently placed face down on the colloidal silica laden pad. The sample then moved with constant velocity in a circular pattern around the disk. For the polishing of the iron samples, a VibroMet 2 by Buehler was used at 60% amplitude for 75 – 90 minutes. This timing was formulated based on previous experiences of members in the metallurgy department (at the Colorado School of Mines) that suggested softer materials such as pure iron would experience significant pitting if subject to vibratory polishing exceeding 90 minutes.

Upon completion of the vibratory polishing, the finished sample was taken off the pad, and then released from the sample holder. At this point, even after a thorough rinsing, the sample surface inevitably still has colloidal silica attached to it. To remove this, an alcohol base (ethanol or isopropanol) was generously applied with a wash bottle for several seconds, followed by a fairly vigorous scrubbing with a cotton ball. This process was repeated several times, each time using a new cotton ball. Finally, to remove any traces of alcohol smudges, the mounted sample was run under water and dried with an air dryer. One then verified that the sample surface was clear of embedded colloidal particles with inspection on an optical microscope.

2.3.6 Etching

Before any significant microscopy was completed on the samples, an initial investigation of the grain structure and substructure of the shot samples had to be
completed. To do this, an etchant specific for pure iron was created, which was found in the LECO Metallography Principles and Procedures guidebook. The name of the etchant was Nital, a common etchant routinely used for etching metals. Using the recipe in the LECO Metallography Principles and Procedures guidebook, a 3% Nital solution (3 mL Nitric acid, 100 mL Methanol) was mixed. With this recipe, as one increases the concentration by adding more Nitric acid, the etching rate is increased. This is an undesired result for the experiment because it was important to have very close control of how fast the material was etched.

After the etchant is produced, a sight glass was placed in a fume hood, and several milliliters of etchant were poured onto the convex side. Being very mindful of time, the mounted sample was placed face down in the solution for five seconds at a time. After each time in the solution, the sample was quickly and vigorously rinsed with ethanol and then cotton swabbed to remove all etchant. The sample was then brought over to an optical microscope and inspected for completeness. This process was repeated until all or most of the grain boundaries were elucidated from each sample. Over-etching is a serious problem that the user should be careful about, as it will produce significant surface deformation. During the course of the etching process, the samples never exceeded 30 seconds of total etching time, and still there was some evidence of over-etching.

2.3.7 Sample Storage

Pure iron readily undergoes an oxidation reaction when exposed to oxygen-rich environments with moisture for long periods of time (i.e. days). This process can lead to the formation of an oxide layer up to 50 nanometers deep. This is commonly seen in
the form of rust. The oxide layer on the surface of the samples can result in significant charging issues while utilizing electron microscopy, and thus should be avoided as much as possible. However, if this does occur, a 30 – 40 minute rebuff on the VibroMet 2 with colloidal silica can remove this oxide layer.

To alleviate this problem altogether and to reliably preserve the high quality polish of the specimens, the experimenters procured a vacuum desiccator and desiccant from Tedpella. 2 DRI-BOX® Reusable Sorbent boxes were used as the desiccant. The desiccant was arranged in the bottom of the vacuum desiccator, with samples lying surface-up on a shelf over the desiccant. A small vacuum pump was used to draw air and moisture out of the vacuum desiccator. There was no vacuum measurement criterion, therefore leaving the vacuum pump on for 2 minutes, and then closing the air valve was sufficient to achieve the vacuum needed for the samples. The vacuum within the desiccator degrades in a matter of weeks, so every week without use, the vacuum pump procedure is completed again. From the moment the samples fully completed the polishing procedure or came out of the electron microscope loading bay, they were put under vacuum in the vacuum desiccator. The samples were essentially in a moisture-free, airtight vacuum all the time when not in use.

2.4 Nano-Indentation

One of the goals of this experiment was to measure material properties at different locations in the samples. To do this, the experiment employed the use of a TI 950 TriboIndenter by Hysitron. This indenter uses load and depth sensing techniques to locally measure the elastic modulus, $E$, and the hardness $H$ of materials at a sub-$\mu$m scale.
The indenter was not a perfectly rigid body; therefore it plays a role in the load displacement behavior [21]. To account for this, the indenter calculated a reduced elastic modulus, $E_r$, defined through equation 2.1.

$$\frac{1}{E_r} = \frac{(1 - v^2)}{E} + \frac{(1 - v_i^2)}{E_i}$$

(2.1)

where $E$ and $v$ are the Young’s modulus and Poisson’s ratio of the specimen, and $E_i$ and $v_i$ are the same parameters for the indenter [21]. Along with the calculation of a reduced Young’s modulus, the indenter also calculated hardness at each indentation using equation 2.2

$$H = \frac{P_{\text{max}}}{A}$$

(2.2)

where $P_{\text{max}}$ is the peak indentation load and $A$ is the projected area of the indenter impression [21].

Due to the extreme strain rate delivered to the targets from the laser, these samples are in relatively uncharted territory as far as material characterization is concerned. It is feasible that the material properties could vary from grain to grain with respect to the location of the laser impact. 3 locations were established for analysis on the shot targets: laser shot center, 300 $\mu$m outside of shot center (still in shot boundary), and outside of the laser impact zone. In addition to testing the sample properties at the surface of the shot specimens, it was also a goal of the experiment to test if the sample properties would change with depth in the targets. Figure 2.6 displays the locations where Fe-2 and Fe-4 were tested with the nanoindenter.
The coining process could have produced a work hardening effect onto the surface of the samples. To test for this, the same nanoindentation process was completed on the unshot iron sample; testing for material properties on the stamp surface, and outside of the stamp near the edge of the sample. Figure 2.5 shows the location of the indentations for the unshot sample.

2.5 Microscopy Techniques

The center focus of the analysis of the shot iron targets was to look at the resulting microstructure utilizing different forms of microscopy. A full range of magnification was required to perform this analysis. There was no single unifying methodology for this, thereby requiring the experiment to utilize a range of microscopy tools. Outlined below is a summary of all methods used to characterize the microstructure of the shot iron samples.

Figure 2.5 – Cross-section illustration of Fe-Unshot showing approximate areas where nanoindentation analysis was done to see if there was work hardening as a result of coining. Dark blue line is under stamp; red line is outside of stamp.
Figure 2.6 - Cross-section illustration of Fe-2 and Fe-4 showing approximate areas where nanoindentation analysis took place for work hardening as a result of the laser shock. The dip at the bottom of the sample represents a spall area where the sample was macroscopically heavily deformed. Lines in blue represent nanoindented patterns under shot center, red represents 300 µm outside of shot center, and yellow represents a location outside of the shot boundary. Note that the analysis took place at several depths within each sample.

2.5.1 Optical Microscopy (OM)

In order to obtain a macroscopic point of view of the damage on the shot samples, an optical microscope (OM) was used. This gave a magnification range from 10x to 1000x. Any micrographs labeled as taken with optical microscopy were taken on PAX-it! Imaging software on the LECO Olympus PMG 3 optical microscope. The OM was also used as the main source of inspection between polishing and etching steps.

2.5.2 Scanning Electron Microscopy (SEM)

The Field Emission Scanning Electron Microscope (FESEM) at the Colorado School of Mines (CSM) was used to make up the entirety of all SEM pictures found in this research (with the exception of the FIB). The SEM at CSM is the JSM-7000F manufactured by JEOL. This SEM has an accelerating voltage range from 10V to 30kV
and a magnification range from 25x to 1,000,000x. Attached to the microscope are a backscatter detector and a charge-coupled device (CCD) sensor used for Electron Backscatter Diffraction analysis.

Several imaging modes were utilized on SEM. First, Secondary Electron Imaging (SEI) was used as a standard for looking at general structure. Second, Topographic Imaging (TOPO) was used to identify topographic features in the samples. TOPO mode often times revealed features not seen in other modes. Lastly, Surface Composition mode (COMPO) was used to elucidate stark contrast differences leading to the observation of deformation structures. In general, greater accelerating voltages displayed better contrast and thus better identification of local features. TOPO and COMPO modes are backscatter modes. COMPO mode was advantageous for viewing contrast differences due to a channeling contrast effect. When special conditions exist, like those in a pure polycrystalline material such as pure iron, the interaction of energetic electrons with the sample surface can be used to detect crystallographic properties and crystal defects [22].

The difference between each of these modes is evident in a side-by-side comparison, but each one was instrumental in characterizing the shot samples. There was no set methodology of what to look for when using these different operating modes, rather the user had to decide locations of interest and exercise discretion as to what classifies as interesting.

Preserving imaging orientation for all samples was of key importance when taking SEM images. Without this preservation, there is no sense of context with respect to the location in the sample or direction of the laser impact. For all images in this
report, one can assume the stamped edge is located upwards with respect to the image location and the laser impact direction is from top to bottom.

2.5.3 Electron Backscatter Diffraction (EBSD)

A pivotal tool for analyzing the microstructure of the laser shot iron targets was the Electron Backscatter Diffraction (EBSD) technique. In general, EBSD can identify crystal orientations, global texture, grain size, phase transformations, slip system activity and fracture analysis etc. Scanning a shot sample with EBSD allows a very powerful analysis to take place whereby identifying local misorientations, twinning, individual grain orientations, and producing maps of inverse pole figures become easy with the help of analysis software. Identifying high angle misorientations is indicative of deformation mechanisms like twinning or grain boundary formation. Lower angle misorientations could be identified as slip bands.

The EBSD data collection software used in this research was produced by EDAX. For analysis of EBSD scans, the Orientation Imaging Microscopy ver. 7.1 (by EDAX) was used.

2.5.4 Focused Ion Beam (FIB)

Repeated observations of banding structures present in Fe-4 led to the need of identifying these planes that the banding structures resided on. To do this, a Focused Ion Beam (FIB) milled perpendicular to the banding structures of Fe-4. A FIB shoots a concentrated beam of Gallium ions with high precision at a targeted area, causing a very localized and highly predictable removal of material.

With respect to Fe-4, the ion beam was aligned directly perpendicular to the sample surface. This provided a 90-degree cut into the sample approximately 7-μm
deep. The stopping criterion for depth was removing enough material so that one could clearly observe a cross-section of the surface banding structure when looking at a sidewall of the cut. An illustration depicting of the location and dimensions of the FIB cut is shown in Figure 2.7. Once the cut was completed, the sample was rotated 90° with respect to the cut, thus enabling imaging of the cross section of the bands. The SEM on the FIB unit then imaged this cross section at 52 degrees with respect to surface of the sample. Figure 2.8 shows how the sample was imaged with SEM.

When observing the finished cut in Figure 2.8, one notices vertical white pieces of material originating at the surface and aligning along the sidewall of the cut. These are essentially tailings as a result of the milling process. A more detailed look at the FIB cut can be found in the results section. The SEM imaging orientation is shown in Figure 2.9.

![Figure 2.7 – Approximate location and dimensions of FIB cut above the spall area in Fe-4. The green arrow points to the face that was used in the two surface analysis. The dark vertical line in the image represents a grain boundary. The cut was 6 µm x 12 µm by 7 µm deep.](image-url)
Figure 2.8 – SEM image of FIB cut shown in Figure 2.7, looking at the face where the green arrow is pointing, imaged at $52^\circ$ with respect to the sample surface. Clearly in view is a contrasted diagonal striation pattern on the face of the cut. This image was used in conjunction with the two-surface analysis.

Figure 2.9 – Imaging orientation picture for the FIB cutting process. The ion column is lined up perpendicular to the sample surface to produce a $90^\circ$ cut. The cut is then imaged at $52^\circ$ with respect to the sample surface.
The FIB was not used in the analysis of Fe-Unshot and Fe-2. The FIB used at CSM is a Helios NanoLab 600i manufactured by FEI.

2.6 Two-Surface Analysis

After numerous EBSD scans and COMPO SEM imaging, prevalent banding structures were found in many locations on Fe-4. A goal of this research was to identify these bands and compare this to the common BCC slip systems. To achieve this, the problem was approached with a two-surface analysis. This analysis required two orthogonal vectors in the plane of the bands, and a crystal orientation at the same location. After finding two vectors that describe the plane, a cross product was then completed, thus identifying the banding plane.

High-resolution EBSD scans were taken across a grain boundary containing these bands. When the scan was completed, it was loaded into the Orientation Imaging Microscopy (OIM) software. Using the OIM software, vectors were drawn parallel to the bands, and normal to the bands in the right grain as seen in Figure 2.10 on page 42. The OIM software outputted the crystal direction parallel to the drawn vectors. There are many banding structures in the right grain, therefore an average direction of 14 vectors drawn parallel to these bands was calculated and used for further calculations. The surface vectors drawn parallel to the bands and the vector drawn perpendicular to the bands provide a basis for the orientation of the bands within the crystal system. The averaged vector describing the direction parallel to the bands in the crystal orientation was found to be [13.64 17.07 2.5] and the vector normal to the bands was found to be [15 -12 -3.66].
Now that EBSD provided the crystal orientation and the directions of vectors plotted along the bands, the last information required is an additional orthogonal vector in the plane of the bands. This required imaging a cross section of the same bands found in the EBSD scan. To do this, the FIB procedure from the previous section was used. The 90-degree FIB cut was imaged on the face where the green arrow points in Figure 2.7. Once this SEM image was taken on this face, vectors were then drawn along this cross section. An illustration of this vector drawing is shown in Figure 2.11 on page 43. Once the vectors have been drawn, angles for each vector were measured with respect to the FIB cut surface edge, which is a known direction in the crystal system because of the normal vector drawn on the surface of the bands. The average angle of each of these bands seen in Figure 2.11 was then used for further calculations.

The first step in calculating the banding plane was establishing a coordinate system to work with. To do this, separate coordinate systems were defined for the banding cross-section as viewed in the 52° viewing angle with SEM, the FIB trench, and the crystal orientation. The coordinate system defined for the trench and viewing angle were called the primed and unprimed systems respectively, as shown in Figure 2.12 on page 44. In order to map the vector as seen in the 52° viewing plane to the FIB trench wall, a transformation matrix was made which defined the transformation from the primed to unprimed systems.

\[
A = \begin{bmatrix}
\cos(i',i) & \cos(i',j) & \cos(i',k) \\
\cos(j',i) & \cos(j',j) & \cos(j',k) \\
\cos(k',i) & \cos(k',j) & \cos(k',k)
\end{bmatrix}
\]  

(2.3)
Figure 2.10 – Illustration of the vector mapping process used for two-surface analysis. The vectors were mapped using OIM Analysis software by TSL. In the software, the “average grain orientations” option was selected before mapping. The mapped coordinates of the vectors can be found in the results section. Green vectors represent vectors drawn along bands on the right hand side of the grain boundary; red vectors represent vectors draw perpendicular to the banding structures.
Figure 2.11 – Vector plotting on the FIB cut face shown in Figure 2.8. Blue vectors represent a coordinate axes which lies in the plane of the 52° viewing angle of the SEM image. Green vectors were drawn along the contrasted striation patterns, where an approximate angle was measured with respect to the viewing angle coordinate axis. The red line represents the grain boundary shown in Figure 2.10.

This transformation matrix is shown in equation 2.3 and uses direction cosines to describe the transformation from one system to another. Evaluating the matrix in equation 2.3 for the transformation shown in Figure 2.12, gives the transformation matrix shown in equation 2.4.

\[
A = \begin{bmatrix}
\cos(-52°) & \cos(38°) & 0 \\
-\cos(38°) & \cos(-52°) & 0 \\
0 & 0 & 1
\end{bmatrix}
\]  \hspace{1cm} (2.4)
After this, a definition for the vector drawn parallel to the cross-section of the bands as observed in the $52^\circ$ viewing plane was defined (this vector is seen as the dashed red line in Figure 2.12 and the green line in Figure 2.11).

The definition of this vector is shown in equation 2.5. The term $x$ is needed to define the vector in this viewing plane because it is nonzero, but it is unknown from looking at the SEM image. Now, to get this vector as defined in the primed system to the unprimed
system the transformation matrix in equation 2.4 was utilized. Equation 2.6 shows how this transformation takes place.

\[ a' = xi' - \sin(62^\circ) j' - \cos(62^\circ) k' \]  \hspace{1cm} (2.5)

\[ a_{cs} = a' \cdot A \]  \hspace{1cm} (2.6)

Evaluating equation 2.6 gives a vector with the variable 'x' as an unknown (as shown in equation 2.7). However, knowing that this cross-section banding vector has no dimension in the i-direction (unprimed system), the variable 'x' can be solved for.

\[ a_{cs} = \begin{bmatrix} 0.616x + 0.696 \\ 0.788x - 0.544 \\ -0.469 \end{bmatrix} \xrightarrow{\text{After Solving for } x} \begin{bmatrix} 0 \\ -1.4345 \\ -0.4690 \end{bmatrix} \]  \hspace{1cm} (2.7)

Knowing the average vector drawn parallel to the bands and the average normal vector to these bands as shown in Figure 2.10, a coordinate system was then devised for the crystal orientation. These two vectors are orthogonal and are drawn with the same orientation as the unprimed system in the FIB trench. Knowing this, basis vectors describing the crystal orientation were calculated as shown in equations 2.8 – 2.10. The basis vector \( \hat{e}_1 \) is aligned with the average vector describing the crystal direction parallel to the surface vector drawn with the bands. Likewise, the basis vector \( \hat{e}_3 \) is aligned with the normal vector describing the normal of the bands as seen in red in Figure 2.10. The basis vector \( \hat{e}_2 \) is unknown because it is coming out of the plane that is shown in Figure 2.10.

\[ \hat{e}_1 = \frac{13.64i + 17.07j + 2.5k}{\sqrt{13.64^2 + 17.07^2 + 2.5^2}} = 0.6203i + 0.7761j + 0.1137k \]  \hspace{1cm} (2.8)
To solve for the components of the basis vector $\hat{e}_2$, dot products were taken with respect to the other basis vectors (as shown in equations 2.11 and 2.12), and because the components of $\hat{e}_2$ are in unit vector form, a unit vector property was used (as seen in equation 2.12). This gave three equations and three unknowns.

Ultimately $\hat{e}_2$ was solved and the result is seen in equation 2.14.

$$\hat{e}_1 \cdot \hat{e}_2 = 0$$  \hspace{1cm} (2.11)

$$\hat{e}_2 \cdot \hat{e}_3 = 0$$  \hspace{1cm} (2.12)

$$x^2 + y^2 + z^2 = 1$$  \hspace{1cm} (2.13)

$$\hat{e}_2 = 0.0758i - 0.2035j + 0.9761k$$  \hspace{1cm} (2.14)

Once all of the unit vectors (basis) describing the crystal orientation were calculated, the basis vectors were then arranged in a matrix as seen in equation 2.15. The vector describing the cross-section of the bands is then dotted into this crystal orientation, and this gave a vector describing the cross-section of the bands within the crystal coordinate system (as shown in equation 2.16).

$$B_{\text{crystal}} = \begin{bmatrix} \hat{e}_1 & \hat{e}_1 & \hat{e}_1 \\ \hat{e}_2 & \hat{e}_2 & \hat{e}_2 \\ \hat{e}_3 & \hat{e}_3 & \hat{e}_3 \end{bmatrix} = \begin{bmatrix} 0.6203 & 0.7761 & 0.1137 \\ 0.0758 & -0.2035 & 0.9761 \\ 0.7670 & -0.6136 & -0.1875 \end{bmatrix}$$  \hspace{1cm} (2.15)

$$a_{\text{crystal}} = a_{cs} \cdot B_{\text{crystal}}$$  \hspace{1cm} (2.16)

Finally, two orthogonal vectors had been calculated which were needed to describe the banding plane in the crystal coordinate system. The last step to describe the plane is to compute a cross product between the vector drawn parallel to the bands on the surface.
and the cross-section of these bands as seen in the FIB cut (as shown in equation 2.16). Now that a normal vector

\[ \mathbf{B}_{\text{normal}} = \mathbf{a}_{\text{crystal}} \times \hat{e}_1 \]

(2.16)

describing the banding plane was found, the vector was then put unit vector form, and divided through by the smallest non-zero component.
CHAPTER 3: RESULTS AND ANALYSIS

A thorough microscopy investigation of the shot samples is outlined in Sections 3.1 – 3.3. Following this are the results from the nanoindentation study in Section 3.4. Lastly, a microbanding plane is calculated utilizing the FIB and two surface analysis in Section 3.5.

3.1 Pre and Post Shot Micrographs

To characterize the post-shot deformities on the sample specimens, it is essential to first look at the pre-shot samples as seen in the stack assembly prior to the shot. After the sample is shot, a macroscopic post-shot investigation takes place followed by a comprehensive microscopy study, which utilizes EBSD, SEM and optical microscopy.

3.1.1 Pre-Shot Stack Assembly

The pre-shot target stack assemblies for each sample are shown in Figure 3.1. In the images, a mounting washer is attached to the surface of each sample, showing an effective circular target region in the center of each specimen across the sinusoid stamped surface. This stamp may be off-centered for different specimens, but a homogenous region of stamping is selected as the effective target region for each laser impact.

3.1.2 Post-Shot Iron Samples

Pictures of the post-shot specimens taken directly after removal from the recovery tube are shown in Figure 3.2 on page 51. Note how both the side and back view of Fe-2 and Fe-4 show macroscopic spall that is not present in Fe-3. It is
speculated that Fe-3 was placed in the recovery tube without a retaining ring behind it. The reason for this speculation is that upon laser impact, Fe-3 was shot back into the aerogel where it was later recovered. Fe-2 and Fe-4 had this retaining ring in place and both were recovered sitting on top of the retaining ring. It follows that if Fe-4, a higher energy shot impact

![Figure 3.1 – Close-up of pre-shot target assemblies. The TOP images show how the mounting washer was attached to the stamped surface of each specimen. The stamp in the middle of each section is the effective target area for laser impact. The BOTTOM images show the underside of the target assembly, showing the raw iron attached to the bottom of the mounting washer. Not shown are the layers of ablator and heat shield between the washer and the sample surface.](image)

than Fe-3, successfully was retained within the recovery tube without shooting back into the aerogel, then Fe-3 did not also have this retaining ring. The use of the retaining ring
was an important part of this experiment, because it effectively amplified the usefulness of the pressure wave induced by the laser striking the ablator. Rather than the pressure wave going straight through the sample with no retaining ring, it is speculated that the ring allowed for wave reflection, sending the pressure wave back and forth several times. It is postulated for this reason that Fe-3 did not exhibit the same spall.

When Fe-3 was sectioned, mounted, and polished, at no point during the polishing procedure was there evidence of spall activity. Fe-3 was the last sample to be polished, and with knowledge of the spall exhibited by Fe-2 and Fe-4, it was expected that a defined spall area would be evident during the coarse grinding steps of the polishing procedure. This was also the expectation knowing that Fe-3 was the mid-range energy shot specimen. Fe-3 continued to be polished, never once revealing this spall area. In the end, the entire shot region of Fe-3 was polishing through in the search for the spall region. It is for this reason that Fe-3 was not included in the post-shot investigation.

After receiving the targets from the recovery tube, the samples still had a layer of ablator, heat shield and aluminum flashing on them. For a thorough removal of said layers, the samples were cleaned with nitromethane. The laser impact surface after cleaning with nitromethane is shown in Figure 3.3 on page 52.

3.2 Post-Shot Diagnostics

Shown in Table 3.1 (page 52) are the experimental results for the laser energy and peak pressure as predicted by Hyades, the 1D hydrodynamic code. Note that although there are simulations for the Fe-3 target shot, it is not used in the microscopy analysis. Hyades predicted a peak pressure of 98 and 196 GPa for Fe-2 and Fe-4 respectively.
Figure 3.2 – Post-shot iron targets after being removed from recovery tube. TOP – a top view of the laser impact surface for each sample. MIDDLE – Backside view of each corresponding sample. BOTTOM – Tilted view of underside of each sample, giving macroscopic view of spall/shot areas. Note how Fe-3, shows no defined spall area on the backside of the sample.

3.3 Microscopy

A range of microscopy techniques was used to characterize the shot specimens. In the following sub-sections, there is a mix of optical microscopy, EBSD, and SEM images. Inverse pole figure (IPF) maps display crystal orientation as shown in the color scheme mapped on the EBSD scanned area. Misorientation maps display local plastic
deformation by calculating the rotation of the crystal lattice at one point with a neighboring point and displaying the difference in a color-coded scheme.

Figure 3.3 – Post-shot iron targets after a thorough cleaning with Nitromethane. This cleaning completely removed the remaining ablator, heat-shield and flashing.

Table 3.1 – Post-shot experimental parameters as predicted by Hyades. All shot samples were shot in a nanosecond, putting the laser power in the gigawatt range. The maximum predicted peak pressure of the laser shot samples reached 196 GPa. Fe-3 was not used in the microscopy analysis.

<table>
<thead>
<tr>
<th>Identifier Name (Fe=Iron)</th>
<th>Shot Energy Requested (J)</th>
<th>Actual Energy Delivered (J)</th>
<th>HYADES Predicted Peak Pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-Unshot</td>
<td>0</td>
<td>0</td>
<td>0 Mbar (0 GPa)</td>
</tr>
<tr>
<td>Fe-2</td>
<td>70</td>
<td>67</td>
<td>0.98 Mbar (98 GPa)</td>
</tr>
<tr>
<td><em>Fe-3</em></td>
<td>100</td>
<td>93</td>
<td>1.23 Mbar (123 GPa)</td>
</tr>
<tr>
<td>Fe-4</td>
<td>150</td>
<td>162.3</td>
<td>1.96 Mbar (196 GPa)</td>
</tr>
</tbody>
</table>

On the misorientation maps, blue indicates the highest areas of misorientation, which in turn, frequently identifies high-angle grain boundaries or significant deformations in blue.
Each sample was initially scanned for the \( \varepsilon \)-phase (HCP) during the course of this research. Even though the phase diagram indicates the \( \varepsilon \)-phase (HCP) cannot be present at standard temperature and pressure conditions, it is feasible at extreme shock conditions that some of this phase could be trapped within the shot regions. After scanning each specimen, the OIM software indicated there was no \( \varepsilon \)-phase present within any of the samples. Upon learning about this revelation, all subsequent EBSD scans used the \( \alpha \)-iron preset for indexing. The \( \alpha \)-iron preset was used for all EBSD scans seen in this report.

3.3.1 Fe-Unshot (0 J, 0 Mbar (0 GPa))

The unshot iron sample needs to be analyzed to act as a baseline for the structural characterization of the laser-shot samples. Seen in Figure 3.4 is a composite image of the unshot iron sample, which was stitched together using four optical microscope images. The sample exhibits a large grain structure (~250 \( \mu \)m) that is evident after etching.

![Figure 3.4 – Composite image of Fe-Unshot cross-section after etching. Stamp is evident on surface. Grains size analysis on OIM software indicates an average grain size of 250 \( \mu \)m. Diagonal contrasting lines are likely artifacts of cleaning with ethanol after etching.](image)

To be as thorough as possible, an EBSD scan of Fe-Unshot had to be taken across the entirety of the sample surface. The scans were started on the left hand side...
region, scanning over the stamp edge along an area that showed a lot of the stamped surface. Seen in Figure 3.5 is the result of an EBSD scan over this area. Clearly seen in the IPF and misorientation map is the stamp edge, located on the upper-left hand side of the image. The area shows a large grain structure, confirming the optical microscopy investigation. Also shown in Figure 3.5 is some pixelation near the bottom of the scan. With knowledge that this sample was not deformed, and only stamped, these objects are thought to be artifacts of the polishing process or the beam rastering and therefore are not real from a mechanistic point of view.

Figure 3.5 – IPF map and associated misorientation map of the left hand side area of Fe-Unshot. Deep scratches are shown as going horizontal. The highly pixelated multicolored area near the top of the IPF is Bakelite. Sporadic pixelation and blue misorientations near the bottom of each image indicate polishing and/or scanning artifacts, which aren’t assumed to be real deformations.
When the electron beam scans over Bakelite, even though it is non-conductive, certain impurities, which didn’t wash off during the cleaning process or beam fluctuations can cause a “charging” effect whereby the beam shifts while it is rastering a scan. This is thought to be the reason why there appears to be a shadowing effect in the Bakelite region at the top of Figure 3.5. The non-conductive nature of the Bakelite also allows the buildup of change in certain areas, also contributing to the charging effect.

Figure 3.6 – IPF map and associated misorientation map near center of Fe-Unshot. A large grain structure is observed directly under the stamped region. Large lines going horizontally through the center and bottom of the images are scratches as a result of polishing.

Moving along the stamped surface of Fe-Unshot, scanning took place near the middle of the sample specimen. The result of this scan is shown in Figure 3.6. Again,
with regard to the last scan on the left side stamp boundary, a large grain structure is seen with no real deformations present as expected. There is however a long scratch across the middle of the IPF and misorientation map, which is a product of polishing error.

Lastly on Fe-Unshot, a final EBSD scan was taken over the right hand stamp boundary as shown in Figure 3.7. The grain size is again large (~250 µm) and the scratch from Figure 3.6 is shown as continuing throughout the scan. Another horizontal scratch is shown near the stamp and stamp boundary. There appears to be a few traces of grain refinement, however, due to the heavy pixelation and charging seen in the previous scans, this refinement is considered inconclusive.

Figure 3.7 – IPF map and associated misorientation map near center of Fe-Unshot. A deep scratch is shown in the center of both images, continuing from the center of the sample as shown in Figure 3.6.
3.3.2 Fe-2 (67 J, 0.98 Mbar (98 GPa))

The mid-range shot sample (Fe-2) is shown as a composite image of 4 stitched together images in Figure 3.8. The laser epicenter and corresponding shot boundaries are shown as a red arrow and red-dashed line respectively. Like Fe-Unshot, a relatively large grain structure is observed. Significant spall is shown near the bottom of the sample where the pressure wave ultimately exited the specimen. Figure 3.8 shows the etched version of Fe-2 to elucidate the grain boundaries of the specimen. The dark region to the left of the shot shows an example of an area that was over-etched. This over-etching seems to exist mostly in individual grains, indicating preferential etching.

![Composite image of Fe-2 cross-section after etching](image)

Figure 3.8 – Composite image of Fe-2 cross-section after etching. A solid red arrow indicates the direction and location of laser impact epicenter. The dashed-red lines indicate approximate locations of the shot boundary based on laser 800-μm diameter laser geometry. Indented region on top is stamped surface. Grains size analysis on OIM software indicates an average grain size of 250 μm. Imaged with optical microscope.

Figure 3.9 shows a close-up SEM image of the entire shot region with a red arrow and dashed lines to indicate the shot epicenter and shot boundary respectively. The sample is etched in this image, giving some indications of relative grain structure. One would assume that Figures 3.8 and 3.9 would be almost identical considering they
are displaying the same region. However, after initially taking the composite image on the optical microscope, it was decided that the over-etched needed to be repolished in order to carry out SEM imaging. For the reason, Fe-2 was repolished, using a finer grit to remove the small deformations induced from the etchant. Following the repolish, Fe-2 was then etched less aggressively and the result is shown in Figure 3.9.

![Figure 3.9 – SEM image of Fe-2 etched cross-section at shot center. Spall is evident at the bottom of the target. Diagonal fracture formations persist near bottom of sample, in and outside of the shot boundary. Stamped sinusoid surface is clearly shown. Red arrow indicates location of laser impact, dashed red line shows shot boundary. Small pieces of material above the stamped surface are mounted tailings from the sectioning process.](image)

Figure 3.10 shows a close-up image of the left side of the spall region on Fe-2. Looking at the image, many diagonal fractures are observed, which appear to reside on
45° planes. Knowing that the grain size is observably large on Fe-2, it is then postulated that many of these fractures are going through grains, causing intragranular fracture. The final pure SEM image taken of Fe-2 shows the grain structure near the top of the sample about 30 µm below the stamped surface as seen in Figure 3.11. This image resides directly in shot center. In the image, the contrasted diagonal lines represent grain boundaries. There is some evidence of small fractures occurring in this

![Figure 3.10 – Close-up SEM image of Figure 3.7 on the left hand side of the spall area in Fe-2. Intragranular fracture is evident. These 45° fractures are visible throughout the bottom side of the shot boundary.](image-url)
area; although this is not conclusive due to the nature and structure of pits and voids that could be the result of polishing. Circular objects within the image can be assumed to be pits, but the ones that appear to be white could be embedded polishing media.

Figure 3.12 shows an IPF overlaid on the SEM image to give texture and context to some of the local deformations seen near the left hand side of the spall area. This scan was taken directly in the shot region over several fracture areas. Initial investigation of this image does not appear to show a preferred orientation with respect to the deformations occurring.
Similar to Figure 3.12, Figure 3.13 on page 62 shows an IPF map overlaid on an SEM image to give context to certain deformation mechanisms near the right side of the spall area. In the scan, three fracture formations are seen which are generally diagonal with respect to the laser impacting the surface from somewhere in the top of the sample. In this scan, more defined striation patterns are observed than in previous scans.

Figure 3.12 – Scan across a large crack located directly above the spall area at shot epicenter in Fe-2. To emphasize local deformations and structure, the inverse pole figure is overlaid on the SEM image.
In the upper left of the image, pink/purple bands are observed, indicating possible twinning deformations. After observing these bands, an EBSD scan was completed over them. Following this scan, an attempt to identify twins common in BCC and FCC materials, along with the rare twin [3] identified by Dougerty et al using the OIM software took place, but the software did not identify these bands as twins. The misorientation map of this scan also showed small misorientations.

Figure 3.13 – Scanned over crack formations residing on the right hand side of the spall area in Fe-2. Shown is an IPF overlaid on an SEM image to give contrasts to structure and deformations.
Figure 3.14 displays a grain identification map and inverse pole figure for a location that resides on the right side shot boundary of Fe-2. The grain ID map shows what the OIM analysis software indicates as individual grains. The heavily pixelated diagonal line going through several grains is most likely a scratch from polishing. Likewise, any areas with heavy pixelation could be pits, or a result of beam rastering.

In the scan, a large grain structure is observed with some sub-grain formation. Near the right side of the scan, defined lines are seen in a few of the grains, which could be indicative of twinning, however, without having the software attempt to identify twins, this speculation is currently inconclusive.

Figure 3.14 – Grain identification and inverse pole figure map of a large area of Fe-2, located on the right hand side shot boundary. LEFT – Auto grain identification, showing large grains with sub-grains located inside. RIGHT – IPF of same region, showing contrasts of crystal orientation within each grain. Stamped surface is shown in upper-left side of image.
Shown in Figure 3.15 is a misorientation map of the same scan shown in Figure 3.14. The stamped surface is evident on the top left of the image. Due to the abundance of red and green areas indicating misorientation angles between 2 – 5° and 5 – 15° respectively, it is assumed the current view is a very deformed structure. However, when comparing this misorientation map to those of the unshot sample, these maps are relatively the same.

![Figure 3.15 – Misorientation map of Figure 3.14. A very misoriented structure is observed with no orderly deformations.](image)

To observe for more local deformations in Fe-2, a higher magnification EBSD scan was taken of an area located within the frame scanned in Figure 3.14. The result
of this higher magnification scan is seen as an IPF in Figure 3.16. This figure shows an area located at the interface between shot and unshot material, across several grains. Many contrasting areas are observed within the grains, but the contrasts again do not appear to have a preferred ordering. The lines located near the bottom right of the scan were initially thought to be scratches, however, this result is inconclusive until future analysis can take place. Regardless, a heavily dislocated structure is observed.

Figure 3.16 – Close-up IPF map of enclosed area of Figure 3.14 showing the interface between shot and unshot material. This close-up shows a heavily dislocated structure with no preferential ordering of deformations.
Figure 3.17 shows a grain ID and IPF map of a location near or on the left side shot boundary. Large grains are observed with some pixelation residing within them. Near the top of the scan, it appears a small pinkish grain formed, indicating the possibility of partial recrystallization. There does appear to be several deformation features appearing across the bottom of the scan, as shown in the inverse pole figure map, however, the pixelation makes this assertion inconclusive.

Figure 3.17 – Grain identification and inverse pole figure map of an area of Fe-2, located just under the stamped surface, in the shot boundary on the left hand side of shot center. LEFT – Auto grain identification, showing a small grain that formed in the center of larger grains. This may be evidence of partial recrystallization. RIGHT – IPF of same region. Pixelation on both images could be attributed to detector noise or polishing remnants.
3.3.3 Fe-4 (162.3 J, 1.96 Mbar (196 GPa))

Throughout the microscopic investigation of the post-shot samples, Fe-4 often proved to be the most interesting. A composite view of Fe-4 showing a heavily deformed grain structure can be shown in Figure 3.18. Figure 3.18 shows the approximate location and boundaries of the laser shot. There is a dramatic change in grain size in the shot and unshot regions. Grains within the shot boundary as analyzed in the OIM software indicate an average grain size of 50 \(\mu\)m. Equiaxed grains pervade the shot region and begin to refine at the shot surface. Outside of the shot region, large grains are observed which appear to be of the same size as the unshot sample.

![Composite image of post-shot Fe-4 cross-section after etching. Significant spall occurred near the back end of the sample, as shown near the bottom of cross section. Prominent evidence of grain refinement is shown. Imaged with optical microscope.](image)

In addition to the grain refinement shown in Figure 3.18, it is also observed that a giant piece of the sample is missing near the bottom. Figure 3.19 and Figure 3.18 show where a large piece of the sample was ejected out of the sample near the spall surface. Half of this piece still remains intact and is still effectively stuck on the sample, however,
it does not maintain many contact points as can be shown with the deep crack formations separating it from the rest of the sample. With reference to Figure 3.19, a characteristic of the shot region is that it is almost defined as being completely within deep fractures going through the material along the shot boundary. These fractures continue from the spall region, all the way along the shot boundary and almost penetrate halfway through the sample. Referring to the spall seen in Fe-4 in Figure 3.2, the missing piece of material is already seen as ejected from the backside of the sample, therefore this removal of material does not appear to be the result of the sectioning and mounting process.

Figure 3.19 – Close-up SEM image of the center shot region in Fe-4. A large piece of material was ejected from the bottom of the sample, however, half of this piece remained intact with the sample and is seen in the bottom right of the image. The sample is etched in this image.
Figure 3.20 shows a close-up image of the top surface of the spall area directly under the shot epicenter of Fe-4. Equiaxed grain formation is shown in this region. It also appears that the sample is fracturing in this region along grain boundaries. Figure 3.21 shows an area of Fe-4 directly above the spall that has many contrasted features. Based on size and formation, these features appear to be microbands. Also shown in Figure 3.21 are several void formations.

Figure 3.20 – Close-up image of Figure 3.19 showing the upper surface of the spall area in Fe-4. There is a dramatic grain refinement in this area with respect to the areas outside of the shot region.
Figure 3.21 – Just above the spall area near the center of the shot on Fe-4, heavily contrasted areas are observed, which appear to have formation. This is evident throughout the sample and exhibits characteristics of microbanding.

Continuing the observations of Fe-4 in the shot region, Figure 3.22 accurately captures the competing microband formations that are present everywhere in the shot region. In some instances, the microband formations terminate at grain boundaries, which fracture along the boundary itself.
Figure 3.22 – Located in an area around the spall region of Fe-4, many competing microband structures are observed. Also in this SEM image, cracks are seen which look like grain boundary fractures (i.e. intergranular fractures).

Figure 3.23 shows a close-up SEM image across several crack features on the left side of the spall area. It is easily visible that the material is fracturing along grain boundaries, however, the far right fracture in Figure 3.23 shows that the fractures also penetrate through individual grains themselves.
Figure 3.23 – Several crack formations present around the left side of the spall area on Fe-4. The dark lines are fractures that appear to run through individual grains and along grain boundaries. This is an example of material exhibiting both inter and intragranular fracturing.

In Figure 3.24, pressure wave epicenter on the surface on Fe-4 is shown. Macroscopic plastic deformation is observed in the form of a dip on the sinusoid surface. There is significant grain refinement seen throughout the entire shot region, starting at the surface of the laser impact. The dip was likely created by the initial onset pressure wave striking this region, causing significant plastic deformation. Fe-2 showed a much less significant dip in the shot region.
Figure 3.24 – Shown is the surface of Fe-4 at the epicenter of the laser shot. The stamped sinusoid is in view. There is a noticeable dip in the sample where the laser shot impacted the surface. Grain refinement is seen starting at the surface of the sample.

Perhaps one of the most interesting microscopy images gathered from Fe-4 is shown in Figure 3.25. The image shows a single deformed grain in the shot region of Fe-4 in the center of the image. Many contrasted features are present in the form of bands. These bands are shown in neighboring grains and within the single grain, which is front and center. This is the highest resolution image of a whole single grain gathered
through the course of the microscopy investigation. The contrasted features represent areas with significant deformation features.

Figure 3.25 – SEM image of a single grain of Fe-4 located in the shot region. The contrasts illustrate the banding mechanisms shown all over the shot region. An equiaxed grain structure is present for many of the grains residing in the shot region.

Not only does Figure 3.25 show an approximate size of grains in the laser impact zone, but also it accurately displays a few interesting similarities between neighboring grains. A closer observation shows very small diagonal lines through the bottom right and bottom left grains in Figure 3.25. These are in fact two completely separate grains,
however, the small lines appear in both grains, and are approximately the same size and appear to be going in the same direction. One would expect these lines to be continuous over the center grain, but they appear to terminate at the grain boundary. Figure 3.25 shows how competing microband formations are evident everywhere and most appear to terminate at grain boundaries.

Figure 3.26 – a) Shown is an SEM image of the right side of the spall area where the material was not ejected from the sample and remained intact. A particular grain on the top right side of the image displays an interesting grain boundary. b) A jagged grain boundary, which could be indicative of a cross-slip mechanism.

In the spall area of Fe-4, just above a piece of material that remained intact while a neighboring chunk of iron was ejected from the sample, an interesting area is seen just inside the shot region. Figure 3.26a shows this area of interest and captures how the material is falling apart at grain boundaries at this location. Figure 3.26b is a close-up image of the enclosed area in Figure 3.26a, and this shows grain boundary with a “jagged” appearance. The formation of this jagged grain boundary has been attributed
to a cross-slip mechanism [13]. However, this appears to be the result of slip bands intersecting a grain boundary, leaving steps from the slip bands. Slip transfer is also taking place across the grain boundary.

Figure 3.27 – An SEM image showing the surface in the spall region. The material in this image is curved under the sample surface and thus has not had direct exposure to the polishing media. Observance of the round structures in this area of the sample infers that the virgin material has voids present.

It was necessary to image locations on the surface of the spall area of Fe-4 to get an idea of how the unpolished, raw material looked. Figure 3.27 shows an SEM image of the spall surface of Fe-4, showing great contouring in this region. Sharp edges are observed where the laser induced pressure wave essentially blew apart the
backside of the sample. These clearly defined sharp edges look almost carved out. Also shown in Figure 3.27 are voids within the spall surface, indicating that although the material is high purity, it also contains a significant number of voids of appreciable size. This could be a plausible reason for the prominence of pitting on final polished samples. In addition to these observations, banding formations are also seen near the top of the image, just before reaching the spall surface.

Figure 3.28 – a) SEM image of Fe-4 near the top left of the spall region showing several crack formations with heavy microbanding structures. The top right of the image shows a grain boundary with these banding structures passing through and then changing orientation. b) A close-up of the enclosed area. Many banding structures are observed, which appear to go horizontally through one grain and then abruptly change direction going through another grain.

Homing in on a region where cracks reside near the left side of the spall area on Fe-4, a stochastic fracture pattern is observed as seen in Figure 3.28a. In the image, microbands are prominent everywhere. Figure 3.28b is a close-up of a region in Figure 3.28a and shows many competing microbands going through a grain boundary and
completely changing direction after traversing the grain boundary. The area in Figure 3.28b was chosen as a location for extensive analysis as seen later in this report. This is the location chosen for the two surface analysis.

Figure 3.29 shows the analysis of an EBSD scan taken over the middle of the shot epicenter and spanning from the stamped surface to just above the spall area. In Figure 3.29a, a grain identification map is shown, which shows dramatic grain refinement in this region. In general, many of the grains appear to exhibit an equiaxed size with the green grain in center view being one of the few outliers. The white region in the top of Figure 3.29a is Bakelite and where the Bakelite contacts the sample surface, there is a multitude of multicolored single pixels. The frequency of pixels in this region with homogenous size and geometry indicate that this could be a product of misindexing and therefore, these should not be considered to be real grains themselves.

Figure 3.29b shows the inverse pole figure map of the associated scan. Figure 3.29c shows the misorientation map of the same scan. This map indicates that over 56% of the pixels in the image are misoriented between 2 – 5° with respect to neighboring pixels. The misorientation map also provides a way to look at the scratches going across the middle of the sample.

Figure 3.30 on page 81 shows the analysis of an EBSD scan taken over the left side shot boundary. For positional context, the white area in the top right of Figure 3.30a is the stamped surface of the sample as it is dipping into the shot epicenter. Figure 3.30a really shows how the grain structure changed outside of the shot region.
Figure 3.29 – Large EBSD scan over the middle of Fe-4 directly in the shot region: a) individual grain identification map; b) inverse pole figure map with legend on top, c) misorientation map with legend on top of figure.
All of the grains on the left side of the figure are very large and the further right one looks in the figure the grains refine as they get closer to shot center. With regards to Figure 3.30a, the large grain formations appear to have a semicircular curvature to them emanating from shot center. An inverse pole figure of the same scanned region is shown in Figure 3.30b. Here many scratch patterns are observed that go through the sample horizontally. Also seen are several lines that appear to have to have differing contrast through the grain. Figures 3.30a and 3.30b both show several structures near the middle left of the grain ID which could either be a sub-grain existing in the material, or a possible twinning mode. More analysis needs to be completed on these areas.

Figure 3.30c confirms that the scratches present in Figure 3.30b are in fact deep scratches as a result of the metallographic preparation and it also shows a dislocated structure. The two features identified as blue in the grain ID map existing within the orange grain are shown to have high misorientation angles, thus also confirming that these regions deserve some further analysis.

Following the EBSD characterization of Fe-4 across the shot region and around both shot boundaries, the investigation continues on the right side shot boundary. Figure 3.31 shows the analysis of a scanned area over the right side shot boundary. Like the maps contained in Figure 3.30, Figure 3.31 on page 83 displays a similar deformation. Figure 3.31a shows the grain ID map for this scan, which again shows dramatic grain refinement in the shot region with very large grains existing outside of the shot area. The large grains again also exhibit a semicircular formation emanating from the shot center.
Figure 3.30 – Large EBSD scan over the left side shot boundary in Fe-4: a) individual grain identification map; b) inverse pole figure map with legend on top, c) misorientation map with legend on top of figure.
Figure 3.31b shows the inverse pole figure map of the same scan, displaying a highly contrasted crystal orientation within each grain. In general, the contrast of crystal orientations within grains tends to stabilize in the blue and light blue grains shown in Figure 3.31a. Some parts of the image contain lines, some of which are thought to be scratches and some of which are debatable. On the lower right hand side of Figure 3.31b, several line patterns are observed that are directed in the diagonal sense. Upon viewing Figure 3.31c, these same line patterns show high angles of misorientation, which leads to a conclusion that they are either deep scratches or a deformation feature such as twins.

Figures 3.29, 3.30, and 3.31 all paint a broad spectrum of the shot epicenter and the shot boundaries of Fe-4. Using these figures in the analysis adds great depth and meaning to the images taken with SEM. It is very obvious that significant grain refinement occurred in the shot region. In addition, it appears that heavy deformation persists not only in the shot region, but also the shot boundaries of Fe-4. All three scans were completed over cracked regions, as can be seen in the bottom of each figure in the misorientation maps. These fractures appear to be all intergranular fractures, as they follow grain boundaries. Figures 3.30 and 3.31 show grains with long lines running through them. These could be indicative of deep scratching as a result of the metallographic preparation, or they real deformations.
Figure 3.31 – Large EBSD scan over the right side shot boundary in Fe-4: a) individual grain identification map; b) inverse pole figure map with legend on top; c) misorientation map with legend on top of figure.
Fe-4 showed intergranular and intragranular fractures taking place around the spall region. Figure 3.32 shows the analysis of an EBSD scan over an area, which exhibited both intergranular and intragranular fracture. Figures 3.32a and 3.32b each
show profuse banding on the upper part of the image. Both figures in Figure 3.32 show how the banded structures are going across a fractured area on the top left of the scan.

Figure 3.33 – Inverse pole figure map of a high resolution EBSD scan taken over a grain boundary that shows a great deal of microbanding. The bands appear to be continuous over the grain boundary; however, they abruptly change direction after traversing the grain boundary. Refer to Figure 3.28 for SEM of this area.

Throughout the course of the investigation of Fe-4, microbands were prevalent nearly everywhere in the shot region. One place in particular showed microbands going through a grain boundary and abruptly changing directions on the other side of the boundary. Figure 3.33 shows this area of interest in the form of an inverse pole figure.
Within each grain on either side of the grain boundary, the bands appear to have slight contrast with respect to the general homogenous color. This represents a slight change in local crystal orientation. The bands have an average thickness of \(~0.4\) \(\mu\)m.

Figure 3.34 – Grain identification map associated with the same EBSD scan as the IPF in Figure 3.33. The OIM software completed an automatic grain identification that picked out several areas alongside the grain boundary, which had the potential to be individual grains.

Figure 3.34 shows a grain identification map associated with Figure 3.33. Here the OIM software has picked out areas on both sides of the grain boundary, which appear to lie in the same direction as the microband formations, and it has identified these areas as individual grains. This can be indicative of real grains, real deformation
features like twins, or bad indexing. A formal analysis cannot be completed on this scan without looking at the misorientation map.

Figure 3.35 – Two different types of misorientation maps associated with the EBSD scan seen in Figure 3.33; a) traditional misorientation map; b) Kernel Average Misorientation (KAM) map.

Shown in Figure 3.35 are two different methods of displaying misorientation of the EBSD scan of Fe-4, associated with Figures 3.33 and 3.34. Figure 3.35a is a standard misorientation map that is identifying high angular misoriented features within the region. The majority of the bands show a green or red misorientation indicating the max angular misorientation associated with the microbands is between 5° and 15°. The Kernel Average Misorientation (KAM) map shown in Figure 3.35b is another way of graphically observing local misorientation. In a KAM map, the relative angular misorientation difference between neighboring points in a scan is plotted in a color-
coded fashion. The KAM map in Figure 3.35b shows the microbands in red as being prominently misoriented with respect to the surrounding material.

Initially, after observance of the misorientation angles seen in Figure 3.35a and the grain identification in Figure 3.34, we thought there was a possibility of twins in this region of Fe-4. Identifying these supposed twins became important to characterizing the deformation mechanisms in the shot region of Fe-4. The OIM analysis software has the ability to identify twins, and software was used for this analysis.

All of the common twins associated with BCC and FCC metals were input into the OIM software along with the rare twin, which was indicative of the $\alpha$-\varepsilon pressure driven phase transformation in iron (a summary of these twin mode inputs can be seen in Table 3.2). Each of these twin modes was given a high angular tolerance ($\sim$5-10°) to capture a wide range of possible twin misorientations. A twin is defined as a 180 rotation about the K1-plane, which is why this is the angle input with each twin orientation in Table 3.2.

Concluding the twin identification study in the heavily banded region, the software only identified several pixels as can be seen in Figure 3.36. These pixels had no real shape, thus concluding that no twins were present within the frame of reference. Backing up this consensus in Table 3.2 is the fraction of twins identified, which never reached greater than 0.01. After observing these results, the misorientation map in Figure 3.35a was revisited and a conclusion was made that at such low misorientations, twinning was likely not the mechanism of deformation in this area of interest.
Table 3.2 – Twin identification inputs used in the OIM analysis software to identify possible twinning mechanisms in Figure 3.36. These inputs are all based on twin modes common for BCC and FCC materials and α-iron and the rare twin found by Dougherty et al [3]. The fraction column indicates the fraction of pixels in the image identified as twins. [23].

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Figure 3.36 – Attempt at twin identification using the OIM analysis software for the heavily banded area along a grain boundary in Fe-4. Only several pixels were identified as having a plane, which could exist as a twin, leading to the conclusion that no twins are present within this reference frame. The color code and specific twin orientations can be seen in Table 3.2.
The heavily banded areas shown in Figures 3.33 – 3.36 continued to be an area of extreme interest due to the banding uniformity and the abrupt change in direction as they proceeded through the grain boundary. This gave the experiment justification to perform a focused ion beam (FIB) cut normal to the banding structures to observe a cross-section of the bands. This cut is shown in Figure 3.37. The shadow like figures observed within the rectangular cut shown in Figure 3.37 are pieces of material which did not cut with cross-sectional uniformity. The cut was performed based on the Helios Nanolab preset for silicon. The FIB at the Colorado School of Mines EM lab often uses silicon as a standard preset for cutting because it generally follows a cutting speed that is conducive for most materials.

If Figure 3.37 is rotated clockwise 90° and the viewing direction is aligned at 52° with respect to the sample surface, one observes the face seen in Figure 3.38. In Figure 3.38, dark contrasted bands are observed as going in the diagonal on the face. These contrasted bands are the cross-section of the bands viewed so prominently in Figures 3.28b, and Figures 3.33 – 3.35. Further evidence that these bands are the same bands shown in these figures is the thickness of the cross-section of each band, which was measured to be 0.4 – 0.5 µm. The thickness of the cross-section of the bands seen in Figure 3.38 and Figure 3.33 are consistent. The sidewall that contains the band cross-sections has vertical white pieces of the material, which are thought to be the tailings of the FIB cut.
Figure 3.37 – Looking into the Focused Ion Beam (FIB) cut, at a 90° angle with respect to the sample surface. The banding structures are clearly shown on both sides of the sample and the grain boundary is seen just to the left of the cut.

Figure 3.38 – Looking at the side view of the FIB cut seen in Figure 3.37. Observed is the cross-section of the banding structures, which are so prevalent in Fe-4. This SEM image was used to draw vectors along the cross-section of the bands for the two surface analysis. The green arrow in Figure 2.7 shows the face and orientation in the current frame of reference.
After attempting the first FIB cut, noting the ease and high precision of the cutting instrument, it was decided to complete a secondary FIB cut normal to the banding structures on the other side of the grain boundary. This secondary FIB cut can be seen in Figure 3.39. In Figure 3.39, the first FIB cut can be seen on the other side of the grain boundary.

When viewing the SEM image in Figure 3.39, a skeleton-like structure is noticed emerging around both of the cuts. This structure is more prominent in some regions more than others. In fact, located on the upper side of the first cut in Figure 3.39, it shows how some of these microbands or the walls of the microbands are clearly defined. This skeleton structure emerges because of the ion beam, which is sending high-energy ions to remove material in the desired cut area. In some cases, these ions either did not hit the area they were intended to, or bounced out of the cut entirely, removing some material around the cut. What is peculiar here is how these stray ions, which are bombarding the microbands, seem to preferentially remove material from some bands, which in turn gives definition to other bands.

A close-up SEM image of the secondary cut can be seen in Figure 3.40. The purpose of this secondary FIB cut was to enable the identification of the banding plane on both sides of the grain boundary. However, this proved to be very difficult due to the poor definition of the observed cross-section banding structures. Seen on the face of this cut is a deep contrast on both sides of the face, which is indicative of a grain boundary. With regards to Figure 3.38, the SEM did not capture easily visible lines marking the microbands on the surface.
Figure 3.39 – Secondary FIB cut on the other side of the grain boundary, cut normal to the banding structures. Note the skeleton-like structure of the bands following bombardment with stray heavy ions from the FIB cut.

Figure 3.40 – Close-up of the secondary FIB cut on the other side of the grain boundary, normal to the banding structures. This face shows a clear separation line between two contrasted bulk areas, indicating a grain boundary exists here. This SEM image was not able to capture the cross-section of the surface bands as easily as was seen in Figure 3.38, therefore this image was not used for two-surface analysis.
3.4 Nanoindentation

A nanoindentation method was used to characterize the reduced Young’s Modulus \( (E_R) \) and the Hardness \( (H) \) at various points within the shot and unshot material. The analysis was comprehensive, taking measurements at differing depths and locations inside and outside of the shot area. Another purpose of this was to observe if there was any work hardening as a result of the stamping process. Shown in Figure 3.41 is an example of the location and size of the nanoindents used in this analysis on Fe-Unshot.

![Figure 3.41 – An example of the nanoindentation on Fe-Unshot. This image was captured on the optical microscope and shows the relative size and location of nanoindentation analysis. In this case, nanoindents were performed starting at the stamped surface to measure work hardening from the stamping process.](image-url)
Figure 3.42 shows the nanoindentation analysis of Fe-Unshot. Fe-Unshot represents the “control” material, therefore it hasn’t had any exposure to the laser shock. A goal of testing the samples for nanoindentation was to get some sense as to whether or not the samples experience any sort of work hardening coming from the stamping of the sinusoid pattern on the surface of each sample.

Work hardening (or cold hardening) is a phenomenon whereby the working and subsequent plastic deformation induced in a material inserts dislocations and causes dislocation movement, thus creating a strengthening effect in the worked region of the material. If work hardening were present in the samples, an increased hardness value would be expected.

Figure 3.42a shows the locations of the nanoindentation inside of the stamped area and somewhere outside of the stamped area in blue and red respectively. The graphs in Figure 3.42b show the calculated hardness and reduced Young’s modulus at each of these locations. Error bars are also shown in the graphs in Figure 3.42b.

From Figure 3.42b, the hardness and reduced Young’s modulus values do not appear to change significantly with depth under the stamp. This can be inferred from the 2.75 – 2.6 GPa change as the hardness is measured to a depth of 75 µm below the stamped surface. This small change could also be attributed to measurement error. Also shown in Figure 3.42b, it appears there is some appreciable hardness change in the unstamped material, which is an interesting result.
Figure 3.42 – Nanoindentation analysis of Fe-Unshot; a) Illustration of Fe-Unshot showing the approximate locations where nanoindentation took place; b) hardness and reduced Young’s modulus values at various locations color-coded with respect to the illustration in part a. The vertical lines shown at each data point are error bars.
The results for the hardening investigation via nanoindentation analysis on Fe-2 are shown in Figure 3.42. There were 3 total locations of interest on the shot samples: directly at shot center, 300 \( \mu m \) outside of the shot, and somewhere outside of the shot boundary. Figure 3.43a shows the approximate regions where each of these studies took place. In an attempt to look for hardening within the material, the nanoindentation studies also looked at each of the locations with varying depth as seen in Figure 3.43a.

The results of the nanoindentation study on Fe-2 are shown in Figure 3.43b. At each measurement location, the measured hardness at the first data point (corresponding to the surface of the sample) is high or the highest with regard to the other data points in the sample. This could indicate either that there is some work hardening occurring at the surface, or that the measurement was so close to the Bakelite that the Bakelite could have been measured instead. At the center of the shot region, there is an evident decreasing trend in hardness and Young's modulus as measured from the surface of the laser impact down to 50 \( \mu m \) below the surface. Also observed is a generally decreasing trend in hardness outside of the shot region measuring down from the sample surface.

Seen in Figure 3.43b, as hardness is measured from 0 to 50 \( \mu m \) deep, there is a hardness change from 3.5 to 2.7 GPa in the center of region. This is not a large amount of hardening by any means, but observing this is still important for a complete and thorough analysis.
Figure 3.43 – Nanoindentation analysis of Fe-2; a) Illustration of Fe-2 showing the approximate locations where nanoindentation took place; b) hardness and reduced Young’s modulus values at various locations color-coded with respect to the illustration in part a. The vertical lines shown at each data point are error bars.
Again referring to Figure 3.43b, there does not appear to be an appreciable change in hardness or Young’s modulus as the nanoindentation is carried out in deeper locations in the sample. For all locations deeper than 150 μm, the hardness appears to level out at around 3 GPa. There are some small fluctuations as shown in sporadic data, however, the sporadic nature of the measurements do not appear to be telling a significant change in hardness.

Fe-4 is the last sample that underwent the nanoindentation analysis. Due to the size of the refined grains observed in this sample, the indentation in these areas could easily spread across several grains at each measurement location. Figure 3.44a shows an illustration of where the nanoindentation took place on Fe-4. In the center of the shot region, the indenter measured a hardness of 3.25 GPa, which remained relatively constant proceeding down to depths of 250 μm from the sample surface (as seen in Figure 3.44b). Figure 3.44b also shows that there was slight decrease in hardness measured at about 600 μm from the surface at center shot. In this region, it is reasonable to speculate that the indenter is indenting over individual bands.

Figure 3.44b also highlights an interesting find whereby the hardness found at 300 μm right of shot center was the highest measured hardness of any sample in the study. This would indicate that work hardening is definitely taking place as a result of the last impact. Generally speaking, there was an increase in hardness found on the surface of each shot sample and steadily decreased to a baseline value in a small depth.
Figure 3.44 – Nanoindentation analysis of Fe-4; a) Illustration of Fe-4 showing the approximate locations where nanoindentation took place; b) hardness and reduced Young’s modulus values at various locations color-coded with respect to the illustration in part a. The vertical lines shown at each data point are error bars.
3.5 Two Surface Analysis

The first part of the two surface analysis was drawing vectors parallel to the banding structures which were observed as being very defined in Figure 3.33. An approximate drawing of these vectors can be seen in Figure 2.10. Completing this in the OIM analysis software, the software automatically identified the average grain crystal orientation in the right grain (orange/red in Figure 3.33) and gave the drawn vectors in coordinates that described the crystal direction parallel to the [001] plane. The raw vector mapping coordinates can be seen in Table 3.3. Averaging the vectors drawn parallel to the bands and the vectors that described the normal of the bands gave two nearly orthogonal vectors (as seen in Table 3.4).

After the FIB cut was completed, the cross section of the banding structures was imaged at a 52° SEM tilt angle. This raw image can be seen in Figure 3.38. Proceeding to draw a line parallel to the cut surface, which roughly aligns with the surface normal vector, vectors were drawn to map the cross-section of the banding planes. After the vectors were drawn, the angle between the normal line drawn on the edge of the FIB cut and the drawn vectors was measured as seen in Figure 2.11. With this average vector angle and the average banding vector direction on the surface along with the average local crystal orientation within the grain, all of the information that is needed to define the microbanding plane is collected.
Table 3.3 – Vector mapping data from the vectors that were drawn on the banding features shown in Figure 3.33. Figure 2.10 shows how the vectors were drawn: 14 vectors drawn parallel to the banding structures seen in right grain in Figure 2.10, and three describing the normal direction of these bands. The vectors were drawn using the OIM analysis software package.

<table>
<thead>
<tr>
<th>Vector Location Identifier</th>
<th>Average Crystal Orientation (hkl)[uvw]</th>
<th>Vector Direction with Respect to Crystal Orientation [uvw]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Parallel with Banding structures</td>
<td>(1 -3 13)[17 -16 -5]</td>
<td>[17 21 3]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[17 20 3]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[15 20 3]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[13 20 3]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[14 19 3]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[16 20 3]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[10 13 2]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[10 13 2]</td>
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<td></td>
<td></td>
<td>[17 21 3]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[6 7 1]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[11 14 2]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[19 22 3]</td>
</tr>
<tr>
<td>Normal to Banding Structures</td>
<td>(1 -3 13)[17 -16 -5]</td>
<td>[15 -13 -4]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[17 -13 -4]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>[13 -10 -3]</td>
</tr>
</tbody>
</table>

Table 3.4 – Averaged Vector data from Table 3.3. These averaged vector directions were used in the calculations for the two surface analysis.

<table>
<thead>
<tr>
<th>Averaged Vector Location</th>
<th>Average Crystal Orientation (hkl)[uvw]</th>
<th>Average Vector Direction with Respect to Crystal Orientation [uvw]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Parallel with Banding structures</td>
<td>(1 -3 13)[17 -16 -5]</td>
<td>[13.64 17.07 2.5]</td>
</tr>
<tr>
<td>Normal to Banding Structures</td>
<td>(1 -3 13)[17 -16 -5]</td>
<td>[15 -12 -3.667]</td>
</tr>
</tbody>
</table>

Following the procedure outlined in the methods section, the banding plane was identified as (-1.4255 1.0 0.9506) (an illustration of this banding plane identification process can be seen in Figure 3.45). In integer form, this result is closely approximated
as \((\overline{3} \ 2 \ 2)\). In fact, the angular difference between these two planes is 1.3°.

Furthermore, this calculated plane is also similar to \(\{2 \ 1 \ 1\}\), which is \(\sim 9°\) misaligned from the calculated band plane. These relatively small misalignments could come from a variety of assumptions that were made as a result of the vector mapping process. For instance, the average of all the vectors drawn parallel to the microband structures on the surface of the EBSD scan was

![Figure 3.45 – Vector Mapping illustration of the identification of the microband plane seen in Figure 3.33. The end result of drawing a vector parallel to microbands on the surface of an EBSD scan, then going through vector transformations to obtain an orthogonal vector aligned with the cross section of the bands in the crystal orientation. These two orthogonal vectors define the plane of the microbands and the cross product between the two defines the normal direction of this plane. Not perfectly orthogonal with the vector drawn perpendicular to these bands on the same scan (91.25° apart). This in turn caused systematic error to propagate through](image)

not perfectly orthogonal with the vector drawn perpendicular to these bands on the same scan (91.25° apart). This in turn caused systematic error to propagate through
the calculations because the crystal basis system was established based on a perfectly orthonormal coordinate system which used the two surface vectors as two orthogonal axes. Furthermore, error in the final banding plane calculation could have come from a protractor measurement taken from a printed out picture of the SEM image showing the sidewall of the FIB cut where the cross-section of the microbands resided. However, with these errors identified, this analysis technique for determining planes of slip bands still gave reasonable results within 10° of precision. Figure 3.46 shows how several common slip systems in BCC materials compare to the calculated banding plane using a two-surface analysis approach.

Figure 3.46 – Inverse pole figure showing comparison of calculated microbanding plane with several common slip planes found in BCC materials (α-iron in particular). The {223} plane was found using a two-surface analysis approach.
CHAPTER 4: DISCUSSION

Hundreds of images and scans were gathered as evidence for the microscopic investigations of these laser shocked samples. Additional evidence comes in the form of a nanoindentation study. Now, like a detective, this report needs to take information from each of these sources and follow the evidence that will lead to logical inferences about what took place after the laser impacted the samples.

4.1 Recovery and Recrystallization

The unshot iron target (Fe-Unshot) is considered the standard as far as unshot microstructure is concerned. Like all of the targets, Fe-Unshot was stamped on the surface and it was measured as having approximately the same dimensions as Fe-2 and Fe-4. Looking at the composite micrograph of Fe-Unshot (as seen in Figure 3.4), it is seen to generally exhibit a sporadic, but large grain structure on the order of several hundred \( \mu m \) in size. When comparing this to the composite micrograph of Fe-2 (98 GPa) (as seen in Figure 3.8), it is observed that the grain size doesn’t appear to be changing by a noticeable amount both in the shot and unshot regions.

Further comparing the unshot grain size to the composite micrograph of Fe-4 (shown in 3.18) a significant reduction in the size of the grains located in the shot region is shown. The unshot material residing on the outside of the shot region in Fe-4 is made up of large grains that appear to form semicircular grain boundaries emanating from the laser impact region. This stark contrast in grain size structure in the shot and unshot regions infers that the pressure wave induced significant grain refinement in the material.
The equiaxed grains present in the shot region of Fe-4 indicate either a dynamic recrystallization process took place or enough thermal energy was delivered through the ablator and heat shield that the sample melted and subsequently reformed grains as the pressure wave rebounded through the sample. This rebounding effect can be explained in the following way: Aside from the ablator and heat shield, each shot sample was held in place between a stainless steel washer and a retaining ring. The circular retaining ring, which held the sample in place as the laser impacted the target stack, had edges that the sample rested on. These circular edges could have acted as reflective surfaces for the pressure wave, that could’ve ultimately caused the pressure wave to rebound several times, ringing within the sample. Circular geometries cause different types of wave reflections that exhibit no preferred direction. This abnormal wave reflection could have also added shear components to the pressure wave, thus deforming the sample in a multitude of directions.

There is further evidence that these boundary conditions holding the sample in place had an effect on the targets. Fe-3 was shot without a rear-retaining washer and this led to the sample being shot back into the aerogel catcher after the initial pressure wave impact. While polishing Fe-3, there was no apparent spall area, only a plastically deformed dipped impact surface. This lack of observance ultimately caused over polishing through the shot region.

An alternative theory to explain the grain structure present in the shot region of Fe-4 is that the sample was impacted at such a fast timescale that it was too fast for substantial grain growth to occur. It is estimated that the time it took to fully complete the process from the laser striking the target stack to the pressure wave reflections
completely dissipating was approximately less than 100 nanoseconds. Though this is a possibility, the relative change in grain size from the unshot material to Fe-4 indicates that recrystallization likely occurred.

4.2 Phase Transformation

Non-diffuse phase transformation is a phenomenon, which is commonly thought to occur in the timeframes associated with the speed of sound. Although this is fast, the timeframes from laser impact experiments are on the order of a few nanoseconds. The reversible pressure driven phase transformation in iron has been studied at constant pressure using diamond-anvil cells, however this has not been well characterized at high pressure using impulse lasers.

It was feasible for the experiment to postulate that with high-pressure shock compression (exceeding 50 GPa) happening on the timescales of a nanosecond, iron could’ve immediately transformed to the HCP $\varepsilon$-phase, and then as it reached ambient pressure conditions and transformed back to the BCC $\alpha$-phase, it could’ve trapped some of the $\varepsilon$-phase within the matrix. Using the EBSD software preset for the $\varepsilon$-phase in an attempt to identify these $\varepsilon$-phase structures trapped in the bulk $\alpha$-phase, the software did not identify any of these in all of the scanned areas. With knowledge that the iron samples greatly exceeded the phase transformation pressure and there was no identification of any remaining evidence of this phase transformation, the results from this experiment confirm the hypothesis that the characteristic HCP $\varepsilon$-phase can only be present at high pressure.

The rare twin identified by Dougherty et al.[3] as being a unique characteristic of the $\alpha$-$\varepsilon$-$\alpha$ phase transformation was important for the deformation characterization of the
Along with several common BCC and FCC twin modes (as seen in Table 3.2), this rare twin was input into the EBSD analysis software in an attempt to identify active twinning mechanisms. In all of the EBSD scans collected as a result of this post-shot investigation, none of the scans identified a defined twin structure of the kind listed in Table 3.2. The absence of any of these common twin modes leads to the conclusion that the primary deformation mode for these samples was slip system activity.

Furthermore, observing the phase diagram in Figure 1.1, it is also feasible that the thermal energy delivered to the stack from the laser could’ve been high enough to transform the shot samples into austenite ($\gamma$-phase FCC). If the samples did experience this austenitic phase transformation at some point during the onset pressure wave, which then continued to transform into the $\varepsilon$-phase, it would be expected to see a low symmetry martensitic structure appearing somewhere in the shot region. In a microscopic investigation, this martensitic structure would be observed as existing in lenticular shaped microstructural features. However, at no point while analyzing the samples were these types of grain geometries observed. In fact, further evidence that this martensitic transformation never took place is the prominence of the equiaxed grain structure in Fe-4, which is not at all a characteristic of martensite formation.

4.3 Plasticity

During the microscopic investigation of Fe-4, a prevalent banding structure was observed all throughout the shot region. Often times these bands would terminate at fractures or grain boundaries, however, they were also observed as being continuous through grain boundaries accompanied by an abrupt change in direction (seen in Figure
3.33). These observations indicate that these bands exist as compatible and incompatible deformation mechanisms within the samples.

These neatly ordered band formations do not appear to be the same size and structure of normal slip bands. They measure approximately 0.3 – 0.5 µm in thickness and have a double wall appearance. Figures 3.35a and 3.35b both show the local misorientation of these banded structures as being very low, on the order of a few degrees. This leads to the conclusion that these are the same structures observed by Gray et al, thus the banded structures that fit these characteristics in Fe-4 are called microbands.

A proposed mechanism to create these microbands is the process of adiabatic shear banding. As the laser strikes the ablator, it delivers both a pressure wave and thermal energy to the iron. This thermal energy locally heats up various areas in the material, creating environments where slip is favorable. Further explaining this concept, the heat cannot dissipate fast enough in these localized areas and as a result, they soften and create areas of high dislocation density favorable for slip. This type of creating shear bands has been known to happen occur in shock loading scenarios. With the higher strain rates observed in laser shock loading, this could be a common occurrence.

The two surface analysis that was performed on the well-defined microband structure shown in Figure 3.33, identified these microbands as having a plane normal direction of [-1.4255 1.0  0.9506] (or with integers roughly \{\(\frac{3}{2} \ 2 \}\}). This calculated normal was found to be \(\sim 8^\circ\) misaligned with a \{2 1 1\} plane and \(13^\circ\) misaligned with a \{1 2 3\} plane. Considering these relatively slight misalignments with common slip
planes in BCC materials, this gives confidence that this two surface approach can be used again to identify other microband planes. Orthogonality assumptions were made between the grain boundary and the surface band vector on the EBSD scan along with a rudimentary angle measurement with respect to a slightly curving grain boundary. Although these assumptions could result in a large range of error, this method generally gives a good idea of plane identification and can be relatively accurate.

4.4 Hardness and Stiffness

The hardening and strengthening results from the nanoindentation study were largely inconclusive for Fe-Unshot and Fe-2 (98 GPa). For the unshot target, there was not a significant amount of hardening imposed on the material as a result of the stamping process. There was however, a small indication of hardening seen outside of the stamped region as seen in Figure 3.42b. For Fe-2, due to the decreasing trend in hardness values measuring from the top of the sample in the shot region, this gave a slight indication of hardening at the surface of the sample at impact surface. This result is deemed inconclusive due to there being no real variation in the hardness bounds seen in other locations in the target (as seen in Figure 3.43b).

In comparison with Fe-Unshot and Fe-2, Fe-4 exhibits the largest hardness value at the surface of any sample (4 – 4.5 GPa). As the hardness is measured with increasing depth in the sample, the hardness value in Fe-4 decreases back down to the baseline hardness seen in Fe-2 and the unshot sample. This makes sense because at these depths in Fe-4, it is very possible that the measurement is taking place over individual grains and certainly over formations of microbands. This is an example of a laser induced pressure wave changing local microstructure resulting in work hardening.
CHAPTER 5: CONCLUSIONS AND FUTURE WORK

To conclude this research, we now take information from all of the analyses completed on these shot iron samples and come up final verdict for what may have happened to these samples under these extreme loading conditions (discussed in Section 5.1). Also, in closing this research, several areas for future work are outlined in Section 5.2.

5.1 The Final Verdict

The microscopy investigation did not identify evidence that would imply a phase transformation took place when these iron samples were shocked at high pressure. Twinning would be a good indication of a martensitic phase transformation, however, the microscopy investigation discovered none of these deformations. Generally speaking, transforming a very stable $\alpha$-phase (BCC) to the $\varepsilon$-phase (HCP) requires a great deal of coordination which could simply not applicable at the extreme strain rates delivered from laser shock compression. The laser impact and subsequent pressure wave delivered such high pressure in a nanosecond that its possible that phase transformation was no longer energetically favorable and instead, the material plastically flowed along slip planes. The thermal energy passed to the sample from the laser inevitably created areas with high dislocation density, further encouraging this slip plane movement.

Conventional crystal mechanics would infer that if one subjects a material to high pressures at a fast rate and then adds additional driving force to achieve this, then one can activate a range of various deformation mechanisms. During the course of this
investigation, various analysis techniques were unable to identify any twin modes common to BCC and FCC materials or transformation twins within the shocked material. This begs the question: if a phase transforming material is shocked to an extreme pressure in such a short duration, why would it take the time to stop plastically deforming and instead go through a phase transformation or form a twinned structure? The answer is fast free energy minimization. Changing crystal structure requires time, that of which is not present during a laser shock loading event.

This experiment showed that even though iron undergoes a pressure induced phase transformation; it is possible to shock iron at such a rate where phase transformation is no longer possible. Plastic deformation as a result of slip system activity is favorable during laser shock compression exceeding 100 GPa.

5.2 Future Work

The microscopy investigation of these shot iron samples is far from being completed in its entirety. There were many instances where EBSD scans were gathered and after creating various analysis maps, scratches were evident on the surface. Hand polishing is not an exact science. Different polishing conditions vary by the day in common user labs. Therefore for additional microscopy work on these samples, using a precise instrument like a cross-section polisher is recommended, which can remove layers of material at very small thicknesses. One could also utilize electro-polishing in the quest to repolish and analyze these iron samples.

To fully characterize the deformation mechanics of iron under shock loading conditions, further testing on a wider array of shot energies must take place. There was a vast difference in grain structure at 98 and 196 GPa shocks respectively. Quantizing
the energy levels of shock where the deformation mechanisms change would be a worthwhile endeavor.

The use of the retaining ring residing behind the shot sample in the target stack proved to be significant. Systematic pressure wave reflections after laser shock created a ringing effect, which clearly had an impact on the microstructure of these samples. It would be interesting to complete this study of iron if one could characterize the effect of this retaining ring at varying shot energies. When this experiment took place without the retaining ring, the sample shot back into the aerogel catcher and was basically only subjected to the onset pressure wave. This event more accurately simulates the shock loading conditions that might happen in an inertial confinement fusion experiment. Without having a shocked iron sample that did not employ the use of a retaining ring, one cannot accurately conclude with certainty that the deformation mechanisms are the same as with the retaining ring.

Iron containing meteorites have traveled enormous lengths at incredible velocities through perilous circumstances. The origins of many of these meteorites are mostly unknown. For all we know, these meteorites could have originated as the result of a supernova event. This extreme event could feasibly put these meteorites in shock-compressed conditions. It would be very interesting for this experiment to look at these iron-containing meteorites and try to identify if they underwent a similar microstructural change to the one characterized in this report. A similar microscopic investigation could be used to identify if iron meteorites contain microband formations.

The analysis showed that it was relatively easy to identify microband planes using a two-surface approach. With a relatively high degree of accuracy, the analysis
identified a \{\overline{3} \ 2 \ 2\} microband in $\alpha$-iron. The two surface approach was useful, but the tedious method of removing localized material with the FIB is not exactly a streamlined process. To better understand these microbanding structures in laser shocked iron, it would be beneficial to utilize a Transmission Electron Microscope (TEM). If the experiment utilized this in the future, it could easily identify microband structures and confirm if all of them reside on primary slip planes.

To further explore planetary science applications, it would be interesting to run a similar experiment and subsequent investigation on a nickel-iron mixture. These elements are thought to be the main constituents of the inner core and it has often been postulated that the inner core is stable and solid. In addition to the Earth’s core, it has been shown that these elements exist abundantly together in the universe as a result of supernova events. Performing laser shock experiments on this alloy would be interesting to observe the microstructural changes and phase transformations that take place during a shock-loading event.

Lastly, it is simply not possible in the time of exposure to these samples for full observance in all corners of each sample for identification of twinning. Twins can be evident in the nano-scale range and the microscopy analysis did not probe this scale in length. A thorough search for twin modes needs to be conducted on repolished iron samples under shock loading conditions.
LIST OF ABBREVIATIONS

BCC – Body-Centered Cubic (crystal system)

CCD – Charge-coupled Device

COMPO – Surface Composition Mode

CSM – Colorado School of Mines

EBSD – Electron Backscatter Diffraction

FCC – Face-Centered-Cubic (crystal system)

FIB – Focused Ion Beam

HCP – Hexagonal-Close-Packed (crystal system)

ID - Identification

IPF – Inverse Pole Figure

OIM – Orientation Imaging Microscopy (software)

OM – Optical Microscopy

RM – Richtmyer-Meshkov (instability)

SEI – Secondary Electron Imaging Mode

SEM – Scanning Electron Microscopy

TOPO – Topographic Imaging Mode

TSL – Company that makes microscopy software used in report

VISAR – Velocity Interferometer System for Any Reflector
REFERENCES CITED


