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**FUNDED PROJECT: NOVEL METHODS FOR FATIGUE LIFE PREDICTION FOR  
TURBINE ENGINE COMPONENTS**

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## **PREFACE**

It is critical to state that the progress and completion of this funded research was significantly hampered by the Covid-19 pandemic. The pandemic created a slowdown particularly since the project had significant experimental components and the researchers involved had limited or no access to the experimental facilities in the University during the pandemic. As a result, the experimental components of this work were not complemented until March 2022. This has led to significant delays in publishing the outcomes of our research activities. In addition, some of the initial tasks of this project were modified (with the approval of the Project Engineer) during the Covid-19 pandemic.

# 1 SUMMARY

This research was funded via the Air Force Research Laboratory Diverse Collegiate R&D Collaboration Program. It was conducted at the Applied Mechanics and Materials Research Laboratory, Howard University, Washington, DC from September 29, 2017, to March 31, 2022. The overall goal of the project was to develop novel methods fatigue life prediction for turbine engine components. The ultimate aim was to improve the mechanical properties and microstructure of advanced materials produced via additive manufacturing and grain refinement. The specific objectives of the funded research were modified due to Covid-19 pandemic to i) study the influence of geometry on the microstructures and mechanical properties of ultrafine grained aluminum alloys produced via severe plastic deformation, ii) investigate the microstructures and mechanical behavior of additively manufactured advanced materials.

For the first objective, grain refinement was accomplished by processing AA5052 alloy via multiaxial forging (MAF) at cryogenic temperature with the forging process repeated in multiple cycles of 2, 4, and 6. The base solution heat treated (SHT) sample was used as a reference to determine the effects of the grain size refinement on the microstructure and mechanical behavior of the ultrafine grained (UFG) alloy. Subsequently, the microstructure and the mechanical behavior of the processed samples at various strain rates were investigated to determine the influence of cryogenic forging (CF) on the alloys' subgrains dimensions, grain boundaries interactions, strength, ductility, and toughness. For the purpose of achieving the second objective, AM samples of 316L SS were of two different thicknesses were fabricated via Laser Powder Bed Fusion (L-PBF) and used to conduct both uniaxial tensile and fatigue tests. Post-deformation microstructural characterizations were subsequently performed to determine the failure modes.

For the UFG AA5052 alloy, the Electron backscatter diffraction (EBSD) maps demonstrated a significant grain size refinement from 350  $\mu\text{m}$  for the base SHT sample to average grain sizes of 1.78 $\mu\text{m}$  for the 2 cycles, 1.3  $\mu\text{m}$  for the 4 cycles and 0.94 $\mu\text{m}$  for the 6 cycles, respectively, as a result of the cryogenic plastic deformation. The average misorientation angles are 10.69°, 12.55°, and 16.72°, for the 2, 4, and 6 cycles, respectively. Relative to the SHT sample, the CF AA5052 alloy showed improve high strain rate mechanical properties in terms of the maximum flow stress and the true strain at failure, although the true strain at failure reduces with increase in number of forging cycles. In addition, microstructure characterizations of the post-deformed samples via optical microscopy show the formation of adiabatic shear band, and shear band bifurcation that result from contention between the initial shear band's propagation direction and the direction of the maximum shear stress.

For the AM 316L SS steel samples, the rapid cooling and solidification of the AM materials result in the formation of cellular subgrain structures that affect the mechanical properties of as-built samples. The cellular boundaries portrayed by the low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs) contribute to the increase in yield strength and ultimate tensile strength values of the AM materials when compared to the as-received samples. The SEM images of the fractured surfaces showed ductile fracture as characterized by the spherical micron-sized dimples. Microcracks, voids, and pores were also observed on the surfaces of the samples. The fractography obtained for the samples tested under fatigue loading showed the presence of numerous defects that reduced the fatigue strengths of the samples.

## 2 INTRODUCTION

### 2.1 Background and Motivation on the Study on Ultrafine Grained Aluminum Alloy

Aluminum is the most plentiful metal in the earth's crust, and the third most plentiful element after oxygen and silicon. Although, aluminum is too reactive chemically to exist in nature as a free metal, it has been discovered that it combines with over 270 different minerals out of which Bauxite is its main source. Aluminum alloys have been the key structural materials in the aircraft, automobile, structural and construction sectors due to their unique combination of good mechanical properties such as high specific strength, excellent ductility, good machinability, and high corrosion resistance. The strength to weight ratio has been dominant consideration and it is particularly important in engineering design where parameters such as specific stiffness or resistances to buckling are involved. Aluminum has a density of  $2.7\text{g/cm}^3$  which is approximately one-third that of steel ( $7.83\text{ g/cm}^3$ ).

Pure aluminum and its alloys have a face-centered cubic (FCC) structure, which is stable up to its melting point of  $657\text{ }^\circ\text{C}$ . Since the FCC structure contains multiple slip planes, this crystalline structure greatly contributes to the excellent formability of aluminum alloys. The yield strength of pure aluminum is  $7\text{--}11\text{ MPa}$ , whereas those of some heat treated commercial high-strength alloys exceed  $550\text{ MPa}$  [1]. Also, the corrosion resistance can be excellent due to a thin surface layer of aluminum oxide that forms when the metal is exposed to air, effectively preventing further oxidation [2]. Aluminum has a stacking-fault energy of approximately  $200\text{ MJ/m}^2$ , which is considerably higher when compared to magnesium or steels [3]. Aluminum is a good thermal and electrical conductor and has lower density than copper.

Aluminum 5052 (AA5052) fits applications where good workability, weldability, high fatigue strength, very good resistance to corrosion, and moderate static strength are desire. AA5052 has significant applications in aircraft, marine, general sheet metal work, architecture, heat exchangers, fuel lines and tanks, rivets, wires, flooring panels, streetlights, and appliances [4]. An ever-growing demand for greater efficiency of materials in the aerospace and the automotive industries and the immediate need to decrease fuel cost motivates engineers and scientists to fabricate novel materials by manipulating microstructure to achieve light materials with superior mechanical characteristics. This can be achieved through several ways including precipitation hardening, solid-solution strengthening, strain hardening and grain boundary strengthening. However, strengthening methods such as annealing for precipitation hardening results in restoration process through recovery and recrystallization. It will have definite negative influence on the strength [5]. The excellent mechanical strength of nanostructured and ultrafine grained (grain size less than  $1000\text{ nm}$ ) has drawn a great interest of the materials scientists to grain refinement in recent times.

Ultrafine grained (UFG) materials are potential candidate for a wide range of structural and functional applications due to their outstanding mechanical and physical properties in terms of monotonic strength, high cycle fatigue strength and improved corrosion strength [6,7]. In recent years, UFG and nanostructure materials with superior strength, ductility, toughness, and corrosion resistance when compared to their bulk alloys have been developed using several severe plastic deformation routes (SPD) [8]. SPD are recognized as metal forming processes in which an intense plastic strain is imposed on a bulk coarse grained solid in order to fabricate the nano/ultrafine

grained morphology in the bulk metals [9]. In fact, the strength of metallic materials can be improved by grain refinement, and this has been very well explained by the Hall-Petch relationship [10]. It has also been reported that the combination of UFG and coarse-grained materials could improve the wear resistance, fatigue crack initiation resistance and corrosion resistance of materials [7].

The commonly employed techniques such as equal channel angular pressing (ECAP) [11], equal channel angular rolling (ECAR) [12], high pressure torsion (HPT) [13], constrained groove pressing (CGP) [14], multi-axial forging (MAF) [15], cyclic and accumulative roll bonding (ARB) [16] have been used for the production of UFG and nanostructures in various metallic materials. Out of these SPD methods, MAF is widely considered as one of the least-costly and most promising deformation approaches primarily due to its ability to produce materials with isotropic properties without the need for expensive dies [17]. MAF involves repeated compression on the sample such that the axis changes to 90° in every pass. This process can be directly associated with grain splitting due to the formation of microbands. Such microbands intersect cause the subdivision of coarse grains into finer domains [18].

Room temperature SPD techniques have not been found highly reliable to producing UFG materials. This is due to the fact that dynamic recovery during the plastic deformation accelerates the dislocation annihilation process. Dynamic recovery restricts the development of dislocation boundaries and hinders grain refinements [19]. However, it has been proven that SPD at liquid nitrogen temperature causes suppression of dynamic recovery by reducing the dislocation mobility and allowing the accumulation of dislocation density to attain a higher steady state level when compared to the conventional room temperature plastic deformations [17]. Accordingly, cryogenic deformation requires less amount of plastic deformation energy for generating UFG structure when compared to the SPD processes at room temperature [19].

The growing number of applications of aluminum alloys in both aerospace and automotive industries subjected to impact loadings such as crash, explosion, and high-speed collision highlights the importance of understanding the mechanical responses of the materials under impact loading conditions [20–22]. Unlike quasi-static deformation processes, impact and dynamic loads often involve high strain rates, large strains, and rapid changes in temperature due to severe plastic deformation and adiabatic heating conditions. The deformation and failure of metallic alloys under quasi-static loading are normally by slip and twinning while a failure at high strain rate is initiated by intense localization of shear strain [23–25]. As observed in metallic materials, aluminum alloys experience heterogeneous deformation leading to the formation of ASBs under dynamic compressive stress [26,27]. ASBs could be defined in areas of intense shear strain localization resulted from thermo-mechanical instabilities induced by localized adiabatic heating during impact load [24,28].

Unfortunately, the literature on the influence of cryogenic SPD on the quasi-static and high strain rate mechanical properties of aluminum alloys is very limited. Therefore, one of the objectives of this funded project was focused on the quasi-static and high strain rate (1000-5,500 s<sup>-1</sup>) dynamic mechanical responses of UFG AA5052 alloy processed by MAF at cryogenic temperature. In addition, the microstructures of the processed materials were investigated to determine the influence of cryogenic forging on their subgrains structure and grain boundaries interactions.

Subsequently, the microstructures of the post-deformed samples after mechanical tests were evaluated in order to analyze the failure mechanisms such as the fracture modes, shear bands and cracks. The experimental data obtained in this research also include important information on the dynamic yield and flow stresses under dynamic loading.

## **2.2 Background and Motivation on the Study of Mechanical Behavior of Advanced Materials produced via AM**

Additive manufacturing (AM) has been recognized as a key technology that could revolutionize manufacturing processes for many industries. Apart from being a faster and cheaper alternative to conventional manufacturing (CM) techniques, it has the capability to produce new advanced materials and components with complicated geometries that are extremely difficult to produce via CM processes. AM also offers great potential for reducing manufacturing costs, in particular, it can reduce post-production activities since sub-assemblies can be consolidated into a single unit [29]. Parts produced via AM have demonstrated a distinct anisotropic behavior [30-39]. This is true for both polymers fused deposition modeling and selective laser metal printing. Additionally, for metal alloys, AM produced parts have demonstrated an increased in strength when compared to traditionally produced metal parts, this includes studies on Inconel, stainless steel, and titanium [40-43]. Such improvements are attributed to the unique and fine microstructure as a result of rapid solidification due to the short laser-material interaction time and high cooling rates of AM processes.

Compared to the investigations on quasi-static tensile properties of AM components, the studies on fatigue behaviors are rather limited with little research being done on stainless steel metals. Other studies have demonstrated that the surface defects are quite detrimental to the fatigue strength as it depends on the surface roughness of a component. The high roughness of the surfaces results in increased number of stress concentrators that then become crack initiation sites. Post processing of AM metals are known to not only improve the surface finish but also decrease porosity and eliminate any residual stresses. Heat treatment, chemical polishing, hot isostatic processing, and laser polishing are some of the commonly used post processing methods for AM materials. Kahlin et al. [44] demonstrated that post processing of the surfaces either through centrifugal finishing, shoot peening, or finishing can enhance the fatigue life of a material. The mechanical properties, both tensile and fatigue strengths, were seen to improve for most components when surface roughness was reduced [45-46]. The most effective way in enhancing the fatigue performance of materials is to ensure that the processing parameters are optimized and thus post processing acts as a secondary additional process.

Additively manufactured materials are known to display anisotropic mechanical behaviors. The anisotropy of these materials can be attributed to many different factors such as build direction, scanning speed, and manufacturing angle, just to mention a few. Mower and Long [47] conducted a study on 316L stainless steel fabricated at different build orientations. The vertically built samples were observed to have lower fatigue lives compared to the horizontally built samples. They also observed that the fatigue strength of L-PBF 316L was lower than that of the wrought materials. The subgrain structures, the grain boundaries, grain sizes and shapes all contribute to the anisotropy of L-PBF 316L metals. Hitzler et al. [48] reported that the elongation to failure of the materials was dependent on the orientation of loading direction with respect to the build

direction. Yadollahi et al. [49] observed that the lack of fusion effects affected the vertically build samples more as compared to horizontally built ones. As such, the horizontal materials had longer fatigue lives compared to the vertically built ones. The effect of angle of print was demonstrated by Alkindi et al. [50] who showed that 17-4 PH stainless steel specimens printed at  $90^0$  exhibited lower tensile properties compared to those at  $0^0$ . The melt pools and grain structures formed differently under different orientations thus affecting the grain angle boundaries as well as the orientation of defects.

In this funded research, L-PBF 316L stainless steel, vertically built, was used to further characterize their mechanical performances. Consequently, the thickness of the samples was considered in understanding the mechanical behavior of materials. The size of a material is generally known to affect its fatigue strength through the size effect criteria. Studies on wrought materials have shown that increasing the size of a material increases its fatigue strength [51-52]. These studies have also shown that number of defects in a given sample can vary depending on the size of the material. It is because of this factor that the distribution of defects was used to establish the grounds of the weakest link theory studies. Defects not only cause premature failures but also cause the scatter in mechanical properties. In this study, special attention was paid to the initiation and occurrence of cracks in explaining the lower fatigue performance of additively manufactured materials.

### 3 TECHNICAL TASKS, MATERIALS AND METHODS

This section of the Final Report presents the materials used in this funded project as well as the quasi-static, fatigue, and high strain rate experiments conducted along with their associated microstructural characterizations and failure analysis. This chapter also includes important information on the materials preparation, materials processing, facilities, and experimental setups.

#### 3.1 Tasks

The improvement of the mechanical properties and microstructures AA 5052 alloy via grain refinement was accomplished by performing the following research tasks:

- i. Developed UFG structure in AA5052 alloy via MAF at cryogenic temperatures,
- ii. Performed microstructural characterization of the processed material to observe the effect of each material processing condition on its microstructure,
- iii. Performed quasi-static and dynamic mechanical experiments on the processed material to characterize its mechanical behavior and deformation mechanisms, and
- iv. Conducted microstructural characterization of the post-deformed samples after the mechanical tests to determine the failure mechanisms.

The study conducted on the influence of geometry on the mechanical and microstructural properties of AM 316L stainless steel produced via L-PBF was accomplished by performing the following research tasks:

- i. Fabricated AM samples of 316L SS with 1.5 mm and 4.0 mm thickness via L-PBF
- ii. Conduct uniaxial tensile and fatigue tests on the AM samples,
- iii. Performed microstructural characterizations of the processed and fractured samples to realize failure mechanisms.

#### Materials and Processing

For the grain refinement part of this project, the detailed chemical compositions of the AA5052 grain refinement specimens were aluminum alloys investigated are summarized in Table 1.. They were received as rectangular plates having a cross section of 200 x 200 mm<sup>2</sup> and a thickness of 40 mm. The specimens were initially homogenized at a temperature of 500°C for 10 h in a controlled atmosphere of argon, and then quenched in water at room temperature. Subsequently, samples with dimensions 33 × 31 × 28 mm<sup>3</sup> were cut from the solution treated samples and processed through MAF.

**Table 1: Chemical Composition of the AA5052 Alloy (wt.%).**

| Elements | Cr        | Cu  | Fe  | Mg      | Mn  | Si   | Ti | Zn  | Zr | Al      |
|----------|-----------|-----|-----|---------|-----|------|----|-----|----|---------|
|          | 0.15-0.35 | 0.1 | 0.4 | 2.2-2.8 | 0.1 | 0.25 |    | 0.1 |    | Balance |

The MAF process was conducted at a cryogenic temperature (i.e., liquid nitrogen temperature of -196 °C) at a strain rate of 10 s<sup>-1</sup> using a friction screw forging machine shown in

Figure 1. The setup for cryogenic MAF consists of a cryo-can for procuring and storing the liquid nitrogen, sample container to hold the sample in the liquid nitrogen and a friction screw forging machine for MAF process. To carry out MAF at cryogenic temperature, the samples were initially dipped in liquid nitrogen for 15 min. The extrusion direction of the starting samples was preferred as the first forging axis. An axial compression at an equivalent true strain of  $(\Delta\epsilon) = \ln(1/1.18) = -0.165$  was applied for one forging pass. Furthermore, quenching in liquid nitrogen for 5-10 min was performed after each pass to keep the samples in a thermal equilibrium with the liquid nitrogen. After this quenching, the samples were taken out from the containers and then immediately forged. The cumulative strain after one cycle (consisting of 3 forging passes) was obtained as  $(\sum \Delta\epsilon_{n=1}) = |\Delta\epsilon_1 + \Delta\epsilon_2 + \Delta\epsilon_3| = -0.495$ . The samples were rotated by  $90^\circ$  after every pass along the axes of the applied strain in three orthogonal directions to maintain a constant dimensional ratio of 1.18: 1.11: 1.0 throughout the process. Eventually, The AA5052 samples were subjected to a cumulative strain of -2.97 under 6 cycles (18 passes). The samples for the alloy were successfully forged at multiple cycles from 2, 4, and 6. SHT processed samples were also produced and used as a reference material for this study. The processed samples were machined for the following purposes:

- 1) Small pieces of  $5 \times 5 \text{ mm}^2$  were cut from the central region of the forged samples for microstructural characterization.
- 2) Cylindrical test specimens having dimensions of 3 mm in diameter, D and 3 mm in length, L (i.e., aspect ratio,  $L/D = 1$ ) were prepared for the SHPB tests with precision machining the processed materials using a lathe machine. The schematics of the whole process from cryoforging sample preparation to SHPB impact test are depicted in Figure 2.



**Figure 1. Friction screw forging machine used for Multi-axial forging**

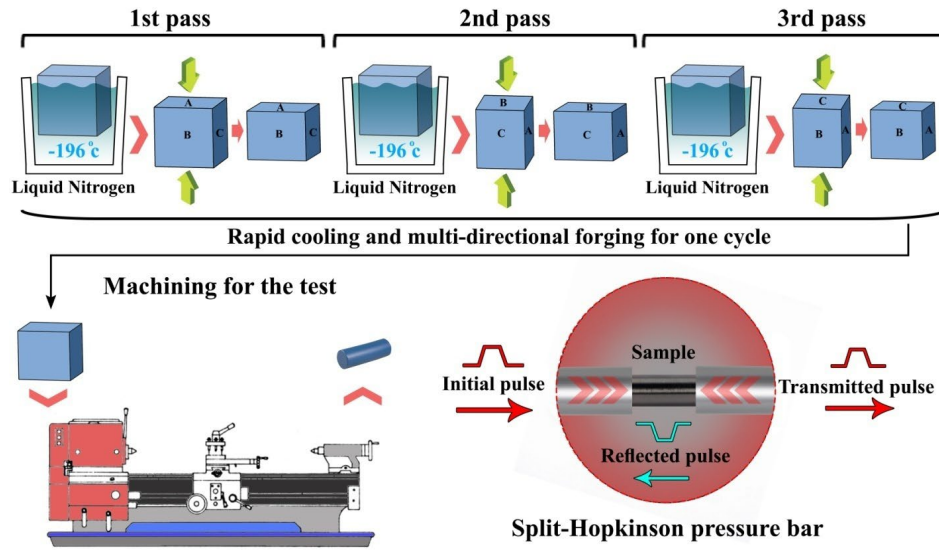


**Figure 2. Schematics of the cryogenic multi axial process followed by SHPB test.**

The AM 316L SS dog-bone-shaped samples used in this study for the second objective were fabricated by Honeywell FM &T using the L-PBF method with a Renishaw's AM 250 machine. The samples were fabricated in accordance with ASTM E8 specifications. The samples used in this study have two different thicknesses, of 1.5 mm and 4.0 mm, with the other dimensions, such as gauge length and width, remaining fixed. The detailed chemical compositions of the 316L sample are given in Table 2.

**Table 2: Chemical Composition of 316 L SS Powders (Wt.%) Provided by Honeywell FM &T.**

| Element                 | Fe   | Cr    | Ni    | Mo   | Mn  | Si   | N    | O    | P     | C     | S     |
|-------------------------|------|-------|-------|------|-----|------|------|------|-------|-------|-------|
| <b>Powder wt.%</b>      | Bal. | 18.2  | 10.2  | 2.57 | 1.1 | 0.8  | 0.07 | 0.02 | 0.009 | 0.013 | 0.001 |
| <b>ASTM 240 316L SS</b> | Bal. | 16-18 | 10-14 | 2-3  | 2.0 | 0.75 | 0.1  | -    | 0.045 | 0.03  | 0.03  |

## 3.2 Mechanical Tests

### 3.2.1 High Strain Rate Tests for Ultrafine Grained 5052 Alloy

The processed AA5052 samples were machined to cylindrical test specimens with 3 mm diameter and 3 mm long (aspect ratio,  $L/D = 1$ ) for the attainment of high strain rates as is the common practice in split-Hopkinson pressure bar (SHPB) [53] experiments. SHPB, also called Kolsky bars, was used for the characterization of the dynamic response of the material at high strain rates which fall within between  $10^2$  to  $10^4$   $s^{-1}$  range. A photograph of the SHPB setup employed in performing

the high strain rate tests is illustrated in Figure 3. This impact machine possesses an incident bar, a transmitter bar, a striker bar, a light gun, and data acquisition system (strain conditioners/amplifiers and a digital oscilloscope).

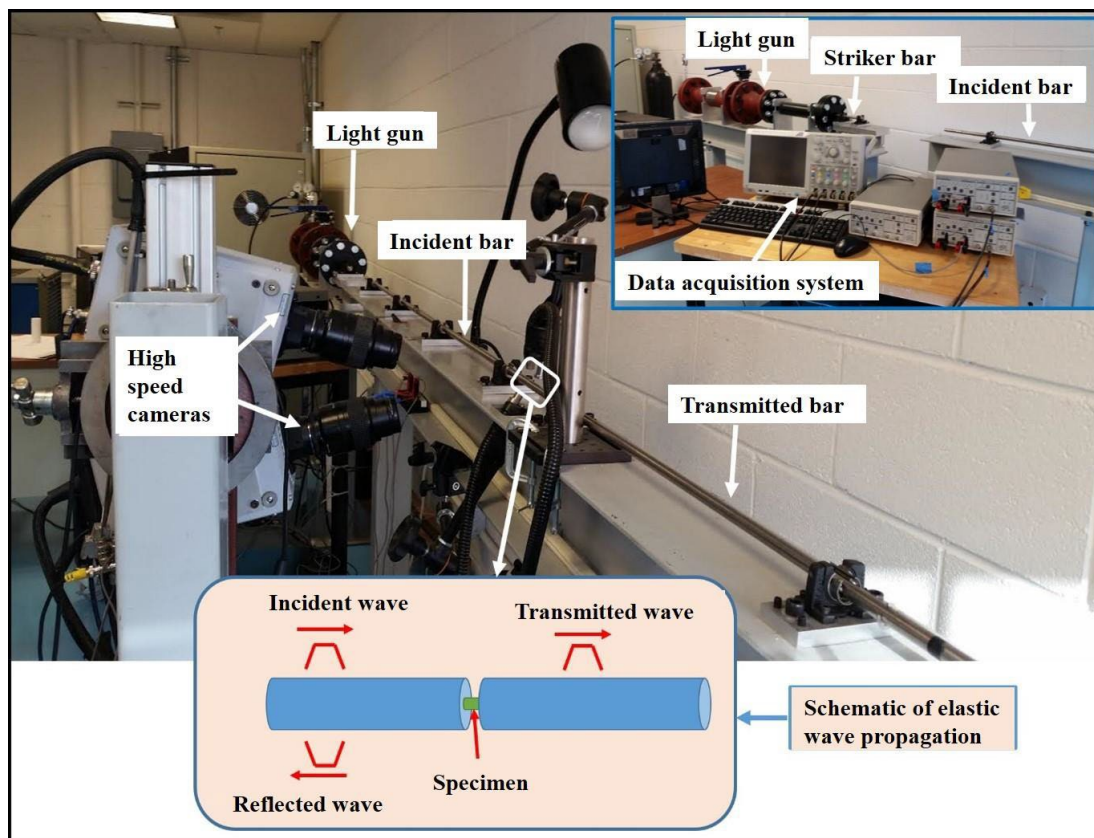


**Figure 3. Experimental set-up of SHPB accompanied with high-speed cameras and data acquisition system.**

The incident and transmitter bars are made of Ti-6Al-4V alloy and have dimensions of 13 mm in diameter and 1900 mm long. The specimens were sandwiched between the incident and transmitter bars. Strain gages of 1000  $\Omega$  resistance are mounted on the incident and transmitter bars at a distance of 940 mm away from the respective bar-specimen interfaces in order to prevent wave interference. The striker bar is fired from a pressurized gas chamber that is charged by a regulated compressed nitrogen gas cylinder. The impact force generates elastic waves which travel through the bars and the sample. The transmitted wave propagating through the transmitter bar is captured as a transmitted wave by the strain gage attached to the transmitter bar. A fraction of the elastic wave is also reflected at the contact between the specimens and the incident bar. These reflected waves are captured by the strain gage attached to the incident bar as reflected waves. The elastic wave data captured by the strain gages are processed by strain conditioners/amplifiers connected to the strain gages and are stored by the connected mixed signal digital oscilloscope. Using these signals, the data are converted to true stress, true strain and strain rate data using a MATLAB code that was provided by the producer of the SHPB. A typical voltage-time graph is indicated in Figure 4 to show the captured signal from the incident, the transmitted and reflected waves. The

compression tests were carried out on each specimen of the MAF processed AA5052 alloy under cryogenic (CF) at various strain rates ranging from  $1,000 \text{ s}^{-1}$  and  $5,500 \text{ s}^{-1}$ . Replicating the experiments ensured its repeatability and the validity of the experiment such that the average of results has been reported.

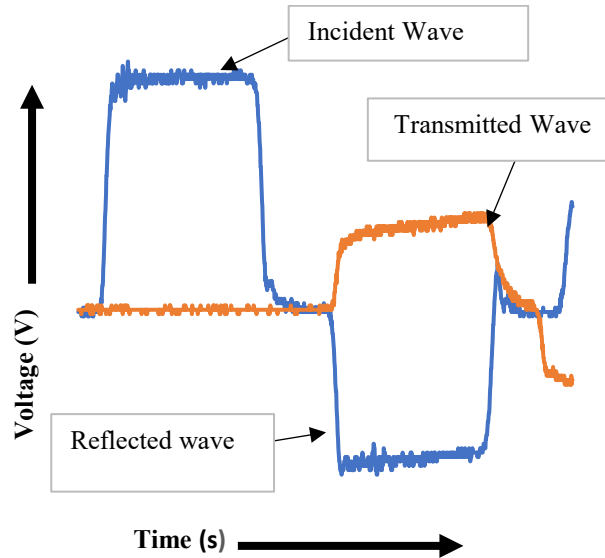


**Figure 4. A Typical Voltage-Time Data of a High Strain-Rate Testing of UFG AA5052 Alloy.**

### 3.2.2 Tensile and Fatigue Tests for the for AM 316L SS produced via L-PBF

Tensile tests were conducted using Instron 5569A dual-column tabletop testing machine at room temperature under a strain rate of  $0.002 \text{ s}^{-1}$ . Five tests were conducted for each thickness, and the average results for each property were reported. The samples did not undergo any surface treatments or post-fabrication processes before being tested. The surfaces did not undergo any polishing or treatments prior to hardness testing. High cycle fatigue tests (HCF) were performed in accordance with ASTM E606 standards using a sinusoidal loading waveform, a frequency of 15Hz, and a load ratio of 0.1. The load ratio,  $R$ , was determined from the ratio of minimum stress divided by the maximum stress applied. The cyclic fatigue loads were carried out at 95 %, 90 %, 95%, 90%, 80%,60%, 50%, 40%, 35%, 30%, and 20 % of the ultimate tensile strength (UTS) for both thicknesses. Tension-tension load controlled fatigue tests were done with a minimum of four samples to ensure adequate data was obtained. The tests were considered runouts when the cycles were above  $2 \times 10^6$ .

### 3.2.3 Hardness Test

Hardness measurements were conducted on both the UFG AA5052 alloy and AM 316L SS materials using a Rockwell hardness tester (B- scale) according to the ASTM E18-20 specifications. A load of 100 kgf was applied for a dwell time of 10 s and indents were made on the surface of gage sections of the samples along a straight line. A minimum of five indents were done on the samples to obtain an average hardness measurement.

### **3.3 Microstructural Characterization**

#### **3.3.1 Metallographic Specimen Preparation**

The microstructures of all the processed MAF samples and the deformed samples after every mechanical test were characterized using optical and scanning electron microscopes. For the UFG AA5052 sample preparation, the samples were first cold mounted using epoxy to avoid structural changes like grain growth which can occur during hot mounting. The standard metallographic grinding and mechanical polishing technique were used for both initial and post-mortem microstructural characterizations. The samples were ground using silicon carbide grinding papers of different grit sizes of 400, 600, 800 and 1200 grit. Water was used to eliminate the embedding of abrasive particles into soft aluminum alloys. These polished samples were then cloth polished using diamond suspension slurry with dimensions of 3  $\mu\text{m}$ , 1  $\mu\text{m}$  and 0.25  $\mu\text{m}$  in water. Different composition of etchants exists but for the best result for the AA alloy, Keller's etchant, which is a solution of 2 ml HF, 1.5 ml HCl, 2.5 ml HNO<sub>3</sub>, and 95 ml DI water, was used.

For the OM and EBSD AM 316L SS samples, the samples were first sectioned along a plane, using a conventional band saw. The faces of the sectioned samples were cold mounted and then mechanically polished using silicon carbide paper of different grit sizes of 320, 600, 800, and 1200 grit. Water was used to remove any abrasive materials from the surfaces of the samples before changing to the next grit size. Subsequently, the samples were cloth polished using a diamond paste of 3  $\mu\text{m}$  and 1  $\mu\text{m}$  before being finally polished using 0.05  $\mu\text{m}$  colloidal silica to obtain a mirror finish. The samples were then cleaned ultrasonically with ethanol and dried under cool airflow.

#### **3.3.2 Optical Microscopy (OM)**

Optical microscopic studies were performed using Zeiss Optical Microscope on the prepared samples after processing and after the mechanical tests. This analysis was used to realize the second phase arrangements in the processed samples as well as to figure out the failure modes in the post-mortem samples. OM with magnification lenses ranging from 5x to 100x and attached with a digital video camera connected to computer was used.

#### **3.3.3 Scanning Electron Microscopy (SEM) & Electron Back Scatter Diffraction (EBSD)**

The microstructures of the initial undeformed, fractured, and impacted samples were also characterized using SEM (Zeiss SUPRA™ 40VP). The samples were cleaned in ultrasonic bath before studies in SEM in order to remove the contaminants from the surface. EBSD was used to estimate the grain size of the processed samples. Zeiss SUPRA™ 40VP Field Emission SEM with TSL-OIM EBSD Data Collection was employed for the EBSD analysis. The EBSD was operated at an accelerating voltage of 20 kV, and imaging was carried out at a step size of 0.05  $\mu\text{m}$  with a 15×15  $\mu\text{m}^2$  map size. The grain size was determined by line intercept method. EBSD samples were prepared by mechanical and cloth polishing followed by electro-polishing at -30 °C at a potential of 11 V for 90–120 s. The electro-polishing was done using solution containing 20% perchloric acid and 80% methanol, DC power supply and reference electrode (stainless steel). Furthermore, fine cloth polishing with colloidal silica was performed to relieve the remaining stresses from the surface. The post-processing of the EBSD data was carried out using orientation imaging microscopy (OIM) analysis software to obtain the Inverse pole figures, Image Quality Figures, and Kernel Averaged Misorientations (KAM) maps.

## 4 RESULTS AND DISCUSSION

### 4.1 Optical Micrographs of the UFG AA5052 Alloy

The optical micrographs of the undeformed AA5052 are shown in Figure 5. These include the solution heat treated (SHT) sample (Fig. 5a) and the cryogenic forged samples at 2 cycles (Fig. 5b), 4 cycles (Fig. 5c), and 6 cycles (Fig. 5d). Figure 5a shows that the grains for the SHT material appear to be equiaxed with an average size measured as around 350  $\mu\text{m}$ . There is also the presence of impurity phase particles ( $\text{MgO}$  and  $\text{Mg}_2\text{Si}$ ). These particles are of various shapes and sizes and measures up to 20  $\mu\text{m}$ . They play a prominent role in pinning the movement of dislocations. Figures 5 (c-d) shows that there is a reduction in the second phase particles as the number of MAF cycles increases. These refined second phase particles align in the forging direction and its presence within the microstructure is an advantage because it promotes dislocation accumulation by impeding its motion. The cryogenic forging refined the grains to become UFGs and therefore the grains become unrecognizable through the optical micrographs.

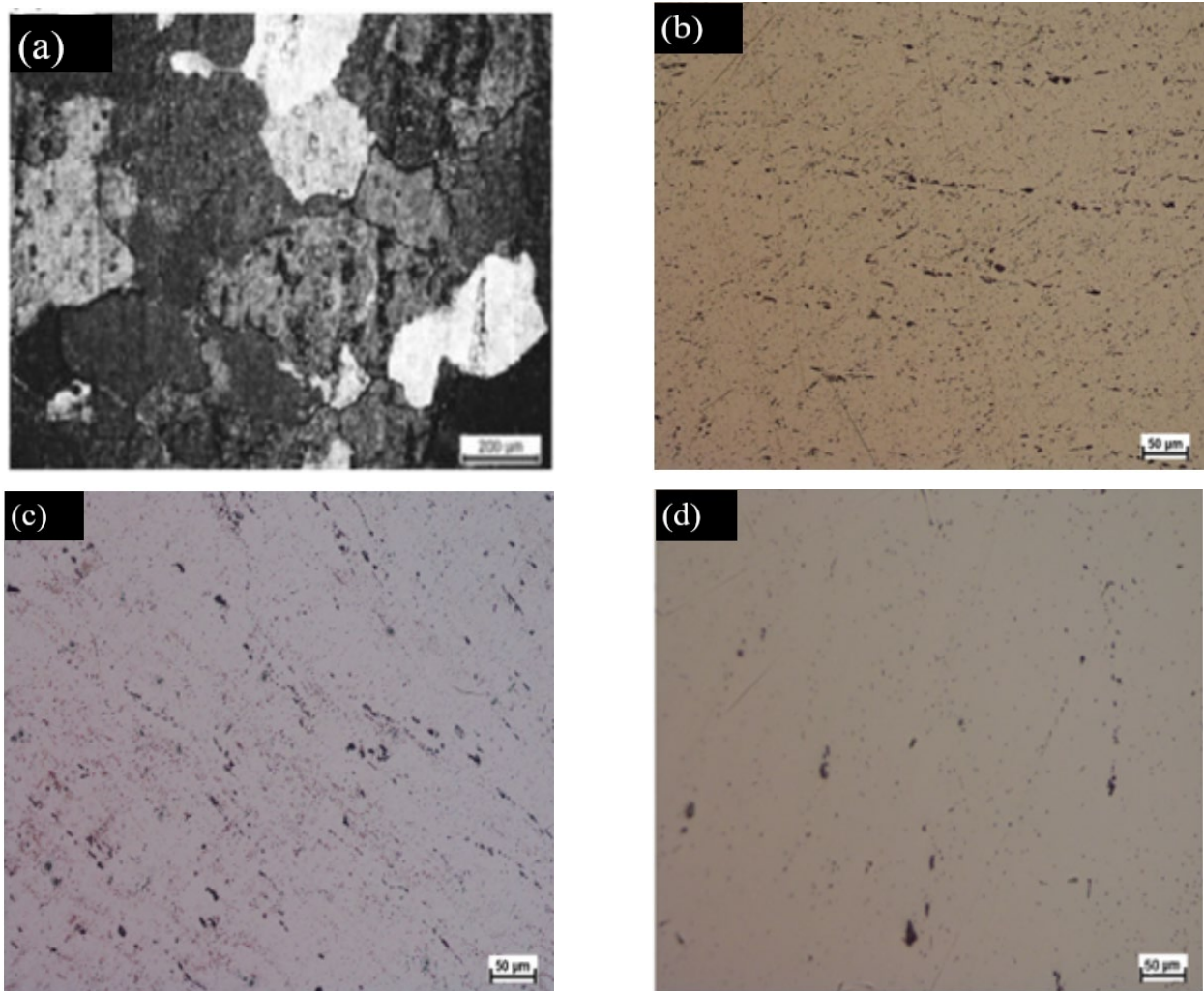


**Figure 5. Optical Micrographs of AA5052 CF (A) Solution Heat Treated. (B) 2 Cycles (C) 4 Cycles (D) 6 Cycles.**

#### 4.2 Inverse Pole Figures and Image Quality Maps of the Initial Microstructure of UFG AA5052 Alloy

The cryogenic forged samples were subjected to electron back-scattered diffraction (EBSD) analyses using a Zeiss SUPRATM 40VP Field Emission SEM with TSL-OIM EBSD Data Collection and the Orientation Image Microscopy data acquisition software to examine the crystallographic texture, grain size distribution, and the distribution of misorientation angles. The Inverse Pole figures of the AA5052 at 2 cycles, 4 cycles, and 6 cycles of CF are presented in Figure 6 to Figure 8. The radial direction of the sample coincides with the vertical axis while the forging direction (tangential direction) is along the horizontal axis. These figures clearly show that all samples produced after MAF possess both elongated and fragmented grains. The grain refinement appears to be best in the samples produced after 6 MAF cycles. This leads to formation of high angle grain boundaries (HAGBs), which are responsible for blocking of dislocations like slip, to increase strength of the material.

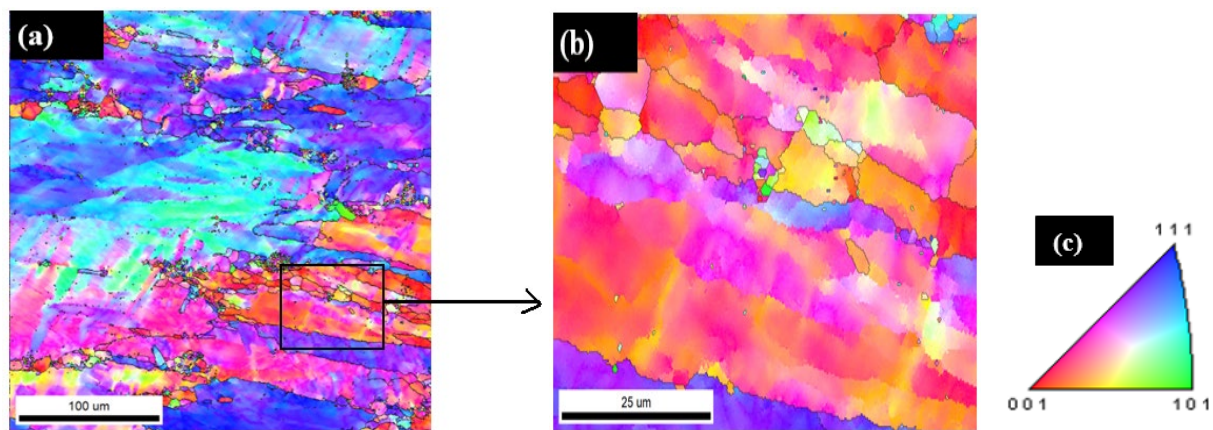


**Figure 6. Inverse Pole Figures of the AA5052-CF 2 Cycles (A) Large Area (B) Small Area (C) Color Code Map.**

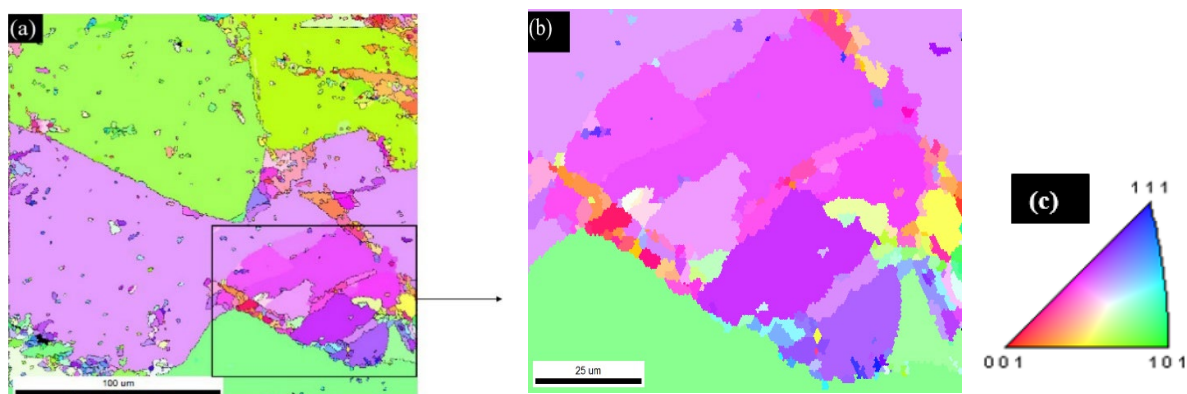


**Figure 7. Inverse Pole Figures of the AA5052-CF 4 Cycles (A) Large Area (B) Small Area (C) Color Code Map.**

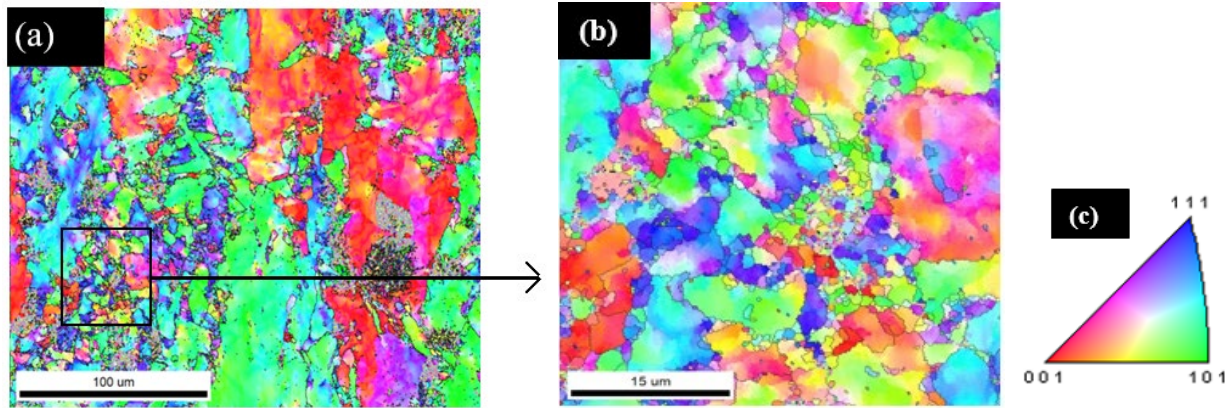


**Figure 8. Inverse Pole Figures of the AA5052-CF 6 Cycles (A) Large Area (B) Small Area (C) Color Code Map.**

A typical method of constructing image quality (IQ) maps is to map the lowest IQ value to black, the highest value to white, and the gray values between these two extremes are linearly scaled [54]. The IQ map for AA5052 2 cycles, 4 cycles, and 6 cycles CF are shown in Figure 9. Based on the local misorientation, it is easy to differentiate the recrystallized grains from the deformed grains. The IQ maps shows that the AA5052 6 cycles CF has more deformed grains than recrystallized ones while the reverse is the case for AA5052 2 cycles CF. This is largely due to the high plastic deformation the 6 cycles. Figure 10 to Figure 12 show the grain misorientation plot, wherein, an increase in the fraction of HAGBs is observed from 2 cycles to 6 cycles conditions. The average misorientation angle for the 2, 4, and 6 cycles are  $10.69^\circ$ ,  $12.55^\circ$ , and  $16.72^\circ$ , respectively.

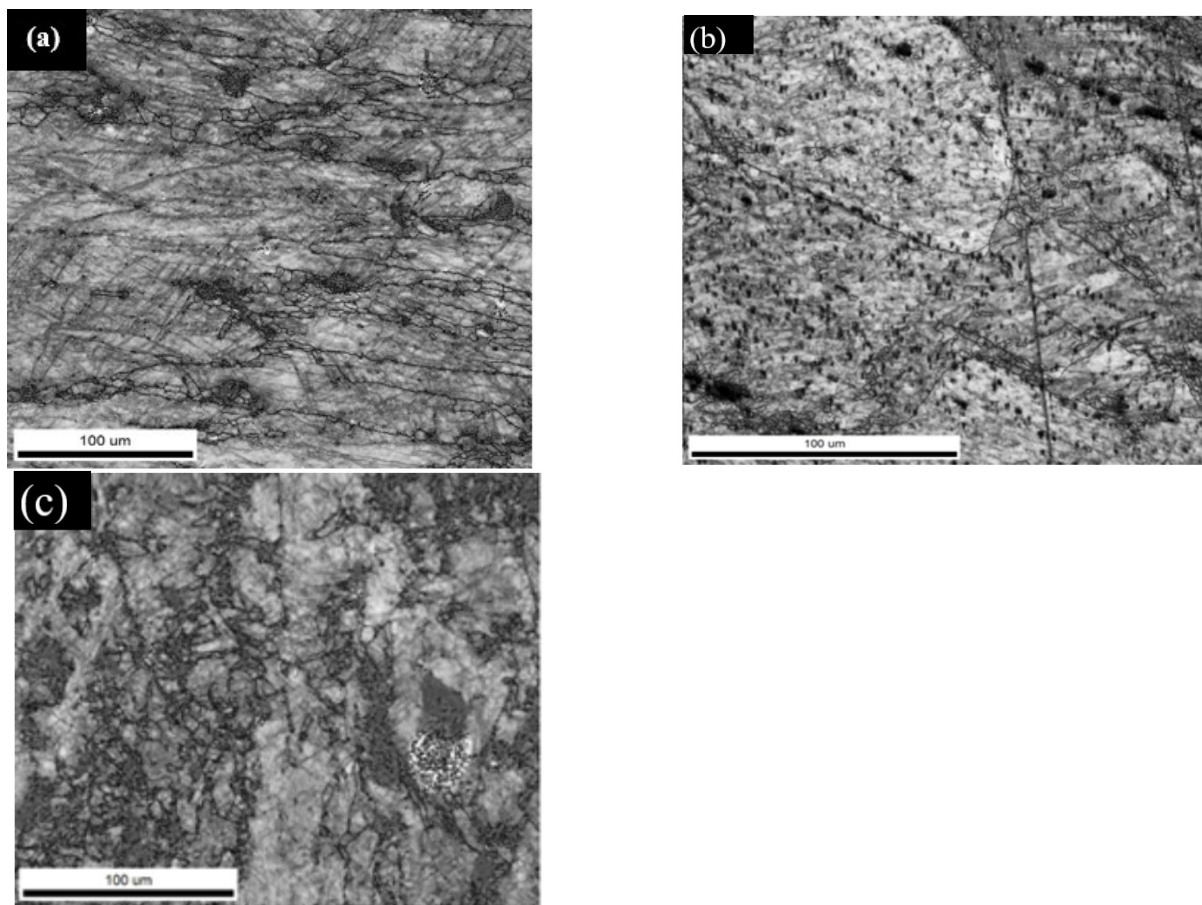


**Figure 9. Image Quality (IQ) Maps for AA5052 CF (A) 2 Cycles (B) 4 Cycles (C) 6 Cycles.**

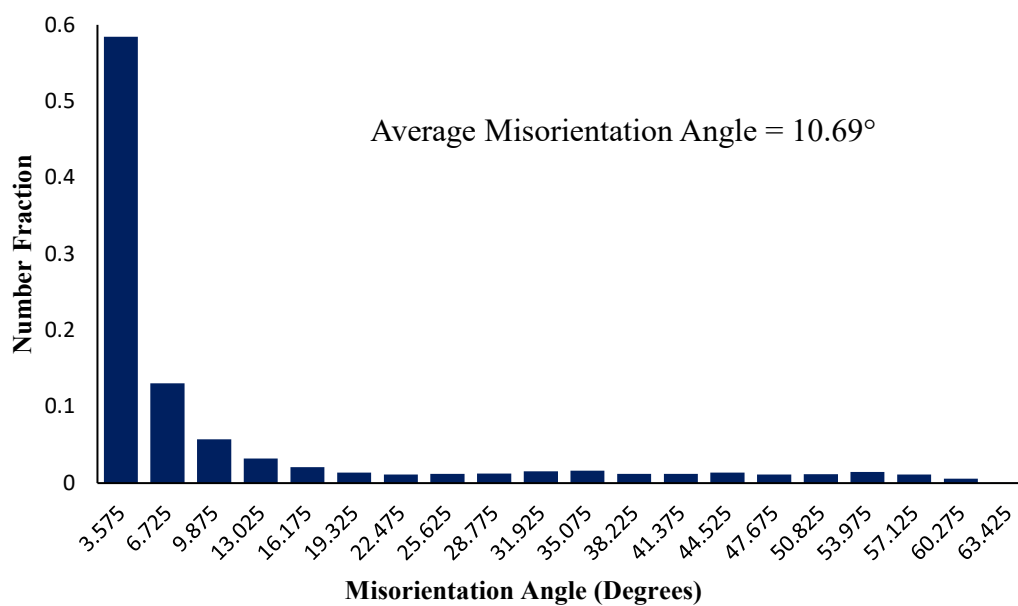


**Figure 10. Misorientation Angle Profile for AA5052 CF 2 Cycles**

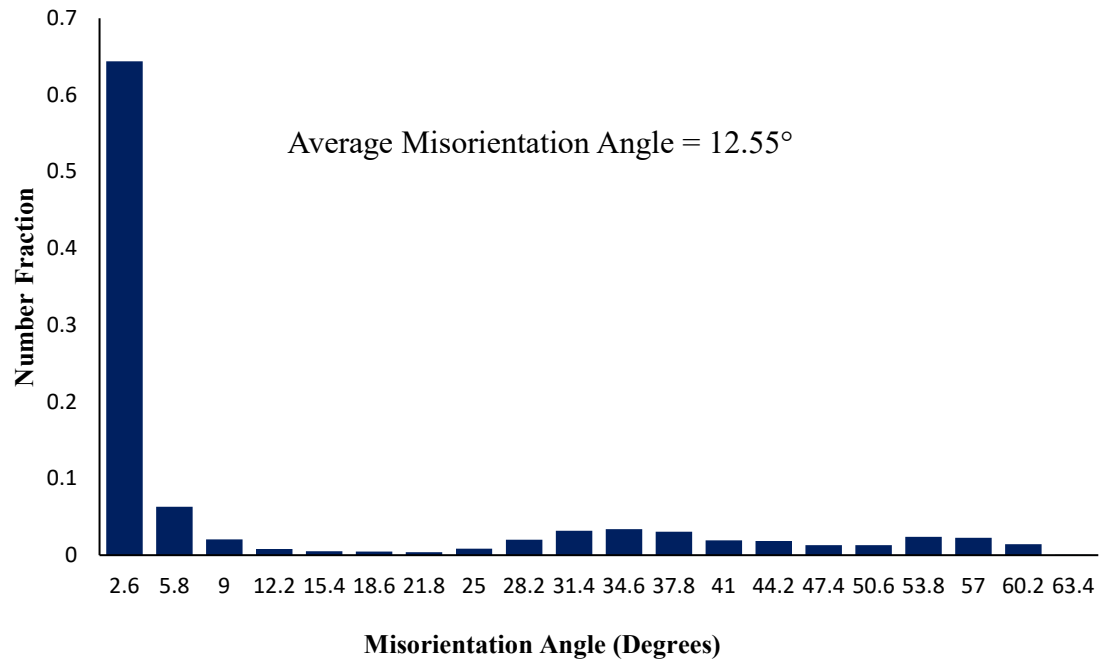


**Figure 11. Misorientation Angle Profile for AA5052 CF 4 Cycles**

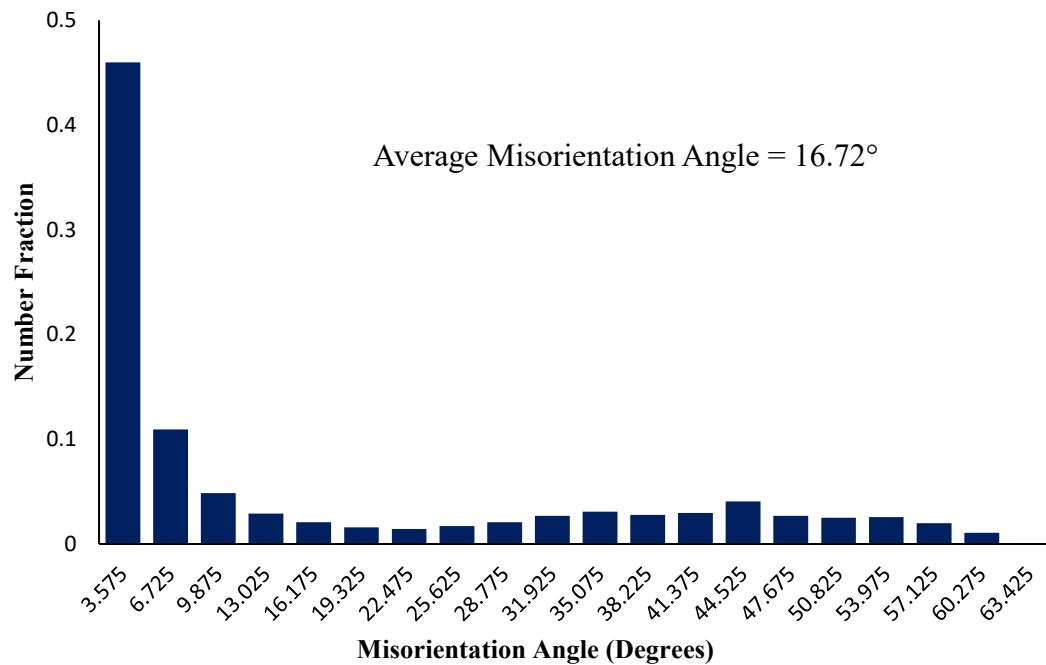


**Figure 12. Misorientation Angle Profile for AA5052 CF 6cycles.**

Significant numbers of grains with low misorientation angle are formed within the microstructures of the processed AA5052 alloy due to the large deformation imposed during the multiaxial forging. Bertolini et. al. [55] reported an increase of the low angle grain boundaries (LAGBs) in AA7075-T6 that was deformed at  $-100^{\circ}\text{C}$  when compared with the as-received alloy. Imposing more plastic strain on the material lead to accumulation of dislocation in the grain boundaries which then lead to formation of incidental dislocation boundaries (IDBs) and geometrically necessary boundaries (GNBs) [56]. As the imposed strain increases so does the misorientation angle of the GNBs, hence transformation from LAGBs to HAGBs [57]. Continuous dynamic recrystallization (CDRX), sometimes referred to as in situ dynamic recrystallization, is the gradual conversion of dislocation boundaries at low plastic strain into high-angle boundaries at large plastic strain [58]. A material's dislocation mobility reduces when the stacking fault energy (SFE) is low [59]. However, aluminum which has high stacking fault energy, undergoes CDRX when strain hardened. This results in an accumulation of dislocations in LAGBs, which increases the misorientation angle and eventually transforms them into HAGBs [60]. This explains why there is higher fraction of LAGBs at the lower number of forging cycles relative to the higher number of forging cycles.

### **4.3 Average Grain Size of UFG AA5052 Alloy**

Further analysis through the Orientation Imaging microscopy (OIM) yields the grain size distribution chart. Grain boundaries were the foundation of the initial model for the Hall-Perch dislocation relation, which was founded on the idea that they act as obstructions to dislocation motion, causing a dislocation pile-up against them [61]. Therefore, reducing the grain size through severe plastic deformation process, which consequently leads to increasing the number of grains, is an attempt to increase the number of grain boundaries to act as trap for dislocation motion, hence, causing dislocation pileup at the grain boundaries. Surface to volume ratio is an important factor in determining the number of grain boundaries in a material. Lower grain size diameter implies higher ratio of grain boundaries to dislocation and thus, higher strength.

The grain size distribution charts for the UFG AA5052 alloy after 2 cycles, 4 cycles, and 6 cycles of MAF are shown in Figure 13 to Figure 18. These charts are particularly useful for estimating the average grain size and the range of grain size that is present within the microstructure. Rather than having approximately small variation in the grain size distribution, the samples produced after 2,4, and 6 MAF cycles have grain size ranging from ultrafine grains to conventional grains which is particularly positive in enhancing the corrosion resistance of this alloy. In a non-passivating environment, it has been demonstrated that a wider grain size distribution offers greater corrosion resistance than a narrower grain size distribution for the same average grain size although the opposite characteristics is true for a passivating environment [62].

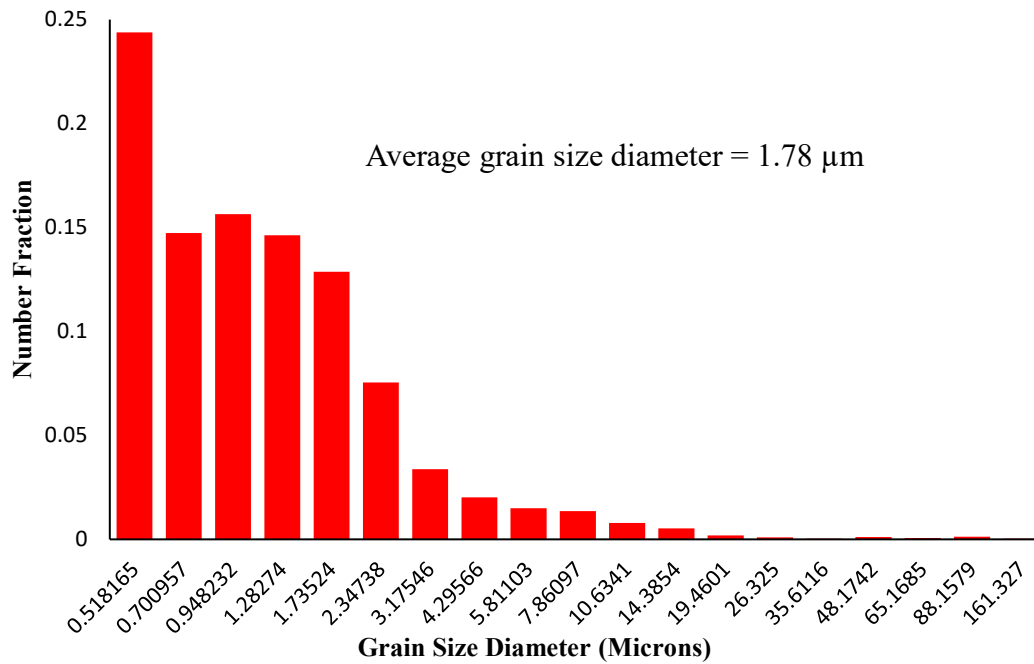


**Figure 13. Grain Size Distribution Chart for AA5052 CF 2cycles (Number Fraction).**

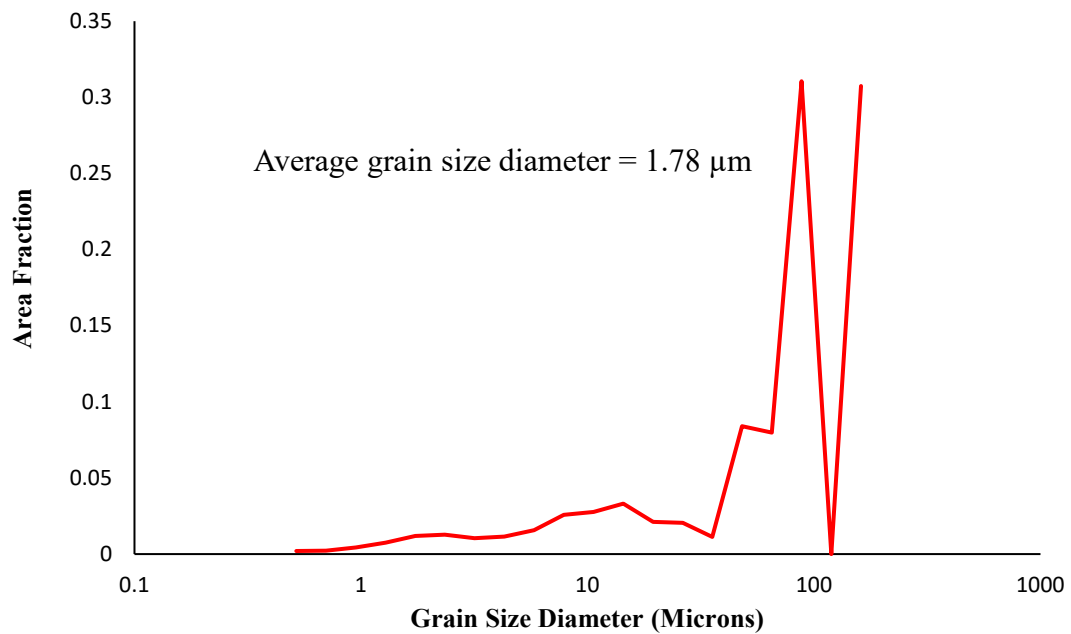


**Figure 14. Grain Size Distribution Chart for AA5052 CF 2cycles (Area Fraction).**

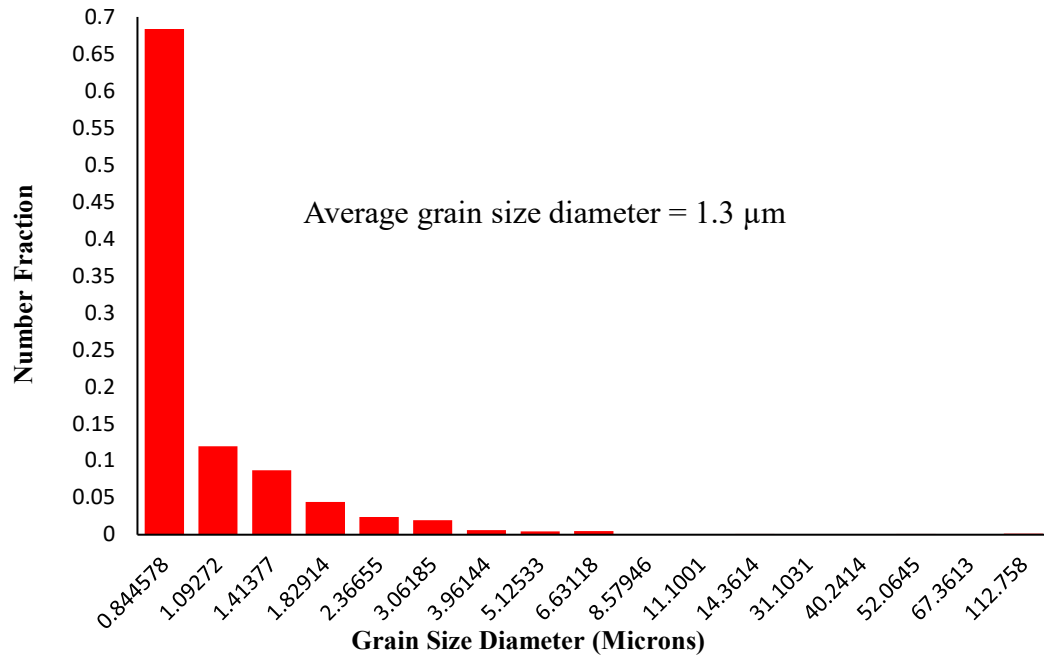


**Figure 15. Grain Size Distribution Chart for AA5052 CF 4cycles (Number Fraction).**

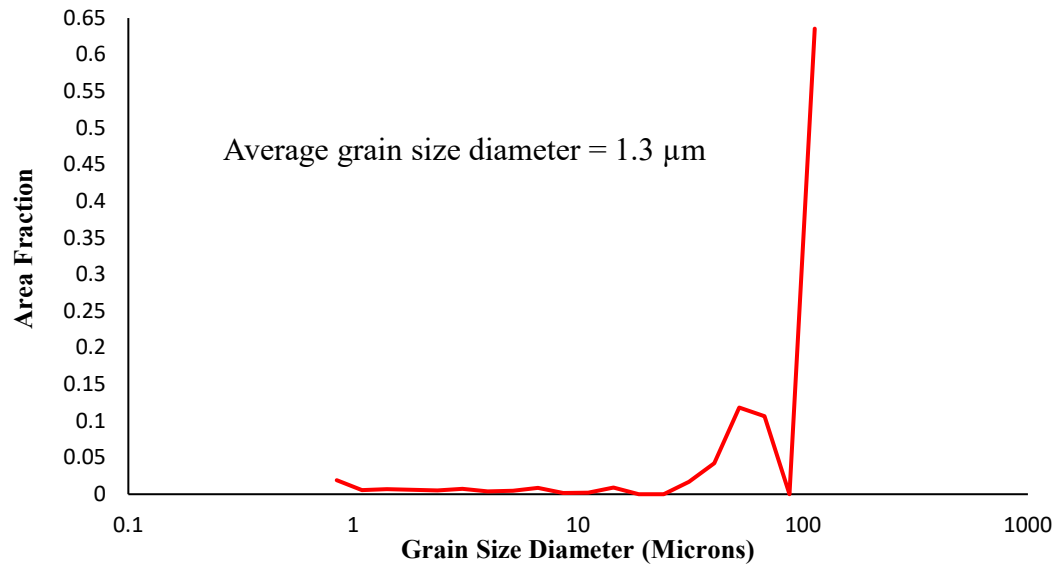


**Figure 16. Grain Size Distribution Chart for AA5052 CF 4cycles (Area Fraction).**

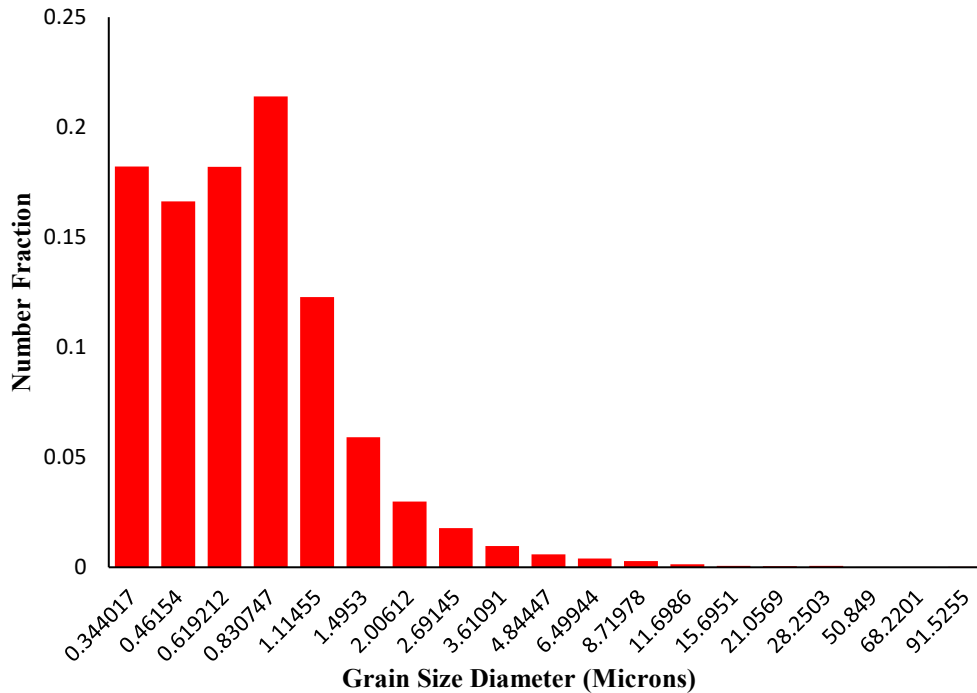


**Figure 17. Grain Size Distribution Chart for AA5052 CF 6cycles (Number Fraction).**

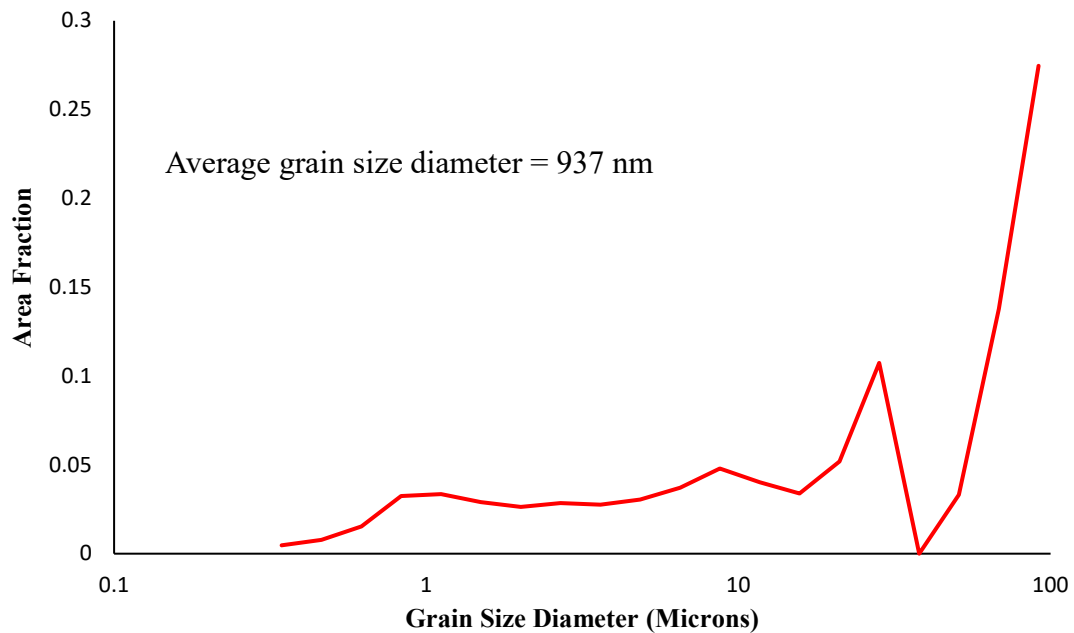


**Figure 18. Grain Size Distribution Chart for AA5052 CF Another 6cycles (Area Fraction).**

The grain size diameter for the 2 cycles ranges from 518 nm to 161  $\mu\text{m}$  while that of the 4 cycles and 6 cycles ranges from 844 nm to 112  $\mu\text{m}$  and 344 nm to 91  $\mu\text{m}$ , respectively. Using statistical analysis, the average grain size for the 2, 4, and 6 cycles were estimated to be 1.78  $\mu\text{m}$ , 1.30  $\mu\text{m}$ , and 937 nm, respectively. As stated earlier, there has been high grain refinement (grain size reduction) relative to the solution heat treated sample that has an equiaxed grain structure with

average grain size of  $350\ \mu\text{m}$ . Although all the samples possess ultrafine grains, based on statistical analysis, the 6 cycles possess more ultrafine grains within its microstructure than the 2 cycles and the 4 cycles samples. The percentages of ultrafine grains present in the microstructure are 54.8 %, 68.4 %, and 74.5 % for the 2 cycles, 4 cycles, and 6 cycles, respectively. While there is a presence of grains of grain sizes in the micron regions, there are clusters of ultrafine grains which provides high volume of grain boundaries to block dislocation motion and thereby strengthening the alloy.

#### **4.4 Mechanical Properties and Microstructures of UFG AA5052 Alloy**

Strain rate in the order of  $10^2$  to  $10^4$  is considered high strain rate and materials behave differently when subjected to high strain rates than as it would under quasi-static conditions [63]. Therefore, it is of utmost importance that material be studied under high strain rate loading for applications where the materials are subjected to such loading. For the purpose of investigating the high strain rate SHPB experiments were conducted, and the results are presented in the following subsections.

##### **4.4.1 True Stress -True Strain Curves**

The high strain rate true stress-true strain curve is the end-product of post-processing the voltage time data gotten from the Split Hopkinson Pressure Bar (SHPB) experiment. These voltage data was first converted into stress-strain data using a MATLAB code provided by the maker of the SHPB. Thereafter, series of data filtering process was done to arrive at the true stress-true strain data that was used for plotting the true stress vs. true strain curves presented in this final report. Figure 19 gives the stress-strain curves of the imparted SHT and UFG samples.

The SHT sample undergoes a strain hardening deformation process and has its maximum flow stress towards the end of the deformation process. However, the MAF processed samples reached their maximum flow stress at the earlier part of the stress strain curves because their deformation is dominated by thermal softening. In addition, the CF samples true strain at failure increases relative to the SHT sample as seen in Figure 20. However, the 2 cycles have the highest true strain at failure of all the cycles. The true strain at failure for the SHT and the samples processed at 2 cycles are 2.04 and 3.54, respectively. The true strain at failure for the 4 cycle and 6 cycle alloys are almost equal and they lie between that of the SHT and 2 cycles. These are 2.91 and 3.0 for 4 cycles and 6 cycles, respectively.

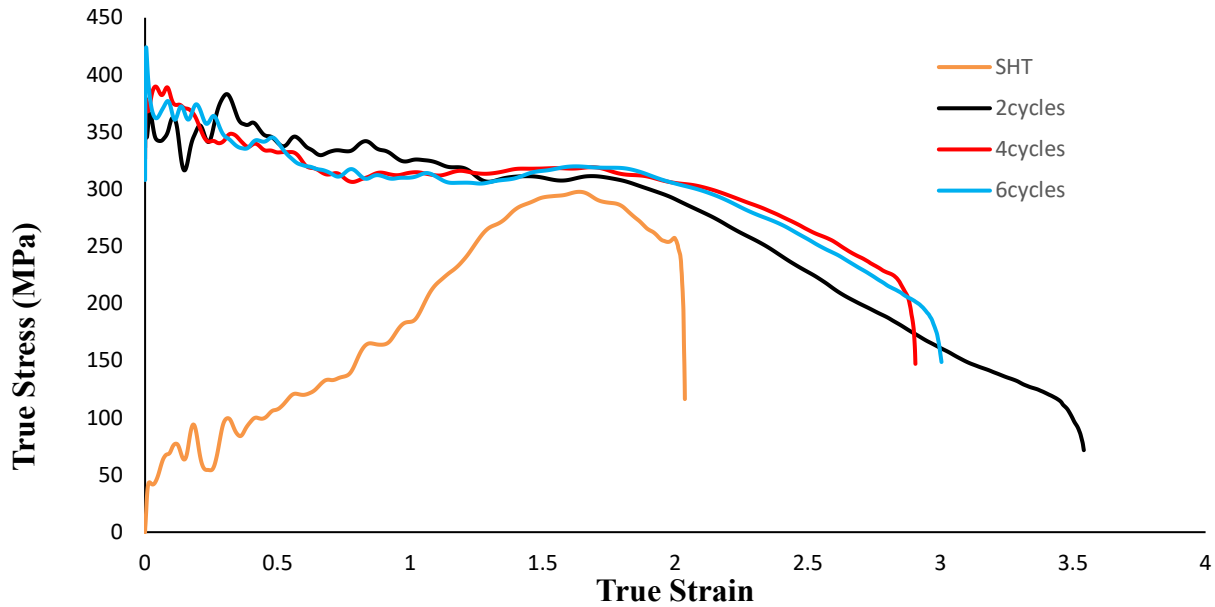


**Figure 19. Dynamic Stress-Strain Curves of AA5052 When Subjected To 5500/S Strain Rate.**

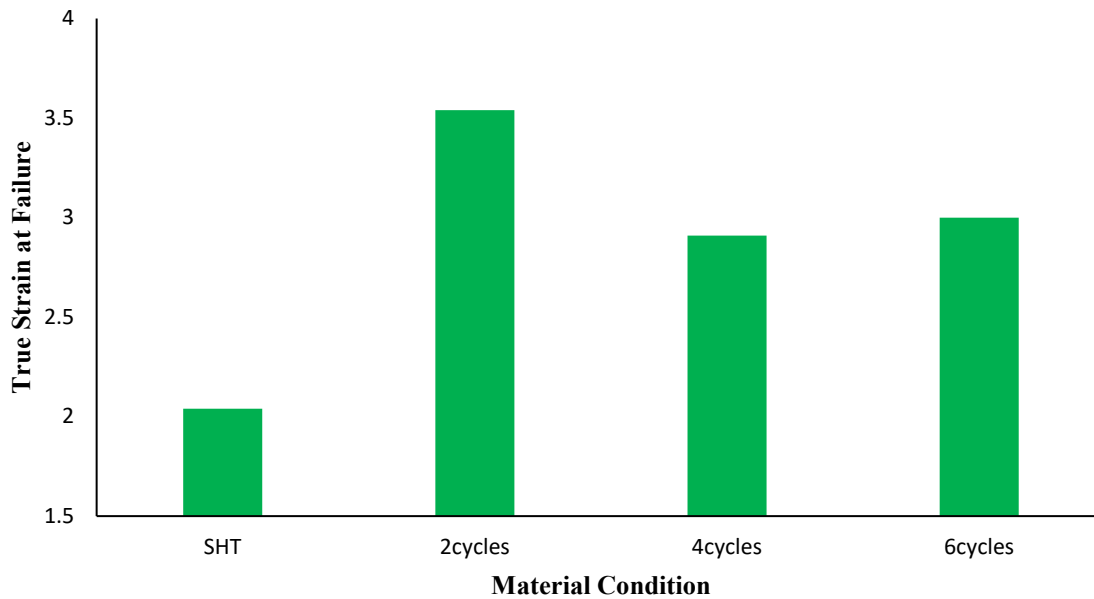


**Figure 20. AA5052 True Strain at Failure When Subjected To 5500/S Strain Rate.**

During the high strain rate deformation, about 90% of the impact energy is converted into thermal energy, and the increased temperature results in softening of the flow. For the high strain rate deformation of the CF samples, it appears that both strain hardening and thermal softening might

have occurred, but the effect of the thermal softening seems to be more predominant in Figure 19. The strain hardening effect during plastic deformation results from the multiplication of dislocations, and strain hardening occurs when dislocations accumulate. However, the CF samples deformation is dominated by thermal softening and not strain hardening because the residual strain in the SPD processed samples makes it almost impossible to accommodate work hardening during deformation therefore resulting to thermal softening.

Despite experiencing some thermomechanical instability and thermal softening, the CF samples have higher maximum flow stress when compared to the SHT sample, which mean there is improvement in the strength of the materials due to the multiaxial forging SPD process (Figure 21). The maximum flow stress for the SHT, 2 cycles, 4 cycles, and 6 cycles are 297.7 MPa, 398.4MPa, 440.9MPa, and 467.4 MPa, respectively. Despite the strain hardening effect that occurs in the SHT samples, its maximum flow stress does not still match the maximum flow stresses of the CF samples. One important factor contributing to the better high strain rate mechanical properties experienced by the CF samples is the lower average grain size diameter relative to the SHT.

The true stress-true strain curves of AA5052-CF 2, 4, and 6 cycles are presented in Figure 22, Figure 23, and Figure 24, respectively. It can be observed that the effect of thermal softening is predominant after the yield point for all the true stress-true strain curves. The thermal softening effect is continuous unto the point of failure. For all the samples tested at different strain rate, there exist little elastic deformation followed by extended plastic deformation. Generally, the final stage of the plastic deformation of aluminum alloys under high strain rate is characterized by the drop in the flow stress because of stress collapse from thermal softening. The thermal softening arises from conversion of about 90% of the plastic work into heat thereby causing thermomechanical instability.

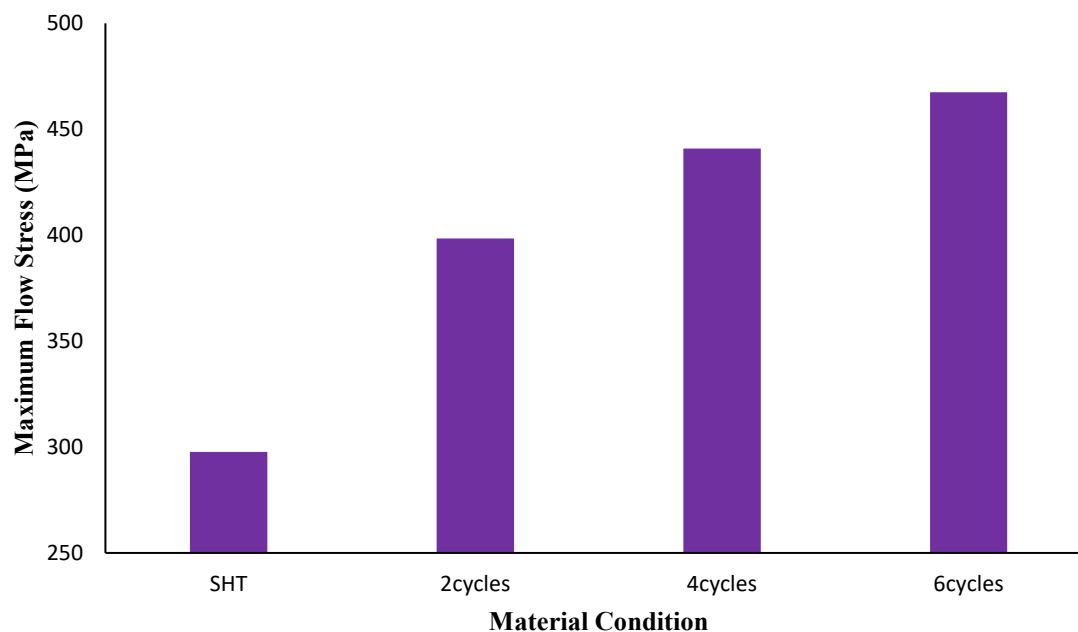


**Figure 21. AA5052 True Strain at Failure When Subjected To 5500/S Strain Rate.**

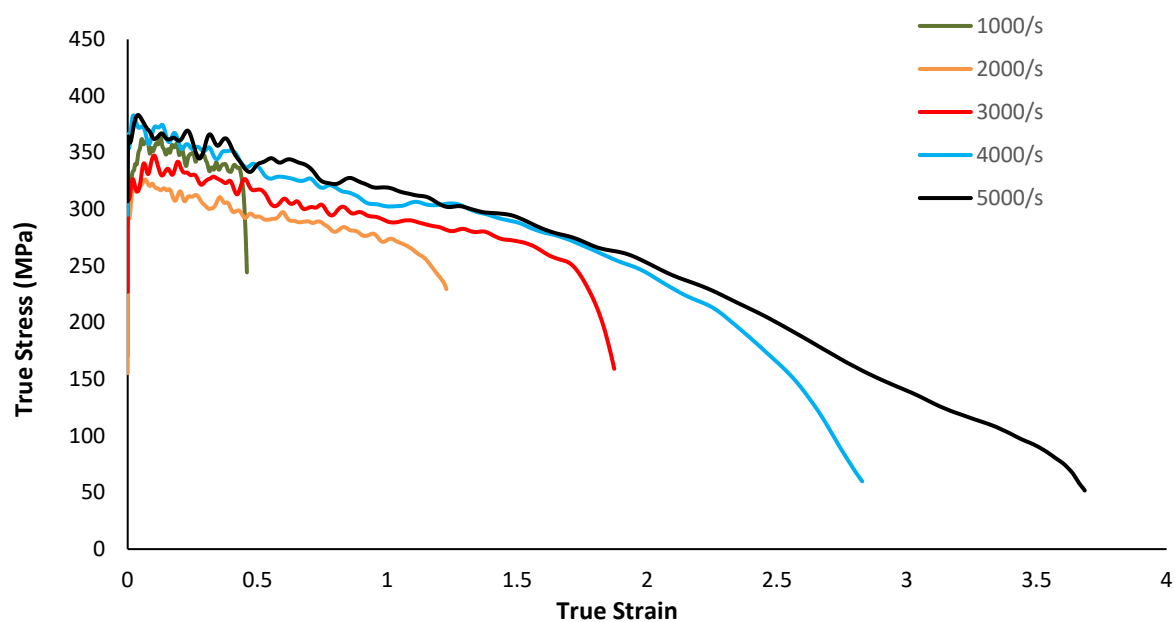


**Figure 22. Dynamic Stress-Strain Curves of AA5052 2cycles CF.**

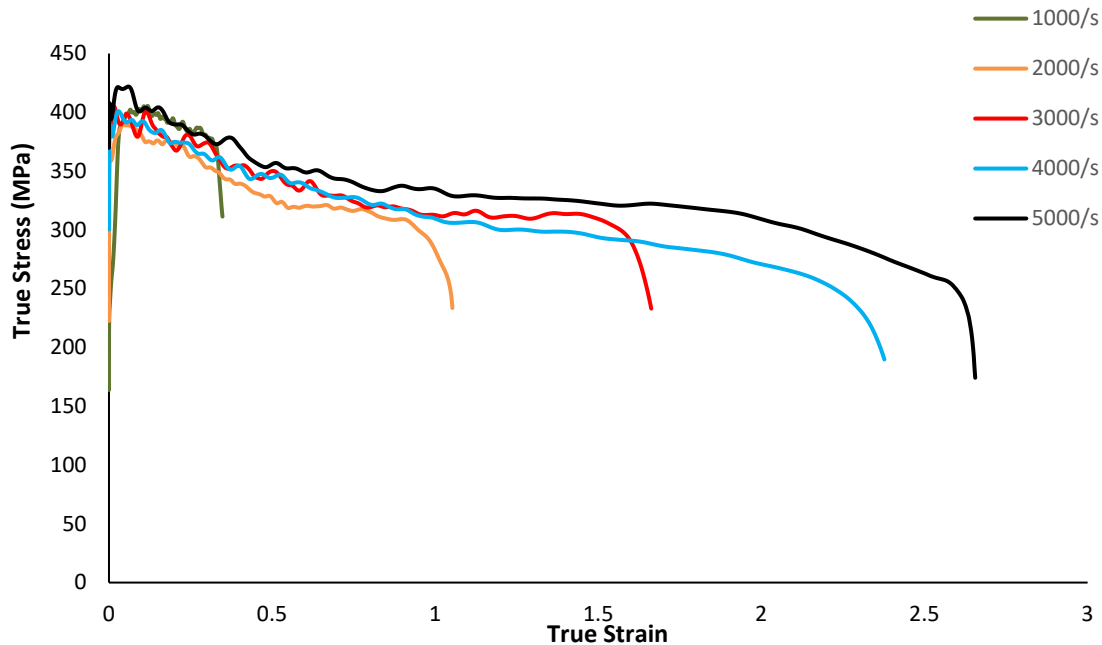


**Figure 23. Dynamic Stress-Strain Curves of AA5052 4cycles CF.**

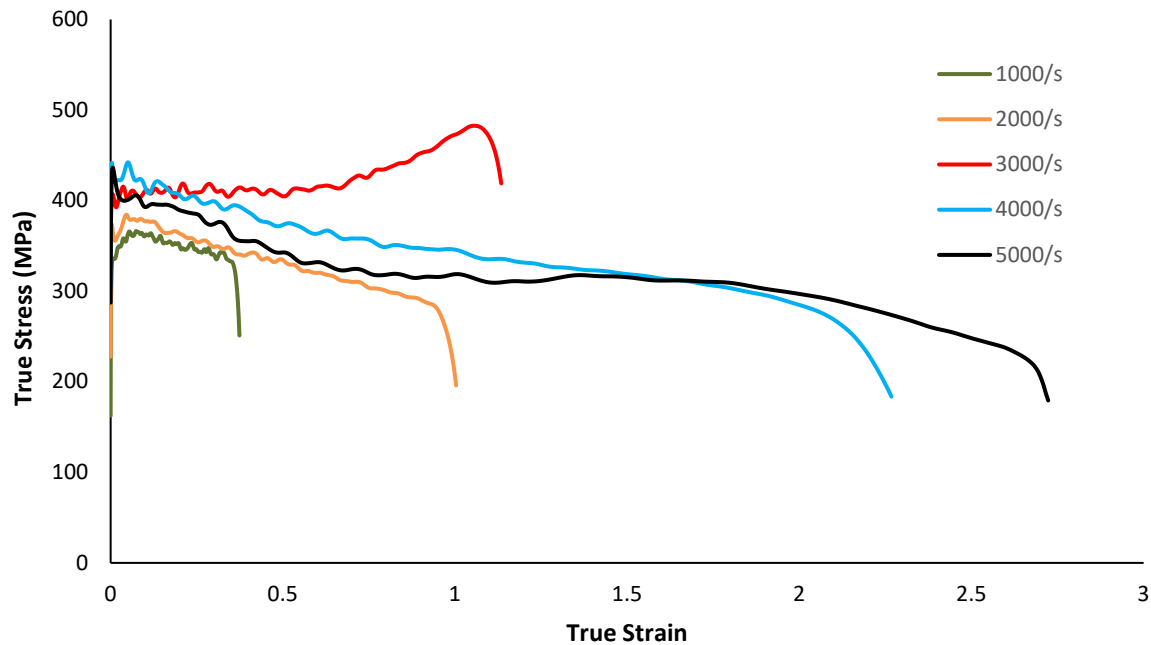


**Figure 24. Dynamic Stress-Strain Curves of AA5052 6cycles CF.**

The total true strain experienced by the samples at various strain rate increases with increasing strain rate. This effect is common to all the samples and the effect of strain hardening decreases with strain rate. In this case, the total strain the materials experience during the high strain rate deformation decreases with the number of cycles for the SPD process. In predicting the strain rate sensitivity index of AA5052, Mohebbi et. al [64] listed the factors that affect high strain rate deformation. These factors include thermally activation dislocation glide, grain boundary sliding,

and dislocation annihilation. These factors could potentially serve as controlling mechanisms for the deformation experienced by the AA5052 CF.

It is not unusual for materials undergoing high strain rate deformation to experience thermomechanical instability [65]. This thermomechanical instability is a consequence of intense adiabatic heating the samples undergo during testing due to the higher thermal energy generated from the impact energy affects the overall performance of the alloys [66-70]. Stress-strain curves with multiple peaks in the figures is a possibility for high strain rate deformation and it may be the consequence of dynamic recovery and recrystallization combined with dislocation accumulation and annihilation [71-72]. As a result of dislocation multiplication at grain boundaries, it is possible for strain hardening to dominated after thermal softening, resulting in an increase in stress up to the second flow stress peak. If the first peak is higher than the second peak, then the second peak does not really have much significance as the first.

The true strain at failure of the 2 cycles is the highest at every strain rate while the difference between the true strain at failure of 4 cycles and 6 cycles is relative lower when compared with the 2 cycles. With increasing strain rate, there is an increase in the true strain at failure and a relative increase in the maximum flow stress. A higher number of cycles also result in increase of maximum flow stress and a reduction of the true strain at failure. Again, this is expected to be the case as higher number of cycles have lower average grain diameter and a more dislocations at the grain boundary. Although the high strain rate deformation of the AA5052 is dominated by the thermal softening, the effect of the thermal softening is less at lower strain rate while it increases with strain rate. At 1000/s strain rate, the stress-strain curves look almost horizontal, meaning the strain hardening effect and thermal softening effect almost canceled each other out. The summary of the maximum flow stress and the true strain at failure at different strain rates is presented in Table 3.

**Table 3: AA5052 CF Maximum Flow Stress and True Strain At Failure**

|                               | <i>Cycles</i> | <i>1000/s</i> | <i>2000/s</i> | <i>3000/s</i> | <i>4000/s</i> | <i>5000/s</i> |
|-------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|
| <b>Maximum Flow Stress</b>    | 2             | 388           | 351           | 395           | 457           | 420           |
|                               | 4             | 425           | 411           | 437           | 426           | 452           |
|                               | 6             | 392           | 400           | 494           | 470           | 462           |
| <b>True Strain at Failure</b> | 2             | 0.454         | 1.242         | 1.835         | 2.759         | 3.791         |
|                               | 4             | 0.365         | 1.057         | 1.662         | 2.410         | 2.664         |
|                               | 6             | 0.370         | 0.990         | 1.152         | 2.347         | 2.758         |

#### 4.4.2 Absorbed Impact Energy

The toughness of a material is the ability of that material to absorb energy when deforming plastically until it fractures or fail. However, “Absorbed Impact Energy” has been chosen in this case because unlike the toughness of the materials that are tested under quasi-static loading and observed unto fracture point, those tested under high strain rate does not necessarily fail by fracture and even if it does, the failure criteria are different as it is characterized with the formation of adiabatic shear bands. The measure of the extent of plastic deformation a material undergoes before fracture is termed ductility. The absorbed impact energy does not rely on high ductility alone but rather combines the effect of strength and ductility. That is, higher strength combined with higher ductility leads to higher absorbed impact energy. In practice, an inverse relationship exists between strength and ductility such that a gain in one leads to a loss in the other which

eventually cancel any potential gain in the absorbed impact energy. Higher strength is often characterized by lower absorbed impact energy and consequently relatively more catastrophic failure[73]. Therefore, for safety-critical applications where untimely or catastrophic failure is not tolerable, materials that has a combination of strength and ductility to produce optimum absorbed impact energy are always the preferred structural material [73].

The absorbed impact energy could be evaluated by trapezoidal rule or by fitting a polynomial curve of second order to the stress-strain curve and integrating the equation of such polynomial within the limits. Irrespective of the chosen method, the result will always be almost identical. Here, the first approach (that is, trapezoidal rule) is chosen and upon estimating the high strain rate absorbed impact energy, the results are presented in Figure 25. Two things can be readily observed from the chart presented in Figure 25. First, the absorbed impact energy increases with strain rate. The increase is caused by the higher impact energy at higher strain rates. At higher strain rates, more energy is absorbed by all the materials at different cycles because the higher strain rates are as a result of higher velocity of the striker bar. There is higher rate of change of momentum at higher velocity provided other factors are constant and, in this case, the controlling factor is the velocity of the striker bar which generates higher impact force and consequently, higher energy [74]. Secondly, there is a reduction in the absorbed impact energy at higher number of cycles. From a previous section, it has been shown that higher number of cycles is characterized by lower average grain size and higher strength due to the number of dislocations at the grain boundaries that has hardened the material. Therefore, it could have been proposed that for the same material subjected to different number of MAF cycles, the one with higher strength will most likely have a lower absorbed impact energy [73]. Two different materials with different flow strength does not necessarily follow the rule of high strength and then lower absorbed impact strength.

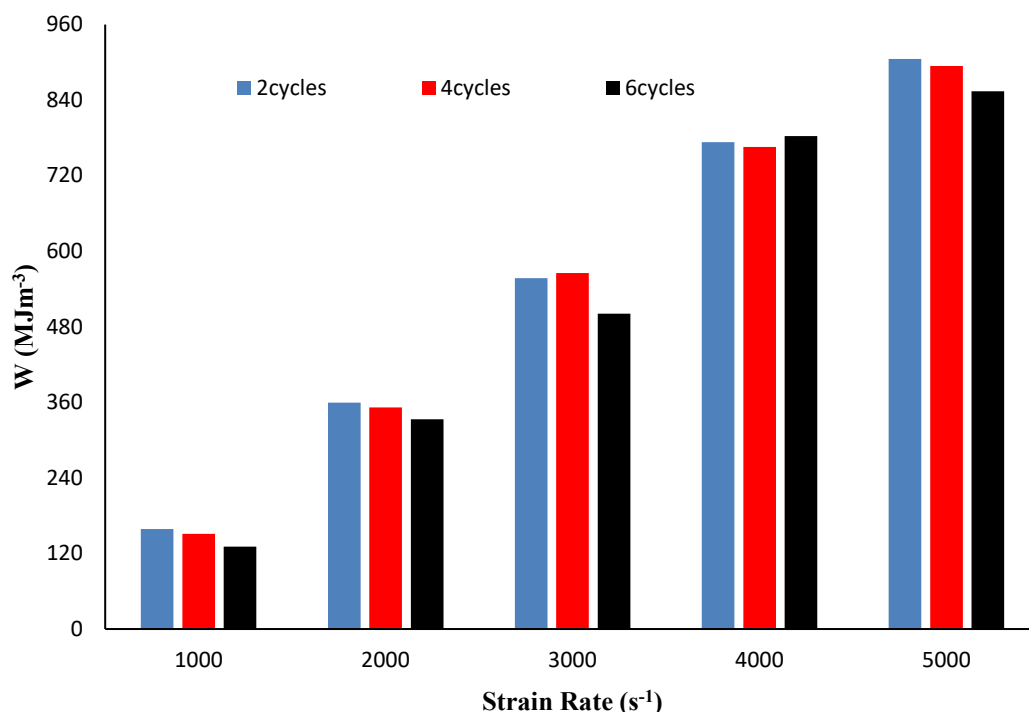


**Figure 25. Absorbed Impact Energy for AA5052 CF At Different Strain Rate.**

Hahn and Rosenfield [75] attributed the reduction in absorbed impact energy of the high strength material to the lower energy required to connect voids, the presence of unstable local plastic region, and the overall loss of strain hardening capacity. The internal energy would have increased at higher number of cycles due to dislocation pileup at the grain boundaries [76]. This will in turn raise the activation energy for dislocation motion of the material and until this activation energy is reached, there is no energy absorption in the material during the deformation process [77]. Therefore, any force that act on the material prior to reaching activation energy of the dislocation motion does not account for the absorbed impact energy. It was demonstrated by Li and Cui [78] that UFG materials have relatively lower energy absorption capacity when compared with conventional grain size and most structural metallic materials have the optimum energy absorption capacity when the grain size ranges from  $1\ \mu m$  to  $10\ \mu m$ .

#### 4.5 Hardness Test Results

The Rockwell hardness test (scale B) was conducted on the AA5052 alloy, and the results of the experiments are presented in Figure 26. There is a significant increase in the hardness value between the SHT and 2cycles CF from 28 HRB to 83.3 HRB. However, further increase in number of forging cycles does not yield much increase in the hardness value with the value improving from 83.3 HRB at 2 cycles to 86.3 HRB at 6 cycles. The average grain size diameters of the samples are  $352\ \mu m$ ,  $1.78\ \mu m$ ,  $1.30\ \mu m$ , and  $937\ nm$  for SHT, 2 cycles, 4 cycles, and 6 cycles, respectively. By relating the average grain size diameters of the samples to the hardness values, it can be easily deduced why there is such improvement in hardness between SHT and 2 cycles and further cryogenic forging after 2 cycles does not yield much improvement in the hardness.

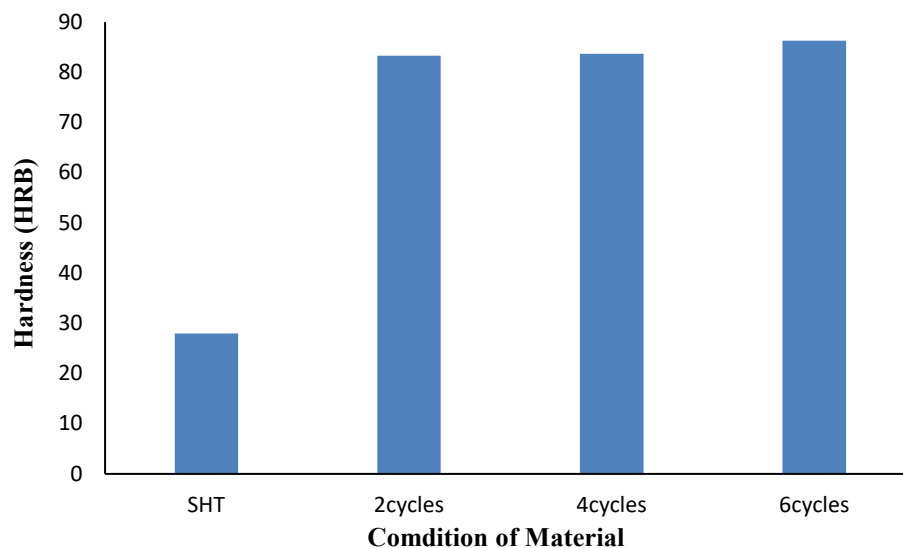


**Figure 26. AA5052 CF Hardness Test Results**

#### 4.6 Metallographic Analysis of the Impacted Samples

The OM images showing strain localization as well as the evolution and formation of the different stages of ASBs in the AA5052 CF are discussed under this section. The failure mode of high strain rate deformation of metallic material is characterized by formation of adiabatic shear bands which can be either deformed bands (DBs) or transformed bands (TBs). The OM image of the AA5052 CF alloy 2 cycles that has been deformed at  $5500 \text{ s}^{-1}$  strain rate is shown in Figure 27. There is a presence of transformed ASBs. As a result of intense strain localization, crack initiation and growth occur through one of the transformed ASBs root and this failure mechanisms are reported for high strain rate deformation of aluminum alloys and steel [79-80]. This transformed bands are therefore most often considered as an element of failure.

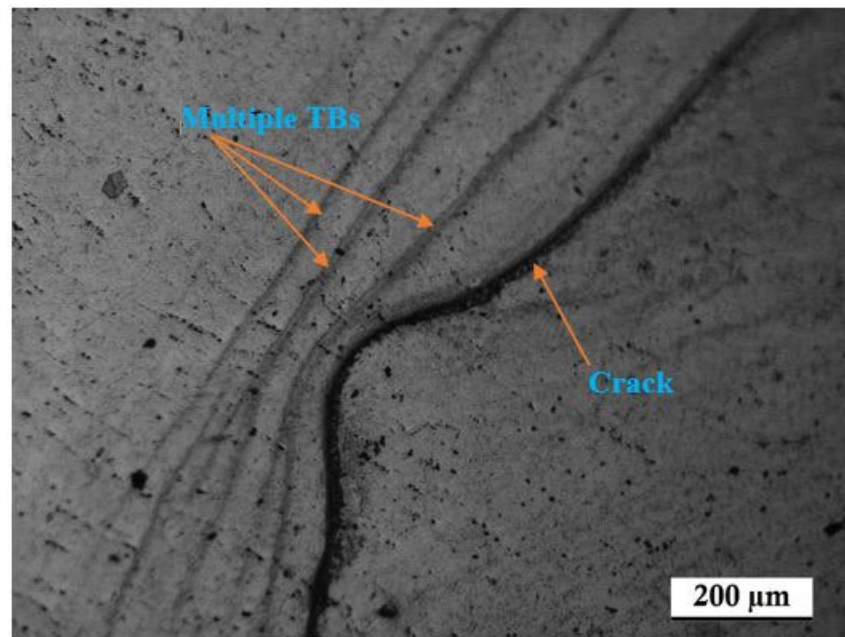


**Figure 27. Adiabatic Shear Band Obtained in AA5052 CF 2cycles At 5500/S.**

The OM image of AA5052 CF 4cycles that is deformed at  $5500 \text{ s}^{-1}$  is presented in Figure 28. Unlike the crack that is visible in the AA5052 CF 2 cycles, there is only the presence of transformed bands in AA5052 CF 4cycles and these transformed ASBs formed multiple shear bands just as it occurs in AA5052 CF 2 cycles. Multiple localizations of deformation are caused by the re-hardening mechanism. In other circumstances, the material changed by successive shear banding developing a differently inclined shear band after a few iterations of localization and results in a sudden softening [81].

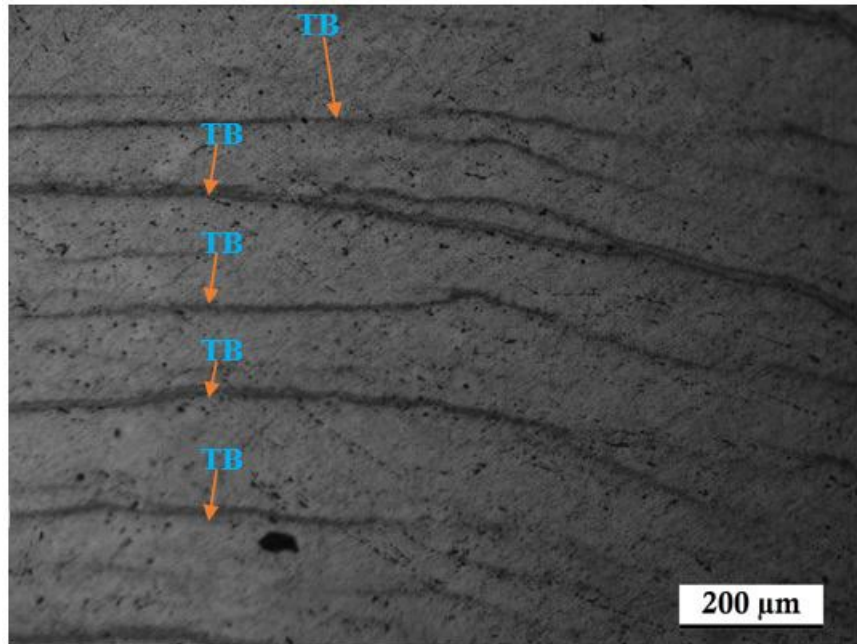


**Figure 28. Adiabatic Shear Band Obtained in AA5052 CF 4Cycles at 5500/s.**

#### **4.7 Mechanical Properties of 316L SS produced via L-PBF**

##### **4.7.1 Mechanical Properties under Tensile Tests**

Figure 29 shows the stress-strain curves for both thicknesses and a chart that compares the yield strength (YS), the ultimate tensile strength (UTS), the elongation to fracture ( $E_f(\%)$ ), and hardness (HRB) from the tested materials with the values of a wrought/standard sample. The ASTM 240/240M specifications for 316L wrought samples were used as a reference value such that; UTS=485 MPa, YS=170 MPa,  $E_f = 40$ , and HRB= 95. The 4.0 mm thick samples exhibited better tensile properties such that the UTS was higher by 5.1 % ( $614 \pm 15$  MPa), higher yield strength by 1.3 % ( $475 \pm 21$  MPa), and 9.2 % higher elongation to fracture ( $72 \pm 3.5$  %) than the 1.5 mm thick samples. For conventionally manufactured samples, the ductility is known to decrease as the strength increases. However, the results of this study showed an increase in strength with an increase in ductility. The ductility of most metals is greatly affected by the presence of defects such as voids, nano inclusions, lack of fusions, and cracks. However, by eliminating these defects, the ductility of metals can be enhanced. The absence of a trade-off between ductility and strength makes AM 316L SS samples suitable for designs that require both high strength and high ductility.

The average hardness of the L-PBF samples were determined to be 87 HRB for the 1.5 mm thick samples and 95 for the 4.0 mm thick samples. These results suggest that the thickness of a material significantly affects its hardness properties. A large scatter in data was observed and could be attributed to the presence of pores and voids. ASTM 240/240M -20a standards specifies that the maximum hardness of 316L SS manufactured either through casting or forging is 95 HRB. The standard values from ASTM F3184-16 for AM components do not specify the hardness values and thus the Renishaw datasheet was used for comparison. The datasheet provides that the hardness value should be 76.5 HRB. With the experimental values obtained, it is clear that the L-PBF samples had higher hardness values than those stipulated.

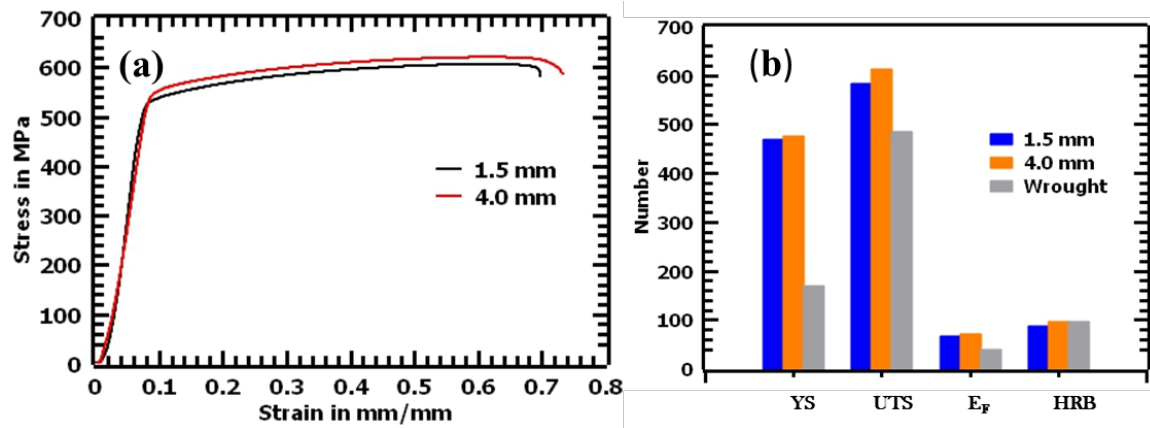


**Figure 29. (a) Stress-Strain Curves of 1.5 mm and 4.0 mm Thick Samples, (b) Average YS, UTS, E<sub>f</sub>, and HRB Values for L-PBF Samples and Wrought Samples.**

#### 4.5.2 Microstructure Characterizations for Tensile Test Samples

The OM images were used to determine the average porosity of the samples. The 1.5 mm thick samples had an average density of 99.972 %, whereas the 4.0 mm thick samples had an average density of 99.986 %. The maximum density was not observed and could be due to the presence of gas-induced and process-induced pores. ImageJ software was used to determine the area fraction of the pores. For both thicknesses, the results showed a porosity of less than 0.2 %. Zhang et al. [82] observed that the area fractions for the pores of L-PBF samples were less than 0.5 %. The samples used in this test, therefore, demonstrated higher density than those of previous works.

The etched samples observed under OM revealed melt pools and melt pool boundaries as shown in Figure 30. The solidification of newly deposited melted material occurred on top of the already solidified layers and thus resulting in molten tracks that overlapped. Figure 30 (a) and (b) of undeformed samples show fine columnar and cellular structure. Some melt pools have a mixture of columnar and cellular structures occurring together. The solidification mode given by the ratio of temperature gradient ( $G$ ) and the growth rate ( $R$ ) influences the microstructures formed. Cellular structures are formed when the ratio of  $G/R$  is intermediate. In addition, increased heat transfer causes finer cell structures to be formed, whereas slower heat transfer results in coarse cell structures. The deformed samples have elongated grains, as shown in Figures 30 (c) and (d). The plastic deformation causes the grains to elongate as they resist the tensile loads hence the observed features.

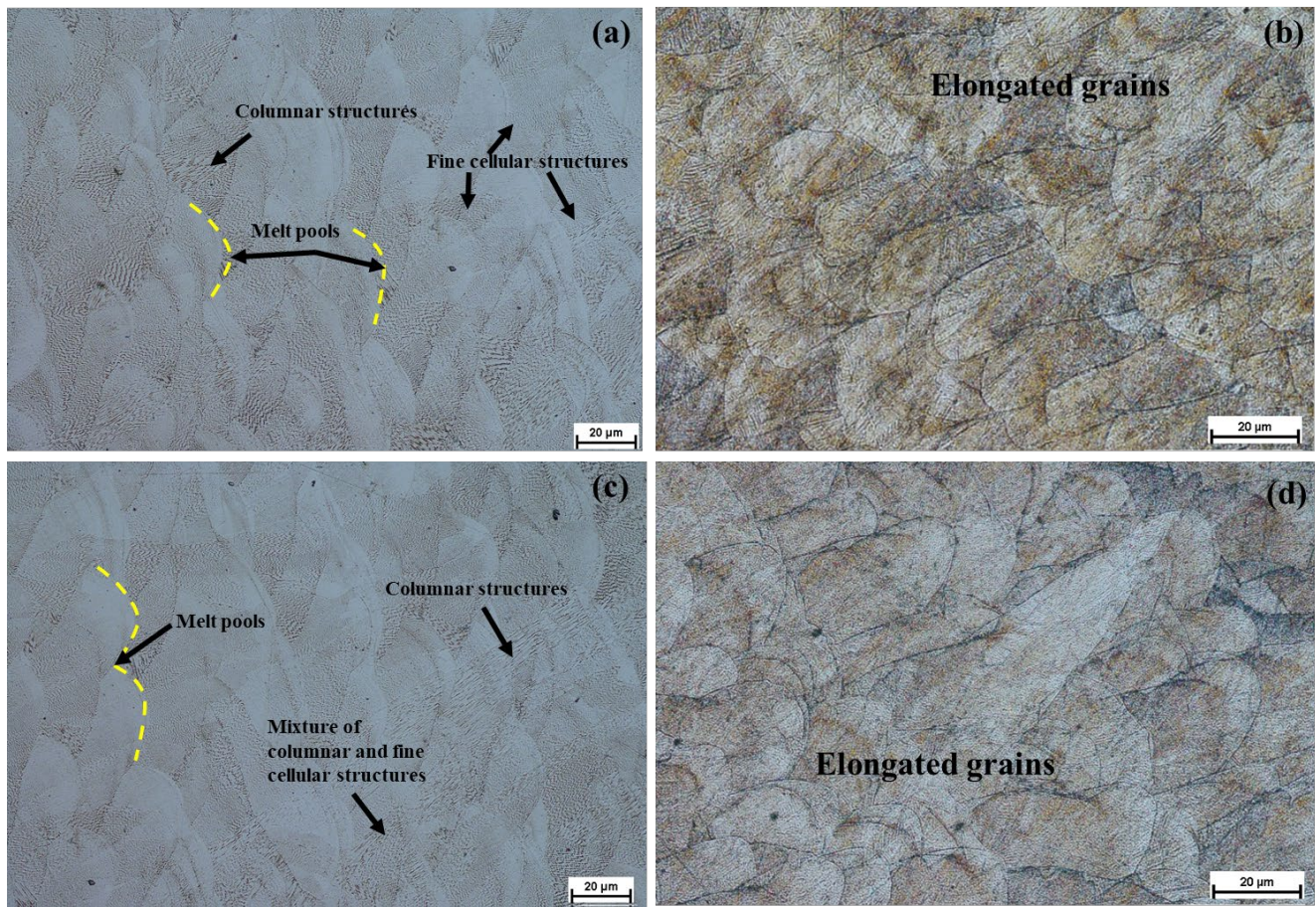


**Figure 30. Micrographs of Etched Samples Showing (a) Undeformed 1.5 mm, (b) Deformed 1.5 mm, (c) Undeformed 4.0 mm, and (d) deformed 4.0 mm, All Observed at 20 X Magnifications.**

The fractured surfaces of tested tensile samples observed under SEM are shown in Figure 31. Both thicknesses revealed various defects. Figures 31 (a) and (b) depict the presence of unmelted materials formed due to insufficient cooling time or the laser power required to sufficiently melt the powder before being deposited on these surfaces. Several pores are also observed on the edges of these samples. These pores could have contributed to the reduced mechanical properties of the samples as they acted as weak spots for the fracture to occur. Figures 31 (c) and (d) show the presence of the balling effect. The size of spherical metallic balls was observed to be smaller but numerous in the thinner sample compared to the thicker sample. Balling effects occur when unmolten material is deposited at high speed with less time to cool properly, resulting in the agglomeration of spherical metallic balls. Li et al. [83] argued that lowering the oxygen content during fabrication helps restrict the balling effect initiation and thus creates much smoother surfaces. Other studies have shown that the defects can be alleviated by remelting the surfaces of the fabricated surfaces.

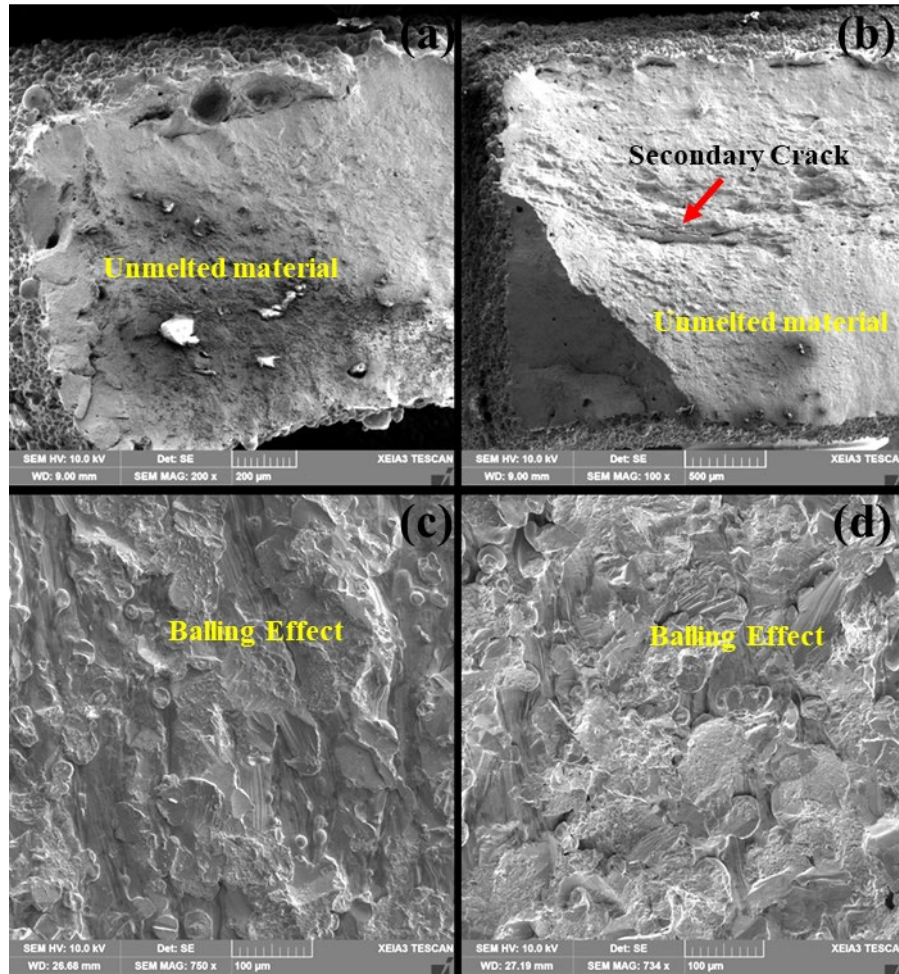


**Figure 31. SEM Images of Fractured L-PBF Samples (a) and (c) Represent 1.5 mm Thick Samples, whereas (b) and (d) Represent 4.0 mm Thick Samples. Images (a) and (b) Show Various Defects at The Edges and On The Sample Surface. The Balling Effects Are Also Depicted in (c) and (d).**

EBSD characterization was conducted to further understand the impact of grain morphology and grain orientation on the mechanical properties of L-PBF 316L SS samples. The IPF maps shown in Figure 32 show the crystal orientations of the undeformed and deformed samples. The undeformed samples were observed to have a more random texture as compared to the deformed samples. Using the inverse figure pole plots shown in Figure 33, the as-built samples showed a strong crystallographic orientation in the  $\langle 101 \rangle$  direction. When tensile loads were applied, the preferred crystallographic orientation of the 1.5 mm thick sample changed to  $\langle 001 \rangle$ , whereas that of the 4.0 mm thick sample changed to  $\langle 011 \rangle$  direction. Both undeformed samples depicted a weaker preferred crystallographic orientation along  $\langle 011 \rangle$  direction, whereas the deformed samples depicted a weaker orientation along the  $\langle 111 \rangle$  direction. These results suggest that grain rotations tend to occur when tensile loadings are applied. In addition, the results suggest that deformation causes the preferred orientation to change with a thickness of a material. Liu and Dupont [84] discussed that the deformation twinning in a material is unfavorable in the  $\langle 001 \rangle$

orientation. The grains' high dislocation pile-ups necessary for the formation of twinning tend to require greater stress to be plastically deformed, hence increased strength.

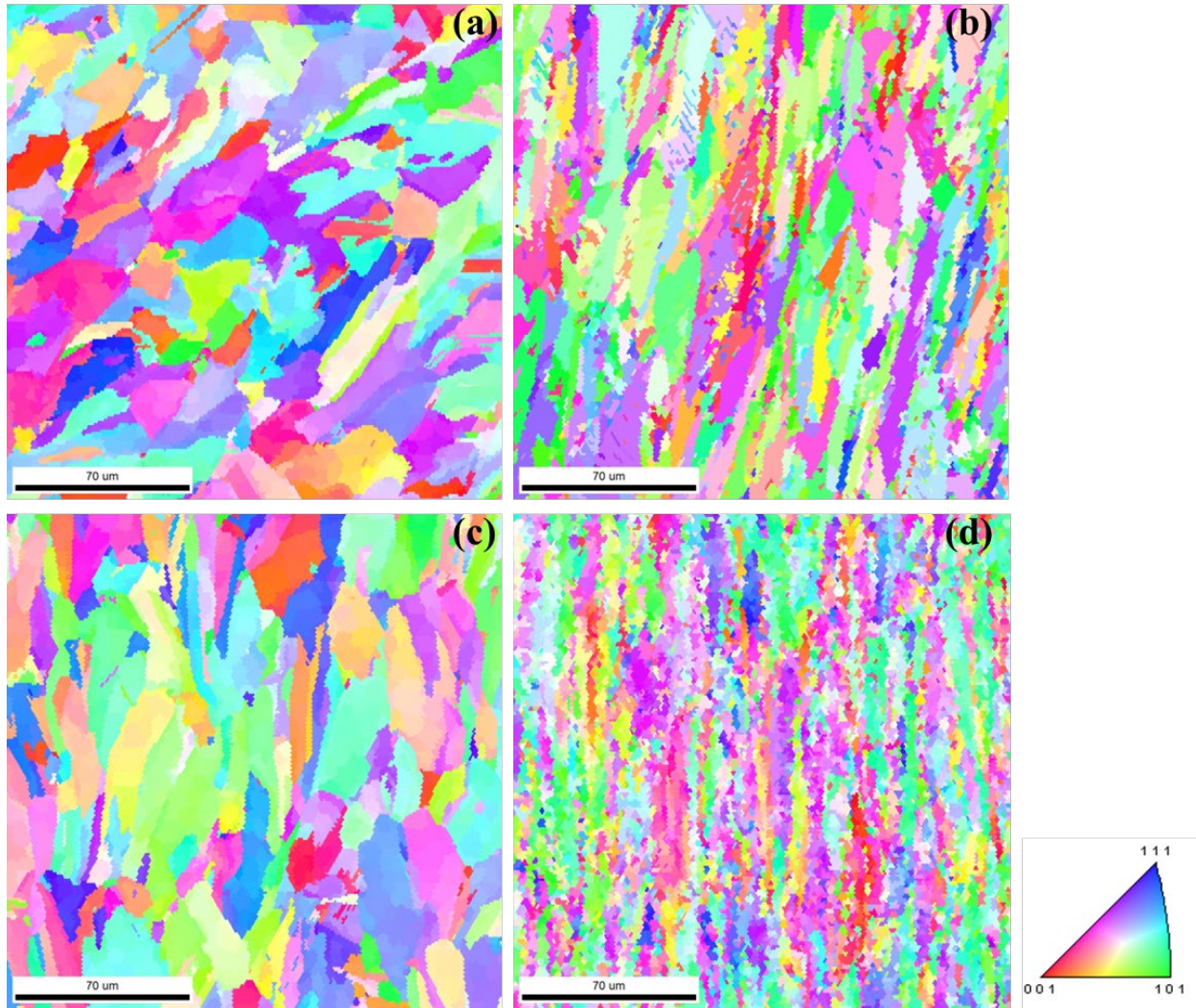


**Figure 32. IPF Maps of the L-PBF Samples Where Different Colors Correspond to the Different Crystallographic Directions; (a) 1.5 mm Undeformed, (b) 1.5 mm Deformed, (c) 4.0 mm Undeformed, and (d) 4.0 mm Deformed.**

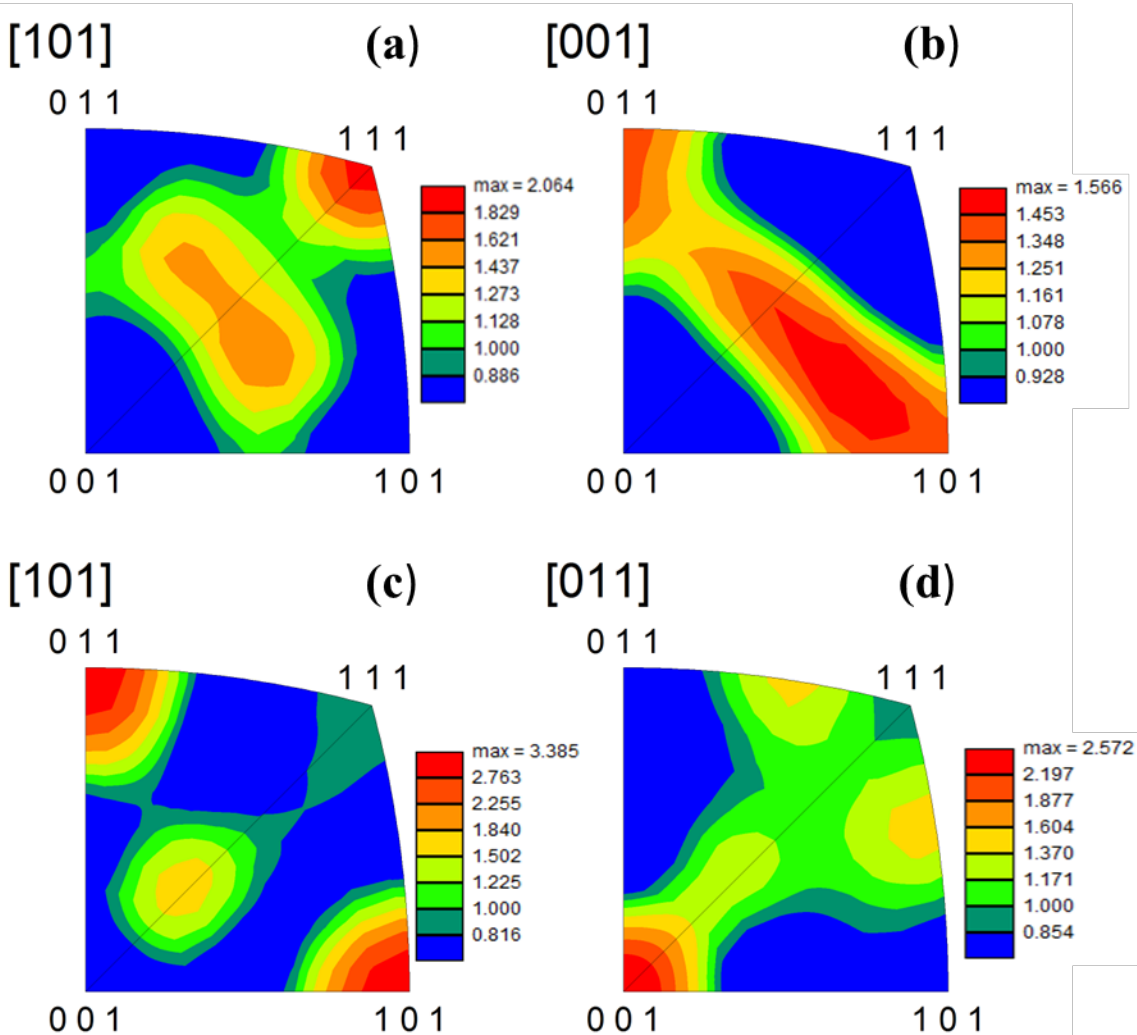


**Figure 33. Inverse Pole Figure Plots for (a) 1.5 mm Undeformed, (b) 1.5 mm Deformed, (c) 4.0 mm Undeformed, and (d) 4.0 mm Deformed.**

The Kernel Average Misorientation (KAM) maps of the observed surfaces are shown in Figure 34. KAM maps are used to represent the average misorientation of a given point (kernel) with respect to that of its neighbors in the same grain, and a misorientation of less than  $5^{\circ}$  is considered. The KAM maps are used to measure the local orientation gradients inside a grain induced by the deformation process. The KAM maps in Figure 34 represent the undeformed and deformed samples for both 1.5 mm and 4.0 mm thick samples. For both thicknesses, an increase in plastic deformation resulted in an increase in the KAM values. The color intensity variations in the KAM maps are observed and could be due to intragranular misorientations. When subjected to deformation, the grains undergo strains that vary from one grain to another. The thermal gradient introduced during the fabrication process also causes varying residual stresses in the grains, thus causing them to behave differently when subjected to loads. The red spots in the KAM maps are more evident along the grain boundaries, suggesting high dislocation densities within these regions.

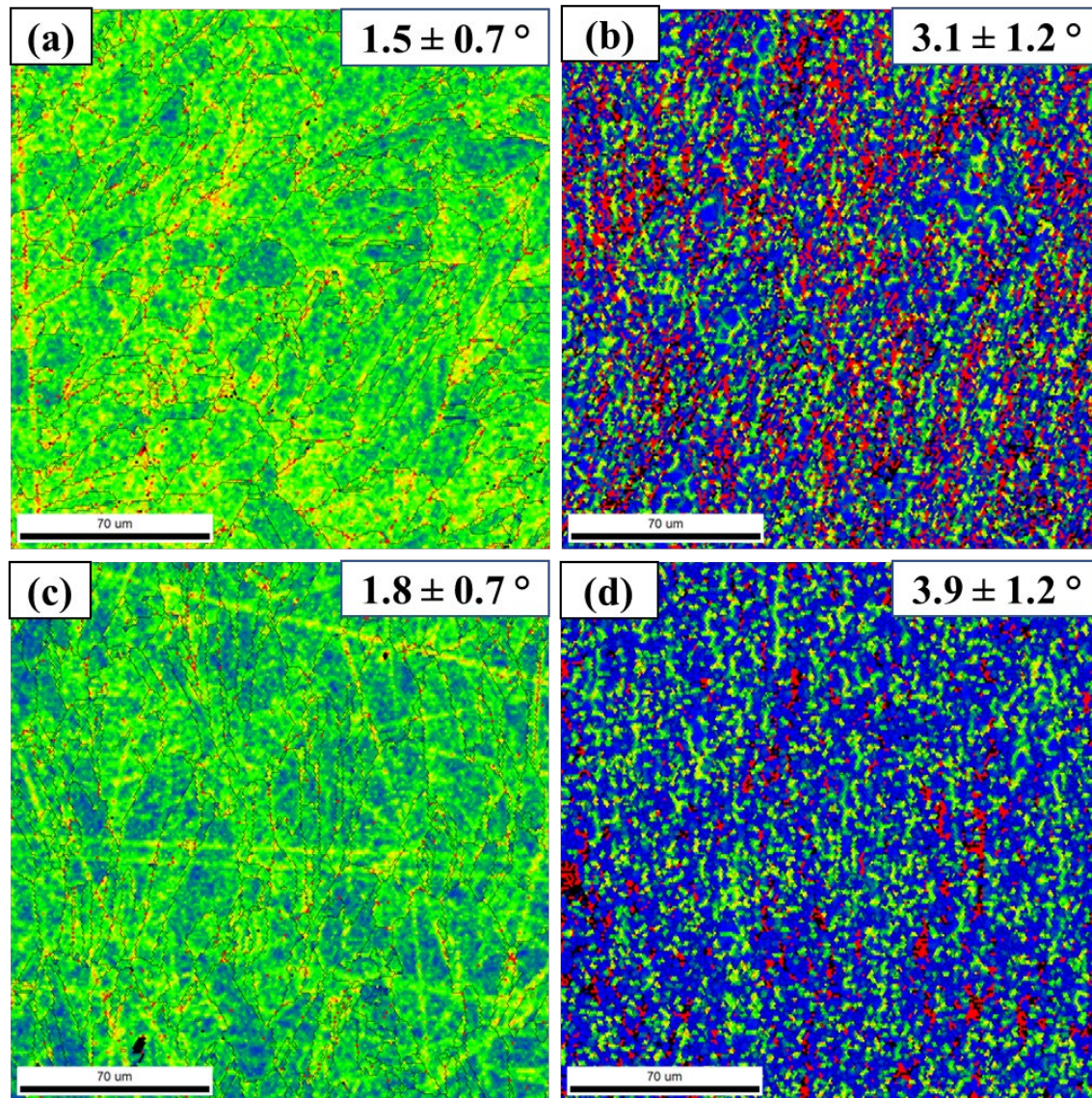


**Figure 34. KAM Maps Where (a) Undeformed 1.5 mm, (b) Deformed 1.5 mm, (c) Undeformed 4.0 mm, and (d) Represents Deformed 4.0 mm Thick Samples.**

The distribution of the KAM charts acquired from the KAM maps is shown in Figure 35. The chart for the 4.0 mm thick sample is quite flat, indicating an increase in the strain variation and, thus an increase in dislocation density within the grains. Before any strain was applied and even after the application of strain, the KAM values of 4.0 mm thick samples appeared higher relative to the 1.5 mm thick samples. In addition, the distribution of the KAM values was observed to be broader for the deformed samples compared to the undeformed samples. Prior to deformation, the samples' grain sizes were larger, as observed in the 1Q maps and KAM maps. However, the grains became more densely formed after deformation and reduced in size.

To understand this phenomenon, the grain size distribution charts in Figure 36 for both thicknesses were obtained. For undeformed samples, the average grain sizes were similar, meaning the thickness of the sample had an insignificant effect on the initial grain size distribution of the

samples. However, upon the deformation process, the 4.0 mm thick samples were observed to have smaller grains than the 1.5 mm thick sample. The average size grain (diameter) for the 1.5 mm was determined to be  $2.8 \pm 2.5 \mu\text{m}$  and  $2.1 \pm 1.3 \mu\text{m}$  for the deformed 4.0 mm thick sample. The scatter in data is due to the edge grains that were included in the data analysis.

The Image Quality Maps were also obtained for undeformed and deformed samples, as shown in Figure 37. The undeformed samples depict a large number of Low Angle Grain Boundaries (LAGBs) compared to the High Angle Grain Boundaries (HAGBs). The deformed 4.0 mm thick sample has a high number of LAGBs compared to the 1.5 mm thick samples, as shown in Figure 36 (b) and (d), respectively. For the deformed 1.5 mm thick samples, as supported by the IQ map, the distribution is as follows;  $1-15^\circ$ ,  $\sim 22.7\%$  of the total GBs, with  $1-5^\circ$  shown by red lines being  $\sim 4.1\%$  and  $5-15^\circ$  shown by green lines being approximately  $18.7\%$ , whereas for the 4.0 mm thick samples,  $1-15^\circ$ ,  $\sim 51.6\%$  of the total GBs, with  $1-5^\circ$  shown by red lines  $\sim 26.2\%$  and  $5-15^\circ$  shown by green lines being approximately  $25.4\%$ . The 1.5 mm thick sample has a higher number of HAGBs,  $\sim 77.2\%$ , while the 4.0 mm thick sample has  $\sim 48.4\%$ . The misorientation charts for the L-PBF samples are shown in Figure 38. The undeformed samples have low values of average misorientation angles compared to the deformed samples. The deformed 1.5 mm thick sample is observed to have undergone higher misorientation than the 4.0 mm thick sample, as shown in Figures 38 (b) and (d).

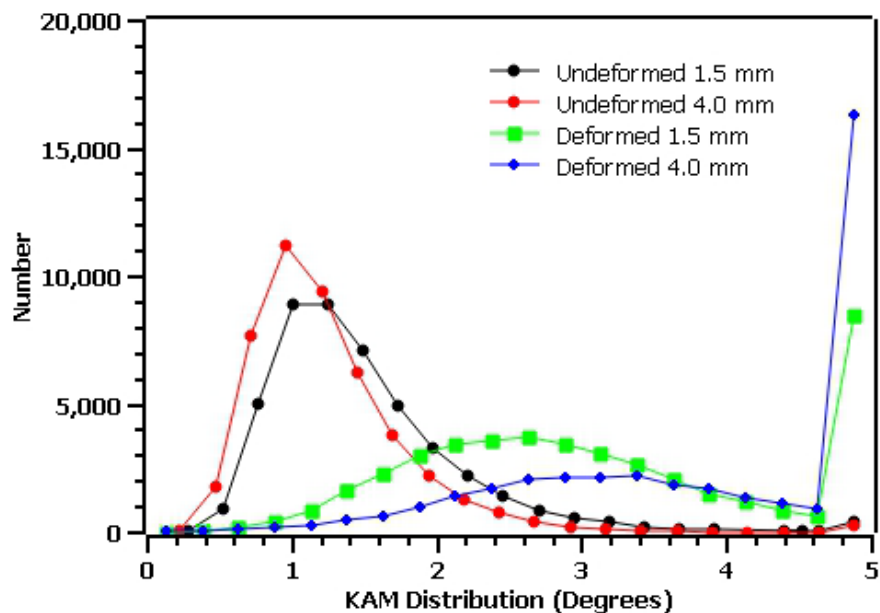


**Figure 35. Distribution of KAM Values of Undeformed and Deformed L-PBF 316L SS Samples.**

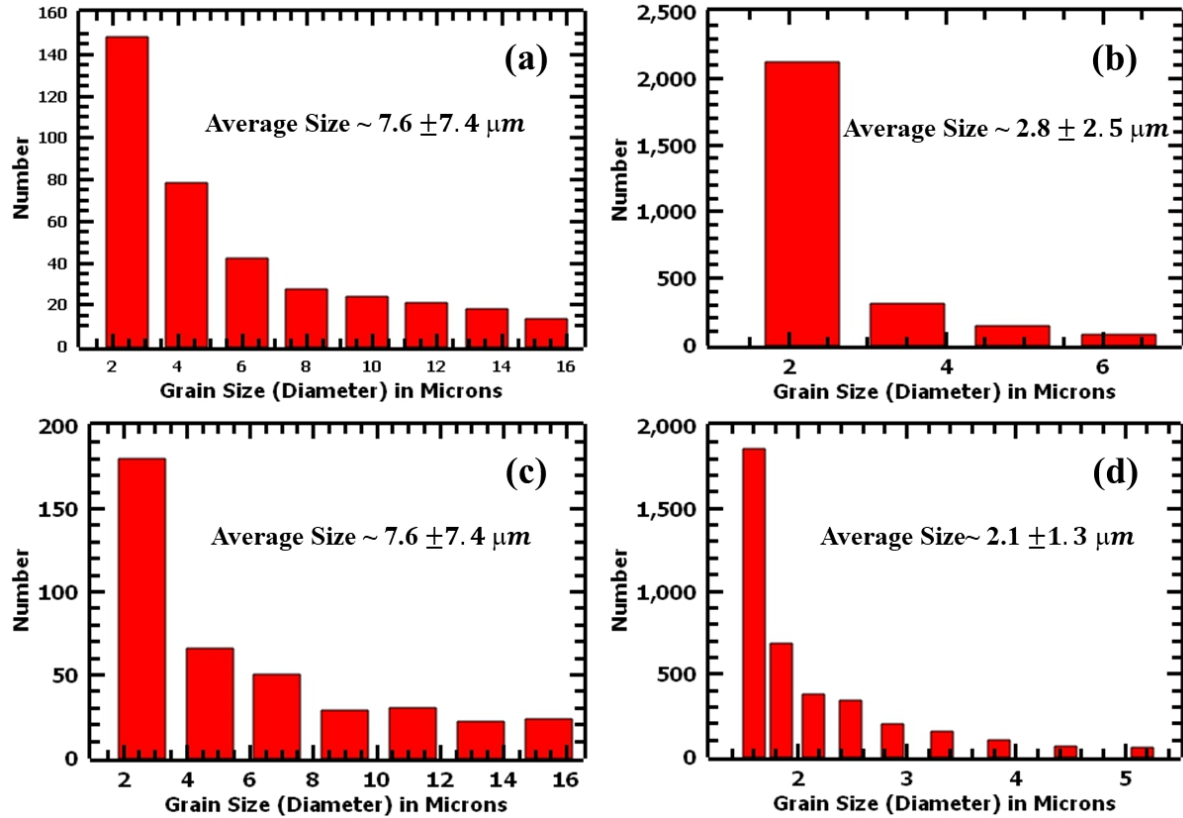


**Figure 36. Grain Size Distribution Charts for AM 316L SS Obtained From EBSD Data (a) Undeformed 1.5 mm, (b) Deformed 1.5 mm, (c) Undeformed 4.0 Mm, and (d) Deformed 4.0 mm Thick Samples.**

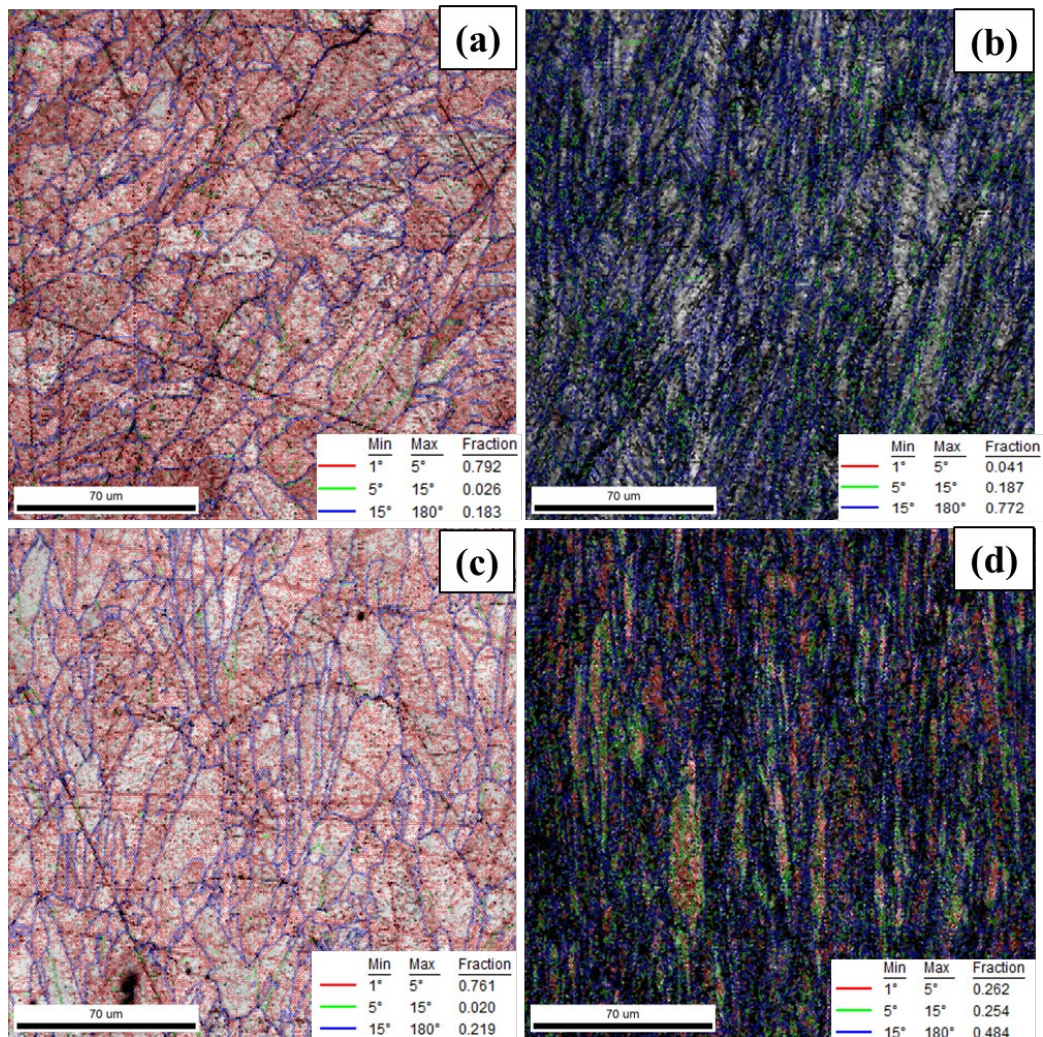


**Figure 37. IQ Maps a) Undeformed 1.5 mm, (b) Deformed 1.5 mm, (c) Undeformed 4.0 mm, and (d) Deformed 4.0 mm Thick Samples.**

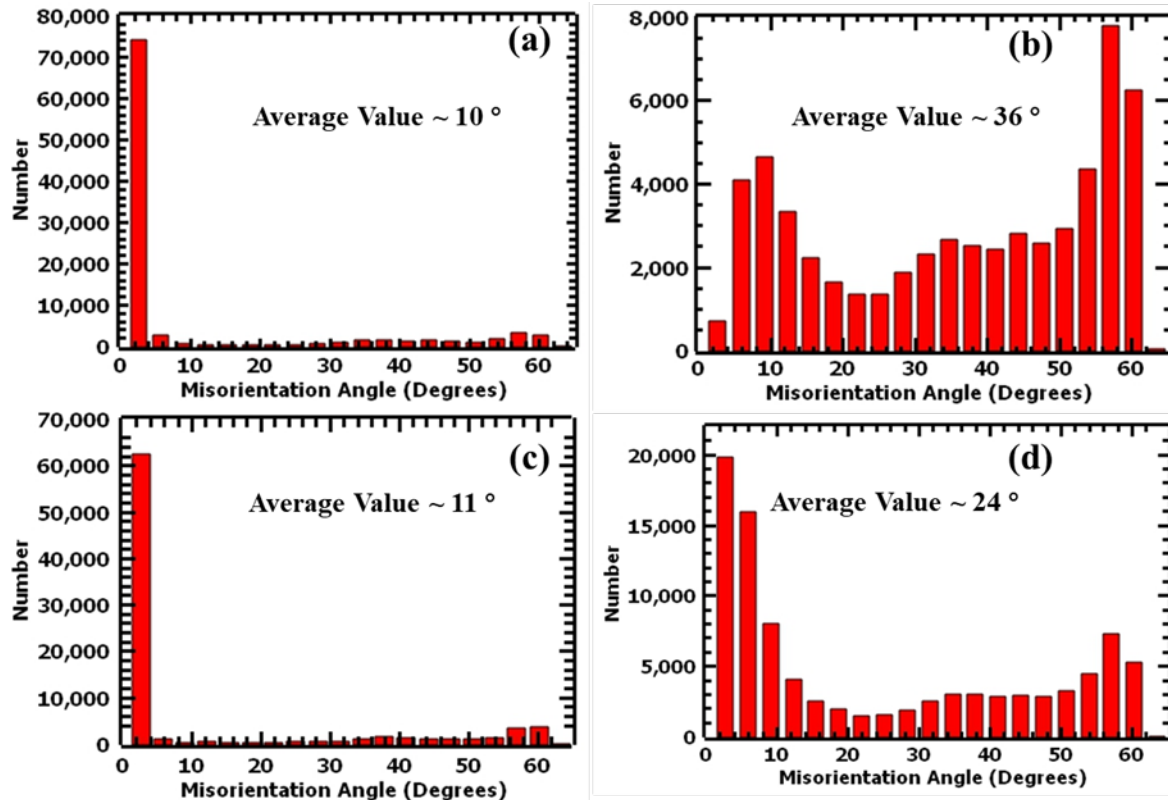


**Figure 38(a) Undeformed 1.5 mm, (b) Deformed 1.5 mm, (c) Undeformed 4.0 mm, and (d) Deformed 4.0 mm Thick Samples.**

#### **4.8 High Cycle Fatigue Test Properties Results and Characterization**

Table 4 summarizes the fatigue lives of as-built samples at different stress amplitudes for each thickness. The S-N curves in Figure 39 also demonstrate the effect of thickness on fatigue lives of L-PBF 316L stainless steel samples. The results show that the thinner material had longer fatigue lives compared to the thicker material at higher stress amplitudes. Both thicknesses incurred runouts at 20 % of the UTS. For the 1.5 mm thick samples, the fatigue strength was determined to be 55 MPa whereas for the 4.0 mm thick samples, the fatigue strength was determined to be 56 MPa. The fatigue lives were observed to increase slowly with decreasing stress amplitude. A significant difference between the 30 % and 20 % of the UTS for both thicknesses was observed. The results were observed to be quite inferior to the conventionally manufactured samples.

**Table 4: Experimental Test Results for Stress Controlled High Cycle Fatigue Tests of as-Built Samples**

| $\%S_{UTS}$ | Average Values<br>(1.5 mm) | Average Values<br>(4.0 mm) |
|-------------|----------------------------|----------------------------|
| 100         | 1                          | 1                          |
| 95          | 432                        | 94                         |
| 90          | 3,262                      | 404                        |
| 80          | 72,186                     | 25,370                     |
| 70          | 93,518                     | 83,145                     |
| 60          | 133,963                    | 97,099                     |
| 50          | 224,907                    | 160,216                    |
| 40          | 384,629                    | 337,965                    |
| 35          | 421,863                    | 467,534                    |
| 30          | 1,058,252                  | 1,501,503                  |
| 20          | 5,922,285                  | > 6,606,161                |

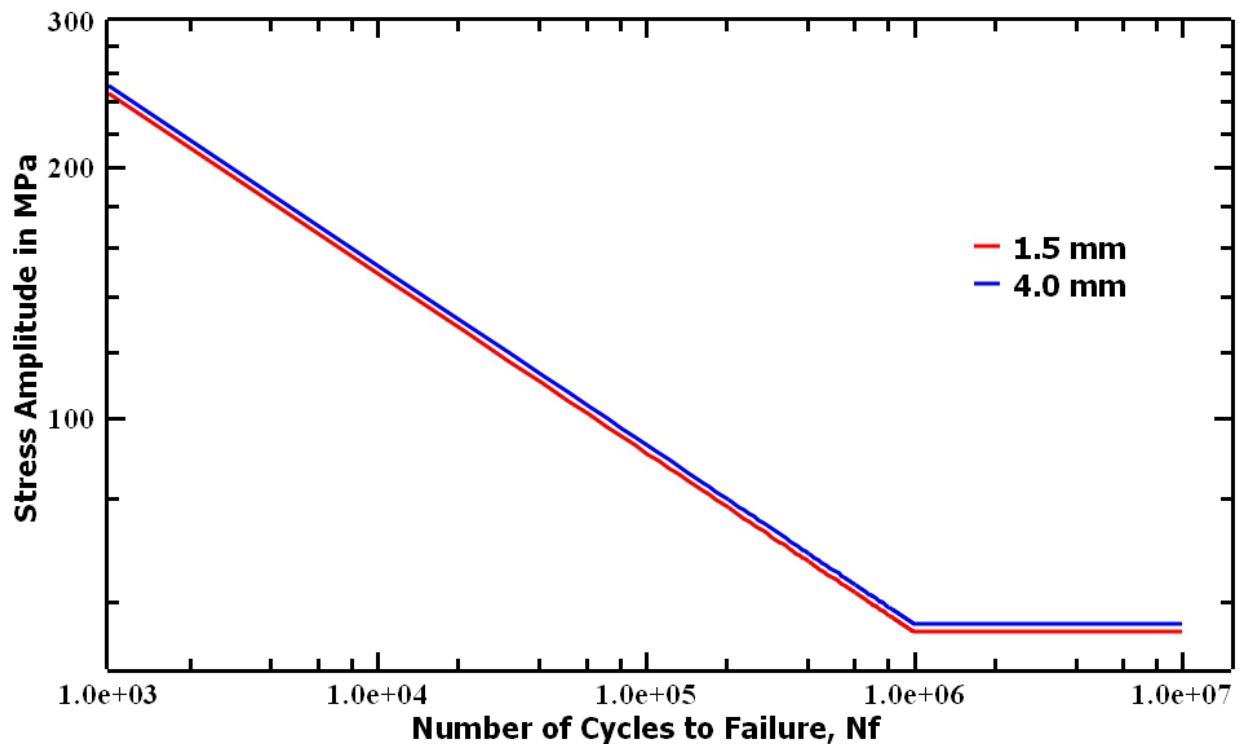


**Figure 39. Comparison of Stress Life Fatigue Data Between the 1.5 mm and 4.0 mm Thick As-Built L-PBF Samples.**

The fracture surfaces of fatigue tested samples are displayed in Figure 40. The displayed images show sample at high stress amplitudes (90 %), medium stress amplitudes (60 %), and low stress amplitudes (40 %). The analysis was done at these stress amplitudes to determine if there was any significant difference in the fractured surfaces at different amplitudes. The three stages of fatigue failure; crack initiation, crack propagation, and fracture, were observed in the samples tested. The

cracks were seen to originate from defects at the surface such as lack of fusion, pores, and voids, depicted in Figure 40. Porosity throughout the bulk materials was observed on both thicknesses. Higher magnifications of the samples show the unique microstructure of the fractured areas. Coalescence of micro-voids and the presence of fine dimples demonstrate the ductile fracture of the material. The micropores can grow into larger dimples that are evidence of localized plastic deformation around the pores. As shown in Figure 40 (a), (c), (e), and (f), striations were observed which are typical fatigue features depicting propagation of the cracks. Figure 40 (b) displays a large void next to the surface edge which then act as a crack initiation site. The presence of unmolten material shown in Figure 40 (e) tends to propagate cracks and thus premature failures. Microcracks and large cracks were also observed in Figure 40 (a), (c) and (f). The presence of fatigue striations is seen to propagate in various planes and with the presence of microcracks, they quickly merge and trigger failure.

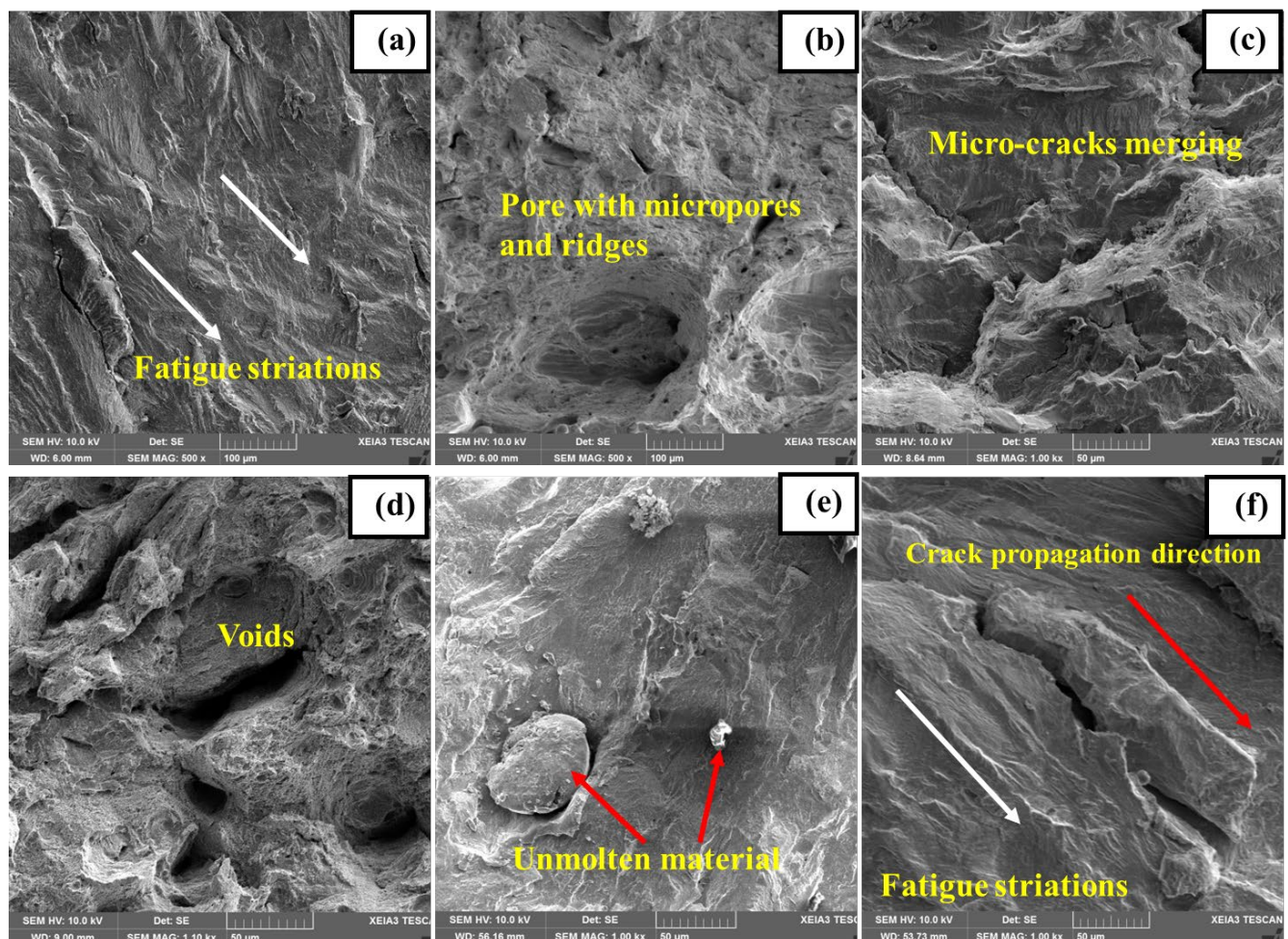


**Figure 40. Fracture Surfaces Of Fatigue Tested Samples: (a), (b), and (c) Representing 1.5 mm Samples, (d), (e), and (f) Representing 4.0 mm Samples, All Observed At 90 %, 60 %, and 40 % of the UTS, Respectively.**

## 5 CONCLUSIONS

### 5.1 Conclusions on Microstructures and Mechanical Behavior of the AA5052 Aluminum Alloys

MAF at high cycles and high cryogenic temperature resulted in crystallite size reduction to nanoscales for the AA5052 alloy as well as heavily deformed grains associated with high density of dislocations and second phase breakage to finer particles which are the crucial factors to increase the mechanical properties of the material. This can be due to the induced intense plastic deformation as well as the suppression of the dynamic recovery and recrystallization mechanisms. The Electron backscatter diffraction (EBSD) maps demonstrated a significant grain size refinement from 350  $\mu\text{m}$  for the base SHT sample to average grain sizes of 1.78 $\mu\text{m}$  for 2 cycles, 1.3  $\mu\text{m}$  for the 4 cycles and 0.94 $\mu\text{m}$  for the 6 cycles, respectively as a result of cryogenic plastic deformation. The inverse pole figure of the AA5052 CF 2 cycles, 4 cycles, and 6 cycles confirmed the presence of large volume of sub-grains. While the average grain size of the AA5052 reduces with number of forging cycles, the average misorientation angle increases with number of forging cycles.

An integrated enhancement in mechanical strength, ductility and toughness of the alloy was achieved as the result of cryogenic MAF process. The dynamic mechanical response of the CF samples was investigated at various high strain rates via stress-strain profile resulted from SHPB test. In this regard, the flow stress as well as the strain rate sensitivity of the CF samples increased. The high level of sensitivity in the CF alloys to strain rates shows the change in the plastic deformation mechanism to grain boundary interactions. Both the maximum flow stress and the true strain at failure of the CF samples are higher than that of the SHT. However, there is reduction in ductility of the CF samples with increase in number of forging cycles while the maximum flow stress increases. At 5500/s, the true strain at failure of SHT, 2cycles, 4cycles, and 6cycles are 2.04, 3.54, 2.91, and 3, respectively while their maximum flow stress at failure is 297.7 MPa, 398.4 MPa, 440.9 MPa, and 467.4 MPa. In addition to the dynamic strength, the refined grain size in the CF samples resulted in the absorption of the higher portion of the deformation energy indicating higher amount of toughness in the fabricated UFG material.

Microstructure characterization of the post-deformed samples via OM and SEM indicated fully transformed and deformed ASBs localized in the 2 cycles and 4 cycles CF sample due to localized shear deformation and thermal instability where the sample had relatively experienced a significant thermal softening at 5,500  $\text{s}^{-1}$  strain rate. The failure mode of the AA5052 CF is characterized by the formation of adiabatic shear band, cracks, and bifurcation of the shear bands due to the competition between the initial shear band's propagation direction and the direction of the maximum shear stress. However, much less significant deformed ASBs and considerable increase in crack initiation strength were observed by morphology and final configuration of the post-deformed UFG samples at 6 cycles.

### 5.2 Conclusions on Microstructures and Mechanical 316L SS Steel produced via L-PBF

This study investigated the influence of sample thickness on the mechanical and microstructural properties of L-PBF 316L SS. The samples exhibited an appreciable yield strength, ultimate tensile strength, elongation to failure, as well as hardness than those of wrought samples. Based on the results obtained, the following conclusions can be inferred:

- a. The characterization of the material revealed that strength and ductility are sensitive to the thickness of the material. The UTS, YS, and elongation to fracture increased as the thickness of the sample increased. Despite the thinner sample having lower strength, its properties were still higher and better than those of conventionally manufactured 316L. In addition, there was no trade-off between ductility and strength, which is an additional advantage of AM components as compared to wrought 316L samples.
- b. The excellent mechanical properties can be attributed to the unique microstructures formed due to rapid cooling and solidification. The dislocation networks, the presence of very fine sub-grain features, and the established grain boundaries provided the necessary resistance to the tensile loadings.
- c. The thickness of the AM 316L samples was observed to affect the grain size, orientation, and percentage of low and high-angle grain boundaries. Therefore, from these results, the optimum parameters can be selected for manufacturing strong and preferred components in industrial sectors.
- d. The KAM values were observed to increase with increased thickness as well as increased deformation. The KAM charts also confirmed the intragranular misorientations due to tensile deformations and was also attributed to the re-arrangement of dislocations.
- e. The fatigue strength of as built materials was observed to be significantly affected by the surface roughness of the materials and process induced defects. The defects were observed to be randomly distributed and to be the highest contributor of the lower fatigue performances. The defects acted as a source of stress concentrators that then nucleated and propagated the cracks. Post processing and stress relieving treatments can be conducted to alleviate the residual stresses.

## 6 LIST OF PUBLICATIONS

### ***Peer-Reviewed Journal Papers***

1. Chepkoech et al. (2022). Mechanical Properties and Microstructure of 316L Stainless Steel Fabricated Via Laser Powder Bed Fusion, Metals, (under Review)
2. Ajao et al (2022), Microstructure and mechanical Properties of Ultrafine Grained AA5052 Aluminum Alloy, Materials Science and Engineering (under preparation).

### ***Conference Presentations and Proceedings***

3. O. J. Ajao, G. M. Owolabi, H.A. Whitworth, O. Scott-Emuakpor. Dynamic Impact Response of Ultrafine Grained AA5052 Aluminum Alloy Processed Via Multiaxial Forging at Cryogenic Temperature. Engineering Technologies (ICIET-20), New York, December 2020.

## 7 GRADUATE/UNDERGRADUATE STUDENTS INVOLVEMENT

### *a. Graduate Student*

1. Oluwafemi Ajao (PhD, Dec. 2022): Microstructures and Mechanical Properties of **Cryo-**forged Ultrafine Grained AA5052 Aluminum Alloy, Department of Mechanical Engineering, Howard University.
2. Melody Chepkoech (PhD, May 2023): Quasi-Static and Fatigue Behavior of 316L SS Produced by L-PBF, Department of Mechanical Engineering, Howard University.

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85. Chepkoech et al. (2022). Mechanical Properties and Microstructure of 316L Stainless Steel Fabricated Via Laser Powder Bed Fusion, Metals, (under Review)
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88. Oluwafemi Ajao (PhD, Dec. 2022): Microstructures and Mechanical Properties of **Cryo-**forged Ultrafine Grained AA5052 Aluminum Alloy, Department of Mechanical Engineering, Howard University.
89. Melody Chepkoech (PhD, May 2023): Quasi-Static and Fatigue Behavior of 316L SS Produced by L-PBF, Department of Mechanical Engineering, Howard University.

## LIST OF ACRONYMS

|       |                                      |
|-------|--------------------------------------|
| MAF   | Multiaxial Forging                   |
| SHT   | Solution Heat Treat                  |
| UFG   | Ultrafine Grained                    |
| CF    | Cryogenic Forging                    |
| L-PBF | Laser Powder Bed Fusion              |
| EBS   | Electron Backscatter Diffraction     |
| LAGB  | Low Angle Grain Boundaries           |
| HAGB  | High Angle Grain Boundaries          |
| SEM   | Scanning Electron Microscope         |
| FCC   | Face Centered Cubic                  |
| SPD   | Severe Plastic Deformation           |
| ECAP  | Equal Channel Angular Pressing       |
| ECAR  | Equal Channel Angular Rolling        |
| HPT   | High Pressure Torsion                |
| CGP   | Constrained Groove Pressing          |
| ARB   | Accumulative Roll Bonding            |
| CM    | Conventional Manufacturing           |
| AM    | Additive Manufacturing               |
| SHPB  | Split Hopkinson Pressure Bar         |
| OM    | Optical Microscopy                   |
| OIM   | Orientation Image Microscopy         |
| KAM   | Kernel Averaged Misorientations      |
| GNB   | Geometrically Necessary Boundaries   |
| CDRX  | Continuous Dynamic Recrystallization |
| SFE   | Stacking Fault Energy                |
| DB    | Deformed Bands                       |
| TB    | Transformed Bands                    |
| UTS   | Ultimate Tensile Strength            |
| G     | Temperature Gradient                 |
| R     | Growth Rate                          |