MICROSTRUCTURAL CHARACTERIZATION OF NANOSTRUCTURED
ALUMINUM EXTRUSIONS

A Thesis

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MICROSTRUCTURAL CHARACTERIZATION OF NANOSTRUCTURED ALUMINUM EXTRUSIONS

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Nanostructured aluminum alloys are advantageous for their high strength properties and low density due to the presence of not only multiple material components, but also the presence of nanocrystalline grains that increase strength through grain boundary strengthening. Microstructural analysis is a crucial step in understanding alloy mechanical properties. The alloys observed in this research were analyzed through transmission electron microscope (TEM) observation and grain analysis to determine the average grain size of each nanocrystalline alloy and determine the distribution of grain sizes through each alloy. Average grain sizes for the alloys, found through number fraction analysis, ranged from 44nm to 624nm. Each alloy had a distribution of grain sizes, where the alloy contained high concentrations of grains close their respective average grain sizes with lower concentrations of grains further from the average size.
I would like to thank Dr. Troy Topping for the opportunity to work on this project. Troy’s initial presentation of the project to me and its objectives piqued my interest in this research project immediately and his knowledge and guidance through the course of the research process was incredibly helpful. In addition to this, Dr. Topping introduced me to many of the following people mentioned below who were a crucial part of this project and its completion. Troy made sure the research process went smoothly and helped me make sure I could complete this thesis on time in a thorough manner. I am extremely grateful to have the opportunity to work with Dr. Troy Topping.

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CHAPTER 1
INTRODUCTION

1.1 NANOCRYSTALLINE ALLOYING

Nanocrystalline alloying of materials is the processing of an alloy that yields a material in a nanocrystalline state, where grains are under 500nm in size [1]. Such alloys are desired as nanostructured alloys have higher strength values than alloys containing larger grains. This is due to the fact that nanocrystalline grain structures are more resistant to plastic deformation due to higher grain boundary area within the alloy structure.

Alloys produced through nanocrystalline processing can be advantageous as they can have higher strength values while maintaining the same weight as similar alloys with larger grain sizes. An alloy that has higher strength without added weight would be desirable for purposes such as incorporation into armor for military vehicles in order to protect the vehicle and its occupants without adding weight, or usage within the aerospace industry for uses such as structural and fuselage components, utilizing the high strength and low density properties of the alloys [2]. Aluminum is often processed to produce nanostructured alloys in order to obtain smaller grain sizes because aluminum is light in comparison to denser, heavier metals such as steel and titanium. Obtaining high strength values from lightweight aluminum can greatly benefit purposes where high strength materials are desired, but weight needs to be low [2].
1.2 THESIS OBJECTIVES

Though the mechanical properties of these nanostructured aluminum alloy extrusions are known, the microstructure of said extrusions has yet to be analyzed. This lack of information prevents the material properties of these alloys from being attributed to specific microstructural properties. Knowledge of the microstructure of these nanostructured alloys is extremely valuable as such knowledge can be applied to further development of nanostructured aluminums. Certain microstructural properties can yield specific mechanical characteristics such as elevated tensile strength or high ductility values.

The purpose of this research work is to examine a set of extruded nanocrystalline aluminum alloy samples to analyze their microstructure characteristics and grain size frequencies. This research is also to complement research in progress into nanostructured aluminum alloys by Troy Topping, Gaunt Murdock, and Dr. Enrique Lavernia. This research into the nanostructure of these alloys is to coincide with their research into the development of these nanostructured aluminum alloys.
CHAPTER 2
BACKGROUND

2.1 ALLOYED ALUMINUM
The materials to be in this research are all aluminum alloys. Aluminum alloys are desirable for research and experimentation as aluminum is a very lightweight metal that can be alloyed with numerous elements. Since aluminum can be alloyed with other elements, specific alloys can be designed to have specific mechanical properties such as high strength while maintaining the lightweight property of aluminum, a property that other metals such as steels lack. In this thesis, elements referred to as “X” cannot be disclosed at this time due to existing non-disclosure agreements with our private sector partners. Such alloys can be used for high stress, high impact, or load bearing applications, such as armor or structural features, without adding significant weight to a structure or machine such as an armored vehicle or aircraft [2].

2.1.1 ALUMINUM-COPPER-X ALLOYS
One type of aluminum alloy in this research is the Al-Cu-X alloy. This is an aluminum alloy that utilizes copper and an undisclosed element (element X) within its microstructure in order to create a lightweight alloy with a high specific modulus and strength along with very low density [3]. Allying the aluminum with proportions of copper and element X lead to the formation of precipitate particles within the alloy’s
microstructure. These particles impede dislocation motion in the material, thus impeding permanent deformation and requiring larger stress levels to strain the material [3]. Al-Cu-X alloys have been studied and developed as materials to be utilized in the aerospace industry for purposes such as structural components [3].

2.1.2 ALUMINUM- MAGNESIUM -X ALLOYS

Al-Mg-X alloys will also be observed in this research. Al-Mg-X alloys are also valuable for purposes such as aircraft components such as fuselage sections and bulkheads due to their alloy properties. Like Al-Cu-X alloys, Al-Mg-X alloys have relatively high strength and lower density. Al-Mg-X alloys also have high toughness and fatigue resistance, making these materials suitable and desirable for uses in areas such as aircraft design where strength, toughness, and material density are crucial in design [4].

2.1.3 NANOPARTICULATE REINFORCEMENT

Particulate reinforcement is another method of altering the properties of an alloy. Nanoparticulate reinforcement particles within an alloy produce metal matrix composites (MMCs). Particulates such as alumina (Al₂O₃), boron carbide (B₄C), and diamatane are utilized with metal matrix materials to yield MMCs with enhanced properties. These composites are advantageous as particulate reinforcement of MMCs can be a relatively low cost process that yields isotropic material properties [5]. The presence of nanoparticulates within MMCs increases the strength and elastic moduli properties of the
matrix alloys. Furthermore, these nanoparticulate reinforcements can improve the properties of alloys at elevated temperatures [5]

2.1.3.1 ALUMINA PARTICULATES

Aluminum oxide (Al$_2$O$_3$), also known as alumina, is a chemical compound often used in nanoparticulate reinforcement of metal matrix composites. Alumina is incorporated into MMCs in order to enhance properties such as strength properties since alumina has very high hardness. Also, alumina is used in MMCs to enable properties, such as strength and elastic modulus, to be maintained under higher temperatures [6].

2.1.3.2 BORON CARBIDE PARTICULATES

Boron carbide (B$_4$C) is a ceramic material utilized in nanoparticulate reinforcement because of properties related to the particulate itself. As a particulate, B$_4$C lends itself to metal matrices to increase the MMC strength properties and wear resistance while maintaining a low density in the MMC. Furthermore, as a ceramic, B$_4$C aides in improving the heat tolerance of the MMC, alloying mechanical properties to remain consistent at elevated temperatures [7].

2.1.3.3 ALUMINUM-DIAMATANE ALLOYS

Aluminum-diamatane alloys incorporate diamatane within the nanostructure of the aluminum alloy. Diamatane is a compound derived from crude petroleum consisting of hydrocarbons arranged in a diamondoid arrangement [8]. Within this arrangement,
fourteen carbon atoms form a diamondoid cage, a formation seen in figure 2.1, with hydrogen atoms branching from the carbon atoms.

![Diamondoid Atomic Structure](image)

**Figure 2.1 Diamondoid Atomic Structure [9].**

Adding diamantine to an aluminum alloy will increase the strength properties of MMCs while also enhancing the thermal stability of the composite’s grains [10].

2.2 NANOCRYSTALLINE ALLOYS AND MECHANICAL ADVANTAGES

Nanocrystalline alloys are desirable due to their enhanced mechanical properties in relation to coarse grained alloys. Alloys with nanocrystalline and ultrafine grain size can be advantageous as the strength properties of these alloys can be enhanced by altering the size of the grains in the alloys and the concentrations of specific grain sizes. Furthermore, nanocrystalline alloys will have greater strength values compared to alloys that are coarse grained. Nanocrystalline alloys have higher strength due to grain boundary strengthening, where strength values, such as yield strength and tensile strength, of an alloy are increased by reducing the size of the grains of the alloy’s nanostructure [11]. Reducing the size of the grains in the microstructure of the alloy increases the grain boundary area
between neighboring grains through the nanostructure. This increase in boundary area in turn increases the strength of the material by impeding dislocation motion, the mechanism of plastic deformation in crystalline materials. Impeding dislocation motion prevents planes of atoms from slipping over one another during the deformation process, yielding a material that requires more stress to deform [11].

Utilizing nanostructure alloying techniques is especially advantageous with low density alloys like aluminum. Aluminum alloys are low in density, so strengthening these alloys through nanostructure processing techniques can give parts made from these alloys very desirable property values, such as yield strength, in relation to part mass. Interest in utilizing grain boundary strengthening has led to the development of alloy processing methods such as cryomilling that reduce the size of grains within alloy microstructures.

2.3 SEVERE PLASTIC DEFORMATION

Severe plastic deformation (SPD) is a type of alloy processing used to develop nanocrystalline materials. SPD processing involves applying large hydrostatic pressures and shear deformations to materials in order to refine the grain sizes within the alloy. Processes of SPD work an alloy, increasing the number of dislocations and defects in an alloy while refining the average grain size of the alloy [12].
2.3.1 HIGH PRESSURE TORSION

One SPD method used in nanocrystalline alloying is high pressure torsion (HPT). In HPT, a disk of an alloy to be worked is placed in a support where a plunger applies a large compressive stress. While applying this stress, a torsional force is also applied by rotating either the plunger of the support. This rotation applies a shear stress in addition to the compressive stress on the sample [13].

2.3.2 EQUAL CHANNEL ANGULAR PRESSING

Equal channel angular pressing (ECAP) is another SPD processing method. ECAP is an extrusion type of processing, as it involves extruding an alloy billet through an angled metal channel. The channel that the billet passes through can be set to a variety of angles, from a 90° bend to an angle near 180° [14]. Forcing the billet through the channel puts very high strains on the alloy, deforming it as it passes through and increasing the dislocation and defect density in the alloy. The billets can also be put through ECAP at different orientations in order to control the deformation of the material and control the changes to the alloy’s nanocrystalline structure [14].

2.4 CRYOMILLING PROCESS

Another method of reducing the size of grains within a microstructure that is utilized is the cryomilling process. Cryomilling is a process that utilizes ball milling to compress powdered alloy particles. Through the compression, the milling balls reduce the size of the grains in the powder [1]. In addition to ball milling the powdered alloy, cryomilling
involves milling within cryogenic conditions. More specifically, this milling occurs in a cryogenic medium such as liquid nitrogen. Cryogenic conditions are utilized to optimize the milled powders as the low temperature conditions yield finer grain sizes through milling than alternate milling conditions. This is because the cryogenic conditions reduce the propensity for recovery and recrystallization within the alloy powder, allowing grains to be reduced to finer sizes much faster [2].

2.5 CONSOLIDATION PROCESSING

After cryomilling, the nanocrystalline powder produced must be consolidated into ingots through consolidation processes. These processes are required to consolidate the cryomilled powders can vary in technique based on desired microstructure properties. Consolidation processes such as spark plasma sintering (SPS) and hot isostatic pressing (HIP) are used to develop materials with certain structural and mechanical properties.

2.5.1 SPARK PLASMA SINTERING

Spark plasma sintering, or SPS, processing is a consolidation process that utilizes heat through electric currents to consolidate cryomilled powders. The SPS process involves containing cryomilled powders in a graphite die and applying a DC current through the die and powder in a pulsing manner. The pulsing current leads the powders to heat up and cool at rapid rates. The SPS process is a very fast sintering and densification process compared to other processes like hot isostatic pressing. The high speed of the SPS process allows the consolidation of powders with little risk of recrystallization and
coarsening of nanocrystalline powders, a risk of other consolidation processes that rely on heat application [15].

2.5.2 HOT ISOSTATIC PRESSING
Hot isostatic pressing, or HIP, is a consolidation process that relies on heat and pressure to consolidate powdered metals. In HIP processing, powders are contained in a vessel where heat and pressure are applied to the powder. This pressure is applied through high pressure inert gasses, thus rendering the process isostatic. This process aids in reducing the porosity and number of defects of the final consolidated metal [16].

2.6 TEM OBSERVATION
Electron microscopy is necessary in order to observe the microstructure of cryomilled aluminum samples due to the ultrafine grain nature of the microstructure. Observing grains and features of this size on a typical optical microscope would be very difficult as optical microscopes are often limited to how high of a magnification they can utilize for which to observe a sample. An electron microscope is ideal, as it is completely capable of magnifying a nanocrystalline microstructure enough to observe and distinguish ultrafine grains.

For this research, a transmission electron microscope (TEM) will be used for nanocrystalline observation. TEM will be utilized as transmission electron microscopy involves passing electrons through a sample and a series of lenses as seen in figure 2.2.
An electron gun fires an electron beam through a condenser lens, where the beam is focused and hits the sample some of these electrons will be able to pass through the material, while some will not be able to. The electrons that do pass through the sample then pass through objective and projector lenses and are projected onto a viewing screen for observation. The distribution of electrons that do pass through the material and the electrons that do not will generate an image reflecting the microstructure of the sample [18].
Other types of electron microscopes are available for usage for ultrafine observation. Scanning electron microscopes are another often-used type of electron microscope. An SEM is similar to a TEM as they fire electrons at a sample being observed, but an SEM generates an image based on the electrons that are do not pass through a sample and reflect off instead, as depicted in figure 2.3.

![Figure 2.3 Scanning Electron Microscope Schematic [17].](image)

Images produced through SEM, while valuable, are not fit for these tests, as scanning electron microscopy is more suited for observing morphological features, while transmission electron microscopy is more suited for microstructure observation [19].
CHAPTER 3
EXPERIMENTAL PROCEDURE

3.1 EXTRUSION CROSS-SECTION CUTTING
The extruded aluminum alloy pieces to be examined through TEM need to be prepared in a way that isolates a section of the alloy structure that is free of dislocations from sample preparation and electron transparent (approximately 100-150nm thick) [19]. Electron transparency is necessary in order for electrons from the TEM to pass through the sample and reveal the microstructure of the alloys tested. Along with electron transparency, the absence of dislocations in sample preparation is crucial in order to determine the structure of the alloy in a state without any work applied to the nanocrystalline structure. These structures represent the state of the alloys upon extrusion.

In order to obtain TEM specimens that are free of dislocations and electron transparent, cross sections must first be obtained from extruded alloy samples [20]. The initial extruded alloy samples were cylindrical pieces with dimensions between 5.5-6mm in diameter, and 5.5-8mm in length. These samples are sliced in a manner parallel to the length of the sample, meaning that the slices were oriented in a manner parallel to the extrusion. This cross-section orientation is selected in order to cut a sample slice where one can observe the changes in the microstructure of the alloy due to the extrusion.
process. To obtain these slices, each alloy sample was loaded into an ISOMET 4000 Linear Precision saw, as seen in figure 3.1.

![ISOMET Linear Precision Saw](image)

**Figure 3.1 ISOMET Linear Precision Saw.**

The samples were sliced with a diamond blade into cross-section slices that were approximately 1mm thick. The desired thickness of each slice was set using a micrometer controlled cross-feed that extended the sample into the path of the saw to cut off a slice of the specified thickness as seen in figure 3.2.
Figure 3.2 Extrusion Cross-Section Cutting.

This saw utilized a diamond blade and a low blade feed rate in order to cut the sample from the extrusion without exerting excessive force that could put work into the sample that could yield dislocations.

3.2 HAND SANDING

Once the extrusion cross-sections are cut into 1mm thick slices, the samples need to be reduced to a thickness of approximately 100 microns (0.1mm). This is necessary in order to make the sample thin enough to dimple and ion mill efficiently [20]. The hand sanding process was performed using a South Bay Technologies Model 150 Hand Lapping tool, seen in figure 3.3.
To sand down the alloy slices, the slices were first attached to the stage of the lapping fixture. This is done by removing the stage of the fixture and placing it on a hot plate at 300°F. This makes the stage hot enough to melt a thermal paste which was applied onto the stage face. With the paste in place, an alloy slice was placed onto the paste on the stage, which is then removed from the hot plate to cool down. Upon cooling, the thermal paste solidifies and holds the slice to the stage. The stage was reapplied to the base of the fixture as seen in figure 3.4.
Figure 3.4 Sample Slice Mounting for Hand Sanding.

Once the mounting process is complete, the sample sanding can begin. The first wet sanding process involves the 600 grit sandpaper. This process was used to remove a majority of material from the slices. After wetting the sandpaper with deionized water, the lapping fixture was set to remove material from the slice by extending the stage outward in 100 increments and sanding away material at each increment. This was performed until the slice was 160 microns thick. Once this slice is at this thickness, wet sanding with the 800 grit sandpaper was performed to remove more material from the sample, as well as produce a better finish on the sample. This second stage in sanding was performed to remove 40 microns of thickness from the samples, leaving them 120 microns thick. Finally, wet sanding was performed with the 1200 grit sandpaper to remove 20 microns of thickness to bring the sample to a thickness of 100 microns and improve the finish of the sample.
Once the sanding process is complete, the thinned samples were removed from the lapping fixture by removing the fixture stage and soaking it in acetone, dissolving the thermal paste and freeing the thinned samples.

3.3 DISC CUTTING

In addition to the alloys samples being required to be thinned for transmission electron microscopy, the samples are required to be in the form of 3mm discs in order to be mounted for dimpling, ion milling, TEM observation [14]. To obtain these discs from the thinned samples, a Gatan 601 Ultrasonic Disc Cutter, as seen in figure 3.5, was utilized.

![Figure 3.5 Gatan Model 601 Ultrasonic Disc Cutter.](image)

Obtaining the 3mm disc from a thinned sample first involves attaching the sample to the disc cutter’s cutting plate. This involves first heating the cutting plate on a hot plate to approximately 300°F. At this point, a thermal paste was applied to a portion of the plate
and the thinned sample is placed onto the paste. The base was then removed and allowed to cool, solidifying the thermal paste and holding the sample as seen in figure 3.6.

![Figure 3.6 Sample Mounting for Disc Cutting.](image)

The cutting plate with the sample attached was then moved to the stage of the disc cutter. The cutting tip of the disc cutter was then moved down until it contacts the sample. At this point, the disc cutter was switched on and the cutting tip cuts a disc from the sample.
Once the disc was cut, the cutting plate was removed and soaked in acetone to dissolve the thermal paste and free the disc and the remaining portion of the thinned sample.

3.4 DIMPLING

In order to prepare the disc cut samples for observation through transmission electron microscopy, ion milling is performed on the sample. However, before ion milling is performed, the disc samples must be dimpled. The dimpling process creates a dimple in one or both side of a sample, thinning the sample in a specific area prior to ion milling. By removing material from the sample in the form a dimple, the thickness at the center of the dimple is significantly smaller. This allows ion milling through the sample to be accomplished faster by leaving less material to mill through. This also allows the sample to be milled through at a specific point, the lowest point of the dimple where the sample is thinnest, while leaving thicker sample edges for handling purposes [20]. To prepare for ion milling, these samples are dimpled to a point where the thickness at the base of the dimple is around 10 microns, as seen in figure 3.7.

![Figure 3.7 Cross-Section of Dimpled TEM Sample.](image)

To perform the dimpling process on the disc cut samples, a South Bay Technologies d500i Dimpler was utilized. This dimpler holds each sample to be dimpled on a
horizontally rotating stage while a dimpling wheel is rotating vertically against the sample, removing material from the sample in the form of a circular dimple.

To prepare the sample for dimpling, the sample was attached to a sapphire flat. The sample was secured to the flat via thermal paste. The sapphire flat is initially set on a specimen mounting jig on a hot plate where it was heated to about 300°F. Once heated, a thermal paste was melted to the center of the flat and the sample was applied to the paste while being aligned with concentric alignment rings on the jig that are visible through the flat. Once aligned, the sapphire flat was removed from the heating stage and allowed to cool down, hardening the thermal paste and gluing the sample to the flat. The sapphire flat was then set within the magnetic platen assembly, seen in figure 3.8.

Figure 3.8 Magnetic Platen Assembly with Sample Mounted.
The platen was then set on the shaft that rotates the sample and platen assembly. At this step, the first of two dimpling wheels, the 3i tool wheel, is set up on the motor at the end of the dimpling arm, as seen in figure 3.9.

![Figure 3.9 3i Dimpling Wheel.](image)

This wheel was specifically for removing material to form the dimple in the sample. Once the platen and tool are set, the dimpler arm is brought down to bring the tool to the surface of the sample to establish a reference point on the sample for the dimpler. Following the establishment of the reference point, the depth the tool was to dimple is set. The depth the tool is set to dimple to is dependent on the solution that is set within the platen. A solution of metadi fluid and diamond paste was applied to the platen area on top of the sample in order to allow the 3i tool to grind away material during dimpling. A series of these solutions were applied during the dimpling process, decreasing the micron
grade of the diamond paste through the dimpling process in order to control the removal of the material and produce a better surface finish. The first solution used was a mixture of metadi fluid and 1 micron diamond paste. This paste was used to remove material quickly from the dimple and decrease sample thickness to 25 microns. Once the solution is applied, the dimpler was started, rotating the platen and tool while lowering the tool to the sample. Once the tool contacts the sample, the dimpling tool rotates within the diamond paste-metadi solution mixture sitting on the surface of the sample, as seen in figure 3.10.

![Figure 3.10 Start of Dimpling Process with Dimpling Wheel and Platen Rotating.](image)

The coating of the tool in the solution creates an abrasive layer between the tool and sample, promoting the dimple cutting by the tool. The tool dimples the sample until the dimpling tool has dimpled to the depth set before the dimpler was started.
Once this first dimpling process is completed, the dimpling solution was rinsed from the platen to prepare for the next dimpling solution. The next dimpling solution uses a 0.5 micron diamond paste in order to remove material at a slower rate and improve the surface finish of the dimple. This stage of dimpling removes 10 microns of material. For this stage, the dimpler tool is set against the sample to set the reference point for the tool again, setting the dimpling depth to 10 microns. After setting the depth, the dimpling arm was raised in order to apply the 0.5 micron paste solution. The dimpler was then started and dimples the sample until 10 microns are removed from the sample.

Following this dimpling process, the 3i tool was removed from the dimpler and the platen is stage. The 4i tool was then applied to the dimpling arm to remove the final 5 microns of material from the dimple and polish the dimple. Along with this tool, a 0.25 micron diamond paste solution was used to remove the final 5 microns from the sample side at a controlled rate. After setting the dimpling depth and applying the diamond solution, the dimpler was switched on and the final 5 microns are removed from the sample. After the final dimpling stage, the sample had a significant dimple, seen in figure 3.11, where ion milling can occur and where TEM observations were performed.
After dimpling is complete, the sapphire flat was removed from the platen and set back on the mounting jig on the hot plate. The heat from the jig softened the thermal paste holding the sample to the flat and allow it to be removed. Any thermal paste remaining on the sample was removed with acetone. The sample was then remounted upside down in order to dimple the bottom side of the sample. This forms a thinned dimple area in the sample that is approximately 10 microns thick, an area that could be ion milled easily.

3.5 ION MILLING

Once dimpling is complete, the disc samples must be ion milled in order to thin a portion of the sample to where it is ion transparent. Ion transparency is necessary in order for electrons to pass through the sample and be detected by the TEM. Ion milling is the ideal process to produce an observable metallographic specimen because ion milling will
gently remove material from the sample dimples without causing dislocations within the samples. This yields samples that are thin enough to be observed by transmission electron microscopy and free of deformation within the microstructure [21].

The ion mill used for the milling process for this research is a Fischione Model 1010. To set up a sample to be ion milled, the disc sample was first be placed in a stage gear. This gear holds and rotates the samples as ion beams were applied to remove material. A clip holds the sample in the center of the gear with the sample dimples exposed. This gear stage was then set in the ion mill to begin ion milling.

The ion mill was initially set up to perforate the samples by applying ion beams to both sides of the sample based on recommended settings from Fischione. The initial ion milling settings included applying both beams with a current of and voltage of 5kV while the stage was set at an angle to the beams of 25°. Once the stage was set, ion milling was performed on each sample until perforation, where the sample is completely milled through as seen in figure 3.12.
Figure 3.12 TEM Sample Ion Milled to Perforation.

Following the perforation of the sample, two more milling cycles were applied to the sample. The stage following the perforation of the sample was milling at a voltage of 2kV with the stage set to an angle of 4° to the ion beams. The final stage of the milling process utilizes beams set to a voltage of 0.5kV and the specimen stage set to an angle of 4° to the ion beams. These last two milling cycles are crucial in order to thin the sample around any perforations. While the first milling cycle will thin the sample dramatically, the low angle milling helps thin down a greater portion of the area around the perforations in the sample, yielding more areas that can be observed through TEM [21].
3.6 TEM OBSERVATION

Once the alloy samples have been ion milled, they are ready for observation through transmission electron microscopy. The TEM used for these observations is a Philips CM120. These samples are set up in the TEM in a way that the microscope is focused on the areas of the samples that had been perforated through ion milling because these are the electron transparent areas of the samples. Any other areas on the samples are too thick for electrons to pass through and be detected by the microscope. The thin areas around the perforations of the samples are have been thinned enough so electrons can pass through and be detected by the TEM, generating images of the microstructures of the samples. These samples are observed in magnifications varying from 20000x to 60000x depending on the number of grains present in each scan of the samples. Each scan would yield at least 10 significant grains to measure. For each sample, a minimum of 20 scans are performed in order to record the size of at least 200 grains per sample.

3.6.1 GRAIN SIZE DETERMINATION

Once collected, the size of the grains in the TEM scans were measured. This was performed through use of ImageJ, an open source image-processing program that is intended for scientific image analysis. To determine the size of the grains within a scan, ImageJ opens up the scan’s image within its program. Once open, ImageJ is set to measure in terms of the scale indicated by the scan. This is accomplished by adjusting ImageJ’s scale to that of the TEM scan. Once the scale is set, grain size is measured by using ImageJ’s measuring tool to measure each grain from one side to the opposite side.
These measurements can be performed in multiple orientations for the grains, but for this study, the grains were measured along the longitudinal axis as this axis indicated the direction of extrusion for the samples. This axis would give an indication of how the processing of the alloys affected their microstructure properties and in turn relate the grain size to the material properties of the alloys.

3.6.2 GRAIN SIZE DISTRIBUTION OBSERVATION

With ImageJ set to measure grains within each scan, grain size distribution analysis can begin. In order to perform this analysis, a large number of grains in each alloy are measured in order to collect enough grain size data to determine the grain size distribution in each alloy. ImageJ allows large quantities of grains to be measured and allows those measurements to be transferred to other programs for analysis. Once a large quantity of measurements are collected (typically 200 or more), the measurements are transferred into a workbook on Microsoft Excel. Excel is utilized to create a visual of each alloys grain size distribution through generation of histograms. Histograms are used as the frequency of specific grain sizes (or grain size ranges) can be visually represented for valuable and convenient analysis.
CHAPTER 4
RESULTS AND DISCUSSION

4.1 OBSERVED ALLOY COMPOSITIONS

For this research, seven different nanocrystalline/ultrafine grain alloys supplied by Boeing are observed through Transmission Electron Microscopy and analyzed in terms of grain size distribution through ImageJ and histogram analysis. The samples are alloyed with multiple elements and nanoparticulate reinforcements. The components within each sample are identified in Table 4.1.

Table 4.1 TEM Sample Alloy Compositions.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Components</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-1</td>
<td>Al with Nanoparticulate Reinforcement</td>
</tr>
<tr>
<td>B-1</td>
<td>Al-Mg with Nanoparticulate Reinforcement</td>
</tr>
<tr>
<td>B-2</td>
<td>Al-Mg with Nanoparticulate Reinforcement</td>
</tr>
<tr>
<td>C-1</td>
<td>Al-Cu-X</td>
</tr>
<tr>
<td>D-1</td>
<td>Al-Cu-X</td>
</tr>
<tr>
<td>E-1</td>
<td>Al-Mg-X</td>
</tr>
<tr>
<td>F-1</td>
<td>Al-Mg-X</td>
</tr>
</tbody>
</table>

This sample refers to the different compositions of the sample by letters “A” through “F”, while referring to their processing methods as “1” and “2”.
4.2 GRAIN SIZE DISTRIBUTION ANALYSIS

Each sample in this research went through multiple TEM scans in order to observe a large portion of each sample’s microstructure. This also allowed a large quantity of grains to be observed and measured in each sample through use of ImageJ. For each sample, 500 grains were observed in order to not only measure a large number of grains in each alloy, but also to see any trends in grain size frequency for specific measurements. The measurement of the longitudinal dimension of each grain is determined and recorded through ImageJ. These measurements are transferred into Microsoft Excel where the frequency of specific grain size intervals are compared through histograms. These representations of grain size distributions are then compared to the material properties of the alloys observed to find correlations between the size distribution and alloy behaviors.

4.2.1 SAMPLE A-1

Alloy sample A-1 was observed through TEM at a magnification of 33000X, as seen in the sample scan of the alloy in figure 4.1.
Figure 4.1 Sample TEM Scan of Sample A-1 (33000X magnification).

This alloy contained oblong grains. Along with these oblong grains, the alloy contained numerous regions that appeared as dark or textured features in the nanostructure of the alloy. These regions could be features such as additional alloy phases or precipitates particles, though the identity of these regions is inconclusive as the scope of this study does not extend into this area of research.

This alloy had some of the largest grains of all of the observed alloys in this research, with an average grain measuring 377nm in the direction of extrusion (longitudinal directions). The distribution of grains of specific sizes is visually and numerically depicted in figure 4.2 and table 4.2.
Figure 4.2 Grain Size Frequency of Sample A-1.

Table 4.2 Grain Size Distribution of Sample A-1.

<table>
<thead>
<tr>
<th>Grain Size (nm)</th>
<th>% Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-100</td>
<td>0.4</td>
</tr>
<tr>
<td>101-200</td>
<td>7</td>
</tr>
<tr>
<td>201-300</td>
<td>23</td>
</tr>
<tr>
<td>301-400</td>
<td>31.2</td>
</tr>
<tr>
<td>401-500</td>
<td>19.2</td>
</tr>
<tr>
<td>501-600</td>
<td>12.6</td>
</tr>
<tr>
<td>601-700</td>
<td>4.2</td>
</tr>
<tr>
<td>701-800</td>
<td>2.2</td>
</tr>
<tr>
<td>&gt;800</td>
<td>0.2</td>
</tr>
</tbody>
</table>
These visual representations revealed that the greatest size frequency of grains (31.2%) were between 301nm and 400nm in size in the longitudinal direction. Grains within 100nm of that size range (201nm-300nm, 401nm-500nm) had a frequency of 42.2%.

4.2.2 SAMPLE B-1

Alloy sample B-1 was observed at a magnification of 60000X, as seen in the sample scan of the alloy in figure 4.3

![Figure 4.3 Sample TEM Scan of Sample B-1 (60000X magnification).](image)

This alloy also contained oblong grains. And like the A-1 alloy sample, this sample contained numerous dark and unevenly textured regions within the alloy’s structure.
This image also displays significant dark regions on the left and right sides of the image. These regions are areas of the sample that were thicker and therefore not as electron transparent as the lighter regions of the sample. The lack of electron transparency blocks more electrons from passing through the sample and being detected by the microscope, thus yielding darker regions.

The frequency of grain of specific size intervals (50nm) is depicted in figure 4.4 and table 4.2.

![Sample B-1 Grain Size Frequency (Longitudinal Direction)](image)

**Figure 4.4 Grain Size Frequency of Sample B-1 in Longitudinal Direction.**
Table 4.3 Grain Size Distribution of Sample B-1 in Longitudinal Direction.

<table>
<thead>
<tr>
<th>Grain Size (nm)</th>
<th>% Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-50</td>
<td>0.2</td>
</tr>
<tr>
<td>51-100</td>
<td>17.4</td>
</tr>
<tr>
<td>101-150</td>
<td>26.2</td>
</tr>
<tr>
<td>151-200</td>
<td>25.2</td>
</tr>
<tr>
<td>201-250</td>
<td>16.6</td>
</tr>
<tr>
<td>251-300</td>
<td>9.6</td>
</tr>
<tr>
<td>301-350</td>
<td>3.4</td>
</tr>
<tr>
<td>351-400</td>
<td>0.2</td>
</tr>
<tr>
<td>401-450</td>
<td>0.8</td>
</tr>
<tr>
<td>&gt;450</td>
<td>0.4</td>
</tr>
</tbody>
</table>

Figure 4.4 and table 4.3 reveal that grains in sample B-1 have an average grain size of 170nm in the longitudinal direction, and a high tendency for grains to be between 101nm and 200nm in length in the longitudinal direction. 51.4% of measured grains in this sample were within that measurement interval.

Unlike other sample, sample B-1 was also analyzed in terms of transverse orientation (perpendicular to the direction of extrusion) as well. This analysis was performed because this research included two samples with the B composition. Each of these samples was produced by alternative processing methods. Analyzing the samples in their longitudinal and transverse orientations would allow more comparison between the two alloys to see how each sample’s treatment affected the alloy’s grain size distribution. The grain size frequency of sample B-1 in the transverse direction is visually represented in figure 4.5.
Figure 4.5 Grain Size Frequency of Sample B-1 in Transverse Direction.

The distribution of grain sizes in the transverse direction in B-1 is very different from the distribution of grains in specific size intervals in the longitudinal direction, as there was a much more gradual spread of grain sizes in the transverse direction in this alloy. The average grain size in sample B-1 in the transverse direction is 93nm, significantly shorter than the average grain size in the longitudinal direction in this alloy.

4.2.3 SAMPLE B-2

Alloy sample B-2 was observed at magnification of 93000X, as seen in the sample scan in figure 4.6.
Unlike other samples, grains in sample B-2 were predominantly equiaxed grains, thus were approximately the same length in both longitudinal and transverse directions. However, these grains could still be measured in terms of specific longitudinal and transverse directions as many grains still maintained a specific extrusion (longitudinal) orientation. The frequency of specific grain size intervals is illustrated in figure 4.7 and table 4.4.
Figure 4.7 Grain Size Frequency of Sample B-2 in Longitudinal Direction.

Table 4.4 Grain Size Distribution of Sample B-2 in Longitudinal Direction.

<table>
<thead>
<tr>
<th>Grain Size (nm)</th>
<th>% Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-30</td>
<td>1.2</td>
</tr>
<tr>
<td>31-40</td>
<td>4.6</td>
</tr>
<tr>
<td>41-50</td>
<td>13.4</td>
</tr>
<tr>
<td>51-60</td>
<td>18</td>
</tr>
<tr>
<td>61-70</td>
<td>20.6</td>
</tr>
<tr>
<td>71-80</td>
<td>16.8</td>
</tr>
<tr>
<td>81-90</td>
<td>13.8</td>
</tr>
<tr>
<td>91-100</td>
<td>6.6</td>
</tr>
<tr>
<td>101-110</td>
<td>3</td>
</tr>
<tr>
<td>111-120</td>
<td>1</td>
</tr>
<tr>
<td>&gt;120</td>
<td>1</td>
</tr>
</tbody>
</table>

The grains in the B-2 alloy sample are predominantly sized between 51nm and 80nm in terms of longitudinal orientation. 55.4% of the measured grains were in this range.
Additionally, the average longitudinal grain size of the alloy was 69nm. This average size was noticeably shorter than the average longitudinal grain size of the B-1 sample discussed earlier. That sample had an average longitudinal grain size of 170nm, 101nm larger than the average longitudinal grain size of the B-2 sample. Since these two samples were of the same composition, this indicates that the extrusion methods of each sample dictated their grain size development. This is also evident when factoring in the transverse grain size frequencies of these alloys. The grain size frequency of sample B-2 is illustrated in figure 4.8.

![Sample B-2 Grain Size Frequency (Transverse Direction)](image)

**Figure 4.8 Grain Size Frequency of Sample B-2 in Transverse Direction.**

The average grain size within alloy sample B-2 in transverse orientation is 59nm. Not only is this shorter than the average transverse grain size of sample B-1 (92nm), but this
average size displays how the two different extrusion processes have affected the alloy grains in terms of a longitudinal size to transverse size ratio. The grains in the B-1 sample were oblong, as is evident when observing the average grain sizes of this sample in both the longitudinal and transverse directions. The average longitudinal size of the B-1 sample was 170nm, while the average transverse size was 93nm. This means that the average B-1 alloy grain was 1.84 times longer than it was wide. The B-2 sample on the other hand had an average longitudinal size of 69nm, and an average transverse size was 59nm. The average B-2 alloy grain was therefore only 1.17 times longer than it was wide. Thus, the process used to produce alloy B-2 produces not only smaller grains than the process used for B-1, but also more equiaxed grains. This was something that was also visibly noticeable in TEM scans of each sample.

4.2.4 SAMPLE C-1

Alloy sample C-1 was observed at 93000X magnification, as see in the sample scan in figure 4.9.
Figure 4.9 Sample TEM Scan of Sample C-1 (93000X magnification).

Alloy sample C-1 stood out from other samples, as it did not contain any dark or differently textured regions that could be identified as components such as additional alloy phases, constituent regions, or other components. The grain size distribution is depicted in figure 4.9 and table 4.5.
Alloy sample C-1 contained grains that were significantly smaller than those of the other samples in this research were. 69.2% of the grains in this alloy had longitudinal sizes...
between 31nm and 60nm. In addition, the average grain size in this orientation for this alloy was 44nm, the smallest average grain size out all of the samples observed.

4.2.5 SAMPLE D-1

The alloy Sample D-1 was scanned and analyzed at 93000X magnification, as seen in the sample scan in figure 4.10

![Sample TEM Scan of Sample D-1 (93000X magnification).](image)

The structure of this sample was similar to that of the B-1 and A-1 samples. The structure contained oblong grains along with dark regions that could represent additional phase
material. The grain size frequency of alloy sample D-1 is depicted in figure 4.11 and table 4.6.

![Sample D-1 Grain Size Frequency](image)

Figure 4.12 Grain Size Frequency of Sample D-1.

<table>
<thead>
<tr>
<th>Grain Size (nm)</th>
<th>% Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-20</td>
<td>0.2</td>
</tr>
<tr>
<td>21-40</td>
<td>5.8</td>
</tr>
<tr>
<td>41-60</td>
<td>24.6</td>
</tr>
<tr>
<td>61-80</td>
<td>26.8</td>
</tr>
<tr>
<td>81-100</td>
<td>19.4</td>
</tr>
<tr>
<td>101-120</td>
<td>13.8</td>
</tr>
<tr>
<td>121-140</td>
<td>5.2</td>
</tr>
<tr>
<td>141-160</td>
<td>2</td>
</tr>
<tr>
<td>161-180</td>
<td>0.6</td>
</tr>
<tr>
<td>181-200</td>
<td>0.4</td>
</tr>
<tr>
<td>&gt;200</td>
<td>1.2</td>
</tr>
</tbody>
</table>

Table 4.6 Grain Size Distribution of Sample D-1.
A large proportion of the grains in sample D-1 are between 41nm and 80nm in length longitudinally. 51.4% of the grains in the scans were measures to be in this range. The average grain size longitudinally for this alloy was 80nm. It is important to note that this sample contained several grains that were significantly larger than a majority of the grains that were present. This alloy contained grains that were well over 141nm in size longitudinally.

4.2.6 SAMPLE E-1

The sample for alloy E-1 was scanned and observed at a magnification of 93000X, as seen in the sample scan in figure 4.12.

![Sample TEM Scan of Sample E-1 (93000X magnification).](image)
This sample appeared to be familiar in structure to sample D-1, containing a grain structure with oblong grains as well as dark unidentified regions that constitute unknown structural features. The grain size frequency and distribution of alloy sample E-1 is depicted in figure 4.12 and table 4.7.

![Sample E-1 Grain Size Frequency](image)

**Figure 4.14 Grain Size Frequency of Sample E-1.**
Table 4.7 Grain Size Distribution of Sample E-1.

<table>
<thead>
<tr>
<th>Grain Size (nm)</th>
<th>% Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-20</td>
<td>0</td>
</tr>
<tr>
<td>21-40</td>
<td>6.6</td>
</tr>
<tr>
<td>41-60</td>
<td>23</td>
</tr>
<tr>
<td>61-80</td>
<td>30.6</td>
</tr>
<tr>
<td>81-100</td>
<td>20.6</td>
</tr>
<tr>
<td>101-120</td>
<td>11</td>
</tr>
<tr>
<td>121-140</td>
<td>5.4</td>
</tr>
<tr>
<td>141-160</td>
<td>1</td>
</tr>
<tr>
<td>161-180</td>
<td>0.4</td>
</tr>
<tr>
<td>181-200</td>
<td>0.8</td>
</tr>
<tr>
<td>&gt;200</td>
<td>0.6</td>
</tr>
</tbody>
</table>

From the scan and measurement data, a high concentration of grains with between 61nm and 80nm size interval is evident. 30.6% of the grains in the alloy have a longitudinal length within that range, with an average grain size of 78nm longitudinally. Furthermore, 74.2% of the grains are within a size range from 41nm to 100nm in sample E-1.

4.2.7 SAMPLE F-1

Sample F-1 was scanned and analyzed at a magnification of 60000X, as seen in the sample scan in figure 4.13.
Figure 4.15 Sample TEM Scan of Sample F-1 (60000X magnification).

This alloy had a similar structure to that of other samples like alloy B-1 with long grains and the presence of dark unidentified regions. However, the grains in alloy sample F-1 are significantly larger than those of other samples. This trend is very prominent in the visual representations of this analysis in figure 4.14 and table 4.8.
Figure 4.16 Grain Size Frequency of Sample F-1.

Table 4.8 Grain Size Distribution of Sample F-1.

<table>
<thead>
<tr>
<th>Grain Size (nm)</th>
<th>% Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-200</td>
<td>0.4</td>
</tr>
<tr>
<td>201-400</td>
<td>15.6</td>
</tr>
<tr>
<td>401-600</td>
<td>36.2</td>
</tr>
<tr>
<td>601-800</td>
<td>30.6</td>
</tr>
<tr>
<td>801-1000</td>
<td>10</td>
</tr>
<tr>
<td>1001-1200</td>
<td>3.8</td>
</tr>
<tr>
<td>1201-1400</td>
<td>1.6</td>
</tr>
<tr>
<td>1401-1600</td>
<td>0.8</td>
</tr>
<tr>
<td>1601-1800</td>
<td>0.8</td>
</tr>
<tr>
<td>&gt;1800</td>
<td>0.2</td>
</tr>
</tbody>
</table>
Sample F-1 had by far the largest grains out of all the alloys observed. The average grain size of this alloy was 629nm in longitudinal orientation. Sample F-1 also had highest concentrations of grains within the size intervals of 401nm to 600nm and 601nm to 800nm. Grains in these size intervals made up 66.8% of the alloys structure. It is notable that the histogram representation of this alloy’s grain size frequency does show a less gradual dispersion of grains of varying sizes. The histogram for F-1 reveals high concentration spikes in the 401nm to 600nm and 601nm to 800nm intervals, unlike the dispersions within samples of alloys like E-1 and B-2.
CHAPTER 5
CONCLUSIONS

This research has revealed a large amount of information about the grain structures of alloyed aluminums scanned through TEM and analyzed in this work. Through grain size analysis, each alloy had their grain size frequency analyzed and average grain size calculated. Through histogram analysis, each alloy was revealed to have a Gaussian distribution of grains through the grain size ranges. Each alloy contained high concentrations of grains in size intervals that included the average grain size of the alloy, with much lower concentrations of grains of much larger and smaller sizes.

A majority alloys observed displayed grain structures containing grains that were significantly larger in their longitudinal orientation than their transverse orientation. This could be due to the nanostructure development processes of the alloys. The exceptions to this trend were alloy samples B-2 and C-1. Sample C-1 had a unique structure with small nearly equiaxed grains, while sample B-2 had a structure that resembled that of alloy B-1, though with equiaxed grains. Though the reason why sample C-1 had the structure it had was inconclusive, sample B-2 had equiaxed grains rather than the long grains in the B-1 sample counterpart because B-2 was subjected to a different production process. This process could have deformed the alloy differently or possibly applied heat that would allow for more equiaxed grains.
The samples observed in this research also revealed prominent dark and textured regions within their microstructures. Each sample, with the exception of that of alloy C-1, contained a uniform grain structure with these standout regions spread through their structure. These regions are likely additional structural features that are present because these extrusions are aluminum samples alloyed with other components. These regions could be additional phases, constituents, precipitate particles, or other structural components. This scope of this research does not include identification of structural components, so this could be the basis of future work.

It is difficult to determine why alloy sample C-1 had a drastically different structure than the other alloys tested. One possibility is that the sample was not prepared for TEM correctly. The sample may have been ion milled too quickly with a beam that was too powerful. This would have led to the sample possibly having deformations or an unevenly milled surface that would not represent the true structure in the TEM scanning process.
CHAPTER 6
FUTURE WORK

Beyond this research, much more work can be done with the alloys observed, such as identification of microstructure components. The TEM scans of this research revealed that the aluminum alloys observed had distinct dark and textured regions within the nanocrystalline aluminum microstructures. The scope of this research did not address the specific components present in these alloys, as this work was focused on grain size. Future research could dwell into the identification of these components. Identifying these features could lead to a greater understanding of microscopic behavior, such as slip systems, and macroscopic behavior of the alloys.

Further analysis of the microstructures of these alloys could also help establish a link between the microscopic properties and macroscopic behavior of these alloys. Mechanical testing can generate macroscopic properties like yield strength, tensile strength, and ductility for many metal alloys. Coupling these mechanical properties with TEM analysis of alloy structures can advance the understanding of nanostructured aluminum alloys and their effect on mechanical behavior.


