Effects of Very High Power Ultrasonic Additive Manufacturing Process Parameters on Hardness, Microstructure, and Texture of Aluminum 3003-H18 Alloy

DISSERTATION

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By

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Abstract

Ultrasonic Additive Manufacturing (UAM) process is a solid-state joining process used to build up a solid part from thin metal foils. In UAM process, a cylindrical roller called sonotrode with rough surface is used to push down on to the foil and roll over it from one end to another. While rolling, the sonotrode also vibrates from side to side at ultrasonic frequency of 20 kHz. After the foil is completely added onto the part, another foil is added on top of the same part. The process is repeated until the desired final thickness is obtained. In addition, the part can be machined while being additively fabricated allowing complex geometry parts to be made. Some current applications of UAM include industrial molds for plastic injection molding, embedding fibers and sensors into a device, and multi-material structure panels. In order to bond harder materials, Very High Power Ultrasonic Additive Manufacturing (VHP-UAM) process was developed with much higher power capability to produce higher normal force and vibration amplitude than UAM process.

The goal of this research study is to investigate the effects of the very large VHP-UAM processing parameters (normal force and vibration amplitude) on hardness, microstructure and texture in the as-processed and heat-treated conditions both at the interface regions and the bulk regions. In this research work, VHP-UAM parts were fabricated from aluminum 3003-H18 (Al3003-H18) foil. VHP-UAM samples were made with different levels of normal forces and vibration amplitudes while the travel speed was
kept constant. Microhardness measurement was used to assess the change in the properties of the microstructures with reference to the original Al3003-H18 foil. The local microstructure across the thickness of layers in VHP-UAM builds was obtained using electron backscatter diffraction (EBSD) technique available in the secondary electron microscopy. In addition, the macro-texture of the entire VHP-UAM builds was obtained using time-of-flight neutron diffraction in the High-Pressure Preferred Orientation Diffractometer (HIPPO) at the Los Alamos National Laboratory.

The first major finding of this research is that softening behavior occurs when higher vibration amplitude is used. This results in better bonding quality at the interfaces. The degree of softening also decreases in the top layers as the build gets higher. The second major finding is that the fine grains are present at the interface region of the VHP-UAM builds as a result of dynamic recrystallization and severe plastic deformation, whereas the bulk regions contain similar elongated grains and microstructures similar to original foil with decreasing rolling textures as the vibration amplitude increases. The final major finding is that the shear textures are present at and below the interface region where large shear deformation takes place due to ultrasonic vibration and interfacial friction and deformation. These shear textures are very stable without decreasing in its volume fraction after heat treatment at high temperature like the rolling texture components which decrease during heating. Also, the change in microstructure during VHP-UAM processing is explained by the change in stored energy within the sample due to different processing parameter ranges used in lower power UAM and VHP-UAM.
The overall results show that with increasing in vibration amplitude in VHP-UAM processing, the microstructure undergoes larger degree of shear deformation not only at the interface region but also in the bulk region of each layer of the build. The thermo-mechanical cycles also generate heat in the subsequent process, which results in the recovery and/or recrystallization and grain growth in the microstructure of the layers below. However, further experimental characterization is needed to identify the natures of the grains and subgrains as well as their boundaries especially at the interface region to identify the correct mechanisms of grain boundary migration involved during the recovery, recrystallization, and grain growth during heat treatment at elevated temperature.

This research also shows the importance of studying not only how to improve the overall bond quality but also on the underlying and complex microstructure and texture evolution both in as-processed and heat-treated condition within the VHP-UAM builds. With better understanding of how the processing parameters affect the microstructure and texture evolution in the interface region and the bulk region, it would be advantageous for future researchers to design and better select material choices and processing condition to optimize the bond quality and the overall and entire microstructure and properties of the VHP-UAM parts. This research data will be useful for input and experimental validations for future development of process modeling and material modeling to predict mechanical properties as well as the microstructure and texture evolution both during VHP-UAM processing and subsequent heat treatment.
Dedication

This document is dedicated to my Father in Heaven, my mom, and my family.
Acknowledgments

I would like to thank my advisor Dr. Sudarsanam Suresh Babu for his guidance, support, and knowledge during the whole course of my Ph.D. education. I also would like to thank Dr. Avraham Benatar for his guidance and support during my last few years of my Ph.D. after Dr. Babu relocated to work at University of Tennessee. I would like to pass additional thanks to Dr. John Lippold, and Dr. Wei Zhang for their serving on my committee and also thanks to Dr. Stanislav Rokhlin for his help as graduate chair for the past six years. I also thank Dr. Anchalee Manonukul for her guidance in writing dissertation.

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Fields of Study

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Additive manufacturing is a process for fabricating three-dimensional object layer by layer from computer aided design (CAD) data instead of removing material out of a solid block in subtractive manufacturing [1]. Additive manufacturing is also referred to as rapid prototyping or 3D-printing. This saves both time and cost because the product can be designed and manufactured from computer-aided design and computer-aided manufacturing (CAM) modules. Different raw materials, including polymer, ceramics, and metal, can be used to create parts using additive manufacturing such as liquid, powder, tape, or sheet. Additive manufacturing is also attractive because it can be used to fabricate multi-material or multiple-component parts with improved mechanical properties, functionality, and performance [2].

Ultrasonic additive manufacturing (UAM) or ultrasonic consolidation (UC) is a solid-state additive manufacturing process, which applies ultrasonic metal seam welding to bond thin metal tapes/foils together. UAM also uses computer numerical controlled (CNC) milling process to create 3D contour surfaces, holes, or channels while building thicker 3D part according to the programmed 3D CAD files. Due to its low processing temperature, UAM is advantageous for manufacturing dissimilar metal laminates and for embedding wires, fibers, or small devices without melting or damaging parts or
components [2-4]. Thus, the final material properties of the fabricated parts are similar to the original material [5].

The traditional UAM machine has limited operating power at 1.5-3.0 kW, which is capable of joining soft FCC metals such as aluminum and copper foils of 0.1-0.2 mm thick. However, its maximum power, normal force, and vibration amplitude are not sufficient to produce good bonding of thicker tapes or harder materials such as titanium alloy, stainless steel, and nickel-based alloys. Therefore, a new UAM system, called very high power ultrasonic additive manufacturing (VHP-UAM) with power capacity of 9.0 kW, was developed by the Edison Welding Institute (EWI) with the capability to bond harder and thicker material while maintaining productivity and performance [6]. The majority of published literature are based on samples made from UAM [7-62] rather than VHP-UAM. The literatures pertaining to microstructure characterization, properties, and process optimization of parts fabricated by VHP-UAM is still relatively limited [63-67].

Therefore, the current research aims to evaluate the effects of VHP-UAM processing parameters (vibration amplitude and normal force) on the changes in hardness, microstructure, and texture of aluminum alloy 3003-H18 (Al3003-H18). The processing parameters were selected such that the effects of the lower power regime (lower vibration amplitude and normal force) and the higher power regime (higher vibration amplitude and normal force) can be compared. Al3003-H18 was selected for the current study because it is good for structural application, has good formability, and most importantly this alloy has been studied by other low-power UAM processing conditions and therefore allows for a good baseline comparison [8, 9, 20, 33, 35, 36, 46, 48, 49, 57, 60-62, 65-69].
In this study, three material conditions were examined including as-received Al3003-H18 foil, as-processed Al3003-H18 VHP-UAM builds, and heat-treated (343°C for 2 hours) Al3003-H18 foil and VHP-UAM builds. The results cover the effects of VHP-UAM processing parameters on the changes in hardness, microstructure, and texture of as-processed VHP-UAM samples as compared to original as-received Al3003-H18 foil. In addition, the effects of the changes in process parameters and as-processed microstructures on heat-treated microstructure and texture evolution after annealing at 343°C for 2 hours is also investigated. Based on these results, an insight into fundamental metallurgical phenomena during VHP-UAM is postulated.

In this thesis, detailed background and literature review of previous research in UAM and VHP-UAM as well as the challenges needed to drive the research and development of this technology forward is discussed in Chapter 2. This work then explains the research objectives in Chapter 3 and the experimental procedures in Chapter 4. The results and discussion sections are divided into Chapter 5 and Chapter 6. First, the effects of VHP-UAM process parameters on the changes in hardness, microstructure, and texture of Al3003-H18 both in as-processed condition and heat-treated condition compared to original foil are discussed in Chapter 5. Then, Chapter 6 discusses the results of the bulk texture analysis using time-of-flight neutron diffraction at room temperature and in-situ annealing. Finally, Chapter 7 addresses the conclusions of this research work and suggested future work.
Chapter 2: Background and Literature Review

2.1 Ultrasonic Additive Manufacturing (UAM)

UAM is a solid-state joining process developed by Solidica Inc., for fabrication of complex geometry parts [4]. The process relies on ultrasonic seam welding of thin foils of 100-200 μm in thickness, with high ultrasonic frequency of 20 kHz, low amplitude of 14-28 μm, normal force of 800-1500 N, and weld speed of 25-50 mm/s, and ability to preheat the substrate at 65-150°C, one layer after another [49]. During UAM process, the first layer of metal foil is added on top of a base plate or substrate to be joined together (see Figure 2.1). The foil is pressed down by a roller-shaped sonotrode in the normal direction (ND). The sonotrode has some texture or surface roughness to cause traction on the foil surface. This prevents slippage between the foil surface and the sonotrode surface during the application of the horizontal ultrasonic vibration. During each welding cycle, the sonotrode rolls over the top surface of the added foil while pressing down vertically and vibrating horizontally in the transverse direction (TD) at the ultrasonic frequency. It is noted that during UAM, the sonotrode vibrations are not in the welding direction, but the sample is moved in the welding or rolling direction (RD). As a result, the oxide layers are broken through the scrubbing at the surfaces between sonotrode and top foil surface and between bottom foil surface and substrate surface. This forms the nascent metal-to-metal surface contacts, which creates a solid-state bond between the foil
and the substrate [4, 56]. After the welding pass, a new layer of metal foil is added on top of the previous layer and the process is repeated until the part reaches its final dimension.

The UAM equipment also has the ability to machine the part, while joining and stacking each layer, to create channels or contour surface. UAM is also capable of adding different materials or embedding wires or fibers to create composite materials. UAM process has been used to fabricate industrial molds for injection molding, extrusion, and vacuum forming. Potential applications include encapsulating sensors and fibers as well as multi-material structures for advanced applications (see Figure 2.2) [4, 13, 17, 29, 32, 45-47, 55]. UAM has advantages for fabricating and embedding parts
with components, which can be damaged at high temperature, because UAM is low
temperature process and no melting occurs compared to other rapid prototyping processes
[70].

Figure 2.2. Examples of engineering parts fabricated by UAM for potential applications
(a) fibers embedded in aluminum matrix, (b) copper block embedded in aluminum part,
(c) fiber optics embedded between aluminum tapes, (d) X-ray image of internal channels
created in UAM parts (Courtesy of Solidica Inc., and EWI)

2.2 Very High Power Ultrasonic Additive Manufacturing (VHP-UAM)

The UAM system, which is operated by a single ultrasonic transducer of 1.5-3.0
kW, is able to produce good bonding in soft FCC materials such as aluminum and copper
[64]. However, there is limitation in the range of UAM process parameters and power,
which are insufficient to produce higher vibration amplitude and normal force to bond harder and thicker materials [6, 71]. Although UAM of harder materials has been done, no mechanical testing was performed to evaluate the actual weld strength of these materials [7, 45, 56]. It was also found that large unbonded regions were present in the microstructure [56]. Thus, the improvement in process parameters is necessary to minimize the unbonded regions. As a result, the upgraded version of UAM machine or VHP-UAM was created by increasing the amount of ultrasonic power (up to 9 kW), capable of producing larger normal force (up to 15 kN) and larger vibration amplitude (up to 52 µm). In order to increase ultrasonic power from 3 kW in UAM to 9 kW in VHP-UAM, two transducers (4.5 kW each), instead of one transducer in UAM, were combined together in a push-pull configuration (see Figure 2.3 (a)). The surface of the sonotrode was imparted by electro-discharge machining process to produce a surface roughness of $R_A = 7 \, \mu m$ (see Figure 2.3 (b)) [64]. Therefore, VHP-UAM can be used to bond thicker foil and harder materials, in which UAM could not achieve good bonding, with higher productivity [6, 64].

2.3 Prior Research on UAM/VHP-UAM

The goal of UAM is to maximize the productivity of fabricating a solid part using the least amount of time possible. Previous researchers have investigated the correlations between process parameters and the bond quality of the UAM parts [20, 32, 33, 35, 46, 49]. By varying different process parameters (normal force, vibration amplitude, and weld speed), and determining the bond quality in terms of linear weld density (the ratio of
bonded area over the entire interface) or bond strength, ranges of optimal process windows for different materials such as aluminum 3003 and aluminum 6061 alloys were determined \[20, 32, 33, 35, 46, 49\]. Prior results showed an increase in linear weld density and bond strength with higher normal force, higher vibration amplitude, and lower weld speed \[20, 35\]. However, the weld speed cannot be too low as it lowers the productivity rate as well as causes localized melting or sticking of the foil material on the
sonotrode surface. This may cause equipment down time because the sonotrode needs to be cleaned and in some case resurfaced again [4].

While the focus of previous UAM researchers was to maximize the linear weld density and achieve flat and smooth bonded interface, there is insufficient evidence to claim that the bond strength significantly increase as a result of higher linear weld density [20, 49]. Therefore, understanding the bonding mechanism at the microstructural level at the bonded interface locations is also an important subject of research. However, limited works have provided insights on how process parameters affect the microstructure and properties across the interfaces and in the bulk regions from top to bottom of UAM and VHP-UAM parts. Thus, the literature review of the effects of UAM/VHP-UAM process parameters on hardness, microstructure, and texture is emphasized.

2.3.1 Effects of UAM/VHP-UAM Process Parameters on Hardness

While the focus of several previous researches was on improving bond quality of UAM and VHP-UAM samples by optimizing the process parameters, little works were devoted to the change in bulk properties, i.e. hardness, of the foils due to UAM/VHP-UAM process parameters. Kong et al. [34] was the first to report the change in microhardness at the weld interface of aluminum 6061 UAM foils with varying contact pressure, vibration amplitude, and weld speed. It was found that hardness increases with increasing contact pressure, increasing vibration amplitude, and decreasing weld speed in UAM of aluminum 6061 (see Figure 2.4) [34]. When compared to the hardness of original foil, softening behavior was found at low contact pressure, whereas hardening
behavior was found at higher contact pressure. However, no result was reported for the change in hardness further away from the interface, i.e. in the bulk region of the UAM foil.

Figure 2.4. Average microhardness result at weld interface of aluminum 6061 UAM sample on aluminum 1050 base plate at various contact pressure levels [34]

Li and Soar [38] derived the Hall-Petch relationship showing the values of hardness obtained from nanoindentation as a function of average grain and subgrain sizes in aluminum 3003-O UAM samples (see Figure 2.5). They observed hardening behavior in the bulk of aluminum 3003 O foil caused by plastic deformation during UAM process. Similar result was found by Schick et al [49] who reported hardening behavior in the bulk of Al3003-H18 foil after UAM (see Figure 2.6). Both results showed trend of hardening
behavior in the foil after UAM. Unlike UAM results which show hardening behavior in the foil, Sriraman et al. [64] reported softening behavior in hard temper copper C11000 foil after VHP-UAM (see Figure 2.7). They proposed that the dynamic recrystallization and dynamic recovery during the temperature rise by VHP-UAM processing caused the softening to occur in the bulk region of original foil [64]. However, none of these works [38, 49, 64] investigated the effect of increasing process parameters (vibration amplitude, normal force, and weld speed) on the degree of hardening and softening in the foil after UAM/VHP-UAM.

![Figure 2.5. Hall-Petch relationship between hardness (measured from nanoindentation) and average grain diameter for Al3003 foil and Al3003 UAM material (vibration amplitude of 12.3 µm, contact pressure of 155.8 MPa, and weld speed of 34.5 mm/s) [38]](image-url)
Figure 2.6. Histograms showing hardness distribution of Al3003-H18 UAM sample showing hardening behavior in foil after UAM (vibration amplitude of 17 µm, normal force of 1150 N, weld speed of 42.3 mm/s, and preheat of 150ºC) [49]

Figure 2.7. Histogram of hardness distribution in copper C11000 (hard temper) VHP-UAM sample showing softening behavior in foil after VHP-UAM (vibration amplitude of 36 µm, normal force of 6.7 kN, and weld speed of 30 mm/s) [64]
2.3.2 Effects of UAM/VHP-UAM Process Parameters on Microstructure

The microstructure results in UAM and VHP-UAM samples have been reported by several researchers [8, 25, 40, 56, 63, 67, 72]. The first subgrain refinement at the interface of annealed aluminum 3003 (Al3003-O) UAM part was reported by Johnson [25, 72] (see Figure 2.8) and was attributed to severe plastic deformation due to the interaction between the surface texture of the sonotrode and the foil surface. His results also show little changes in the bulk microstructure of the foil after UAM process indicating little effect of UAM process parameters on the bulk properties of the bonded layers [25, 72]. They also reported the localized persistent oxide layers dispersed along the wavy bonded interface affected by plastic flow of material at the interface [25, 72]. Similar results were reported in aluminum 6061 O (Al6061-O) (see Figure 2.9) by Mariani and Ghassemieh [40], aluminum 6061 H18 (Al6061-H18) (see Figure 2.10) by Shimizu et al. [63], and Al3003-H18 alloys (see Figure 2.11) by Dehoff and Babu [8]. Their results show that most local interface locations contain very small and fine recrystallized grains as compared to original equiaxed grains in Al6061-O foil, and thin and elongated grains in Al6061-H18 and Al3003-H18 foils [8, 25, 38, 40, 63, 66-68, 73, 74]. When harder Ni foil was selected for UAM, the results of Yang et al. [56] show that the volume of the interface region containing fine and equiaxed grains is much smaller (see Figure 2.12). This means that the amount of material, which undergoes the microstructural change via severe plastic deformation and dynamic recrystallization, varies with the types and properties of the original metal foil in addition to UAM/VHP-UAM process parameters and surface roughness of the sonotrode.
Figure 2.8. Ion beam induced SEM image showing persistent oxide layer and different grain sizes across the bonded interface of Al3003-O UAM sample (vibration amplitude of 13 μm, normal force of 1400 N, weld speed of 42.3 mm/s, and preheat of 149°C) [25]

Figure 2.9. Inverse pole figure maps of aluminum 6061-O (a) original foil and UAM samples processed at different contact pressures of (b) 135 MPa, (c) 155 MPa, and (d) 175 MPa (vibration amplitude of 50% maximum, and weld speed of 34.5 mm/s) [40]
Figure 2.10. Inverse pole figure map showing the void and microstructure along the interface region of aluminum 6061-H18 VHP-UAM sample (vibration amplitude of 31 \(\mu\)m, normal force of 5.6 kN, and weld speed of 35.6 mm/s) [63]

Figure 2.11. Inverse pole figure maps (with overlay of image quality) of along the bonded interfaces of Al3003-H18 UAM (vibration amplitude of 12 \(\mu\)m, normal force of 1150 N, weld speed of 59.3 mm/s, and preheat of 149°C) [8]
The interface microstructure of VHP-UAM material was first reported by Sriraman et al. [64] in copper C11000 build (see Figure 2.13). Finer grains exist at the interface regions showing some shear and flowing phenomena because of ultrasonic vibration during VHP-UAM process. The interface and the bulk microstructures at different locations from top to bottom of Al3003-H18 VHP-UAM sample were first reported by Fuji et al. [67]. The sample was made using vibration amplitude of 26 µm, normal force of 5.6 kN, and weld speed of 35.6 mm/s and composed of eight bonded layers. Their results showed fine equiaxed grains with shear texture at all selected interface locations from the top, the middle, and the bottom regions of the sample (see Figure 2.14). A similar microstructure was also found in the sample containing two layers using the same set of process parameters. Little changes in the bulk microstructure were reported and elongated grains with rolling texture components were similar to those
found in as-received Al3003-H18 foil [67]. It was believed that dynamic recrystallization was aided by adiabatic heating during high strain rate plastic deformation, especially where asperity collapse occurs [8, 40, 64, 68, 73].

It is also important to note that there seems to be no significant difference between the microstructures in the bulk regions of Al3003-H18 foils processed by UAM (see Figure 2.11) and VHP-UAM (see Figure 2.13). This implies that the processing parameter (vibration amplitude of 26 µm, normal force of 5.6 kN, and weld speed of 35.6 mm/s) used by Fujii et al. [67] may not high enough to induce the microstructure change in the bulk region away from the interface of Al3003-H18 foil subjected to VHP-UAM. However, the distinct softening behavior found in the bulk of copper C11000 VHP-UAM samples by Sriraman et al. [64] indicates that the effects of VHP-UAM process parameters on microstructure changes exist. In order to understand the overall changes in properties (i.e. hardness), and microstructure in UAM and VHP-UAM samples, the change in crystallographic textures at the interface and bulk regions is reviewed.

Figure 2.13. Inverse pole figure maps of (a) as-received copper C11000 foil, and (b) interface region of as-processed copper C11000 after VHP-UAM (vibration amplitude of 36 µm, normal force of 6.7 kN, and weld speed of 30 mm/s) [64]
Figure 2.4. Inverse pole figure maps around the interface regions at different locations in Al3003-H18 VHP-UAM samples at different interface locations: (a) in two-layer build between 1st layer and 2nd layer, and in eight-layer build (b) between 7th layer and 8th layer, (c) between 4th layer and 5th layer, and (d) between 1st layer and 2nd layer (vibration amplitude of 26 µm, normal force of 5.6 kN, and weld speed of 35.6 mm/s) [67]

2.3.3 Effects of UAM/VHP-UAM Process Parameters on Texture

In crystallographic materials, there exists crystal symmetry as well as sample symmetry. The common way to describe sample symmetry is illustrated in Figure 2.3 by using notation axes: ND (normal direction), RD (rolling direction), and TD (transverse direction). Texture is very important because several material properties are specific to texture including Young’s modulus, Poisson’s ratio, strength, ductility, toughness,
magnetic permeability, electrical conductivity, and thermal expansions. In general, a completely new texture is developed after deformation, recrystallization, and phase transformation. Macro-texture represents the overall or the average texture of bulk sample without information on grain size or grain structure. Micro-texture reveals statistical data of grain orientation of individual grains and how individual grains are distributed within the sample. Thus, micro-texture has advantage of gaining knowledge of both microstructure and crystallography, while macro-texture can provide information on volume fraction of particular grain orientation but not the detailed microstructure. The rolling textures in metals are produced by reduction in thickness during rolling process, in which the texture strength increases with larger strain or larger reduction in thickness. It is known that rolling texture varies in pure metals and alloys. For examples, the texture of pure copper is copper texture, whereas the texture of copper-zinc alloy (brass) is brass texture.

Table 2.1 provides list of major rolling texture components of FCC metals with their respective Euler angles and Miller indices. There are two main texture fibers in FCC metals: β-fiber and α-fiber. The β-fiber runs from copper component to brass component through S orientation, while the α-fiber runs from brass component to Goss component. The simple shear texture is represented in the form of shear plane/shear direction with the ideal shear orientations including: the A-partial fiber {111}(uvw), B-partial fiber {hkl}(110), and C-orientation {001}(110) (see Figure 2.15) [75, 76].
<table>
<thead>
<tr>
<th>Name</th>
<th>Indices</th>
<th>Bunge ($\varphi_1$, $\Phi$, $\varphi_2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>${1\ 1\ 2}\ &lt;1\ 1\ 1&gt;$</td>
<td>90, 35, 45</td>
</tr>
<tr>
<td>S1</td>
<td>${1\ 2\ 4}\ &lt;2\ 1\ 1&gt;$</td>
<td>59, 29, 63</td>
</tr>
<tr>
<td>S2</td>
<td>${1\ 2\ 3}\ &lt;4\ 1\ 2&gt;$</td>
<td>47, 37, 63</td>
</tr>
<tr>
<td>S3</td>
<td>${1\ 2\ 3}\ &lt;6\ 3\ 4&gt;$</td>
<td>59, 37, 63</td>
</tr>
<tr>
<td>Brass</td>
<td>${1\ 1\ 0}\ &lt;1\ 1\ 2&gt;$</td>
<td>35, 45, 0</td>
</tr>
<tr>
<td>Taylor</td>
<td>${4\ 4\ 1\ 1}\ &lt;1\ 1\ 1\ 8&gt;$</td>
<td>90, 27, 45</td>
</tr>
<tr>
<td>Goss</td>
<td>${1\ 1\ 0}\ &lt;0\ 0\ 1&gt;$</td>
<td>0, 45, 0</td>
</tr>
</tbody>
</table>

Table 2.1 Major rolling texture components in FCC metals [75]

![Figure 2.15](image)

Figure 2.15. (a) Typical rolling textures in FCC metal, (b) Ideal simple shear textures in FCC metals [76]

The texture changes in Al6061-O UAM samples, Al3003-H18 VHP-UAM samples, and Al6061-H18 VHP-UAM samples were reported using $\{111\}$-pole figures and ND and RD inverse pole figures to represent the texture [40, 63, 67]. Mariani and Ghassemieh [40] studied the changes in crystallographic textures in different regions of Al6061-O UAM foils at three different contact pressures. From their results (see Figure 2.16), the texture is weakest at the interface region in all three conditions. The original foil possesses strong cube texture, which gets weaker with increasing contact pressure and in the bottom foil as compared to the top foil. In addition, the traces of rolling
texture fibers along the $\alpha$-fiber and $\beta$-fiber are also observed in some of the bulk foil regions and interface regions implying some amount of work hardening takes place in the bulk of Al6061-O foil during UAM processing.

![Schematic of data collection for monolithic and fibre samples](image)

Figure 2.16. ND and RD inverse pole figures of a monolithic aluminum 6061 O UAM sample in the upper foil, interface, and lower foil regions (see inverse pole figure maps in Figure 2.9) with ideal locations of rolling textures in FCC metal along $\alpha$ and $\beta$ fibers (vibration amplitude of 50% maximum, contact pressure of (a) 135 MPa, (b) 155 MPa, and (c) 175 MPa, and weld speed of 34.5 mm/s) [40]

Similar texture results were reported in VHP-UAM of Al6061-H18 (see Figure 2.17) by Shimizu et al. [63] and Al3003-H18 (see Figure 2.18) by Fujii et al. [67] where little changes in textures take place in the upper bulk regions and bottom bulk regions at different interface location across the build heights. These bulk regions possess the
original rolling texture from original foil microstructure, which barely changes after VHP-UAM processing at selected processing parameters. Therefore, it is not obvious from the literatures whether there exist the effects of increasing in power and processing parameters (vibration amplitude and normal force) on the changes in microstructure and texture in the bulk regions. The evidence in the difference in hardening and softening behaviors from literature reviews indicate that more study is necessary to understand the nature of softening and hardening behaviors occurring in the UAM/VHP-UAM foils.

Figure 2.17. \{111\}-pole figures of upper bulk regions, interface regions, and lower bulk regions in Al6061-H18 VHP-UAM sample at different locations: (a) 29th layer and 30th layer, (b) 15th layer and 16th layer, and (c) 1st layer and 2nd layer (vibration amplitude of 31 μm, normal force of 5.6 kN, and weld speed of 35.6 mm/s) [63]
Figure 2.18. \{11\}\textsubscript{1}-pole figures derived from top region, interface region, and bottom region of areas at different locations (see inverse pole figure maps in Figure 2.14) of Al3003-H18 VHP-UAM samples at different interface locations: (a) in two-layer build between 1\textsuperscript{st} layer and 2\textsuperscript{nd} layer, and in eight-layer build (b) between 7\textsuperscript{th} layer and 8\textsuperscript{th} layer, (c) between 4\textsuperscript{th} layer and 5\textsuperscript{th} layer, and (d) between 1\textsuperscript{st} layer and 2\textsuperscript{nd} layer (vibration amplitude of 26 \textmu m, normal force of 5.6 kN, and weld speed of 35.6 mm/s) [67]
2.4 Challenges in UAM/VHP-UAM Research

Post processing heat treatment is another approach used to improve the linear weld density. Results by Schick showed that the linear weld density of Al3003-H18 UAM samples improved from 62.7% to 80.6% after heat treatment [73]. The results also showed difference in microstructure evolution across the UAM samples, where large grains occurred above the interfaces, whereas small grains existed below the interfaces as shown in Figure 2.18. Two hypotheses for the persistence of small grains even after heat treatment were proposed [73]. The first hypothesis was the pinning of grain boundaries by intermetallic particles during heat treatment. Another hypothesis was that the dynamic recrystallization occurs during UAM, which is followed by particle-stimulated nucleation of new grains around broken oxides at the interfaces. However, no scientific evidence exists to support the above hypotheses. Several fundamental characterization and modeling works have been done in the areas of recrystallization and grain growth in the deformed metals [77-123], but very few have been referenced to UAM processing [23, 40, 63, 64, 67]. The goal of the current research is to formulate a better understanding of the microstructure evolutions in the UAM and VHP-UAM builds by further examining the change in hardness, microstructure, and texture as a result of changes in VHP-UAM processing parameters (vibration amplitude and normal force) and post-processing heat-treatment.
Figure 2.19. Comparison of Al3003-H18 microstructure in (a) as-processed, and (b) 343°C for 2 hours heat-treated UAM samples [73]
Chapter 3: Research Objectives

In previous chapter, it is found that there are limited studies and understanding on the change in vibration amplitude and normal force on the overall bulk microstructures of foils and parts processed by VHP-UAM. Although it has already been known that fine and equiaxed grains are present at the interface regions of UAM and VHP-UAM samples, no study has reported a clear change in microstructure and texture in the bulk regions because of higher vibration amplitude and higher normal force in VHP-UAM. Therefore, the present work is aimed to perform a fundamental study of VHP-UAM of Al3003-H18 alloy with the following objectives:

1. Develop a fundamental understanding of the effects of vibration amplitude and normal force during VHP-UAM of Al3003-H18 alloy on the changes in hardness, microstructure, and texture in the as-processed condition as compared to the as-received Al3003-H18 foil.

2. Investigate the microstructure and texture evolution of Al3003-H18 VHP-UAM samples after post-processing heat-treatment at 343°C for 2 hours compared to heat-treated Al3003-H18 foil and as-processed VHP-UAM samples.

Chapter 4: Experimental Procedures

4.1 Introduction

This chapter contains a description of experimental procedures used for this research. The chapter is divided into materials section, sample preparation section, hardness measurement section, and microstructure characterization section. Figure 4.1 details the outline of the samples made from two different VHP-UAM machines as well as the characterization methods used to evaluate. It is noted that each type of characterizations and tests was not performed for all VHP-UAM samples.

4.2 Materials

This work evaluates the changes in hardness, microstructure, and texture of the commercial Al3003-H18 foil during VHP-UAM processing and heat-treatment. The original as-received Al3003-H18 foil has the dimension of 25.4 mm wide and 150 µm thick. Its chemical composition in weight percentage is Al-1Mn-0.7Fe-0.12Cu. The VHP-UAM samples were made on top of a 25.4 mm thick aluminum 3003-H14 (Al3003-H14) substrate, which was also used in previous works [49, 66]. The ultrasonic sonotrode horn, which is in contact with the deposited tape, is made of Ti-6Al-4V with surface roughness $R_A$ of 7 µm.
4.3 Sample Preparation

All VHP-UAM samples in this study were made of 150 µm thick Al3003-H18 foil. The VHP-UAM samples were fabricated using two different machines: the Test-Bed machine and the commercial SonicLayer-7200 machine (see Figure 4.2). The Test-Bed machine, which was available earlier, was used to fabricate three ten-layer VHP-UAM samples to study the effects of vibration amplitude and normal force on the changes in hardness, microstructure, and texture of Al3003-H18 both in as-processed condition and in heat-treated condition. The SonicLayer-7200 machine, which was available later during the course of this research study, was used to fabricate thicker (up to 80 layers) Al3003-H18 VHP-UAM samples. These large samples were needed to study the variations in hardness across the samples and the overall bulk texture of the
samples in as-processed and heat-treated conditions. The main difference between the two machines is the fully-automated tape feeder, which was available in the SonicLayer-7200 machine but is not available in the Test-Bed machine. This fully-automated SonicLayer-7200 machine allows the fabrication of thicker samples required for the bulk texture analysis using neutron diffraction (see Figure 4.3).

Figure 4.2. Two VHP-UAM systems used to fabricate samples in this study: Test-Bed machine and commercial SonicLayer 7200 machine [6]

Table 4.1 lists all the conditions and the processing parameters used to fabricate Al3003-H18 VHP-UAM samples in the current study using the two different machines. Each sample is assigned by sample ID, which is designated as Name of Machine (TB =
Figure 4.3. Aluminum 3003-H18 tapes deposited on top of Al3003-H14 substrate using the SonicLayer 7200 very high power VHP-UAM system.

Test-Bed, SL = SonicLayer-7200) – Number of Layer – Vibration Amplitude (in µm) – Normal Force (in N). VHP-UAM samples made from Test-Bed machine has 10 layers, whereas VHP-UAM samples made from SonicLayer-7200 machine has 80 layers (except sample SL-66-28-5340, where its 67th layer could not be bonded because of delamination). The ultrasonic frequency and the welding speed was kept constant at 20 kHz and 35.6 mm/s, respectively, for both machines, with the exception of the 51st and above layers in the SonicLayer-7200 machine, where the welding speed was adjusted to 42.7 mm/s instead of 35.6 mm/s. The initial processing parameter set was selected for sample TB-10-28-5340 and sample SL-66-28-5340 because it was found that this processing condition results in good bond strength during peel test [124]. The other two
parameter sets for each machine were selected at larger vibration amplitude, i.e. 34 µm for SonicLayer-7200 machine and 38 µm for Test-Bed machine. These amplitudes are at maximum allowable by these machines without problem of deposited material sticking onto the sonotrode surface. One welding pass ran at lower normal force (4000 N), and another welding pass ran at higher normal force (8000 N for Test-Bed machine and 5340 N for SonicLayer-7200 machine). It is noted that the normal forces of 4000 N, 5340 N, and 8000 N are more than twice larger than the maximum normal force of 1500 N possible in UAM machine. Thus, normal force greater than 8000 N in Test-Bed machine and 5340 N in SonicLayer-7200 machine was not evaluated in this study. During VHP-UAM, it is noted that the amplitude was input as the percentage of maximum amplitude the machine capable of produce at its highest power. Appendix A provides the calibration curve between the input amplitude in percentage and the measured amplitude in µm as listed in Table 4.1.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Number of Layers</th>
<th>Vibration amplitude (µm)</th>
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<td>28</td>
<td>5340</td>
</tr>
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<td>TB-10-38-4000</td>
<td>10</td>
<td>38</td>
<td>4000</td>
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<tr>
<td>TB-10-38-8000</td>
<td>10</td>
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<td>SL-66-28-5340</td>
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<td>28</td>
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<tr>
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</tr>
<tr>
<td>SL-80-34-5340</td>
<td>80</td>
<td>34</td>
<td>5340</td>
</tr>
</tbody>
</table>

Table 4.1. All Al3003-H18 VHP-UAM samples with sample ID, number of layers, vibration amplitude, and normal force used to fabricate the samples

To prepare heat-treated or annealed samples, one small piece per sample was obtained by sectioning through the ND-TD plane for the three Test-Bed samples as well
as the as-received Al3003-H18 foil for comparison (see Figure 4.4). Each sample was heated in the Lindberg Blue Tube furnace in an argon atmosphere at 343°C for 2 hours, followed by furnace cooling to room temperature.

Figure 4.4. Schematics of machined Al3003-H18 VHP-UAM and foil stack specimens for neutron diffraction experiment using HIPPO diffractometer

Standard metallography technique was used to prepare specimen for optical microscopy, scanning electron microscopy (SEM), and hardness measurement. Sectioning of samples was performed using water-cooled abrasive saw on Leco CM-15 followed by diamond blade on Leco VC-50 or Allied Tech 5 to get ND-TD cross section. Samples were mounted in the Leco Bakelite thermoset powder or Buehler Konductomet conductive mounting powder. For original tape, stacks of cut tape were clamped using SampKlip specimen support springs before mounting. Samples were ground and coarse polished sequentially with 400, 600, and 800 grit SiC papers in water, followed by 1200...
grit SiC paper in ethanol to minimize surface pitting. Samples were polished using 3 µm, and then 1 µm Leco Microid diamond compound on the Lecloth polishing cloth with Leco Microid diamond compound extender as lubricant. Cleaning was performed using an ultrasonic bath in isopropanol between polishing steps. Final polishing was performed using vibratory polishing with 0.05 µm Buehler MasterMet-2 non-crystallizing colloidal silica on Buehler MaterTex pad for 12 hours to achieve deformation-free surface necessary for EBSD analysis. After final polishing, samples were carefully rinsed with deionized water followed by ultrasonic cleaned in isopropanol for 2 minutes and dried with hot air.

Figure 4.4 displays the schematic of Al3003-H18 samples prepared for neutron diffraction experiment using the SonicLayer-7200 machine. The VHP-UAM samples were machined into rectangular blocks with the cross section area on the RD-TD plane of 10.25 × 10.25 mm² with the thickness of 9.6 mm from the top surface using the wire electro discharge machining (EDM) process. This 10.25 × 10.25 × 9.6 mm³ sample was referred to as the ‘whole sample’. In addition to the ‘whole sample’, the VHP-UAM samples were also machined into thinner ‘top layers’ sample and ‘bottom layers’ sample with dimensions of 10.25 × 10.25 × 5.4 mm³ for ‘top layers’ sample and 10.25 × 10.25 × 4 mm³ for ‘bottom layers’ sample. Similarly, the stacks of the as-received Al3003-H18 tape were EDM machined into 10.25× 50 mm² rectangular shape with two 10 mm holes positioned at 10 mm away from the length side (the tape stacks were tightened and held together using bolts and nuts). All the samples were prepared such that the incident neutron beam would hit the sample on the top surface, i.e. RD-TD plane.
4.4 Microhardness Measurement

Microhardness measurements were performed using a pyramidal Vickers indenter on a fully automated Leco AMH-43 system. The load range was set at 10 and 25 g and the spacing between each indent was typically 100-150 µm. Vicker diamond indenters was used to create arrays of 20 indents along the middle area of each deposited foil layer in order to create a hardness map that represents the hardness distribution of each Al3003-H18 VHP-UAM sample. In this study, microhardness measurements were performed on all samples including as-received foil and heat-treated foil as well as as-processed and heat-treated VHP-UAM samples fabricated from Test-Bed machine and SonicLayer-7200 machine.

4.5 Microstructure Characterization

4.5.1 Light Optical Microscopy

Light Optical Microscopy was performed using an Olympus GX-51 microscope with a 12MP Olympus DP71 digital imaging system. This microscope has magnification ranging from 12.5X up to 1000X. Several images were taken along the interfaces of VHP-UAM samples to calculate the values of linear weld density which is the ratio of the bonded interface over the entire interface length [33]. In addition, the measurements of the final build heights were performed to determine the average reduction in thickness of the foil after VHP-UAM processing.
4.5.2 Scanning Electron Microscopy (SEM)

SEM was performed on three different electron microscopes including FEI Quanta 200 SEM, FEI/Philips XL-30 Field Emission ESEM, and FEI/Philips Sirion Field Emission SEM. All scanning electron microscopes are equipped with both secondary electron (SE) detector and solid-state back-scattered electron (BSE) detector and have capability to perform X-ray energy dispersive spectroscopy (XEDS), which can be used to perform chemical composition analysis. In addition to imaging, FEI Philips XL-30 FE SEM was also used to perform electron backscattered diffraction (EBSD) analysis.

4.5.3 Electron Backscattered Diffraction (EBSD)

Electron backscatter diffraction (EBSD) or Orientation Imaging Microscopy (OIM) is used to gather crystallographic orientation information of the materials from Kikuchi diffraction patterns, which are generated by the diffraction of backscattered electrons. EBSD was used to analyze both microstructural data and micro-texture data of as-processed and heat-treated Al3003-H18 VHP-UAM samples as compared to original Al3003-H18 foil. In this work, EBSD was performed using the FEI/Philips XL-30 FE SEM. The data were collected at the step sizes ranging from 0.2-1 µm using TSL OIM DC software. During EBSD, the SEM was set up at 15-20 kV, 4-5 for the spot size, 200X magnification, and 22-26 mm working distance. Data analyses were performed using EDAX TSL OIM v5 software. The data analyzed include grain orientation, grain shape, grain size, misorientations, types of boundaries, and crystallographic texture. To obtain good results, data were cleaned using grain dilation and neighbor CI correlation
methods. In this study, 5 degree or higher misorientation was used to define the grain boundary between adjacent grains or subgrains.

4.6 Texture Measurement using Time-of-Flight Neutron Diffraction

Neutron diffraction experiment was performed using the neutron time-of-flight (TOF) the high pressure preferred orientation or HIPPO diffractometer at the Los Alamos Neutron Science Center (LANSCE) [125, 126]. The macro-texture was obtained in the form of an orientation distribution function (ODF), which is extracted from multiple neutron TOF histograms using Rietveld analysis [127]. This technique allows the measurement of the bulk texture or crystallographic orientations of the overall grains in the VHP-UAM samples as opposed to the localized or micro-texture obtained from EBSD.

Figure 4.5 illustrates the HIPPO instrument at LANSCE. HIPPO has a 9 meter incident flight-path using the high-flux neutron moderator of a pulsed spallation neutron source available at LANSCE. The beam spot sizes are controlled by an adjustable rotating collimator. The detector banks are composed of 1360 Helium tubes on five conical rings or detector banks in the three-dimensional arrangement. The scattering angle in HIPPO ranges from $2\theta = 10^\circ$ to $150^\circ$ around the incident beam direction [125]. In this study, the five detector banks were slightly modified from Figure 4.5 with the locations of detector panels collecting diffraction data at $2\theta = 150^\circ$, $120^\circ$, $90^\circ$, $60^\circ$, and $40^\circ$. To increase the coverage on the pole figures, the sample was rotated at three
different angles of $\omega = 0^\circ$, $67.5^\circ$, and $90^\circ$. The beam diameter was 10 mm. The total time of 15 minutes were used to collect data at each angular position.

Figure 4.5. Schematic of HIPPO diffractometer at LANSCE [125]

For in-situ texture measurement, the sample was glued to the sample holder and insert vertically downward into the high temperature vacuum furnace (1800°C ILL furnace), which can be installed into the large sample chamber [126]. The sample was wrapped and shielded with vanadium foil, which helps reduce the contamination of the diffraction peaks so that only peaks diffracted from the sample were displayed in the spectra [128]. The annealing experiment was performed at 343°C for 2 hours with
samples rotated with respect to the vertical axis at three different angular positions of $\omega = -45^\circ$, $22.5^\circ$, and $45^\circ$ for better quality of the pole figure.

Rietveld refinement using the E-WIMV method available in the Materials Analysis Using Diffraction (MAUD) was used to fit neutron diffraction patterns simultaneously [127, 129]. The orientation distribution functions and the pole figures were determined for each sample. The data were exported into MTEX to adjust the alignment and then to generate the ODF, pole figures, inverse pole figures, and fiber plots for macro-texture analysis in this study [130, 131].
Chapter 5: Effects of Process Parameters on Hardness, Microstructure, and Texture

5.1 Introduction

This chapter explains the changes in hardness, microstructure, and texture in the bulk of Al3003-H18 VHP-UAM samples due to the changes in vibration amplitude and normal force. The chapter begins with the hardness results of each layer in as-processed Al3003-H18 VHP-UAM samples compared to as-received foil and the description of ultrasonic power data measured during VHP-UAM processing. The chapter also explains how the hardness results of each layer are correlated to the average power used to weld each layer. The chapter then discusses the effects of vibration amplitude and normal force on the changes in the microstructure and texture from the original as-received foil. These changes include the microstructure changes and the texture changes at the interface regions (the region of 15-20 µm below the bonded interface undergone plastic deformation at the asperity level) and the bulk regions (the region of unaffected or partially affected from plastic deformation below the interface region) of one selected layer in both the as-processed and the heat-treated conditions. This chapter ends with the emphasis on the implication and the significance of the current results on the contribution of the knowledge for UAM and VHP-UAM research area.
5.2 Effects of Process Parameters on Hardness

5.2.1 Hardness Results

Figure 5.1 plots the average Vicker hardness data for each layer of VHP-UAM samples fabricated from the Test-Bed machine with the first layer representing the bottom-most layer or the first layer deposited on the substrate, and the tenth layer representing the top-most layer or the last layer deposited. The reference value of the average hardness of the as-received Al3003-H18 foil of 70.5 VHN is also plotted as dashed line in Figure 5.1. The plot displays the average hardness values both before and after heat-treatment at 343°C for 2 hours. It is found that the overall hardness values of the 28 µm (lower) vibration amplitude sample TB-10-28-5340 are almost equivalent to the hardness of the as-received Al3003-H18 foil. However, the hardness values of the 38 µm (higher) vibration amplitude samples TB-10-38-4000 and TB-10-38-8000 are 10-18 VHN lower than the as-received Al3003-H18 foil. Between the 38 µm vibration amplitude samples, the hardness of the sample with the 8000 N (higher) normal force (TB-10-38-8000) is slightly larger than the sample with the 4000 N (lower) normal force (TB-10-38-4000), although the normal force is double.

The average hardness of sample TB-10-28-5340 tends to decrease from the first (bottom) layer to the last or the 10th (top) layer (bottom-up) of the sample. In contrast, the average hardness of sample TB-10-38-8000 tends to increase from bottom-up of the sample. However, this trend of decreasing or increasing in the average hardness from bottom-up of the VHP-UAM build is not observed in sample TB-10-38-4000. It is believed that the hardening behavior takes place in the bulk of the bottom layers of
Figure 5.1. Plot of the average Vicker hardness measured at the middle of the bulk region of each deposited layer in three Al3003-H18 VHP-UAM samples fabricated from the Test-Bed machine compared to the average original Al3003-H18 tape hardness sample TB-10-28-5340 as they undergo additional deformation due to large compressive normal force during several thermo-mechanical cycles. This result of the hardening behavior is similar to those reported in the bulk microstructure of Al3003-H18 UAM
samples fabricated using the lower power (lower vibration amplitude and lower force) machine [49]. However, the average hardness decreases more in the bottom layers of sample TB-10-38-8000 as compared to the top layers. This indicates that the bulk microstructure in the bottom layers undergoes more cycles of heating and annealing phenomena (recovery and recrystallization), and thus leads to lower hardness. For TB-10-38-4000, it is speculated that the hardening behavior and softening behavior in the bulk during VHP-UAM processing balances each other resulting in neither increasing nor decreasing in the average hardness values bottom-up the sample. Other researchers also reported transient temperature rise at the bottom interfaces, while another layer is deposited during VHP-UAM [65]. This suggests that the temperature rise in subsequent passes plays a significant role in decreasing hardness and alters the microstructure in the bottom layers of Al3003-H18 VHP-UAM samples. As vibration amplitude increases, there may be more accumulated dislocation contents and more stored energy, which is the driving force for dynamic recovery (DRV) and dynamic recrystallization (DRX). The increasing temperature together with increasing stored energy result in softening behavior, which is supported by a decrease in the hardness.

After heat-treatment at 343°C for 2 hours, the overall average hardness values of all VHP-UAM samples and original foil drop closed to 40 VHN. The difference between the lowest and the highest hardness values among these heat-treated samples is only 3 VHN. Hence, there is no significant difference between the average hardness of heat-treated Al3003-H18 VHP-UAM samples and heat-treated original foil. There is also no
difference in the average hardness in different layers of all heat-treated samples, and every sample possesses the lowest hardness values in annealed condition.

Figure 5.2 illustrates the hardness maps of Al3003-H18 VHP-UAM samples fabricated from the SonicLayer-7200 machine. The maps were generated from the arrays of Vicker indents at the middle areas of each deposited layer with 20 indents from left to right of each layer. The legend displaying the range or the spectrum of the hardness values was automatically selected based on the minimum and the maximum hardness values of each sample. It was found that the higher hardness regions are concentrated in the top portion of the samples, whereas the lower hardness regions reside in the bottom portion of the samples. This makes sense because the welding speed was higher (42.7 mm/s) during the welding of the 51st and subsequent layers as compared to lower welding speed of 35.6 mm/s used in the first 50 layers. It was also observed that VHP-UAM sample SL-66-28-5340 processed with vibration amplitude of 28 µm showed more uniform hardness distribution from bottom-up. This means very high vibration amplitude of 34 µm results in larger accumulated heating and thus more softening in the bottom layers as showed in low hardness in the bottom parts of the samples. Furthermore, the hardness values in the map seems to be slightly lower towards the left-hand side at the same layer, as the area of the minimum hardness are located near the bottom left corner of each sample. This could be explained by the uneven compressive force pressing on the top surface and dragging during welding resulting in different hardness distribution from left to right of each sample.
Figure 5.2. Hardness map obtained from Vicker hardness measurements at the middle of each layer in Al3003-H18 VHP-UAM samples fabricated using the SonicLayer 7200 machine
Figure 5.3 plots the average hardness values for each layer of the three VHP-UAM samples processed with the SonicLayer-7200 machine similar to the plot in Figure 5.1. The plot intends to reveal the trend of hardness distribution from bottom-up in VHP-UAM samples. Although the average hardness values oscillate from one layer to the next, there exists the overall trend of increasing in hardness from bottom-up of the VHP-UAM samples processed at 34 µm vibration amplitude but not in sample SL-66-28-5340. In addition, the overall hardness values are lower in the VHP-UAM samples processed with the SonicLayer-7200 machine as compared to those processed with the Test-Bed machine. To be more precise, the overall hardness values of sample SL-66-28-5340 is significantly lower than those of sample TB-10-28-5340 despite similar processing parameters used aside from different build heights and different machines. It is also observed that at the layer above the 50th layer, where the weld speed was changed from 35.6 mm/s to 42.7 mm/s, the hardness of the three VHP-UAM samples are closer to one another and remain relatively constant. The average hardness values in the 34 µm (higher) amplitude samples SL-80-34-4000 and SL-80-34-5340 also seem to saturate at the values between 55-60 VHN, which is the same hardness range of the 28 µm (lower) amplitude sample SL-66-28-5340. It is also interesting to note that there is no significant change in hardness at higher normal force in sample SL-80-34-5340 as compared to sample SL-80-34-4000. It is hypothesized that the increasing weld speed in the last 30 layers causes the adiabatic heating to increase at the bottom layers, i.e. less time for heat to dissipate, and thus increase the amount of softening effect in the bottom layers. The higher softening behavior in VHP-UAM samples made from the SonicLayer-7200
machine leads to the lower hardness in the bulk of Al3003-H18 layers in the as-processed condition, compared to samples made from the Test-Bed machine.

![Graph showing hardness vs. layer number](image)

Figure 5.3. Plot of the average Vicker hardness measured at the middle of the bulk region of each deposited layer in three Al3003-H18 VHP-UAM samples fabricated from the SonicLayer 7200 machine

5.2.2 Relationship between Process Parameters and Ultrasonic Power

During UAM and VHP-UAM, when the levels of vibration amplitude and normal force are higher, the measured electrical power drawn from the UAM and VHP-UAM machines rises [65]. The electrical system in UAM and VHP-UAM machines needs sufficient power level to maintain the constant input vibration amplitude and normal force during one welding pass. The power levels as a function of time during the VHP-
UAM of each layer using the Test-Bed machine are plotted in Figure 5.4. It can be seen from the figure that the overall average power values are almost the same for the two 38 µm vibration amplitude samples, i.e. 1188.61 W for TB-10-38-4000 (see Figure 5.4 (b)) and 1234.01 W for TB-10-38-8000 (see Figure 5.4 (c)). Thus, unlike vibration amplitude, the difference in normal force has little effect on the amount of power required as the average power drawn increases only 3.8% while the normal force is double as large in sample TB-10-38-8000 as compared to sample TB-10-38-4000. The average power used to make sample TB-10-28-5340 is only 549.51 W (see Figure 5.4 (a)), which is significantly less than the other two samples. This confirms that the vibration amplitude greatly affect the values of power drawn from UAM and VHP-UAM systems. Furthermore, the power versus time curve also seem to possess some periodic or sinusoidal behavior, as the curves for each layer seem to superimpose well with one another especially when the vibration amplitude is lower in sample TB-10-28-5340. It is believed that this cyclic behavior of the power versus time curve is related to the natural frequency or vibration of the whole VHP-UAM system. Figure 5.5 shows the average power levels of each layer as obtained from Figure 5.4 for the three VHP-UAM samples fabricated by the Test-Bed machine. It is found that the power required for vibration amplitude of 28 µm in sample TB-10-28-5340 is nearly half of the power required for vibration amplitude of 38 µm in samples TB-10-38-4000 and TB-10-38-8000. It can also be seen that the required power seems to decrease gradually from bottom-up.
Figure 5.4. Plot of ultrasonic power versus time during VHP-UAM processing of Al3003-H18 samples using Test-Bed machine
Figure 5.5. Average ultrasonic power used to weld each layer during VHP-UAM of Al3003-H18 using Test-Bed machine

The average power levels consumed during the fabrication of each layer of VHP-UAM samples using the SonicLayer-7200 machine are displayed in Figure 5.6. Figure 5.6 (a), (b), and (c) plot both the scattered data (instantaneous required power used to maintain both vibration amplitude and normal force during VHP-UAM processing) and the average values of the power drawn at each layer from the first layer (bottom most) to the last layer (top most). The average power levels are plotted again in Figure 5.6 (d) for better comparison among the samples. From Figure 5.6 (d), there is slight difference
between the power levels used to fabricate samples SL-80-34-4000 and SL-80-34-5340, where normal force is different by 1340 N, but large difference between samples SL-66-28-5340 and SL-80-34-5340, where vibration amplitude is different by 6 µm. Therefore, the vibration amplitude has large influence on the power level, whereas the normal force has significantly less effect. In addition, the power levels show exponential decays with increasing numbers of layers or increasing build height. Above the 50th layer when the weld speed increases from 35.6 mm/s to 42.7 mm/s, the power levels remain relatively steady with the exception of sample SL-80-34-5340 where power seems to increase slightly with increasing build height, i.e. the average power in the 80th layer is 9% higher than the average power in the 50th layer.

5.2.3 Correlation between Ultrasonic Power and Hardness Results

The previous two sections discussed the resulting hardness measured in the bulk of each deposited layers in the VHP-UAM samples and the power level that VHP-UAM required to maintain the input process parameters. This section discusses the correlation between the average ultrasonic power used to weld each layer and the average hardness of the same layer. Figure 5.7 plots the average hardness versus the ultrasonic power used in the first 50 layers of samples fabricated from the SonicLayer-7200 machine and the 10 layers of the samples fabricated from the Test-Bed machine, all of which are welded at a constant speed of 35.6 mm/s. Note that the data for the subsequent layers after the 50th layers in the samples fabricated using the SonicLayer-7200 machine were not displayed here. This is because the weld speed was increased, and the focus of this analysis is on
Figure 5.6. Scatter plot of ultrasonic power with the average ultrasonic power used to weld each layer during VHP-UAM of Al3003-H18 using SonicLayer-7200 machine
the variation of vibration amplitude and normal force at constant weld speed of 35.6 mm/s. At the similar power levels, it was found that the hardness values of the VHP-UAM samples processed with the Test-Bed machine are larger than those processed with the SonicLayer-7200 machine. In addition, Figure 5.7 also shows lower hardness or larger softening when larger vibration amplitude was used. It is obvious that for a given machine, the values of the hardness in the bulk region of the as-processed VHP-UAM samples decrease with increasing ultrasonic power.

However, the accumulative effects must also be considered. The total softening in the bottom layers is due to the results of several thermo-mechanical cycles during VHP-UAM. The current result showing decreasing in hardness at higher power level implies that the power level greatly influence the softening behavior of Al3003-H18 foil after VHP-UAM. It is believed that the accumulative thermo-mechanical cycles during the latter passes also cause the softening behavior in Al3003-H18 foil after VHP-UAM but does not significantly affect the power level. Sriraman et al. reported the peak temperature at the interface between bonded layers decreases with increasing build height due to the decreasing amount of ultrasonic energy going into the sample [65]. This agrees well with the current result where ultrasonic power decreases with increasing build height. Thus, with increasing build height, the amount of ultrasonic energy going into the system is less because less power is required at higher build height. In addition, the vibration of the whole system during VHP-UAM when the ultrasonic sonotrode is in contact with the samples with different build height could also affect the variation in power values. The modes of vibration as well as the mode of stick and slip between the
Figure 5.7. Correlation between average hardness and average ultrasonic power used to weld Al3003-H18 tape at constant weld speed of 35.6 mm/s using Test-Bed machine and SonicLayer-7200 machine

sonotrode surface and top foil surface could play a critical role in determining the amount of ultrasonic energy, which is dissipated and is going into the bonded interface [14, 15, 62]. This may explain why variations of hardness values between the two machines exist even though the power levels are similar as showed in Figure 5.7.
5.3 Effects of Process Parameters on Microstructure and Texture

5.3.1 Microstructure and Texture of As-Received Al3003-H18 Foil

The microstructure of the as-received Al3003-H18 foil was characterized using SEM and EBSD. Figure 5.8 displays the ND-TD and ND-RD cross-section micrographs of the original foil sample revealing two phases: aluminum matrix and secondary intermetallic Al₆(Mn,Fe) particles. The shape of the intermetallic phase is ellipsoid/cylindrical shape (equiaxed in ND-TD plane and rectangular in ND-RD plane). The result from the image analysis using ImageJ software with binary filter reveals 4-6% volume fraction of the intermetallic particle with the average equivalent diameter of 1.75 µm in the ND-TD plane and 2.43 µm in the ND-RD plane (the difference in average diameter is due to the analysis in different planes).

Figure 5.9 shows the inverse pole figure map (ND-TD plane) of the as-received Al3003-H18 tape analyzed with EBSD. The aluminum matrix is represented by the colored grains with the intermetallic particles represented by the black particles. The volume fraction of the intermetallic particle was found to be 5.2% with the average particle diameter of 0.6 µm and the maximum particle diameter of 3.2 µm. In the matrix, the aluminum grains are mostly elongated along the rolling direction of the foil. The color representation indicates that most grains have crystallographic plane near \(\{111\}\)-pole with blue-purple shade colors. Other random orientations of aluminum grains are found mostly around the large intermetallic particles. The aluminum grains in the matrix have equiaxed shapes formed by particle stimulated nucleation during fabrication of Al3003-H18 foil in as-received condition [132].
Figure 5.8. SEM micrographs of as-received aluminum 3003-H18 foil: (a) ND-TD plane, (b) ND-RD plane
Figure 5.9. Inverse pole figure map of as-received Al3003-H18 foil

Figure 5.10 displays the map of grain boundaries and grain orientation spread (GOS) of the as-received Al3003-H18 foil. It is clearly shown that the aluminum grains are elongated along the TD direction. There are two kinds of grain boundaries analyzed: the low angle grain boundary (LAGB) with the misorientation angle between 2 and 15 degree (grey line) and the high angle grain boundary (HAGB) with the misorientation angle larger than 15 degree (black line). The volume fraction of recrystallized grains is calculated using the GOS data. The pink-shaded grains represent the recrystallized grains with GOS of 3 degree or lower whereas the white grains represent the non-recrystallized grains with GOS larger than 3 degree. The GOS less than 3 degree means the average misorientation within a single grain is less than 3 degree which is selected as acceptable criteria for analyzing recrystallized grains, which are dislocation-free grains [133]. The
quantitative results show the average grain size of aluminum of 2.083 µm, the fraction of LAGB of 0.211, the fraction of HAGB of 0.789, and the fraction of recrystallized grains of 0.612. These values are used to compare the change in the microstructure of Al3003-H18 after VHP-UAM processing.

Figure 5.10. EBSD map of low angle grain boundaries and high angle grain boundaries in conjunction with the map of recrystallized grains and non-recrystallized grains of as-received Al3003-H18 foil

Figure 5.11 shows the map of major FCC rolling texture components in the as-received Al3003-H18 tape. In this study, five texture components are analyzed including copper, S3, brass, Goss, and rotated cube components [134]. The volume fraction of each texture component was calculated based on the 15 degree radius allowance from the ideal position of each texture component (0.216 for copper, 0.339 for S3, 0.149 for brass,
0.011 for Goss, 0.004 for rotated cube, and 0.281 for other orientations). It can be seen that most of the grains have orientations along the copper, S3, and brass components, all of which lie along the $\beta$-fiber (one of the major texture fibers in rolled FCC metal). Figure 5.12 displays the inverse pole figures in the ND, RD, and TD directions of the as-received Al3003-H18 sample. The $\beta$-fiber can be clearly seen on the ND and the TD inverse pole figures running from copper through S3 and brass components respectively. Figure 5.13 shows the $\{111\}$-pole figure of the as-received Al3003-H18 tape. The result matches well with the typical $\{111\}$-pole figure of FCC metal in the rolled condition [75]. The results of texture components, inverse pole figure, and $\{111\}$-pole figures described here is used as reference data for determining the change in texture in as-processed Al3003-H18 VHP-UAM samples both at room temperature and after post-processing heat-treatment. The same information is used to compare the data obtained from the bulk texture measurement using neutron diffraction discussed in Chapter 6.

5.3.2 Microstructure and Texture of As-Processed VHP-UAM Samples

In this section, the microstructure and texture of Al3003-H18 VHP-UAM samples processed by the Test-Bed machine are discussed. Figure 5.14 displays the optical micrographs of VHP-UAM samples showing the voids or un-bonded regions along the interfaces. It is noticed that the voids or the pores are elongated along the TD direction with relatively flat surface on the top (top layer’s bottom surface) and curved surface on the bottom (bottom layer’s top surface). The values of linear weld density were
Figure 5.11. EBSD map of major rolling texture components of FCC metals in as-received Al3003-H18 foil calculated from several similar optical images taken from different locations in the samples. The results show the values of linear weld density of approximately 98% in sample TB-10-28-5340 and more than 99% in samples TB-10-38-4000 and TB-1-38-8000. The value of near 100% linear weld density, which is much higher than 55-85% linear weld density reported in Al3003-H18 samples processed with the lower power UAM [20], indicates that the bond quality does improve significantly with increasing ultrasonic power.
Figure 5.12. Inverse pole figures of as-received Al3003-H18 foil

Figure 5.13. \{111\}-pole figure of as-received Al3003-H18 foil
In addition to linear weld density, Table 5.1 lists the average reduction in thickness of the 150 µm thick tape after being VHP-UAM processed, as calculated from the optical micrographs of the VHP-UAM samples made using Test-Bed machine and SonicLayer-7200 machine. It was found that the average reduction in thickness is as large as 10% in sample TB-10-38-8000, where both vibration amplitude and normal force are relatively large as compared to only 2% reduction in samples TB-10-28-5340 and TB-10-38-4000. It is speculated that most plastic deformation takes place at the interface region, i.e. 15-20 µm below the interface except in sample TB-10-38-8000, where the bulk region below the interface region also undergoes the plastic deformation due to high normal force of 8000 N. The results from the SonicLayer-7200 machine reveal that the average reduction in thickness increases with increasing vibration amplitude and increasing normal force, as the values of sample SL-80-34-5340 is greater than sample SL-80-34-4000 and SL-66-28-5340 respectively.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Average Reduction in Thickness (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TB-10-28-5340</td>
<td>2%</td>
</tr>
<tr>
<td>TB-10-38-4000</td>
<td>2%</td>
</tr>
<tr>
<td>TB-10-38-8000</td>
<td>10%</td>
</tr>
<tr>
<td>SL-66-28-5340</td>
<td>4%</td>
</tr>
<tr>
<td>SL-80-34-4000</td>
<td>5%</td>
</tr>
<tr>
<td>SL-80-34-5340</td>
<td>6%</td>
</tr>
</tbody>
</table>

Table 5.1. Average reduction in thickness in percentage of aluminum 3003-H18 VHP-UAM samples

Figure 5.15 (a) shows the SEM micrograph of the microstructure of Al3003-H18 VHP-UAM sample TB-10-38-8000. The selected interface region that undergoes large plastic deformation and become wavy contains more condensed of fine intermetallic particles $\text{Al}_6(\text{Mn,Fe})$ as compared to the bulk region. It was observed that the particle sizes are larger and spread wider in the bulk regions as compared to the interface regions. This indicates possible break-up of intermetallic particles during severe plastic deformation and the smaller break-up intermetallic particles highly cluster in certain location along the interfaces. However, the clusters of intermetallic particles may not be so obvious in the region, where plastic deformation at the asperities along the interface is relatively small as displayed in Figure 5.15 (b).

Figure 5.16 illustrates the inverse pole figure maps showing the microstructure along the ND-TD planes of three VHP-UAM samples made from Test-Bed machine from top to bottom interfaces of the second layer from the top or the ninth layer deposited. The top and bottom arrows indicate the location of the bonded interfaces. The microstructures at and below the interface regions composes of the transition from the elongated and pancake-like structure (with colors on the spectrum around the $\{111\}$ corner belonging to the $\beta$-fiber in the legend) to the tiny and equiaxed grains (with
Figure 5.15. SEM micrographs showing the interface morphologies of as-processed Al3003-H18 VHP-UAM sample TB-10-38-8000: (a) wavy interfaces, (b) smooth interfaces (Arrow marks the cluster of break-up intermetallic particles)
random orientation, i.e. different colors of grains). The interface region refers to the area, in which grains are equiaxed and below which elongated grains similar to original as-received microstructure exist. The rest of the microstructure within the layer and below the interface region is called the bulk region.

Figure 5.17 shows the map of rolling texture components of VHP-UAM samples corresponding to Figure 5.16. It can be seen that the rolling texture components (copper, S3, brass, Goss) remain and dominate the bulk texture but not the interface region. At the interface region, the former elongated grains with rolling texture component transforms to fine and equiaxed grains with other random orientations such as recrystallized textures and shear textures aside from original rolling textures. In sample TB-10-28-5340, which is processed with low vibration amplitude, the change in texture components is limited to the small interface regions. In sample TB-10-38-4000, which is processed with high vibration amplitude and lower normal force, there exists large amount of rotated cube component \{111\}\{11\overline{2}\} in grains at the interface regions. This rotated-cube texture component has \{111\} plane parallel to rolling plane and has shear vector in (11\overline{2}) direction. This means the new grains with the rotated-cube texture component undergoes some kinds of shear deformation and are not present in the as-received foil before VHP-UAM processed or in the bulk region where shear deformation does not take place. In addition, the overall volume fraction of the overall rolling texture components also seems smaller in the upper portion of the bulk region as compared to the bottom portion (see Table 5.2). This gradient in the rolling texture components in the bulk region is very
distinct and almost depleted in the upper portion of the bulk region in sample TB-10-38-8000, which is processed with high vibration amplitude and larger normal force.

Figure 5.16. Inverse pole figure maps of as-processed Al3003-H18 VHP-UAM samples across one layer: (a) TB-10-28-5340, (b) TB-10-38-8000, and (c) TB-10-38-4000
In order to analyze, three different regions were selected for quantitative analysis of the variation in volume fractions of each texture components across the 9th layer (from the bottom) of the VHP-UAM samples. These three regions include the interface region, the middle bulk region (selected area from middle portion of the layer), and the bottom bulk region (selected area from the bottom portion of the layer). Table 5.2 details the
volume fraction of each texture component of different regions in three VHP-UAM samples compared to the original as-received foil. The table also includes the overall texture component of the entire selected layer including the interface region and the entire bulk region. It can be seen that the overall volume fraction of each texture component in sample TB-10-28-5340 is the closest to the original foil, in particular the middle bulk and the bottom bulk regions. This agrees with the inverse pole figure map showing the grain orientation and microstructure in Figure 5.16, in which the bulk microstructure looks almost identical to the original foil.

<table>
<thead>
<tr>
<th>Sample</th>
<th>copper</th>
<th>S3</th>
<th>brass</th>
<th>Goss</th>
<th>Rotated cube</th>
</tr>
</thead>
<tbody>
<tr>
<td>Foil</td>
<td>0.216</td>
<td>0.339</td>
<td>0.149</td>
<td>0.011</td>
<td>0.004</td>
</tr>
<tr>
<td>TB-10-28-5340 -</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Whole layer</td>
<td>0.154</td>
<td>0.364</td>
<td>0.127</td>
<td>0.013</td>
<td>0.020</td>
</tr>
<tr>
<td>Interface</td>
<td>0.129</td>
<td>0.194</td>
<td>0.032</td>
<td>0.011</td>
<td>0.054</td>
</tr>
<tr>
<td>Middle bulk</td>
<td>0.205</td>
<td>0.461</td>
<td>0.113</td>
<td>0.002</td>
<td>0.004</td>
</tr>
<tr>
<td>Bottom bulk</td>
<td>0.198</td>
<td>0.409</td>
<td>0.150</td>
<td>0.008</td>
<td>0.001</td>
</tr>
<tr>
<td>TB-10-38-4000 -</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Whole layer</td>
<td>0.126</td>
<td>0.256</td>
<td>0.111</td>
<td>0.038</td>
<td>0.084</td>
</tr>
<tr>
<td>Interface</td>
<td>0.036</td>
<td>0.055</td>
<td>0.008</td>
<td>0.056</td>
<td>0.314</td>
</tr>
<tr>
<td>Middle bulk</td>
<td>0.112</td>
<td>0.230</td>
<td>0.078</td>
<td>0.081</td>
<td>0.028</td>
</tr>
<tr>
<td>Bottom bulk</td>
<td>0.161</td>
<td>0.374</td>
<td>0.109</td>
<td>0.012</td>
<td>0.037</td>
</tr>
<tr>
<td>TB-10-38-8000 -</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Whole layer</td>
<td>0.102</td>
<td>0.204</td>
<td>0.083</td>
<td>0.023</td>
<td>0.050</td>
</tr>
<tr>
<td>Interface</td>
<td>0.066</td>
<td>0.116</td>
<td>0.041</td>
<td>0.031</td>
<td>0.081</td>
</tr>
<tr>
<td>Middle bulk</td>
<td>0.062</td>
<td>0.120</td>
<td>0.026</td>
<td>0.034</td>
<td>0.103</td>
</tr>
<tr>
<td>Bottom bulk</td>
<td>0.184</td>
<td>0.358</td>
<td>0.117</td>
<td>0.012</td>
<td>0.031</td>
</tr>
</tbody>
</table>

Table 5.2. Volume fraction of rolling texture components in aluminum 3003-H18 foil and as-processed VHP-UAM samples obtained from EBSD

At the interface region, the volume fraction of copper, S3, and brass components significantly decrease from the original values in the as-received foil, while the volume fraction of the rotated cube component increase as expected. The results of the interface
region in samples TB-10-38-4000 and TB-10-38-8000 show the similar trends, where the volume fractions of copper, S3, and brass components decrease, while those of Goss and rotated cube components increase. The amount of change in volume fractions is the greatest in the interface region of sample TB-10-38-4000, which shows very large fraction of rotated cube component. Unlike sample TB-10-28-5340, the middle bulk regions of sample TB-10-38-4000 and TB-10-38-8000 show large drop in volume fractions of copper, S3, and brass components, especially in sample TB-10-38-4000. The Goss and rotated cube components are also higher in the bulk of both samples TB-10-38-4000 and TB-10-38-8000. However, in the bottom bulk regions of samples TB-10-38-4000 and TB-10-38-8000, the volume fractions of each texture component are the closet and not much different from the as-received foil.

Figure 5.18 details the \{111\} pole figures of ten smaller cross sections divided from top to bottom of the 9th layer of the VHP-UAM samples. The overall \{111\} pole figures of the entire area of the selected layer are also provided for comparison of the overall texture change from the as-received foil. The \{111\} pole figure results from top to bottom of each selected layer reveal the gradient in microstructure and texture exist as a result of VHP-UAM processing. In sample TB-10-28-5340, only the first or the top-most section displays the distinct \{111\} pole figure showing high intensity of shear texture \{111\}\textlangle uvw\textrangle where the microstructure is sheared along the \{111\} plane in the interface region. The remainders of the bulk possess the \{111\} pole figure similar to those of rolled as-received foil, i.e. rolled FCC metal. In sample TB-10-38-4000, the microstructure containing the shear texture covers two sections out of the ten sections in
the selected layer. Similarly, five sections out of ten sections in sample TB-10-38-8000 shows trait of shear deformation leaving only the bottom five sections with retained rolling texture. This result indicates that shear deformation in the bulk is larger with increasing vibration amplitude and increasing normal force. Further, away from the top interface, where plastic deformation is not significant, the microstructure retains most of the initial crystallographic rolling textures.

The quantitative EBSD analysis data including the average grain diameter, the volume fraction of recrystallized grains, the volume fraction of low angle grain boundaries, and the volume fraction of high angle grain boundaries are displayed in Table 5.3-5.7 respectively. Three different regions are selected for the quantitative analysis including the interface region, the middle bulk region, and the bottom bulk region. The interface region covers the first section from the ten sections in Figure 5.18. Similarly, the middle bulk region covers the fifth section from the top and the bottom bulk region covers the eighth section from the top.

The number average grain diameters and the area average grain diameters of as-received foil and different regions of VHP-UAM samples are displayed in Table 5.3 and Table 5.4 respectively. It is found that the number average grain sizes of VHP-UAM samples are larger than the as-received foil in all regions of all samples. In contrast, the area average grain sizes of the bulk of sample TB-10-28-5340 and all regions of sample TB-10-38-4000 are larger than the as-received foil, whereas the average grain sizes of the interface region of sample TB-10-28-5340 and every region of sample TB-10-38-8000 are smaller than the as-received foil. This result shows that there are some effects from
<table>
<thead>
<tr>
<th>Section in 9th layer</th>
<th>TB-10-28-5340</th>
<th>TB-10-38-8000</th>
<th>TB-10-38-4000</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st section from top</td>
<td>5.250</td>
<td>2.625</td>
<td>6.633</td>
</tr>
<tr>
<td>2nd section from top</td>
<td>4.953</td>
<td>4.847</td>
<td>7.159</td>
</tr>
<tr>
<td>3rd section from top</td>
<td>5.318</td>
<td>4.582</td>
<td>5.540</td>
</tr>
<tr>
<td>4th section from top</td>
<td>6.974</td>
<td>4.766</td>
<td>7.667</td>
</tr>
<tr>
<td>5th section from top</td>
<td>4.304</td>
<td>6.038</td>
<td>6.586</td>
</tr>
<tr>
<td>6th section from top</td>
<td>6.816</td>
<td>5.386</td>
<td>5.767</td>
</tr>
<tr>
<td>7th section from top</td>
<td>6.405</td>
<td>5.103</td>
<td>9.182</td>
</tr>
<tr>
<td>8th section from top</td>
<td>7.681</td>
<td>6.945</td>
<td>4.929</td>
</tr>
<tr>
<td>9th section from top</td>
<td>6.889</td>
<td>4.224</td>
<td>6.904</td>
</tr>
<tr>
<td>10th section from top</td>
<td>4.114</td>
<td>3.792</td>
<td>5.668</td>
</tr>
</tbody>
</table>

Figure 5.18. \{111\}-pole figures of ten smaller cross sections divided from top to bottom of the selected layer of the VHP-UAM samples with maximum texture strength in mrd
<table>
<thead>
<tr>
<th>Foil</th>
<th>TB-10-28-5340</th>
<th>TB-10-38-4000</th>
<th>TB-10-38-8000</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.833</td>
<td>Interface</td>
<td>1.187</td>
<td>1.314</td>
</tr>
<tr>
<td></td>
<td>Middle bulk</td>
<td>1.398</td>
<td>1.396</td>
</tr>
<tr>
<td></td>
<td>Bottom bulk</td>
<td>1.518</td>
<td>1.302</td>
</tr>
</tbody>
</table>

Table 5.3. Number average grain size (diameter in µm) of as-received aluminum 3003-H18 tape and as-processed VHP-UAM samples obtained from EBSD

<table>
<thead>
<tr>
<th>Foil</th>
<th>TB-10-28-5340</th>
<th>TB-10-38-4000</th>
<th>TB-10-38-8000</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.083</td>
<td>Interface</td>
<td>1.405</td>
<td>2.478</td>
</tr>
<tr>
<td></td>
<td>Middle bulk</td>
<td>2.321</td>
<td>2.224</td>
</tr>
<tr>
<td></td>
<td>Bottom bulk</td>
<td>2.732</td>
<td>2.107</td>
</tr>
</tbody>
</table>

Table 5.4. Area average grain size (diameter in µm) of as-received aluminum 3003-H18 tape and as-processed VHP-UAM samples obtained from EBSD

<table>
<thead>
<tr>
<th>Foil</th>
<th>TB-10-28-5340</th>
<th>TB-10-38-4000</th>
<th>TB-10-38-8000</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.612</td>
<td>Interface</td>
<td>0.884</td>
<td>0.896</td>
</tr>
<tr>
<td></td>
<td>Middle bulk</td>
<td>0.710</td>
<td>0.900</td>
</tr>
<tr>
<td></td>
<td>Bottom bulk</td>
<td>0.606</td>
<td>0.860</td>
</tr>
</tbody>
</table>

Table 5.5. Fraction of recrystallized grain of aluminum 3003-H18 tape and as-processed VHP-UAM samples obtained from grain orientation spread data in EBSD

<table>
<thead>
<tr>
<th>Foil</th>
<th>TB-10-28-5340</th>
<th>TB-10-38-4000</th>
<th>TB-10-38-8000</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.293</td>
<td>Interface</td>
<td>0.186</td>
<td>0.226</td>
</tr>
<tr>
<td></td>
<td>Middle bulk</td>
<td>0.229</td>
<td>0.198</td>
</tr>
<tr>
<td></td>
<td>Bottom bulk</td>
<td>0.268</td>
<td>0.232</td>
</tr>
</tbody>
</table>

Table 5.6. Fraction of low angle grain boundaries of aluminum 3003-H18 tape and as-processed VHP-UAM samples obtained from EBSD

<table>
<thead>
<tr>
<th>Foil</th>
<th>TB-10-28-5340</th>
<th>TB-10-38-4000</th>
<th>TB-10-38-8000</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.708</td>
<td>Interface</td>
<td>0.814</td>
<td>0.774</td>
</tr>
<tr>
<td></td>
<td>Middle bulk</td>
<td>0.771</td>
<td>0.802</td>
</tr>
<tr>
<td></td>
<td>Bottom bulk</td>
<td>0.732</td>
<td>0.768</td>
</tr>
</tbody>
</table>

Table 5.7. Fraction of high angle grain boundaries of aluminum 3003-H18 tape and as-processed VHP-UAM samples obtained from EBSD
higher normal force at higher vibration amplitude on the average grain size reduction in sample TB-10-38-8000. This is because the average grain size is larger when normal force is less, while the vibration amplitude is still high in sample TB-10-38-4000. It is also interesting to note that the average grain sizes in the interface region is smaller than those in the bulk regions except in sample TB-10-38-4000, where the interface region shows larger average grain size as compared to the bulk region due to high temperature rise during VHP-UAM processing at larger vibration amplitude.

The volume fractions of recrystallized grains, low angle grain boundaries, and high angle grain boundaries are provided in Table 5.5, Table 5.6, and Table 5.7 respectively. It can be seen that the bottom bulk region of sample TB-10-28-5340 is the closet to the as-received foil for all three categories analyzed here. In contrary, the interface region of sample TB-10-38-8000 has the largest difference from the as-received foil. In addition, sample TB-10-38-8000 also has the highest overall fraction of recrystallized grains, which can be explained by its highest fraction of high angle grain boundaries. It can also be noticed that the fraction of high angle grain boundaries at the interface region is the lowest in sample TB-10-38-4000 as compared to other two samples. This means less dynamic recrystallization occurs in the interface region, but more dynamic recovery by subgrain coalescence occurs in the interface region of sample TB-10-38-4000. This same result can also be illustrated in Figure 5.19, showing the maps of low angle and high angle grain boundaries along with the recrystallized and non-recrystallized grains of the interface regions of the three VHP-UAM samples. Most of the grain boundaries are high angle grain boundaries, which indicate the occurrence of
dynamic recrystallization due to severe plastic deformation. It is noted that the grains are
the finest in the interface region of sample TB-10-38-8000, and the largest in the
interface region of sample TB-10-38-4000. It is also observed that large portion of grains
in sample TB-10-38-4000 seem to merge or coalescence together, producing large
network of grains with similar orientation likely due to dynamic recovery. This explains
why there are more non-recrystallized grains in the interface region of sample TB-10-38-
4000 as compared to the other two samples.

5.3.3 Microstructure and Texture of Heat-Treated Condition

In previous section, the microstructure and texture of as-processed Al3003-H18
VHP-UAM samples compared to the as-received Al3003-H18 foil are discussed. The
microstructure and texture of the heat-treated foil is used to compare the degree of
microstructure and texture changes in the bulk region in VHP-UAM samples during heat-
treatment. This section discusses the effect of heat-treatment on VHP-UAM samples
made with different process parameters and how the microstructure and texture in the as-
processed condition contribute to the changes in microstructure and texture after heat-
treatment.

Figure 5.20 displays the inverse pole figure map revealing the equiaxed aluminum
grains with intermetallic particles Al₆(Mn,Fe) on the ND-TD plane of Al3003-H18 foil
after heat-treatment at 343°C for 2 hours. The number average grain diameter and the
area average grain diameter of aluminum grains are 6.24 μm and 10.61 μm respectively.
The average particle size of Al₆(Mn,Fe) is 1.93 μm. There seems to be no difference in
Figure 5.19. Low angle grain boundaries (grey lines) and high angle grain boundaries (black lines) map with grain orientation spread color map showing fraction of recrystallized grains (white area) versus non-recrystallized grains (pink area) of the interface region in Al3003-H18 VHP-UAM samples

the sizes and shape of intermetallic particles after heat-treatment, whereas the aluminum grains undergo recrystallization and grain growth, i.e. rolled and elongated grains in Figure 5.9 transformed into larger equiaxed grains in Figure 5.20.
Figure 5.20. Inverse pole figure map of Al3003-H18 foil after heat-treatment at 343°C for 2 hours

Figure 5.21 illustrates the map of texture components of heat-treated Al3003-H18 foil similar to Figure 5.11. It can be observed that the volume fractions of rolling texture components are much less compared to the as-received foil before heat treatment with 6.1% copper, 21.2% S3, 5.6% brass, 3.1% Goss, and 0.4% rotated cube. The relatively high fraction of high angle grain boundaries of 0.867 together with the larger fraction of random oriented grains indicates that the recrystallization is the dominant mechanism at this annealing temperature. Figure 5.22 and Figure 5.23 compares the inverse pole figures and the \{111\} pole figures of the Al3003-H18 foil after heat-treatment to the foil before heat-treatment. The results show weak intensity of the \(\beta\)-fiber, which goes through copper, S3, and brass components, still exists, but is much weaker than before heating. The decreasing maximum intensity in the \{111\} pole figure of the foil after
heat-treatment along with the trace of the original texture components confirms the retained but weaker rolling texture components.

Figure 5.21. Texture component map of heat-treated Al3003-H18 foil showing major rolling texture components found in FCC metal with color label indicating selected major texture components found in rolled FCC metals

Figure 5.24 illustrates the inverse pole figure maps of Al3003-H18 VHP-UAM samples after heat-treatment at 343°C for 2 hours. The locations of the interfaces above and below the selected layer are indicated by the arrows. It can be seen that the microstructure at the interface region after heat-treatment consists of small equiaxed grains with larger diameter than the fine and equiaxed grains found at the interface regions of the VHP-UAM samples in as-processed condition showed in Figure 5.16.
However, it is the bulk microstructures, which vary from sample to sample and are greatly affected by the different VHP-UAM processing parameters used. In sample TB-10-28-5340, where the microstructure change is mostly limited to the interface region, large grain growth occurs and the bulk region contains several grains with diameter larger than 10 µm. It is also found that the large grain growth rate in the bulk also consumes parts of the interface region leaving the area or the volume that define the interface region (with different microstructure from the bulk region) much smaller than the as-processed condition showed in Figure 5.16.

Figure 5.22. Inverse pole figures showing grain orientations in normal direction (ND), rolling direction/weld direction (RD), and transverse/vibrational direction (TD) of Al3003-H18 foil before and after heat-treatment at 343°C for 2 hours
Figure 5.23. {111}-pole figure of Al3003-H18 foil before (left) and after (right) heat-treatment at 343°C for 2 hours

In samples TB-10-38-4000 and TB-10-38-8000, where the larger vibration amplitude greatly affects the as-processed microstructure in the bulk region, large grain growth seems to be limited to smaller portion of the bulk region. As seen in Figure 5.24, very large grains exist above the top interfaces of the selected layers in both samples TB-10-38-4000 and TB-10-38-8000, even though large grain growth does not always occur above the bottom interface in the selected layer. To demonstrate this, it is observed in the studied area of sample TB-10-38-4000 that there is no large grain growth in the 9th layer, but there is very large grain in the 10th layer. In contrast to sample TB-10-38-4000, sample TB-10-38-8000 has very large grain in the second layer slightly above the bottom interface with cluster of small equiaxed grains surrounded, as well as very large grains present above the top interface. In addition to different grain growth rates in the bulk regions of VHP-UAM samples, the grains in the bulk regions of samples TB-10-38-4000 and TB-10-38-8000 also seem to possess the same or similar orientations as the grains in the same bulk regions in the as-processed condition. Thus, it is speculated here that
recrystallization is not the dominant mechanism in the microstructure change in the bulk region of these two samples during the selected heat-treatment, but rather the recovery. During recovery, subgrain coalescence takes place and the orientations of original grains are preserved. It is hypothesized that during VHP-UAM processing at very large vibration amplitude, the driving force for recrystallization, which is the stored energy, is
less than original Al3003-H18 foil or the bulk of VHP-UAM sample processed with lower vibration amplitude. When the driving force is lower, it would take more time or higher temperature to initiate the recrystallization, whereas the recovery can happen at lower temperature.

Figure 5.25 displays the inverse pole figure maps of the 10th layer adjacent to the top surface of Al3003-H18 VHP-UAM sample TB-10-28-5340 both before and after heat-treatment at 343°C for 2 hours. It can be seen that the grains, which are located next to the top surface and was in contact with the sonotrode texture, are fine and equiaxed. The region, which contains these fine and equiaxed grains, is 10-15 µm depth from the top surface. It is noted that the severe localized plastic deformation from the sonotrode results in higher stored energy. However, since dynamic recrystallization already occurs at the interface, the amount of stored energy decreases, and thus no static recrystallization occurs at the interface region like in the bulk region during heat-treatment. The results after heat-treatment reveal that the grains, which are located just below the top surface, grow at a slower rate compared to very large grains at some distance below the interface. The result, which shows fine and equiaxed grains remained below the top surface, agrees well with the sluggish grain growth occurs in the interface region in the other layers already deposited discussed in Figure 5.24. Similar results are also found where large grain growth occurs in the bulk regions of other layers deposited.

Figure 5.26 shows the maps of texture components with low angle grain boundaries and high angle grain boundaries of the three VHP-UAM builds made from the Test-Bed machine. It can be seen that most of the grains in sample TB-10-28-5340 have
Figure 5.25. The inverse pole figure maps of the material adjacent to the top surface of Al3003-H18 VHP-UAM sample TB-10-28-5340 (a) as-processed condition, and (b) after heat-treatment at 343°C for 2 hours

other random orientations aside from the original rolled textures. This result is different in samples TB-10-38-4000 and TB-10-38-8000, where most of the equiaxed grains in the bulk possess rolled texture components similar to the as-processed condition. In addition, it can also be seen that the rotated cube components, which dominate most of the interface region of the as-processed sample TB-10-38-4000, still exist in the same region after heat-treatment. This means that the microstructure and texture in sample TB-10-38-
4000 is relatively stable during heat-treatment compared to the as-processed condition. It is also found that the volume fraction of grains with brass texture component also increases in the bulk of the same sample.

Figure 5.26. Texture component maps of heat-treated Al3003-H18 VHP-UAM samples showing major rolling texture components found in FCC metal.
Table 5.8 lists the volume fraction of texture components in Al3003-H18 foil and VHP-UAM samples made from the Test-Bed machine after heat-treatment at 343°C for 2 hours. It is noted that the volume fractions of selected texture components in sample TB-10-28-5340 may not be accurate because the number of grains within the area is relatively small and does not provide a good statistical representation. In sample TB-10-38-4000, the results show high volume fraction of rotated cube component in the interface region and large increase in the volume fraction of brass component in the bulk compared to as-processed condition. In addition, the volume fractions of the overall rolling texture components are also higher in the bottom bulk regions of samples TB-10-38-4000 and TB-10-38-8000 as compared to the middle bulk regions. This implies that the grains with retained rolling texture component in the bulk regions of the as-processed condition in samples TB-10-38-4000 and TB-10-38-8000 are relatively stable and grow larger via subgrain coalescence during heat-treatment. This results in higher volume fraction of some rolling texture components, especially brass component in sample TB-10-38-4000, even after heat-treatment at 343°C for 2 hours.

Figure 5.27 illustrates the \{111\} pole figures of ten smaller sections of the selected layers and the \{111\} pole figures of the whole layers of Al3003-H18 VHP-UAM samples after heat-treatment at 343°C for 2 hours similar to those of as-processed VHP-UAM samples showed in Figure 5.18. It can be seen that the section analysis does not describe the result well for sample TB-10-28-5340 as discussed earlier because of the area contain small number of grains to represent the accurate statistics. The result of sample TB-10-38-4000 in heat-treated condition reveals similar traits of \{111\} pole
<table>
<thead>
<tr>
<th>Sample</th>
<th>copper</th>
<th>S3</th>
<th>Brass</th>
<th>Goss</th>
<th>Rotated cube</th>
</tr>
</thead>
<tbody>
<tr>
<td>Foil</td>
<td>0.061</td>
<td>0.212</td>
<td>0.056</td>
<td>0.031</td>
<td>0.004</td>
</tr>
<tr>
<td>TB-10-28-5340</td>
<td>0.009</td>
<td>0.108</td>
<td>0.067</td>
<td>0.001</td>
<td>0.015</td>
</tr>
<tr>
<td>- Whole layer</td>
<td>0.023</td>
<td>0.019</td>
<td>0.011</td>
<td>0.005</td>
<td>0.087</td>
</tr>
<tr>
<td>- Interface</td>
<td>0.049</td>
<td>0.107</td>
<td>0.017</td>
<td>0.000</td>
<td>0.043</td>
</tr>
<tr>
<td>- Middle bulk</td>
<td>0.004</td>
<td>0.205</td>
<td>0.010</td>
<td>0.002</td>
<td>0.038</td>
</tr>
<tr>
<td>- Bottom bulk</td>
<td>0.074</td>
<td>0.195</td>
<td>0.184</td>
<td>0.198</td>
<td>0.064</td>
</tr>
<tr>
<td>TB-10-38-4000</td>
<td>0.049</td>
<td>0.065</td>
<td>0.030</td>
<td>0.081</td>
<td>0.179</td>
</tr>
<tr>
<td>- Whole layer</td>
<td>0.058</td>
<td>0.185</td>
<td>0.324</td>
<td>0.060</td>
<td>0.004</td>
</tr>
<tr>
<td>- Interface</td>
<td>0.093</td>
<td>0.362</td>
<td>0.301</td>
<td>0.005</td>
<td>0.009</td>
</tr>
<tr>
<td>- Middle bulk</td>
<td>0.063</td>
<td>0.250</td>
<td>0.082</td>
<td>0.004</td>
<td>0.028</td>
</tr>
<tr>
<td>- Bottom bulk</td>
<td>0.057</td>
<td>0.164</td>
<td>0.078</td>
<td>0.013</td>
<td>0.068</td>
</tr>
<tr>
<td>TB-10-38-8000</td>
<td>0.082</td>
<td>0.267</td>
<td>0.086</td>
<td>0.002</td>
<td>0.025</td>
</tr>
<tr>
<td>- Whole layer</td>
<td>0.210</td>
<td>0.348</td>
<td>0.093</td>
<td>0.002</td>
<td>0.003</td>
</tr>
<tr>
<td>- Interface</td>
<td>0.210</td>
<td>0.348</td>
<td>0.093</td>
<td>0.002</td>
<td>0.003</td>
</tr>
<tr>
<td>- Middle bulk</td>
<td>0.210</td>
<td>0.348</td>
<td>0.093</td>
<td>0.002</td>
<td>0.003</td>
</tr>
<tr>
<td>- Bottom bulk</td>
<td>0.210</td>
<td>0.348</td>
<td>0.093</td>
<td>0.002</td>
<td>0.003</td>
</tr>
</tbody>
</table>

Table 5.8. Volume fraction of rolling texture components in aluminum 3003-H18 foil and VHP-UAM samples after heat-treatment at 343°C for 2 hours obtained from EBSD figures in as-processed condition in Figure 5.18, both in the interface regions and the bulk regions. At the interface region, i.e. the top two sections in Figure 5.18 and Figure 5.27 as well as the bottom section in Figure 5.27, the shear texture with \{111\} plane parallel to the ND-TD plane or horizontal plane dominates. Similarly, the \{111\} pole figures of the bulk regions contain retained rolling texture components after heat-treatment with similar orientation to the as-processed condition, but with larger equiaxed grains. The result is also similar in sample TB-10-38-8000 in most of the sections, not containing the very large grain, where the top portion of the build possess the same shear texture with \{111\}\langleuvw\rangle orientation in both as-processed and heat-treated conditions.

Figure 5.28 illustrates the map of low angle grain boundaries and high angle grain boundaries along with the map of recrystallized grains and non-recrystallized grains of
Figure 5.27. \{111\}-pole figures of ten smaller cross sections divided from top to bottom of the selected layer of the VHP-UAM samples after heat-treatment at 343°C for 2 hours with maximum texture strength in mrd.
heat-treated Al3003-H18 VHP-UAM samples made from the Test-Bed machine. The quantitative results of volume fraction of recrystallized grains and high angle grain boundaries in heat-treated VHP-UAM samples compared to the heat-treated foil are displayed in Table 5.9. It can be seen that sample TB-10-28-5340 has the highest volume fraction of recrystallized grains and high angle grain boundaries, while sample TB-10-38-4000 has the lowest fraction of recrystallized grains and high angle grain boundaries. It is also noted that the volume fraction of recrystallized grains and high angle grain boundaries in the heat-treated foil are relatively closed to sample TB-10-28-5340. This means that the recrystallization of new oriented grains and grain growth occurs during heat-treatment of original foil and sample TB-10-28-5340. In contrast, the recovery exists more in samples TB-10-38-4000 and TB-10-38-8000 because the fraction of recrystallized grains and high angle grain boundaries are lower. This means that not all the grains are recrystallized after heat-treatment at 343°C for 2 hours.

<table>
<thead>
<tr>
<th></th>
<th>Foil</th>
<th>TB-10-28-5340</th>
<th>TB-10-38-4000</th>
<th>TB-10-38-8000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fraction of recrystallized grains</td>
<td>0.965</td>
<td>0.986</td>
<td>0.771</td>
<td>0.862</td>
</tr>
<tr>
<td>Fraction of high angle grain boundaries</td>
<td>0.867</td>
<td>0.874</td>
<td>0.670</td>
<td>0.724</td>
</tr>
</tbody>
</table>

Table 5.9. Fraction of recrystallized grains and high angle grain boundaries of Al3003-H18 foil and VHP-UAM samples after heat treatment at 343°C for 2 hours obtained from grain orientation spread data in EBSD
Figure 5.28. Low angle grain boundaries (grey lines) and high angle grain boundaries (black lines) map with grain orientation spread color map showing fraction of recrystallized grains (white area) versus non-recrystallized grains (pink area) of the heat-treated Al3003-H18 VHP-UAM samples.

5.4 Implications and Significance of the Results

Figure 5.29 and Figure 5.30 illustrates the crystal direction maps displaying the shear \{111\} plane normal parallel to ND and the \{110\} plane normal parallel to RD of VHP-UAM samples before and after heat-treatment at 343°C for 2 hours. Table 5.10
provides the volume fractions of the \{111\} \parallel ND and \{111\} \parallel RD crystal directions for both original foil and VHP-UAM samples before and after heat-treatment. It can be seen from Figure 5.29 and Table 5.10 that the volume fraction of aluminum crystals with \{111\} \parallel ND increases with vibration amplitude and normal force during VHP-UAM processing. The volume fraction of \{111\} \parallel ND increases in the following order: as-received foil, TB-10-28-5340, TB-10-38-4000, and TB-10-38-8000 respectively. From Figure 5.29, there are large fractions of crystals with \{111\} \parallel ND in the interface regions of all VHP-UAM samples as well as in the top portion of the bulk region in sample TB-10-38-8000. This confirms the extent of shear deformation, which is increasing with larger vibration amplitude and higher normal force. Similarly, aluminum grains with \{110\} \parallel RD crystal direction, whose \{110\} plane normal is parallel to RD, are found more in the interface region and part of the bulk region with large shear deformation.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fraction of {111} \parallel ND</th>
<th>Fraction of {110} \parallel RD</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before Heating</td>
<td>After Heating</td>
</tr>
<tr>
<td>Foil</td>
<td>0.024</td>
<td>0.042</td>
</tr>
<tr>
<td>TB-10-28-5340</td>
<td>0.063</td>
<td>0.059</td>
</tr>
<tr>
<td>TB-10-38-4000</td>
<td>0.142</td>
<td>0.099</td>
</tr>
<tr>
<td>TB-10-38-8000</td>
<td>0.182</td>
<td>0.175</td>
</tr>
</tbody>
</table>

Table 5.10. Volume fraction of crystal directions of grains with \{111\}-shear plane normal parallel to ND and \{110\}-plane normal parallel to RD in Al3003-H18 foil and VHP-UAM samples obtained from EBSD before and after heat-treatment at 343°C for 2 hours

The results after heat-treatment at 343°C for 2 hours displayed in Figure 5.30 show that the grains with crystal directions \{111\} \parallel ND and \{110\} \parallel RD seem to remain in the same regions similar to the as-processed condition, i.e. at the interface region and
Figure 5.29. Crystal direction maps of as-processed Al3003-H18 VHP-UAM samples showing grains with \{111\}-plane normal parallel to ND and grains with \{110\}-plane normal parallel to RD
Figure 5.30. Crystal direction maps of Al3003-H18 VHP-UAM samples after heat-treatment at 343°C for 2 hours showing grains with \{111\}-plane normal parallel to ND and grains with \{110\}-plane normal parallel to RD

in the bulk region undergone large shear deformation. The volume fraction of grains with \{111\} \parallel ND after heat-treatment is relatively closed or slightly less than before
heating. For examples, the volume fraction after heating is 0.175 compared to 0.182 before heating in sample TB-10-38-4000, and 0.063 before heating compared to 0.059 after heating in sample TB-10-28-5340. Thus, it can be concluded that grains with shear texture and crystal direction \( \{111\} \parallel ND \) are relatively stable during heat-treatment. In the next chapter, the stability of the shear texture, in particular the \( \{111\} \parallel ND \) crystal direction will be examined using neutron diffraction texture analysis. This technique makes it possible to confirm whether these \( \{111\}\{uvw\} \) shear oriented grains are stable in the interface region as well as the bulk region of Al3003-H18 VHP-UAM during heat-treatment at 343°C for 2 hours.

Li et al. reported the deformation twinning of type \( \Sigma 3 \) twin boundary or coherent \( \{111\} \) plane boundary in high purity aluminum alloy under high strain rate shear deformation (see Figure 5.31) [135]. In their experiment, the strain rate is in the order of \( 10^5 \) sec\(^{-1}\), which is about the same order estimated by Schick for UAM of Al3003-H18 [73]. Schick calculated the approximate subgrain sizes to be in the order of 500 nm to 2 \( \mu m \) using temperature range of 300-600 K, which has been measured during UAM and VHP-UAM of Al3003-H18 [48, 65, 66, 73]. The estimated subgrain sizes agree well with the results of average grain size in the interface region of VHP-UAM samples reported in Table 5.3 and Table 5.4. It is hypothesized here that this type of twin boundary, which has an extremely low mobility [135] and if exist at the interface and the bulk regions causes the sluggish grain growth during heat-treatment at 343°C for 2 hours in Al3003-H18 VHP-UAM samples.
Figure 5.31. High resolution TEM images of deformation twinning near the grain boundary of high strain rate shearing of high purity aluminum showing multiple deformation twins (DTs) near a grain boundary (GB) with the corresponding diffraction pattern [135]

The other hypothesis that could explain the sluggish grain growth in the bulk region of VHP-UAM processed at 38 µm (higher) vibration amplitude is the amount of stored energy remains after VHP-UAM processing. Figure 5.32 schematically describes the amount of stored energy as a function of time after being subjected to hot-working and cold-working followed by static recrystallization. Initially, the original as-received Al3003-H18 has higher stored energy level than the annealed aluminum 3003-O (Al3003-O) after cold-work or work-hardening. During heat-treatment at high temperature, static recrystallization takes place resulting in the lower energy state of the
stored energy again. The amount of decreasing in stored energy depends on the annealed temperature as well as the duration of time the material is at the elevated temperature. For hot-working of Al3003-O, the stored energy increases because additional work-hardening is applied into the material at its lowest energy state. This steady state after hot-working during dynamic recrystallization is called intermediate energy state or steady state.

![Diagram of change in stored energy during cold working, hot working, and static recrystallization of metal](image)

Figure 5.32. Schematic of change in stored energy during cold working, hot working, and static recrystallization of metal [136]

It is important to note that recrystallization and normal grain growth seem to occur at a much faster rate in foil and in the bulk of sample TB-10-28-5340 as compared to in the bulk regions of samples TB-10-38-4000 and TB-10-38-8000. This is probably
because larger amount of stored energy is being released during dynamic recovery, dynamic recrystallization, and hot deformation processes within the bulk of TB-10-38-4000 and TB-10-38-8000. Therefore, the driving force for static recrystallization at 343°C for 2 hours is much less compared to original foil and sample TB-10-28-5340, and thus requiring higher temperature or longer annealing time for recrystallization and grain growth to occur at a rate observed in heat-treated foil and TB-10-28-5340. One hypothesis attempted to explain why small grains remained at the interface after heat treatment is the dispersed of small second phase particles Al₆(Mn,Fe) and oxides at the interface regions [73]. Fuji et al. [67] reported finer and higher number of Al₆(Mn,Fe) intermetallic particles near the bonded interfaces as compared to the bulk regions, which would inhibit grain boundary movement and limited final grain sizes in the interface regions. It was also known that these particles could serve as nucleation sites for random oriented nucleus during annealing of aluminum alloys [137]. However, the higher density of finer intermetallic particles does not explain why sluggish grain growth would occur in the bulk of heat-treated VHP-UAM samples TB-10-38-4000 and TB-10-38-8000.

Figure 5.33 explains what happens during UAM and VHP-UAM of Al3003-H18 foil, which is in high energy work-hardened state (higher than intermediate state in hot-working of Al3003-O). Depending on the processing parameters (vibration amplitude, normal force, and welding speed) used, the stored energy level may go up if the added energy due to hot working is higher than the released energy due to dynamic recrystallization and dynamic recovery during UAM and VHP-UAM. For lower power,
i.e. lower vibration amplitude and normal force, the stored energy is expected to increase as reported by hardening behavior in the bulk of Al3003-H18 UAM sample [49]. In contrast, for higher power, i.e. higher vibration amplitude in VHP-UAM of Al3003-H18, the stored energy decreases from the high energy state in as-received Al3003-H18 foil. This release of stored energy is caused by dynamic recrystallization in the interface region and dynamic recovery in the bulk region during adiabatic heating. The lower intermediate energy level in Al3003-H18 VHP-UAM samples processed at higher vibration amplitude means smaller driving force for static recrystallization during post-processing heat-treatment at 343°C for 2 hours. This means static recrystallization does not occur as fast when the stored energy is low in higher vibration amplitude VHP-UAM samples as compared to lower vibration amplitude UAM and VHP-UAM samples. Thus, this explains why very large grains with random orientation are observed in the bulk of Al3003-H18 UAM and VHP-UAM processed with 28 µm vibration amplitude (TB-10-28-5340), while small grain growth with retained rolling texture resides in the bulk of Al3003-H18 VHP-UAM samples processed with 38 µm (TB-10-38-4000 and TB-10-38-8000). It also explains why it is easier for TB-10-38-8000 to have recrystallization and grain growth in some area of the bulk as compared to TB-10-38-4000 because there is additional stored energy from work hardening due to larger normal force during VHP-UAM processing.

Current results indicate that microstructure stability across VHP-UAM builds depends strongly on the thermomechanical cycle used to make the build. Varying
Figure 5.33. Schematic of change in stored energy during UAM and VHP-UAM processing

Vibration amplitude and normal force results in significant changes in both subgrain diameters and textures. The subgrain size \( (d_{sub} \text{ in } \mu m) \) during thermomechanical processing of aluminum alloy, i.e. hot working is related to Zener-Holloman \((Z_h)\) parameter and peak temperature \((T_p)\) achieved \([138]\) as:

\[
d_{sub} = [-0.60 + 0.08 \log(Z_h)]^{-1}
\]

\[
Z_h = \dot{\varepsilon} \exp \left[ \frac{18,772}{T_p} \right]
\]

Schick approximated strain rate during UAM processing to be in the order of 105 rad/sec. Giving approximate peak temperature range between 300 K and 900 K, the subgrain size diameter was approximated to be around 500 nm to 2 \( \mu m \) \([49]\). For VHP-UAM, the
strain rate was expected to be approximately twice larger depended on processing parameters, and thus the subgrain size diameters are within the same order of UAM processing. The current results show the subgrain diameters between 0.89-1.15 μm, which match well with the calculated range even though the actual strain rate and peak temperature may vary depending on the processing parameters used.

The degree of changes in rolling textures and random textures in as-received foil and VHP-UAM build implies the degree of plastic deformation varies across VHP-UAM samples. Changes of rolling texture components and cube texture during hot working of high purity aluminum alloys have been reported and indicate variations of texture components with strain, and Zener-Hollomon parameters [139, 140]. It was found that Zener-Hollomon parameter could not be used to explain cube texture evolution during hot deformation of Al1050 properly. In addition, it was reported that deformation zones near large particles of only 2-3% volume fraction could result in random orientations apart from cube and rolling texture components during hot working [140]. Whether this is the sources of high random textures observed in the bulk of VHP-UAM builds when the amplitude is large is not yet clear.

At the interface, severe plastic deformation results in ultrafine grains of shear texture components, which are also found in other thermomechanical processing [141-143]. The study by Woo et al. [143] showed that severe plastic deformation during friction stir processing results in ultrafine grain structure and shear textures, while the heating alone from the tool shoulder (without stirring pin) had little effect, i.e. no dynamic recrystallization [143]. This is similar to the VHP-UAM samples, where the
interface regions were subjected to severe plastic deformation due to sonotrode texture on the top surface and asperity collapse during the subsequent passes. During VHP-UAM processing, severe plastic deformation is expected as both vibration amplitude and normal force increase. This can be seen as the random textures at the interfaces increase when both normal force and vibration amplitude increase (from 27.2% in original foil to 54.3% in the bulk of sample TB-10-38-8000, and 33.4% in sample TB-10-38-4000 build).

In order to predict both subgrain size distribution and complete textures of VHP-UAM builds, complete thermo-mechanical finite element model is needed to obtain accurate strain, strain rate, peak temperature, as well as temperature profile at a very precise details involving localized severe plastic deformation. Crystalline plasticity model such as those developed by Sarma and Radhakrishnan [144] is needed to simulate texture evolution during plastic deformation. Currently none of these modeling works are available for UAM and VHP-UAM. Even though thermo-mechanical modeling of UAM has been attempted, the model is still not validated with the actual strain rate and peak temperature occurred during UAM and VHP-UAM cycles. In addition, the materials modeling of recrystallization kinetics such as those by Brahme et al. [145] and Radhakrishnan et al. [146] are also needed to understand how microstructures and textures evolved during dynamic and static recrystallization.
5.5 Summary

Microhardness measurement and EBSD analyses were used to study the effects of different VHP-UAM processing conditions on mechanical properties and microstructures of as-received cold-rolled Al3003-H18 foils in as-processed and heat-treated conditions. Important results and findings are summarized here.

- Vibration amplitude has significant effect on softening of VHP-UAM microstructure of VHP-UAM build. The bulk of sample TB-10-28-5340 processed with 28 μm vibration amplitude has almost the same hardness as original foil (69 VHN), whereas the bulk of samples TB-10-38-4000 and TB-10-38-8000 processed with 38 μm vibration amplitude has much lower hardness (54-57 VHN).

- The interfaces of as-processed VHP-UAM builds consist of fine equiaxed grains with average subgrain size around 1μm diameter. The grains at interface regions are mostly random oriented grains with rolled texture component around 30% or less as compared to 72% rolled texture in original foil. The shear texture grains have common (111) plane parallel to ND direction with their perpendicular planes rotated around ND axis. The shear texture strength gets weaker (i.e. highly random oriented) when both vibration amplitude and normal force are very large (TB-10-38-8000).

- During post-processing heat treatment at 343°C for 2 hours, the texture evolutions and grain growth vary significantly with different processing parameters used. Grain growth is slowest in TB-10-38-4000 build processed with lower normal
force (4 kN) and very large vibration amplitude (38 μm). In addition, the grains within the bulk of sample TB-10-38-4000 retained large fraction of rolled texture components as compared to other VHP-UAM samples, where most grains have random orientations and much lower rolled texture components.

- Grain growth seems very sluggish near the bonded interfaces as compared to the bulk of VHP-UAM builds. It was suggested that the size and distribution of second phase particles as well as relative misorientation between the grains play roles in controlling grain growth rates. The different grain growth rates within the bulk, however, was believed to be associated with the different in stored energy which is the driving force for recrystallization during heat treatment.
Chapter 6: Study of Bulk Texture of Al3003-H18 UAM using Neutron Diffraction

6.1 Introduction

In the previous chapter, the results of hardness, microstructure, and texture of Al3003-H18 VHP-UAM samples were discussed. The results showed that there were variations in hardness, microstructure, and texture at the interface region and the bulk region of Al3003-H18 layers deposited using VHP-UAM. However, the microstructure and texture analysis were localized in a small region across 1-2 layers and not the overall larger bulk sample. This chapter is aimed to explain the bulk texture or macro-texture of Al3003-H18 VHP-UAM samples processed with the SonicLayer-7200 machine as compared to the original as-received tape both at room temperature condition and in-situ annealing at 343°C for 2 hour conditions. By the end of the chapter, the readers can expect to understand how the overall change in macro-texture and bulk properties occurs and then able to relate the findings to the knowledge obtained from the microstructure and micro-texture analysis discussed in the previous chapter.

6.2 Macro-Texture Analysis at Room Temperature

6.2.1 As-received Al3003-H18 Foil

The texture representation of as-received Al3003-H18 foil obtained from neutron diffraction is displayed as \{111\}-pole figure in Figure 6.1. It can be seen that the texture
is the same as the rolling texture of FCC metal and is similar to the result obtained from EBSD discussed in previous chapter. The inverse pole figures were also calculated for neutron diffraction texture data of Al3003-H18 foil as illustrated in Figure 6.2. It is noticed that there are extremely low density of grains with \{111\}-plane normal parallel to ND. This intensity at \{111\}-pole of the ND inverse pole figure will be used to determine the existence of grains with \{111\}(uvw) shear texture in Al3003-H18 VHPUAM builds in the next section.

Figure 6.1. \{111\}-pole figure of as-received Al3003-H18 foil stack obtained from neutron diffraction (log scale)

In addition to \{111\}-pole figure and inverse pole figures, the orientation distribution sections in Euler angle space (\(\phi_1\), \(\Phi\), \(\phi_2\)) of Al3003-H18 foil obtained from neutron diffraction is also provided in Figure 6.3. The ODF representation of texture of Al3003-H18 foil shows the nature of \(\beta\)-rolling fiber running from brass to S3 and copper texture components. It can be seen from the ODF of Al3003-H18 foil that such
\{111\}(uvw) shear fiber (intensity along horizontal line at $\Phi = 54.7^\circ$ in $\varphi_2 = 45^\circ$ ODF section) does not exist in as-received Al3003-H18 foil, where the intensity of ODF along the \{111\}-fiber is almost zero.

Figure 6.2. Inverse pole figures showing grain orientations in normal direction (ND), rolling/weld direction (RD), and transverse/vibrational direction (TD) of as-received Al3003-H18 foil obtained from neutron diffraction

Figure 6.3. Orientation distribution function in Euler angle space of as-received Al3003-H18 foil obtained from neutron diffraction
6.2.2 As-Processed VHP-UAM Samples

Similar to the \{111\}-pole figure of Al3003-H18 foil in Figure 6.1, the \{111\}-pole figures of Al3003-H18 VHP-UAM builds processed with SonicLayer-7200 machine at different sections (top layers, bottom layers, and whole sample) are illustrated in Figure 6.4. The result clearly shows that there is difference between the textures of the top layers and the bottom layers of all three VHP-UAM samples as well as difference between each VHP-UAM samples in similar sections.

The results obtained from pole figures using neutron diffraction reveal that the texture of the top layers of the lower amplitude sample SL-66-28-5340 is most similar to the as-received foil with similar highest intensity of 4.0 as compared to 4.1 in the as-received foil but with larger minimum intensity of 0.21 as compared to 0.05 in the as-received foil as showed in Figure 6.4. The increasing minimum intensity in pole figure is likely due to the interface region because the interface region is mainly composed of random textures in addition to shear texture and recrystallized texture as discussed in EBSD results earlier. The grains in the bulk regions is mainly composed of retained rolling textures and thus the similar pole figure as the as-received foil is obtained in top layers of sample SL-66-28-5340. In the top layers of sample SL-80-34-5340 and sample SL-80-34-4000, where the vibration amplitude used was higher, the maximum texture strength or maximum intensity in the pole figures decreases to lower values of 3.5 for sample SL-80-34-5340 and 3.7 for sample SL-80-34-4000. Although the samples are different from those used to obtained microstructure and micro-texture results from
EBSD, the nature of the results are similar where less change in overall bulk texture is expected in lower vibration amplitude build.

Figure 6.4. \{111\}-pole figure of as-processed Al3003-H18 VHP-UAM samples obtained from neutron diffraction (log scale)

The results of \{111\}-pole figures of the bottom layers illustrate that there is some shifting and changes of intensity towards the middle or center point or the ND pole of \{111\}-pole figure, i.e. where \{111\}-plane normal align or is parallel to the ND axis.
especially in higher vibration amplitude sample SL-80-34-5340 and sample SL-80-34-4000. It is noted that though the higher intensity is observed at and near the ND pole, the overall core texture fiber or texture tube representing rolling texture component of the bulk rolled Al3003-H18 in VHP-UAM samples is still retained in all samples. This result obtained from neutron diffraction is consistent with EBSD results obtained earlier.

The results of {111}-pole figure of the whole sample, i.e. thicker sample including top layers and bottom layers reveal combination of the results of the top layers and the results of bottom layers discussed earlier. One could simply think of this as the average results although this is not precise way to address it. It is interesting though that the shapes of the pole figures are more similar to the pole figures of the top layers rather than the bottom layers in sample SL-66-28-5340 and sample SL-80-34-5340 where distinct intensity of {111} || ND is not obvious unlike the pole figure of the whole sample of sample SL-80-34-4000 where {111} || ND texture are much stronger. The quantitative texture analysis using volume fraction calculation of each texture components obtained from neutron diffraction is provided in Table 6.1 showing the rotated cube {111}(112) and shear texture {111}(110) of the whole sample is highest in sample SL-80-34-4000 rather than sample SL-80-34-5340.

The calculated volume fraction of the major texture components showed in Table 6.1 also reveals that there is further decrease in rolling texture components (copper, S3, brass) in bottom layers as compared to the top layers and the whole samples of each sample. The data also indicates that, in general, the quantitative volume fraction of the whole sample lie between the values in the top layers and bottom layers and not exactly
Table 6.1. Volume fractions (in percentage) of rolling texture components in Al3003-H18 foil and as-processed VHP-UAM samples processed with SonicLayer 7200 machine as obtained from neutron diffraction

<table>
<thead>
<tr>
<th>Material</th>
<th>copper</th>
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<th>brass</th>
<th>Goss</th>
<th>Cube</th>
<th>Rotated cube</th>
<th>Shear</th>
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<td>1.73</td>
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<td>2.29</td>
<td>2.10</td>
<td>1.36</td>
<td>1.38</td>
</tr>
</tbody>
</table>

The inverse pole figures of Al3003-H18 VHP-UAM samples obtained from neutron diffraction, similar to those of the original foil, are displayed in Figure 6.5 (SL-
It is noticed that there is not much difference in the TD inverse pole figures of all VHP-UAM samples and the as-received foil, and thus the emphasis will be placed on ND and RD inverse pole figures. It has been known earlier that the high intensity of texture in the ND and RD inverse pole figure belongs to β-fiber of rolled FCC metal and the position of the β-fiber is according to the inverse pole figures of the as-received foil from EBSD result and from neutron diffraction result. However, the results of inverse pole figures of the three VHP-UAM samples in Figure 6.5, Figure 6.6, and Figure 6.7 reveals the changes of intensity at and near the {111} || ND, {111} || RD and {110} || RD crystal directions.

Figure 6.5. Inverse pole figures showing grain orientations in normal direction (ND), weld direction (RD), and transverse/vibrational direction (TD) of different sections of Al3003-H18 VHP-UAM sample SL-66-28-5340 obtained from neutron diffraction.
Near the \{111\} \parallel ND crystal direction, it can be seen that the intensity of texture strength increases in all VHP-UAM samples compared to the as-received foil in Figure 6.2, particularly the bottom layers samples of high amplitude samples SL-80-34-5340 and SL-80-34-4000 as well as the whole sample of SL-80-34-4000. Similarly, the texture strength near the \{110\} \parallel RD is also clearly higher in the bottom layers of VHP-UAM samples indicating the higher volume fraction of shear textures in \{111\} \parallel ND and \{110\} \parallel RD as previously discussed in EBSD results. This result also agrees with the quantitative data of calculated volume fraction of shear texture components displayed in Table 6.1, in which the volume fraction of \{111\}(110) shear texture is at least 3-4 times
Figure 6.7. Inverse pole figures showing grain orientations in normal direction (ND), weld direction (RD), and transverse/vibrational direction (TD) of different sections of Al3003-H18 VHP-UAM sample SL-80-28-5340 obtained from neutron diffraction.

higher than those found in the as-received foil in the bottom layers of each VHP-UAM samples as compared to about 2-3 times higher in the top layers of each VHP-UAM builds. The result of \{111\} \parallel RD in the inverse pole figure determines the change in rolling texture strength along the \(\beta\)-fiber in which the intensity of \(\beta\)-fiber is weaker in the bottom layers of each VHP-UAM sample compared to the original foil, top layers, and the whole sample. This indicates that there is indeed some major change in texture transition within the bulk from rolling texture components towards shear texture components as observed in the EBSD results of the interface and the bulk region right below the interface, especially of the higher vibration amplitude build.
For the ODF of Al3003-H18 VHP-UAM sample obtained from neutron diffraction, only the ODF sections at $\varphi_2 = 45^\circ$ are illustrated in Figure 6.8. As mentioned earlier, the focus of this analysis is to investigate the intensity of the $\{111\}uvw$ shear texture along the $\{111\}$-fiber at the constant $\Phi = 54.7^\circ$ and $\varphi_2 = 45^\circ$ section while $\varphi_1$ ranges from $0^\circ$ to $90^\circ$. It can be seen that, in general, the position of the peak intensity or the core of the main rolling $\beta$-fiber is diminishing from the peak intensity in original foil as vibration amplitude increases with much less intensity in the bottom layers similar to previous sections. Along the $\{111\}$-fiber i.e. at $\Phi = 54.7^\circ$ line (the red lines showed in Figure 6.8), the overall intensity of $\{111\}$-fiber increases from original foil in all VHP-UAM samples as the intensity of $\{111\}$-fiber in original foil is almost zero. In VHP-UAM samples, the intensity along the $\{111\}$-fiber seems to be uniform in most of the samples, except the bottom layers of sample SL-80-34-5340 and sample SL-80-34-4000, in which the edge of the main rolling $\beta$-fiber almost touches the $\{111\}$-fiber at $\Phi = 54.7^\circ$ line. With the tolerance of $10^\circ$ to $15^\circ$, this would add to the overall intensity or increasing in volume fraction of $\{111\}$-fiber especially in the bottom layers of samples SL-80-34-5340 and SL-80-34-4000. This result addresses why the volume fraction of the $\{111\}uvw$ shear texture is higher in large vibration amplitude build, and in particular in bottom layers, where lower weld speed was used and thermo-mechanical cycles are greater. However, the results can also be better interpreted using the texture fiber plots, which will be discussed in the following section.

The texture fiber plots displayed in Figure 6.9 and Figure 6.10 illustrate the $\beta$-fiber and the shear $\{111\}$-fiber of Al3003-H18 VHP-UAM samples obtained from the
Figure 6.8. Orientation distribution function displaying $\phi_2=45^\circ$ sections in Euler angle space of different sections of Al3003-H18 VHP-UAM samples compared to as-received Al3003-H18 foil obtained from neutron diffraction.

ODF of neutron diffraction. For reference, both the $\beta$-fiber and the shear $\{111\}$-fiber of as-received Al3003-H18 foil sample obtained from neutron diffraction were also plotted for comparison and identify how much the rolling texture decreases, and how much shear texture increases after VHP-UAM processing. Similar plots obtained from EBSD results, in which the interface regions have the largest drop in the intensity of rolling $\beta$-fiber, while it has the highest intensity along the $\{111\}$-shear fiber especially in the higher amplitude samples (SL-80-34-5340 and SL-80-34-4000). Previous results also show that the middle bulk region of SL-80-34-5340 also possesses unusual high intensity of the $\{111\}$-shear fiber indicating higher intensity of $\{111\}$-shear fiber is to be expected in a similar fashion in higher amplitude SL-80-34-5340 and SL-80-34-4000.
Figure 6.9. Plots of β-fiber (rolling textures) of as-received Al3003-H18 foil and as-processed VHP-UAM samples in different bulk regions

Figure 6.10. {111}-fiber or A-fiber plots (shear textures) of as-received Al3003-H18 foil and as-processed VHP-UAM samples in different bulk regions

The intensity of the β-fiber of the top layers is the most close to the as-received foil as expected, particularly the lower vibration amplitude sample SL-66-28-5340, while
the bottom layers of sample SL-80-34-5340 has the lowest intensity similar to the quantitative volume fraction results provided in Table 6.1. When compared the absolute $f(g)$ of the $\beta$-fiber obtained from the ODF of neutron diffraction to $f(g)$ of the EBSD results, it can be seen that the orientation distribution $f(g)$ is about twice smaller in neutron diffraction as compared to EBSD analysis. This also show why the volume fraction of each texture component obtained from EBSD results were higher than the volume fraction obtained from neutron diffraction as discussed earlier. The typical shape of the $\beta$-fiber with roughly uniform intensity between the copper $\{112\}\langle1\bar{1}1\rangle$ texture component and the S3 $\{123\}\langle6\bar{3}4\rangle$ texture component following by gradually decrease in intensity towards the brass $\{011\}\langle2\bar{1}1\rangle$ texture component in the as-received foil is very similar in EBSD result compared to neutron diffraction result in Figure 6.9 despite the relative $f(g)$ is higher in EBSD result.

The results from the shear $\{111\}$-texture fiber plots in Figure 6.10 reveal that the intensity along the $\{111\}\langleuvw\rangle$ fiber is much higher than original foil in all VHP-UAM samples despite the absolute magnitude of $f(g)$ is less than 1.0. As showed earlier in Table 6.1, the estimated calculated volume fraction of the $\{111\}\langle110\rangle$ shear texture component is 0.43% for the as-received foil and from 1.25% to 1.63% in the bottom layers of VHP-UAM samples, whereas the calculated volume fraction of $\{111\} \parallel$ ND crystal direction is 2.4% in the as-received foil, but could go up to 6.3% in lower amplitude sample SL-66-28-5340 and 14.2%-18.2% in higher amplitude sample SL-80-34-4000 and sample SL-80-34-5340 as discussed in previous chapter. This large gap between EBSD result and neutron diffraction result imply that it is best to analyze the
relative change in volume fraction or \( f(g) \) data rather than the absolute values as discussed earlier. Therefore, the results of the shear \{111\}-texture fiber indicate that higher volume of grains with shear texture of \{111\}-fiber exists in VHP-UAM samples in particular in the higher amplitude sample SL-80-34-5340 and sample SL-80-34-4000 and in the bottom layers of the samples where the relative \( f(g) \) is much higher than the rest of the sample. This bulk shear texture result confirms the EBSD result that when larger vibration amplitude is used, grains undergo shear deformation even in the bulk region below the interface region of VHP-UAM samples. With shear deformation and higher recrystallized grains at the interface region, the overall volume fraction of grains with rolling texture components, i.e. along the \( \beta \)-fiber would decrease from the as-received foil. In the top layers, it was found that there are not much difference in the intensity of the \{111\}-shear texture fiber whereas in the whole sample, SL-80-34-4000 contain the largest overall shear \{111\}-textures similar to quantitative result showed in Table 6.1 discussed earlier.

This texture has a trait similar to shear textures found in other thermomechanical processing i.e. friction stir welding [147-149]. Fuji, et al. [67] also reported recrystallized grains at the interface regions with \{111\}(110) component due to shear deformation of micro-asperities. These two texture components share the same common \{111\}-plane parallel to the ND direction. Similar shear texture with \{111\}-plane parallel to ND axis was also observed in friction stir welding literatures [147-149]. Sato, et al. [148] studied micro-texture in friction stir welding of aluminum 6063 alloy (Al6063) and found that the shear texture rotated about the ND at different locations within 3.3 mm away from the
center line. This is somewhat similar to the rotation of shear texture as seen in \{111\}-pole figures of the two interfaces above a layer of sample TB-10-38-4000, where shear texture in the top region is approximately 180° away (about ND) from the shear texture direction in the bottom interface region. It is noted that the shear texture in the interface regions is strongest in sample TB-10-38-4000 and weakest in sample TB-10-38-8000. However, grains also seems to possess some form of shear texture components with \{111\}-plane parallel to ND direction up to 60 µm below the bonded interface of sample TB-10-38-8000, where plastic deformation occurs.

6.3 In-situ Macro-Texture Analysis

6.3.1 Heat-Treated Al3003-H18 Foil

During in-situ neutron diffraction experiment, the crystallographic texture of the Al3003-H18 foil stack was collected and plotted in form of the \{111\}-pole figure as showed in Figure 6.11. The \{111\}-pole figure before heat-treatment in Figure 6.11 was obtained from the in-situ experiment in furnace before the temperature is ramped up. The data collection time was 10 minutes. The reasons why these pole figures look slightly different from previous result in Figure 6.1 may include some sample angular position misalignment errors, which can be adjusted during data analysis, and sample being placed in vanadium foil sample holder.

The \{111\}-pole figure before heat-treatment reveals the trait of as-rolled FCC textures including β-fiber with weaker texture strength compared to textures obtained from EBSD and room temperature neutron diffraction. After heat-treatment, the pole
Figure 6.11. \{111\}-pole figure of Al3003-H18 foil stack obtained from neutron diffraction in-situ heat-treatment at 343°C for 2 hours (log scale)

Figure display much weaker retained rolling texture in β-fiber and has shape similar to one obtained from EBSD result of heat-treated Al3003-H18 foil. In addition to retained rolling texture, the cube texture component is also observed in the pole figure. The quantitative data of volume fraction of each major texture components were also calculated and provided both for the foil and VHP-UAM builds in the next section.

The inverse pole figures of Al3003-H18 foil stack before and after in-situ heat-treatment at 343°C for 2 hours are illustrated in Figure 6.12. The results before heat-treatment are very similar to EBSD inverse pole figures. The retained rolling β-fiber can be observed in both ND and RD inverse pole figures of the foil stack before the start of in-situ heat-treatment. The texture strength is weaker compared to EBSD results similar to \{111\}-pole figures mentioned earlier. After heat-treatment, i.e. the foil is cooled down to below 100°C, the result shows similar strong textures near \{001\}-pole and very weak near \{111\}-pole in ND, while in RD, strong texture is found near both \{001\} and \{111\}-
poles. In TD, the texture is very weak, which means there is no preferential orientation along TD.

![Inversion pole figures showing grain orientations in normal direction (ND), weld direction (RD), and transverse/vibrational direction (TD) of Al3003-H18 foil obtained from neutron diffraction in-situ heat-treatment at 343°C for 2 hours.](image)

Figure 6.12. Inverse pole figures showing grain orientations in normal direction (ND), weld direction (RD), and transverse/vibrational direction (TD) of Al3003-H18 foil obtained from neutron diffraction in-situ heat-treatment at 343°C for 2 hours.

6.3.2 Heat-Treated VHP-UAM Samples

The results macro-textures of Al3003-H18 VHP-UAM samples processed from the SonicLayer-7200 machine during in-situ neutron diffraction are discussed in this section. For each build, the “bottom layers” and the “whole sample” specimens were
measured except for sample SL-80-34-5340 of which only the “whole sample” is measured. The {111}-pole figures before heating and after heating are plotted in Figure 6.13 and the inverse pole figures of VHP-UAM samples are illustrated in Figure 6.14, Figure 6.15, and Figure 6.16 respectively. The volume fractions of major texture components before and after heating are calculated using MTEX from neutron diffraction data and listed in Table 6.2 similar to Table 6.1.

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<thead>
<tr>
<th></th>
<th>copper</th>
<th>S3</th>
<th>brass</th>
<th>Goss</th>
<th>Cube</th>
<th>Rotated cube</th>
<th>Shear {111}(110)</th>
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Table 6.2. Volume fractions (in percentage) of rolling texture components in AI3003-H18 foil and VHP-UAM samples during in-situ heat-treatment at 343°C for 2 hours obtained from neutron diffraction (top = before heating, bottom = after cooling down)
Figure 6.13. \{111\}-pole figure of Al3003-H18 VHP-UAM samples obtained from neutron diffraction in-situ heat-treatment at 343°C for 2 hours (log scale)

The results from the \{111\}pole figure results before and after heat-treatment in Figure 6.13 reveals the presence of the \{111\} || ND in all the samples. This \{111\} || ND textures can be seen from the intensity spot located at the center of \{111\} pole figures or at ND poles. This result is obvious because the same \{111\} || ND is not present in the \{111\}-pole figure of original foil stacks showed earlier. Aside from this \{111\} || ND texture, the \{111\}-pole figures of VHP-UAM samples contained retained rolling textures along the \(\beta\)-fiber as expected. This \{111\} || ND textures are also stronger in the “bottom layers” compared to the “whole sample” meaning larger amount of shear deformation taking place in the bottom layers and less shear deformation taking place in the top
Figure 6.14. Inverse pole figures showing grain orientations in normal direction (ND), weld direction (RD), and transverse/vibrational direction (TD) of Al3003-H18 VHP-UAM sample SL-66-28-5340 obtained from neutron diffraction in-situ heat-treatment at 343°C for 2 hours.

layers. This result agrees with the hardness distribution discussed in Chapter 5, where the hardness values gradually decrease from top to bottom of these three VHP-UAM samples, especially when the vibration amplitude is large in samples SL-80-34-5340 and SL-80-34-4000.

After heat-treatment, the \{111\} || ND shear texture remains and can be observed in the \{111\}-pole figures after heat-treatment. One can see this \{111\} || ND textures clearly at the center of the \{111\}-pole figures, especially in the “bottom layers” sample of SL-80-34-4000 after heat-treatment. This result confirms the fact that the shear textures,
Figure 6.15. Inverse pole figures showing grain orientations in normal direction (ND), weld direction (RD), and transverse/vibrational direction (TD) of Al3003-H18 VHP-UAM sample SL-80-34-4000 obtained from neutron diffraction in-situ heat-treatment at 343°C for 2 hours.

which are largely present at and near the interface of VHP-UAM samples, are very stable and remain even after heat-treatment at 343°C for 2 hours. Other interesting textures that are clearly observed from the \{111\}-pole figures in Figure 6.13 include the cube texture in the “bottom layers” of SL-66-28-5340, and the brass texture in the “bottom layers” of SL-80-34-4000. Apart from these, the pole figures are similar to retained rolling textures seen in heat-treated foil with some texture components along the β-fiber getting stronger and some texture components getting weaker after heat-treatment.

The quantitative data of volume fraction of the selected texture components are listed in Table 6.2. The first value (top value) is the calculated volume fraction of each
Figure 6.16. Inverse pole figures showing grain orientations in normal direction (ND), weld direction (RD), and transverse/vibrational direction (TD) of Al3003-H18 VHP-UAM sample SL-80-34-5340 obtained from neutron diffraction in-situ heat-treatment at 343°C for 2 hours.

texture component before heating whereas the second value (bottom value) indicates the volume fraction after heating. Again, this calculated volume fraction may not be accurate as the values seem to be relatively low compared to EBSD result. Therefore, the values will be used as relative value rather than absolute values for comparison between samples as well as before and after heat-treatment.

In original foil stack, the volume fraction of major rolling texture components, i.e. copper, S3, and brass decrease after heating while Goss, cube, rotated cube, and shear \{111\}\(\langle 110 \rangle\) increase after heating. The volume fraction of the cube texture component of the foil stack increase the most, i.e. almost 4 times from 1.39 before heating to 5.58.
after heating, while the volume fraction of each rolling texture components slightly decrease from about 5-6 before heating to about 3-4 after heating. In VHP-UAM samples, the cube texture increase the most in SL-66-28-5340 after heating which confirms the more similar microstructure stability in the bulk to original foil stack when lower vibration amplitude was used. The result of VHP-UAM samples shows consistent higher stability of brass texture component compared to copper and S3 textures. This is very interesting result because before heating, the volume fraction of the brass component is lower than both copper and S3 but after heating, the volume fraction of the brass component is higher than both components. Furthermore, this result is also found to be opposite in original foil in which the brass component is higher than both copper and S3 before heating, but is lower after heating. Therefore, it is likely that VHP-UAM processing produce higher stability of grains with brass texture components which are much more likely to remain in the bulk after heating at 343°C for 2 hours.

The result of the \{111\}\{110\}-shear texture, however, doesn’t seem to correlate well with what is known from the EBSD result, in which the “whole sample” of SL-80-34-5340 (high amplitude, medium/high force) seem to be lower than the “whole sample” of SL-66-28-5340 (low amplitude, medium/high force), and SL-80-34-4000 (high amplitude, low force). Nevertheless, the increasing volume fraction of the \{111\}\{110\}-shear texture after heating implies that the interface grains possessing this \{111\}\{110\}-shear texture are relatively stable after heating as showed earlier in the pole figure results and EBSD results.
Figure 6.14, Figure 6.15, and Figure 6.16 illustrate the inverse pole figures of VHP-UAM samples before and after in-situ heating neutron diffraction experiment. It can be seen that the rolling β-fiber is clearly seen in all samples similar to the result of original foil stack. The very high intensity at the \{111\}-pole in the RD inverse pole figures of the “bottom layers” of SL-66-28-5340 belongs to the high volume fraction of copper texture component as indicated in Table 6.2, where the volume fraction of copper is much higher than S3 and brass components. Similar results were also found in the room temperature textures or as-processed VHP-UAM samples discussed in previous chapter. After heating, the original high intensity near \{001\}-pole and \{110\}-pole of the ND inverse pole figure found in foil stack can also be seen in all heat-treated VHP-UAM samples. In addition, the increase in the intensity of textures near \{111\}-pole of ND inverse pole figure in VHP-UAM samples compared to original foil both before and after heating means that the \{111\} || ND shear textures are stable. This \{111\} || ND shear texture seems to be highest in the “bottom layers” of SL-80-34-4000. This means that the \{111\} || ND shear textures, which is present at and near the interface region (known from EBSD results) do not diminish even after heat treatment. Because this shear texture is rarely seen in EBSD results of heat-treated foil, the in-situ neutron diffraction confirms stable microstructure and texture at the interface region with larger volume of grains composed of shear texture especially in the bottom layers where more accumulative thermo-mechanical deformation takes place and when vibration amplitude used is larger.

While volume fraction of recrystallized cube texture during isothermal annealing at 350-375°C of cold-rolled aluminum alloy can reached up to 50% in single phase
aluminum 1050 alloy (Al1050), the maximum volume fraction of cube texture is only 10% in two phase aluminum 3015 alloy (Al3015) [133, 145, 150]. This implies that secondary phases play a significant role in recrystallization kinetics in Al3003-H18 foil and VHP-UAM builds. On the other hand, VHP-UAM processing also changes the stability of the cold-rolled structures and thus immediately alters recrystallization and grain growth kinetics. Based on the current results, plastic deformations may occur by crystal slipping, shearing of grains, or reorientation of grains along different paths. Difference in shear strains in the bulk of VHP-UAM builds as well as some different in temperature rise result in different orientations of grains and textures of as-processed VHP-UAM builds. The difference in misorientations i.e. grain boundary energy and mobility in addition to the difference in stored energy may lead to changes in recrystallization and grain growth kinetics. For examples, it has been known that grains grow very rapidly with the orientation relationship of 40°〈111〉 orientation relative to the deformed matrix [137]. The very low volume fraction of random texture components (23.6%) in heat-treated TB-10-38-4000 build indicated that oriented growth is evident during recrystallization process in sample TB-10-38-4000. However, the volume fractions of random texture components are much higher, i.e. 77.5% in heat-treated TB-10-28-5340 and 65.1% in heat-treated TB-10-38-8000 build. This implies somewhat very different annealing kinetics from sample TB-10-38-4000.
6.4 Summary

The bulk texture measurement using neutron diffraction reveals both shear texture of \( \{111\}\langle 110 \rangle \) orientation and retained rolling textures along the \( \beta \)-fiber in the bulk of Al3003-H18 VHP-UAM builds. The interface region consists of the fine equiaxed grains as a result of dynamic recrystallization taking place during the shear and plastic deformation at the microasperities. It is apparent that there are accumulative thermomechanical effects on the bulk of VHP-UAM builds as the lower layers of the build demonstrate higher fraction of shear texture, lower fraction of certain rolling texture components, as well as some increase in random textures. When larger vibration amplitude is used to fabricate VHP-UAM samples, the shear texture components increase indicating larger shear deformation taking place at the interface when higher vibration amplitude is used. The texture results after heat treatment show the high stability of shear texture in the interface region as opposed to the significant decrease in rolling texture components in the bulk. On other hand, the sluggish grain growth at the interface was believed to be due to the pinning of grain boundaries by small particles whereas the stronger brass texture components and possible lower number of low angle boundaries could result in slower grain boundary motion in the bulk of VHP-UAM samples processed at higher vibration amplitudes resulting in slower grain growth as compared to original Al3003-H18 foil or VHP-UAM samples processed at smaller vibration amplitude. At 343°C, the texture evolution occurred so fast that we could not resolve the kinetics of recrystallization of Al3003-H18 VHP-UAM builds and their texture components.
7.1 Effect of VHP-UAM Processing Parameters on Hardness of Al3003-H18

In the current study, only vibration amplitude and normal force are varied and studied. It was found that the change in vibration amplitude has the greater effect on the change in hardness than the change in normal force does. By increasing vibration amplitude from 28 µm to 38 µm in the Test-Bed machine, the average hardness in samples TB-10-38-4000 and TB-10-38-8000 are 15 VHN lower than sample TB-10-28-5340, whose hardness is slightly less than the as-received Al3003-H18 foil. The results are similar in the SonicLayer-7200 machine, where samples SL-80-34-4000 and SL-80-38-8000 have the average hardness 5-10 VHN smaller than sample SL-66-28-5340. It was also found that the average hardness seem to be exponential decay with the ultrasonic power used during VHP-UAM processing.

7.2 Effect of VHP-UAM Processing Parameters on Microstructure of Al3003-H18

In this current study, the microstructure analysis was performed mainly using EBSD in VHP-UAM samples made from the Test-Bed machine. In the as-processed condition, the interface region contains fine and equiaxed grains in the order of 1-2 µm average diameters in all samples. The change in the bulk microstructure is obvious when the vibration of 38 µm is used. In sample TB-10-38-4000 with lower normal force, the
grains in the bulk undergo dynamic recovery through subgrain coalescence, which causes softening behavior observed in the hardness result. In sample TB-10-38-8000 with higher normal force, large shear deformation and plastic deformation occurs as observed by 10% reduction in thickness of a foil layer after VHP-UAM. There is also a gradient of microstructure change, which increases from bottom to top of the layer, where the bottom microstructure has little change and is relatively similar to the microstructure of as-received Al3003-H18 foil.

7.3 Effect of VHP-UAM Processing Parameters on Texture of Al3003-H18

In the current study, the texture analysis is performed in the localized region of samples made from the Test-Bed machine using EBSD, and in the overall bulk sample made from the SonicLayer-7200 machine using time-of-flight neutron diffraction. The major change in texture is observed at the interface region and in the partial bulk region where large shear deformation occurs during VHP-UAM. The grains possessing the \{111\}(uvw)-shear texture in the interface region and part of the bulk region is found to be stable and remains even after heat-treatment at 343°C for 2 hours.

7.4 Suggested Future Work

In order to identify the exact mechanisms of sluggish grain growth in the interface regions and the bulk regions of Al3003-H18 VHP-UAM samples after heat-treatment at 343°C for 2 hours, it is suggested that the high resolution TEM analysis should be performed. This would allow the identification of the type of boundaries exist in
different regions. In addition, it would be of benefit to determine the dislocation contents in the grains and subgrains to estimate the change in stored energy in different VHP-UAM and heat-treatment conditions.

In addition, the microstructure analysis of VHP-UAM samples processed by the Sonic-Layer-7200 machine could be analyzed with EBSD to study the microstructure change in different layers with different hardness values. This will help the understanding of why there is hardness deviation within the samples as well as in the different layers of the samples processed with higher vibration amplitude. It is also recommended that nano-indentation would be a better technique to obtain hardness data to obtain hardness distribution within a single layer both at the interface regions and the bulk regions.

Finally, due to the limited time and experiment set-up in the current work, the temperature and displacement measurement at the interfaces and in the sample during VHP-UAM processing was not performed. In the future experiment, where VHP-UAM samples are to be made, it is advised to measure both the transient thermal response both in the interface and the bulk region using thermo-couple, infrared camera, or other temperature measurement techniques. Recently, Foster et al. [151] developed a methodology to track the UAM horn, substrate, and foil displacements at very good time resolutions using photon Doppler velocimeter. Similar measurements at various heights can confirm the consistency of deformation at different build heights. It is also very important to identify the temperature at the contact between the sonotrode surface and the top foil surface because this seems to be the time when the largest severe deformation
and microstructure change takes place. With comprehensive measurement of thermal and mechanical processing conditions, it is possible to understand hardness variations across the build height, as well as the power variations measured by the equipment.
References


Appendix A: Calibration of Vibration Amplitude in VHP-UAM

The vibration amplitude data was typically set in the percentage unit of maximum amplitude according to the voltage and power input. In order to obtain the actual vibration amplitude in micron, the calibration is needed. The amplitude test was performed using laser vibrometer by EWI and the data was provided for reference [152]. Figure A.1 displays the modes of vibration in the sonotrode when the ultrasonic power is applied. In this analysis, only horizontal vibration (mode A) is considered as the vibration amplitude parameter. Test results of the amplitude calibration curves are plotted in Figure A.2 for each data point being measured. The correction of actual vibration amplitude from percent to micron is showed in Table A.1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Input Amplitude (%)</th>
<th>Actual Amplitude (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TB-10-28-5340</td>
<td>50%</td>
<td>28.46</td>
</tr>
<tr>
<td>TB-10-38-4000</td>
<td>70%</td>
<td>38.05</td>
</tr>
<tr>
<td>TB-10-38-8000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SL-66-28-5340</td>
<td>60%</td>
<td>27.61</td>
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<tr>
<td>SL-80-34-4000</td>
<td>75%</td>
<td>34.18</td>
</tr>
<tr>
<td>SL-80-34-5340</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table A.1. Amplitude correction for current study
Figure A.1. Displacement modes during ultrasonic vibration of the VHP-UAM sonotrode (Courtesy EWI)

Figure A.2. Amplitude calibration chart for current VHP-UAM work

\[
y = 0.4796x + 4.4821 \\
R^2 = 0.9995
\]

\[
y = 0.438x + 1.33 \\
R^2 = 0.999
\]