Ultra Fine Grained Production in RRR Niobium for RF Cavities by ECAE

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ULTRA FINE GRAINED PRODUCTION IN RRR NIOBIUM FOR RF CAVITIES BY ECAE

By

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Dedicated to my mother who always told me that I could do anything I put my mind to. To my family for giving me the encouragement to help me get where I am today. To my friends who have given me their support through all the times, trying and not so. Thank you for always being there. Your support and friendship have helped me get to where I am today. I love you and thank you all.
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ABSTRACT

Efficient fabrication of seamless RF cavities by hydroforming requires the use of very fine microstructure. In this study, grain refinement of commercial purity (99.99%) niobium was carried out by equal channel angular extrusion (ECAE) for up to 8 passes via route B_C using a die with a channel intersection angle of 90º at a rate of 0.25mm/sec. Samples were sectioned (top, middle and bottom) from the as received material and the billets of each pass and then analyzed using various analytical tools, which includes Vickers microhardness, X-ray diffraction and Orientation Imaging Microscopy (OIM).

In the as-received condition, the material was characterized by equiaxed microstructure and <100> and <111> duplex texture at the top, middle and bottom sections. In spite of the billet rotation employed in the B_C route, the bottom section showed a consistently different microstructure and texture from the top and middle sections upon processing. Both the top and middle sections of the 1 to 6-pass billets were found to retain most of the <100> and <111> duplex texture, while the bottom region was essentially <110> with weak <100> and <111>. The 8-pass exhibited primarily <110> texture in all the three sections. Microstructural evolution resulting from the processing can be classified into two regimes: cell band formation in the early stages (passes 1-3) of processing and orientation splitting, which occurred during passes 4 to 8. Grain size analysis showed that the grains were reduced in diameter in the middle section from an average of 32.6µm in the AR sample to 0.60µm for the 8P sample. The Nb hardened significantly after the first pass and then gradually increased thereafter. A plot of the hardness value vs. d^{-1/2} revealed that Hall-Petch relation was valid for the top, middle (up to 6-pass) and bottom sections, with the top and middle section utilizing the same friction stress and “locking parameter” constants. The simulated texture of the middle section using a combination of ABAQUS FEM and Lapp softwares was found to be in good agreement with the experimental macro- and micro-texture. The top and bottom sections were not accurately predicted by this simulation.
CHAPTER 1

INTRODUCTION AND BACKGROUND

Introduction

Properties of materials depend greatly upon the type of processing the material has undergone. These different processing routes have an effect on the materials microstructure, crystallographic texture, and crystal structure. The changes in these parameters impart changes in the material properties.

Severe plastic deformation (SPD) has been shown to generate unique physical, microstructural, and crystallographic properties in materials. SPD is defined as a type of plastic deformation that can impart a true strain of greater than 4.00. There are many different types of SPD that can be used to process materials to generate large strains these include traditional types of SPD such as wire drawing and rotary swaging, and recent processing techniques such as three axis deformation, friction stir processing, and equal channel angular extrusion (ECAE).

In wire drawing (Figure 1.1a) the material, usually of a round cross section, is pulled axially through a round die that is slightly smaller than the diameter of the material. To achieve SPD the procedure must be performed several times to a small final diameter. This reduction in diameter of the material increases the materials length dramatically. Wire drawing has been shown to develop what is called a wire fiber texture in the material where all of the grains elongate and run parallel to the drawing direction [1].

In rotary swaging (Figure 1.1b) a cylindrical shaped material is inserted into a machine which contains a die made of two to four pieces with a tapered area from the wire diameter down to a smaller diameter. These dies rotate around the wires axis and cyclically compress the wire radially while the material is fed through the dies. Rotary swaging deforms the material into a shape similar to wire drawing and also generates textures similar to wire drawing [1].
While traditional SPD methods cause significant shape change, recent SPD techniques have been developed to cause minimal change in the materials overall geometry. One of these new SPD techniques is called multidirectional cold forging also known as three axis forging (Figure 1.1c). In this type of deformation, the material, usually of cube or off cube (where one edge is longer than the other two edges) shape, is deformed via compression on all three primary axes independently. This type of SPD has been shown to maintain the close to the original shape of the starting material, however it has been shown to be prone to inhomogeneous deformation and barreling or bulging [2].

Friction stir processing (FSP) is another newer method of producing SPD materials. FSP (Figure 1.1d) is a processing technique derived from the friction stir welding process. The technique involves plunging a non-consumable tool which is spinning at a high angular velocity into the material to be processed and moving the tool along the materials surface. In FSP the material being processed is typically a flat plate, however FSP has also been used to process cylinders and forged parts. One of the main drawbacks of FSP is inhomogeneous deformation developed due to the leading and trailing edges of the tool generate different microstructures as the tool moves across the material. Some advantages of FSP include the ability to perform the deformation with very little loss of material and also the ability to generate materials that can exhibit superplastic forming behaviors.

ECAE is another type of SPD that has garnered a lot of attention recently (Figure 1.1e). ECAE deformation involves a die with two channels of equal cross sectional area that intersect at an angle usually between 90° and 120°. The main mode of deformation in ECAE is simple shear which occurs parallel to the plane of intersection of the die channels [3]. This deformation occurs when a sample of material, called a billet, is forced through the angled channel in the die under high pressure. The material that emerges from the die after the extrusion process is of the same cross-sectional area as the material that was inserted into the die. This brings up the most intriguing part about this type of deformation, which is the material can be cold worked to high levels of strain with very little change in the materials bulk size and shape. This can be extremely useful in many different industrial applications that require a material to have a refined grain structure with a certain texture developed while still being in “bulk” form. A combination of ECAE and heat treatment has been shown to generate materials with superplastic forming
behaviors [4]. The ability to control the microstructures during subsequent extrusions by way of changing the orientation of the billet between each pass is of great beneficial use in ECAE.

![Diagram](image)

**Figure 1.1**: Types of SPD (a) wire drawing, (b) swaging, (c) three axis deformation, (d) friction stir processing, and (d) ECAE

Due to the complex nature of ECAE processing more research is needed to determine what the effects ECAE has on all types of materials. These factors require the need to examine the ECAE process to determine if this process has any distinct advantages over other SPD methods.

ECAE was developed in 1972 by V.M. Segal [4] and there has been a lot of research applied to this type of SPD however not much research has been completed on body centered cubic (bcc) materials and even less has been performed on reactor grade niobium. This research deals with characterizing the results of ECAE on reactor grade niobium (Nb) for a better understanding of the deformation and how the materials parameters change during this deformation process. The processing route that was chosen to be studied was route B\(_C\), which
involves a 90° clockwise rotation of the billet about the long axis between each pass up to 8 passes.

**Purpose of Thesis**

The purpose of this research is:

1. To understand the microstructure and texture evolution of 99.99% pure Nb processed by ECAE.
2. To determine if the ECAE process of Nb up to eight passes results in Ultra Fine Grains (UFG) or Ultra Fine Sub-Grains (UFSG) production, this then may lead to superplastic forming behaviors upon heat treatment.
3. Develop an FEM representation of ECAE process and simulate the texture evolution through eight passes in order to be able to compare it to the strain development and texture evolution of the actual process for scalability purposes.

The resulting SPD Nb billets were then sectioned and analyzed using X-ray diffraction (XRD) for macro texture, orientation imaging microscopy (OIM) for microtexture, and Vickers microhardness testing for progression of the work hardening of the Nb as shown in chapter 3. The result of the texture analysis was compared with the simulated ECAE texture development of bcc materials using a combination of an ABAQUS Finite Element Model (FEM) program in conjunction with Los Alamos polycrystalline plasticity (LApp) program. The variation of the microhardness developed across the billet from the top to the bottom was also compared to FEM simulation results of the strain distribution through the billet.
Background

Material Properties

The reactor grade niobium used for this study was of 99.99% purity, and is also known as residual resistivity ratio (RRR) niobium. The term RRR is a number that is acquired by taking the ratio of the materials electrical resistance at room temperature to its resistance at superconducting temperatures. Hence the higher the RRR number the lower the resistance at superconducting temperatures and the better overall superconductor that can be made from the material. Pure Nb has the highest known critical temperature of any pure element, which is one of the main reasons this type of Nb is commonly used for the production of radio frequency (RF) cavities. Some basic material properties for Nb can be seen in Table 1.1.

Table 1.1: Properties of Annealed niobium.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus of Elasticity</td>
<td>1.05 GPa</td>
</tr>
<tr>
<td>Poisson’s Ratio</td>
<td>.38</td>
</tr>
<tr>
<td>Hardness</td>
<td>60-100 Hv</td>
</tr>
<tr>
<td>Resistance to thermal shock</td>
<td>Good</td>
</tr>
<tr>
<td>Recrystallization temperature</td>
<td>750-850º C (dependent on purity)</td>
</tr>
<tr>
<td>Stress Relieving temperature</td>
<td>649-663º C (dependent on purity)</td>
</tr>
<tr>
<td>Density</td>
<td>8.57 gm/cc (3 x Al and ½ of Ta)</td>
</tr>
</tbody>
</table>

Radio Frequency Cavity Production

High purity Nb has been used for the production of RF cavities for many years. There have been many different techniques used to produce RF cavities each of which has certain advantages and drawbacks. The most traditional type of fabrication used consists of deep
drawing half cells of the cavities and seam welding the half cells together along the equator [5] as shown in Figure 1.2.

**Figure 1.2:** Traditional RF cavity production (a) deep drawing the half cell and (b) welding half cells together to generate the RF cavities.

Although this type of production technique is able to produce RF cavities quickly it has been shown that within the welded areas of the conventional RF cavities there is RRR degradation which can be critical to the performance of the RF cavity [6]. This problem creates the need to generate a fabrication technique that would avoid the seam weld in RF cavities. There is a recent type of RF cavity production that involves hydroforming the cavities in one piece generating a more efficient and ultimately cheaper production method shown in Figure 1.3.

**Figure 1.3:** Seamless RF cavity produced by hydroforming.
The material needed for hydroforming RF cavities must be a seamless tube with a rather small uniform grain size and elongation at break of greater than 30% [5]. The types of production investigated for making such a tube of Nb have been achieved using a sheet of material and deep drawing and spinning the material into a tube shape. This process requires large sheets of pure niobium with characteristics suitable for deep drawing while having a uniform grain structure throughout the material.

One novel and unique technique that has not had much success due to material limitations is back/container extrusion. Back extrusion involves placing an ingot of material in a heavy cylinder and pushing a ram of slightly smaller diameter into the ingot causing the material to extrude back up the ram in the final shape of a tube, as shown in Figure 1.4. The problem with this type of production is the rather large grain size retained from the starting ingot material interfering with the hydroforming technique resulting in necking at the reduced diameter iris area of the RF cavity [5].

![Figure 1.4: A diagram showing the back/container extrusion process](image)

Material characteristics needed for performing hydroforming and achieving a uniform thickness RF cavity is fine grain size, random or homogeneous texture, and large fraction of high angle grain boundaries [5]. This leads to the need to be able to generate a large billet of material with these characteristics and the capabilities to undergo deformations of rather large strains.
without fracture. A material with these characteristics can be achieved by applying the ECAE process on Nb billets.

**Equal Channel Angular Extrusion process**

**Overview**

The Equal Channel Angular Extrusion (ECAE) process is a SPD process developed by V.M. Segal in Soviet Russia around 1972 [4]. The deformation in the ECAE process is primarily a result of simple shear and is known to produce near-nano and nano sized grains with a homogeneous dispersion of orientations in the material [7]. One of the main attributes of the ECAE process is that the material shape post-deformation is similar to its initial shape thus enabling multiple passes upon the sample, allowing extremely high strains to be achieved. Being able to generate high strains and fine grains in a material while maintaining the materials bulk shape has the unique advantage of making large amounts of materials for the production of structural components with unique properties. This is unachievable with traditional techniques.

The ECAE process involves pressing a material through two channels of equal cross section and geometries. The most commonly used die angles between the intersections of these channels are 90°, 120°, and 135°. The smaller the angle of intersection the higher the strain induced on the material per pass. The die used for this study had one half inch diameter circular cross section channels intersecting at an angle of 90°. The results of the ECAE process can be influenced by several different variables during the experimental process. The main variables affecting the outcome of the ECAE process are primarily, the rate of extrusion, the temperature at which the extrusion was carried out, and the force applied to perform the extrusion. For this study the extrusion rate was kept at a constant rate of 0.25mm/sec for each pass. The temperature at which the extrusion was performed was approximately 70°F, or the room temperature of the lab. The force of the extrusion was varied to maintain the proper extrusion rate, the extrusion force was plotted in real time versus the extrusion distance as shown for one pass in Figure A.1. To reduce the effects of the friction forces both the die and billet were polished and lubricated with a high viscosity Permatex® brand lubricant.
The affect of die geometry

Assuming the billet moves at a constant rate through a sharp cornered die with no friction and the deformation zone is a single plane at the channel intersection, the slip-line field can be resolved into a simple diagram as seen in Figure 1.5 [8]. From this diagram some simple operating equations for the ECAE process can be developed as shown in Equations 1 and 2.

![Figure 1.5: Schematic of the (a) slip line diagram and (b) velocity hodograph for ECAE Processing without friction [8].](image)

\[ \sigma = -k \cot \theta \]  
\[ p = 2k \cot \theta \]  

The equations are valid only if no other forces other that the pushrod forces are applied. The constant k is the materials yield shear stress and the value \( \theta \) is the semi-angle between the die channels. From these values the stress \( \sigma \), along the line AO, and p, the pressure exerted by the pushrod can be determined.

As stated if friction is apparent, then this diagram and the resultant equations are no longer valid and another slip line solution technique is required. For sharp corner dies when friction is apparent the deformation zone expands into a fan shape and the material deformation no longer fills the die channel at the point of channel intersection as shown in Figure 1.6 [9].
From Figure 1.6a, the slip lines include a central fan AOB, the ‘dead’ metal zone AO_1B and two zones of uniform stress distribution OBC and OAD. The plastic flow of the material is concentrated inside the fan AOB while the stress is distributed into the rigid material is within zones OBC and OAD. The angle between the channels outer walls and the slip lines OA and OB is given by equation 3.

$$\eta = \frac{\pi - \arcsin(\eta/k)}{2}$$

Where \(k\) is the materials yield shear stress and \(\tau\) is the friction force. From this angle the angle of the central fan AOB can be found using equation 4.

$$\varphi = 2(\eta - \vartheta) \geq 0$$

With low friction the filling of the channel corner section may require additional back pressure. However in the case of \(2\vartheta = 90^\circ\) the slip line solution of Figure 1.6a remains correct for any amount of friction with no need for the application of back pressure.

From the corresponding velocity hodograph shown in Figure 1.6b, the discontinuity of tangential speed \([v]\) and the normal speed component \(v_n\) on OA and OB are:

**Figure 1.6:** Schematic of the (a) slip line field and (b) velocity hodograph for ECAE processing with friction [9].
Upon crossing the lines AO and BO the trajectories of an element of material change abruptly and the element undergoes simple shears $\gamma_1$ and $\gamma_3$ defined as:

$$\gamma_1 = \gamma_3 = \frac{M}{
u_n} \cot \eta$$

(6)

Inside the AOB area the speed components on the $\alpha$ and $\beta$ slip lines are given as:

$$v_n = V \sin \eta, \quad v_\beta = 0$$

(7)

During the passage through AOB the material elements are exposed to rigid rotation on the same angle $\varphi$ into a flow direction and to simple shear $\gamma_2 = \varphi$.

These formulas are valid for most cases until there is an introduction of a round corner of the die at the channel intersection upon which point there is a need for a new set of slip line solutions. A slip line field for ECAE through channels of unit thickness ($h = 1$) with a round corner angle $\psi$ of radius $R$ is shown in Figure 1.7a [9].

![Figure 1.7](image)

**Figure 1.7**: Schematic of the (a) slip line field, (b) velocity hodograph, and (c) material distortion for ECAE processing with round corner channels [9].

The diagram includes the central fan of BOA with a radius of $\rho$ and an angle of $\varphi$, also there is a rigid zone of $C_1BAC$ rotating about $O$ with an angular speed of $\omega$. From the
In kinematics consideration in Figure 1.7b there are separated slip lines CA and C₁B that are formed by circular arcs of radii r. The centers of these radii O₁ and O₂ are located at perpendiculars from O to the speed vectors in each channel. The slip line field is defined by the angles ψ, θ, and γ in Figure 1.7a while the other characteristics can be calculated from the formulae:

\[ R = \frac{1}{\sin(\theta + \psi)} \]
\[ R = \frac{\cot(\theta + \psi) - r}{\sin \gamma} \]
\[ \rho = r \tan(\theta + \varphi) \]
\[ \cos(\theta + \varphi) = \frac{r}{(1 - r \cos \gamma)^2}, \quad \theta_1 = \pi - (\gamma + \theta + \varphi) \]

The angles ψ and θ are defined by the tool geometry and the angle γ can be found from the relationship in equation 8:

\[ \frac{\tau}{k} = \frac{[\rho(\rho^2 + \rho^2) + ry - (1 - \cos \gamma)(\sqrt{\rho^2 - \rho^2} + \rho)]}{\rho \cos \gamma} \quad (8) \]

Assuming that the tangential stress τ is prescribed, then equation 8 gives a definite correlation between the angle γ and friction τ. For a die with a tool angle of 2θ = 90° with different τ/k values and varying the angle ψ the angle γ can be plotted as seen in Figure 1.8. The diagram is confined between γₘₐₓ = (3π/4 − ψ) for maximum friction and γ₀ for zero friction. The pressure pₐ can be found when γ is defined by integrating stresses along the boundary CAO into X₁ direction:

\[ \sum X₁ = 0 \]

From this all slip lines can be calculated and stresses can be checked at points C and C₁. One more restriction for the solution is τ/k ≤ cos2ψ. For any larger frictions the rigid zone C₁BAC disappears and the slip line solution becomes identical to Figure 1.6a [9]. As can be seen in Figure 1.7c the material deformation is not homogeneous across the cross-section of the material being extruded. The shear deformation apparent at the top two thirds of the material rotates towards lower levels of shear over the bottom third of the material.
The effect of processing route and number of passes

ECAE is used to generate high strains in bulk material. Multiple passes of the billet through the die are required in order to achieve strains high enough to be considered SPD. Due to the fact that the geometry of the billet is preserved after extrusion it can then be reinserted back into the die in many different combinations. There are many different configurations in which this can be accomplished however the most common types, or routes, are Route A, Route B_A, Route B_C, and Route C. Each route involves rotations about one or two of the primary axes of the billet. The primary axes of the billet are shown in Figure 1.9. As can be seen the three primary directions are the Extrusion Direction or ED, the Normal Direction or ND, and the Transverse Direction or TD.

Figure 1.8: An effect of round corner angle $\psi$ on slip line characteristics for a die with $\theta = 90^\circ$: (1) $\tau/k = 1$; (2) $\tau/k = 0.95$; (3) $\tau/k = 0.9$; (4) $\tau/k = 0.75$; (5) $\tau/k = 0.5$; (6) $\tau/k = 0$), $\theta_1$ and $\phi$ [9].
As shown in Figure 1.10 the result of one pass through the die is that the material is sheared in the ED ND plane [10]. The first pass for all of the said processing routes is the same.

Figure 1.9: Diagram representing the primary directions of the billet in the ECAE process.

After the first pass the resulting billet is always rotated about the TD direction 90° before the ensuing pass as to maintain the leading edge of the billet as being reinserted back into the die. The second rotation is about the ED. As can be seen in Figure 1.11, the four primary rotations about the ED are, 0° for Route A, 90° clock wise (CW) for even numbered passes and 90°

Figure 1.10: Diagram of the result of one pressing of ECAE [10].
counter clock wise (CCW) for odd numbered passes for Route BA, 90° in the same sense about the ED after each pass for Route BC, and 180° rotation for Route C after each pass.

Figure 1.11: Diagram of the Four primary processing routes of the ECAE process

The accumulation of the shear after the second pass of each of the routes is shown in Figure 1.12. The second pass of Routes BA and BC are the same and are shown as Route B in Figure 1.12b.

Figure 1.12: Diagram showing the accumulation of shear for the second pass for the main deformation routes (a) Route A, (b) Routes BA and BC, and (c) Route C. [10]
Table 1.2: Shearing characteristics associated with the four main processing routes. [10]

<table>
<thead>
<tr>
<th>Route</th>
<th>Plane</th>
<th>Number of Pressings</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0 1 2 3 4 5 6 7 8</td>
</tr>
<tr>
<td>A</td>
<td>ED</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td></td>
<td>TD</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td></td>
<td>ND</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td>B_A</td>
<td>ED</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td></td>
<td>TD</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td></td>
<td>ND</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td>B_C</td>
<td>ED</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td></td>
<td>TD</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td></td>
<td>ND</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td>C</td>
<td>ED</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td></td>
<td>TD</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
<tr>
<td></td>
<td>ND</td>
<td>□ □ □ □ □ □ □ □ □</td>
</tr>
</tbody>
</table>

To understand how the billet deforms for passes greater than two refer to Table 1.2 [10]. The planes ED, TD, and ND represent the shape of a square material element when viewed from that direction after each pass as labeled in Figure 1.9. In Table 1.3 the route name, definition of the route, and the affect of the route on the element or grains are summarized.
Table 1.2: Shearing characteristics of four different processing routes.

<table>
<thead>
<tr>
<th>Route</th>
<th>Definition</th>
<th>Deformation</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>No rotation about ED</td>
<td>Elongating in the direction of the ECAE shear plane towards the ED in the shape of a rhombohedron</td>
</tr>
<tr>
<td>B_A</td>
<td>Alternating CW and CCW 90° rotation about ED after each pass</td>
<td>Elongating along the ED with continuous changes in all three perpendicular planes</td>
</tr>
<tr>
<td>B_C</td>
<td>Either CCW or CW 90° rotation about ED after each pass</td>
<td>Deforming on all three perpendicular planes while returning to a cubic element after every 4n passes</td>
</tr>
<tr>
<td>C</td>
<td>180° rotation about ED after each pass</td>
<td>Shear reversal every 2n passes causing the element to ideally return to cubic on the even passes</td>
</tr>
</tbody>
</table>

The amount of shear developed by each pass of ECAE must now be quantified. Knowing the geometries of the ECAE die the shear developed in each pass of ECAE can be determined by equation 9 [11]:

$$ \varepsilon_n = \frac{N}{\sqrt{3}} \left( 2 \cot \left( \frac{\Phi + \psi}{2} \right) + \psi \right) $$

(9)

where N represents the number of passes, $\Phi$ represents the die intersection angle, and $\psi$ represents the outer bend or sweep angle as shown in Figure 1.13. Using the die geometry of 90° for $\Phi$ and 40° for $\psi$ in this study the strain produced per pass of ECAE is 0.93 mm/mm.
Microstructure and texture evolution during ECAE

As has been stated, the ECAE process imparts SPD by way of shear. One of the main proponents of ECAE is that it has been shown to refine the grain size and structure of the material to the near nano scale [7, 12, and 13]. The SPD of the material is reflected in the microstructure and texture which determines the mechanical behavior such as hardness and tensile strength [14]. It has been shown that ECAE can have substantial effects on the tensile strength and anisotropy of materials [15]. The microhardness is also greatly influenced by the ECAE process and has been documented by Sandim et al [16]. The texture evolution of materials subjected to ECAE has been documented for many Face Centered Cubic (FCC) materials and a few Body Centered Cubic (BCC) materials and was shown to generate textures similar to simple shear obtained by way of torsion [3, 17, and 18]. A typical texture developed by ECAE is shown in Figure 1.14.

Figure 1.13: Schematic of an ECAE die with a curved outer corner defining the locations of the $\Phi$ and $\psi$ angles.
The formation of the submicron grains has been shown to be very complex and involves many mechanisms in FCC materials [12]. The first mechanism of grain refinement is the accumulation of dense dislocation walls or cell bands within the grain boundaries aligned with the shear plane of the die. Upon further deformation another mechanism became apparent, which is accompanied by the appearance of shear bands cutting across the cell bands. The final mechanism as described in [12] is the orientation splitting of the unstable highly strained cell and shear bands of the material. These mechanisms are apparent in materials deformed by route B\textsubscript{C} processing and result in a more chaotic microstructure containing lower misorientation subgrain islands within the ultra-fine grain matrix [12] compared to those deformed by route A.

Figure 1.14: Typical texture developed after one pass of ECAE in BCC materials.[3]
CHAPTER 2

EXPERIMENTAL PROCEDURE

Material

This research was performed using niobium of 99.99% purity. The material was supplied by Black Labs LLC. The bulk material came in six 12.7mm (1/2 in) diameter and approximately 150mm (6 in) long pieces. Each rod was sectioned into three pieces and of approximately 50mm (2 in) long.

These billets were then ground round on one end into a bullet shape to lower stress concentrations on the die wall at the start of the extrusion process created by the leading edge of the billet. Each billet was then polished with 4000 grit SiC paper and then 6 µm diamond suspension on an MDPan polishing pad to decrease frictional effects during the extrusion process.

ECAE Procedure

Equal Channel Angular Extrusion is a severe deformation process that involves the intersection of two channels of equal cross-sectional area. The die, shown in Figure 2.1a, used for this research had a round cross-section with a diameter of 12.7 mm (1/2”) and a channel intersection angle, $\phi$, of 90°. The outer sweep angle, $\psi$, was 45°. This geometry resulted in an average strain of approximately 0.93mm/mm per pass as per Equation 9. The die was made out of silicon bronze material. The die halves were held together using four 3/4”-16 x 4 1/2” grade 8 hex cap screws and four 3/4”-16 grade 8 hex nuts.

In preparation for extrusion the channel was polished with 3 µm diamond suspension with a high speed rotary tool to a mirror finish to reduce the frictional effects during processing. Prior to the extrusion process the billets and die channels were coated with a high viscosity Permatex®
red engine assembly lubricant to further reduce the frictional effects between the billet and the die.

Figure 2.1: (a) ECAE die half used for study and (b) MTS testing apparatus used for ECAE

The billet was then placed between the die halves and the bolts secured the halves together. A pushrod was inserted into the die onto the top of the sample. The die was subsequently placed on the mechanical testing systems (MTS) axial displacement table seen in Figure 2.1b. The billets were extruded at a rate of 0.25 mm/sec and the load and displacement were recorded in real time.

In this research, Route Bc was used for ECAE processing. This particular route involves a 90° rotation of the billet about the extrusion axis in the clockwise direction prior to each ensuing pass. Upon completion of each pass the billet was removed from the die and the die was inspected for any damage or wear. The billet was then either sectioned for analysis or prepared for an additional extrusion by grinding and polishing. If necessary the billet was passed through a resizing die.

After the first three passes, the billet was cleaned, ground, and polished to the proper shape for subsequent extrusions. Grinding involved removal of all flashing material that occurred between the die halves and reshaping the trailing end of the billet that was not extruded so as to
fit back into the die channel properly. Repolishing was done with the 4000 silicon carbide paper and 6µm diamond polish suspension to return the billet back to the smooth surface for reduced frictional effects.

The use of a resizing die was required after the fourth pass and each of the following passes to return the billet to the proper diameter. This was required because the stress required to extrude the billet through the ECAE die caused the ECAE die to elastically deform allowing the billet to increase in diameter plastically (to approximately 13.0-13.5mm after extrusion). The resizing die made it possible to return the diameter of the billet to 12.7mm with no loss of material as opposed to grinding the billet back to the correct diameter for subsequent extrusions, which might have decreased the amount of usable material for analysis.

**Sample Preparation**

In order for proper analysis of microstructure and texture in materials the samples must be mounted and prepared properly. Each analysis technique used has different requirements for the sample preparation. Improper sample preparation can yield incorrect data and lead to invalid conclusions.

In order to analyze the results of the ECAE process samples were cut from the uniform deformation zone along the extrusion direction of the billets after the 1\textsuperscript{st}, 2\textsuperscript{nd}, 3\textsuperscript{rd}, 4\textsuperscript{th}, 5\textsuperscript{th}, 6\textsuperscript{th} and 8\textsuperscript{th} pass. These samples were labeled as 1P, 2P, 3P, 4P, 5P, 6P, and 8P respectively. The uniform deformation zone is defined as the zone located more than one diameter of the billet from the leading and trailing ends of the billet. One sample was also taken from the center of the as received (AR) billet. The samples were sectioned from the billet as shown in Figure 2.2. Analysis of the samples was done at the locations labeled top, middle, and bottom also shown in Figure 2.2. The analysis of the top and bottom of the billet was performed within the outermost two millimeters from the top and bottom edges of the sample and the middle analysis was performed within the middle five millimeters of the samples.
Material Sectioning and Mounting

Material sectioning was performed using a Struers high speed wet saw. The billets were first sliced along the extrusion direction at 18mm from the leading edge of the billet. The next slice was taken from the billet approximately 8mm from the first slice toward the trailing end of the billet. This disk shaped piece of material was then sliced in half along the ND-ED plane.

The sectioned samples were then mounted to facilitate the handling of the samples during the grinding and polishing stages of the sample preparation. The samples were mounted in Buehler epoxy resin and epoxy hardener. It is of great importance to keep track of the extrusion and normal directions when mounting the samples into the epoxy mount. The epoxy resin and hardener were chosen because of their low temperature mount. This type of mount is typically used for cold worked materials. It does not introduce any increased temperatures that could cause stress relaxation or recrystallization of the material being mounted as opposed to typical hot mounting techniques. This epoxy resin mounting system typically took around 8 hours to fully cure before grinding and polishing could commence.

Figure 2.2: Representation billet orientations, sectioning, and areas of interest for analysis
**Mechanical Grinding and Polishing**

After the epoxy hardened, the samples were ground and polished for analysis. The first step was to plane grind the sample until a flat uniform surface was achieved using SiC grinding papers with grits ranging from 1000 grit to 2400 grit paper. It was important to ensure that the surface to be analyzed remained level so as to avoid introducing any false sample orientation directions. Furthermore, samples were cleaned with metallography soap and de-ionized water in between each grinding and polishing step. This ensured the removal of any residual particles from the previous grinding or polishing step.

Subsequent to the plane grinding steps, the samples were polished using different polishing cloths and diamond polish suspensions and lubricants. The first polishing step used an MDLargo polishing cloth with a nine micron diamond polish lubricated with ethyl alcohol until all scratches from the 2400 grit SiC grinding paper were removed. The next step of polishing was done using a three micron diamond polish on an MDDur polishing cloth. The DP blue lubricant was used as a suspension lubricant to remove all the scratches left from the 9μm DP polish.

The samples were then polished using an MDChem polishing pad with a four parts 0.04 micron OP-S to one part 30% hydrogen peroxide mixture for five minutes changing the orientation of the sample after each minute. They were then cleaned in deionized water and quickly dried with a blast of compressed air as to prevent any water spots from forming. At this stage the samples were ready for X-ray diffraction (XRD) and micro-hardness testing.

In order to perform orientation imaging microscopy (OIM) the samples were further polished. Using a vibratory machine called a vibromet polisher in conjunction with a chemical etchant. This was done for one hour to remove any minor directional scratches on the sample.

Next the samples were etched in a mixture of one part hydrofluoric acid (HF), 2 parts nitric acid (HNO₃) and 4 parts phosphoric acid (H₃PO₄) for one minute then removed and fully rinsed and dried with a blast of compressed air again. Once the samples had been etched they were placed back on the vibratory polisher for one hour to remove any surface topography that would interfere with the OIM scans they were then cleaned with de-ionized water and dried with a blast of compressed air to prevent any water spots from forming on the sample surface.
Material Characterization Techniques

Micro-hardness Testing

Micro-hardness test measures the materials resistance to localized plastic deformation. The test consists of an indenter of known shape and size which is pressed into a material under a set load for a set period of time. The indenter tip can be any known geometry typically a ball or pyramid shaped. The indentation made in the material being tested is then measured and the volume of displaced material is used to calculate the hardness of the sample.

Micro-hardness testing in this research was performed using a Newage Testing Instruments C.A.M.S. computer assisted micro hardness tester (Figure 2.3) with a Vickers hardness tip. This tester had a pyramidal shaped tip and left a diamond shaped indentation in the sample. A load of 100g with a dwell of 10s was used to perform the indentations. This load and dwell setup was used because it was determined that the indentation made encompassed a large number of individual grains thus removing any anisotropy within each grain and giving the average hardness of the material. Vickers microhardness number (Hv) was calculated from the average lengths of the diagonals along with the load used. Three rows of fifty indents were performed on each sample starting 0.25mm from the top of the sample and spaced 0.25mm apart all the way to the bottom of the sample. This spacing was used because it was at least five times the diameter of the largest indentation. These indentations were used to determine the variation of the hardness from the top to the bottom of the sample.

Figure 2.3: Newage Testing Instruments C.A.M.S. Microhardness tester
X-ray Diffraction

X-ray diffraction was used to measure the macro-texture of the specimen. The X-ray diffractometer used in this research was the Philips X’pert XRD equipped with a copper target k-alpha X-ray tube this x-ray tube emits x-rays at a known wavelength of 1.5406 Å. Using X-ray diffraction (XRD) is a common way to determine the macro-texture development in bulk materials.

To perform this type of analysis the samples were lightly ground to provide a level surface and were positioned in the goniometer with the ED aligned with the y-direction of the X’pert XRD. The x-ray beam was then rotated around the sample along a plane that was perpendicular to the plane of the surface of the material at an angle of 2\(\theta\). When the beam hits the surface of the material some of the x-rays diffract off of the sample which were collected at a receiving slit. The intensity of the x-rays was plotted versus the angle 2\(\theta\). When the intensities were plotted for a polycrystalline material the plot showed peaks at certain angles.

The angle of the diffracted x-rays is related to Bragg’s law, and depends on the atomic spacing of the atoms in the material as shown in Figure 2.4. To determine the planes that are causing the diffraction, the following Bragg’s law is solved for the interplanar spacing of the diffracting planes [1].

\[
n\lambda = 2d \sin(\theta)
\]

(10)

where \(n\) is an integer, \(\lambda\) is the wavelength of the emitted x-rays, \(d\) is the interplanar spacing of the atoms of the material and \(\theta\) is the diffraction angle or angle between the incident rays and the scattering planes For the case of Nb in this study the first three primary peaks were at 2\(\theta\) angles of roughly 38.4°, 55.5°, and 69.6°, which compared to the (110), (200), and (211) planes respectively.
When the families of planes were determined a texture measurement was made of the material. This texture measurement sets the \( 2\theta \) value constant at the locations of the peaks during the \( 2\theta \) scan and moves the sample through the other degrees of freedom in the XRD, such as wobbling the sample in the plane of the surface and spinning the sample about its normal direction. This was done in order to generate a large amount of data from the bulk of the material for the assembly of pole figures. The (110), (200) and (211) pole figures are generated from the texture measurements at those peaks using the preferred orientation package Los Alamos (popLA). The program popLA is a free shareware program developed by Los Alamos National Laboratory to facilitate in generating pole figures from XRD raw data. PopLA uses the data collected from texture measurements of the individual poles and compiles them into one set of pole figures to determine the macro-texture.

**Scanning Electron Microscopy/Orientation Imaging Microscopy**

Scanning electron microscopy (SEM), is a widely used type of analysis in the materials analysis field. It is used to obtain high resolution images of the microstructure of processed materials. In this study, Zeiss 1540 XB FE-SEM (Figure 2.5) was used to collect data from samples for microstructure and micro-texture analysis. Electron back scatter diffraction (EBSD) or OIM is what is used to determine the micro-texture of the materials. During OIM
measurement, the material is inclined to an angle, usually 70 degrees, to the electron beam. This angle allows the beam to reflect off of the lattice planes within the first 30-40nm of the material.

![Zeiss 1540 XB Field Emission Scanning Electron Microscope with OIM](image)

**Figure 2.5**: Zeiss 1540 XB Field Emission Scanning Electron Microscope with OIM

The reflections are then captured on a phosphorous screen in the form of bands and poles. The bands represent the different reflecting planes of the material. The thicknesses of the bands are inversely proportional to the interplanar spacing of the atoms of the grain. The poles are the intersection of the bands. These bands and poles form Kikuchi patterns on the phosphor screen and the orientation and shape of these patterns are used to determine the orientation of the atomic structure in the scan area.

These patterns determine the orientation of the spot being scanned with respect to the global orientation of the material. This is useful in gathering information to determine multiple different parameters from the micro-texture of the material to grain boundary character distribution of the material.

All OIM data in this study were gathered and analyzed using EDAX-TSL OIM v5.2 software. In the EDAX software, the user sets up scans to traverse a certain area in a point by point manner to determine the orientations of the materials microstructure in that area. The user sets the scans to move along the sample at a certain spacing which is much smaller than the grain size of the material. The orientation imaging software takes the data from each point and maps it
out on a grid which then gives the user a map called the orientation map of the scan of the material.

The scans performed in this experiment consisted of 200x magnification scans for the AR, 250x magnification scans for the 1P and 2P samples. For the 3P sample the scan magnification was 500x. The magnification used for the 4P, 5P, 6P, and 8P was 1000x. The reason for these magnifications was to acquire enough data within the scan area to generate useful and reliable results within the area scanned in a reasonable amount of time.

Finite Element Modeling and Texture Simulation

ABAQUS Model

Finite element modeling (FEM) of this particular deformation process was performed at Brown University Department of Mechanical Engineering.

Using ABAQUS 6.1 FEM software, a model of the die, billet, and pushrod were built. The die halves were modeled using discrete rigid non-deformable shells which were of the same geometry as the actual die used for the experimental extrusions.

These die halves were meshed using R3D4 (4 node 3-D bilinear rigid quadrilateral) elements. The billet was made using C3D4 (4 node linear tetrahedron) deformable elements and was given similar geometries as the actual billets and die used in the experimental ECAE process. The material parameters used were that of reactor grade niobium, and the hardening characteristics of the material were taken from a typical stress strain curve from tensile testing with the hardening linearly extrapolated out to a strain of 2. The pushrod was simulated using a discrete rigid wire element and only interacted with the billet so as to allow the pushrod to be larger than the channel area for complete coverage of the billet on the die.

After the simulation finished multiple data sets were collected, such as the force required to perform the deformation, the strain distribution maps across the billet in all three mutually perpendicular planes (i.e. ND/ED, ED/TD, and ND/TD planes), and each strain component (i.e. ε11, ε12, ε13, ε22, ε23, and ε33) for the top, left, right, bottom, and middle locations (as shown in Figure 2.6) for use in texture evolution simulation.
Texture Evolution Simulation

In order to simulate the texture evolution of niobium during route B_C processing multiple programs were used. Los Alamos polycrystalline plasticity (LApp) was used for the texture development from each pass, and popLA was used to analyze the data. In popLA multiple steps were required to properly process and analyze the data and to convert the data for use back in LApp. Figure 2.7 shows a flow chart used for texture simulation.

Figure 2.6: Locations of strain data acquisition where (1) top, (2) right, (3) bottom, (4) left, and (5) middle of the billet

Figure 2.7: Flow chart used for texture simulation.
The first step was to convert the strain components from the results of the FEM model into an incremental strain tensor file (STRAIN.txt) for each location individually for use in LApp program. Then using the initial texture of the as received (AR) Nb a TEXIN file was created for use as the initial input texture. LApp used this data together with the STRAIN.txt to simulate the texture of the first pass for each location.

Before the resulting texture can be used for the next pass, it was converted into popLA pole figure files. Using popLA, the files were then smoothed to 5 degrees and the pole figure images were captured. Also using popLA the data set for each location was tilted 90 degrees about the horizontal to simulate the ED rotation and rotated 90 degrees about the center axis to simulate the reinsertion into the die that was performed for the B_C processing route. After performing the rotations on the data set the pole figure files were then converted back into weights files and renamed for the proper location (for example 1p output top was renamed 2p input right) for the next pass in LApp. Then this process was repeated until there were simulated texture files up to the 8th pass for each of the five locations shown in Figure 2.6.
CHAPTER 3

RESULTS AND DISCUSSION

Effect of ECAE processing

Effect of ECAE on microhardness

The Vickers microhardness (Hv) results for each pass are shown in Figure 3.1. The starting microhardness for the as-received niobium samples averaged 55 ± 5 Hv. The microhardness after one pass showed a substantial increase for all sections. The hardness of the top and middle sections increased by 127% to a value of about 125 ± 7 Hv. The bottom section showed an 87% increase in hardness to about 103 ± 12 Hv after one pass. After two passes there was only a slight 4% increase in the hardness across all sections. The top and middle sections increased to about 133 ± 4 Hv while the bottom section increased to about 111 ± 13 Hv. After the third pass of ECAE the top and middle sections showed again only a slight 4% increase in hardness to about 139 ± 4 Hv while the bottom section showed a more substantial increase of about 15% to 129 ± 9 Hv. The fourth pass of ECAE caused an increase in hardness to the middle and the bottom section while showing little change in the top section. The middle section increased to a value of about 145 ± 3 Hv and the bottom section increased in hardness to about 138 ± 7 Hv. The top section remained at a hardness of 140 ± 3 Hv after the fourth pass. The microhardness of the fifth pass showed a slight decrease (about 2%) in the middle and bottom sections with an increase (about 2%) at the top section. The middle section decreased slightly to about 144 ± 3 Hv while the bottom decreased to about 136 ± 6 Hv, the top section increased to a hardness of about 143 ± 3 Hv after the fifth pass. The 6p sample showed an increase in the hardness at the top and bottom with another decrease in the hardness of the bottom of the sample. The top and middle sections showed a microhardness number of about 150 ± 3 Hv after the sixth pass while the bottom hardness number fell all the way to about 129 ± 14 Hv. The 8p sample Vickers microhardness numbers increased across the entire sample. The microhardness for the
middle section increased to about 169 ± 2 Hv while the top section increased to a value of about
165 ± 4 Hv and the bottom section increased substantially to about 155 ± 11 Hv.

Figure 3.1: Plot of the Vickers microhardness for the top middle and bottom sections
with respect to the pass number

As received Nb microstructure

The AR Nb was analyzed to determine the starting microstructure, macrotexture,
 microtexture, and Vickers Microhardness. From these starting properties the influences that the
ECAE SPD process has on the materials microstructure could be determined.

Figure 3.2 showed the micrograph of the AR Nb. It was evident that there were large
equiaxed grains with an average grain size of 32.6μm. The grains showed little to no
deformation within the grain boundaries. The crystal direction of the grains varies primarily
from <001> to <111>.
Figures 3.3a and 3.3b show the macrotexture and microtexture IPF’s respectively. These figures show a similar strong (001) peak blending to a slightly weaker (112) peak concurrent with the OIM micrograph. These characteristics are common in wire drawn and annealed Nb rod stock. The calculated texture strength of each of the micro and macrotextures are shown to be around 20.

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Effect of ECAE on microstructure

One pass. The microstructure of the cross section of niobium after one pass of ECAE revealed two primary regions of deformation as shown in Figure 3.4. The first region of deformation was roughly the top two thirds of the sample where the grain structure is deformed mostly by simple shear at the plane created by the intersecting channels of the die (the shear plane). This region is called the top section shown in Figure 3.4a. The grains show preferential alignment with this shear plane and deformation bands called shear bands are aligned with both the shear plane and the extrusion direction. The primary direction of the grain orientation showed mostly a (112) crystal direction with some (001), (111), (102) and (212) directions dispersed throughout.

The second section called the bottom is shown in Figure 3.4b showed little shear deformation and has been called the rigid zone (9) shown in Figure 1.4. As can be seen the bottom section showed no preferential alignment of the grains shape in any direction. There were shear bands seen within the grains however that are oriented parallel and perpendicular to the shear direction. The crystal orientation within the grains however showed more (102) direction with a few (101) and (112) directions mixed throughout when compared to the top section.

Figure 3.4: OIM map showing the deformed microstructure of Nb after 1 pass of ECAE ($\varepsilon \approx 0.93$) where (a) is the top section and (b) is the bottom section
Two passes. Again the microstructure of the cross section of ECAE deformed niobium subjected to two passes route B_C processing revealed two primary deformation zones as seen in Figure 3.5. The top section (Figure 3.5a) again showed the grain shapes elongated along the shear plane with shear bands within the HAGB aligned with the shear plane and the ED. The primary crystal orientation of the material within the grains showed mainly (001) directions with some (111), (112) and (102) directions dispersed throughout.

The bottom section (Figure 3.5b) showed no grain shape orientation preference. The shear bands within the grains show some cross hatching aligning with and perpendicular to the shear direction within the grains. The orientation of the crystal direction showed primarily (102), (101), and (112) directions with a few (212) and (111) directions mixed throughout.

Three passes. After the third pass of ECAE on the niobium billets there was a noticeable change in the microstructure across the cross section. As can be seen in Figure 3.6 there are three distinct regions across the billets cross section. These three regions were given the names top, middle, and bottom and were present for each subsequent pass of the ECAE process. The top section is at a position within the top quarter of the billet while the middle section has a unique microstructure prevalent in the middle half of the cross section and the bottom section has its very own microstructure located within the bottom quarter of the billet cross section.
The top section shown in Figure 3.6a showed a very heavily feathered subgrain microstructure within each grain. This feathered subgrain microstructure is a result of the shear deformation material distortion reversal of the first passes shear plane with the shear plane of the third pass as shown in Figure 3.7a. The microstructures crystal orientation was shown as being close to random.

The middle section (Figure 3.6b) showed a crystal orientation of mostly (102) directions with some (212) and (001) directions mixed in. The middle section also showed the grains to align with their long axis oriented parallel with the shear plane with a subsequent decrease in the amount of shear banding within each grain boundary.

The bottom section shown in Figure 3.6c showed a very unique microstructure. There was mostly (212) crystal orientations with some (112) and (101) directions and very few (111) directions visible. The shear plane material distortion reversal prevalent in the top section was again visible in the bottom section a graphical representation is shown in Figure 3.7b

![Figure 3.6](image)

**Figure 3.6**: OIM map showing the deformed microstructure of Nb after 3 passes of ECAE ($\varepsilon \approx 2.79$) where (a) is the top section (b) is the middle section and (c) is the bottom section
Four passes. The microstructure from the fourth pass of ECAE is represented in Figure 3.8. The top section shown in Figure 3.8a showed a reemergence of the heavily feathered rather large elongated grains aligned with the shear direction. There is a large quantity of (111) crystal orientations with a few (001), (102), (212), and (112) directions throughout.

The middle section shown in Figure 3.8b showed a substantial amount of grain refinement and orientation splitting grain subdivision. The main crystal orientation apparent in the middle section was that of (112) and (111) directions with a random dispersal of all other directions throughout the microstructure. The smaller grains still showed an apparent preferential to align with the shear direction of the fourth pass.

The bottom section shown in Figure 3.8c showed a low amount of shear deformation associated with the ECAE process however it did show some grain refinement. The crystal orientation of the bottom section showed a mixture of mostly (112) and (212) directions with small amounts of (100), (111), and (101) directions spread throughout.

Figure 3.7: Graphical representation of shear plane reversal from one pass to three pass for the top (a) and bottom (b). The first element represents the material distortion after one pass of ECAE, the R+2p+R arrow represents a 90º rotation after the first pass plus the second pass of ECAE plus another 90º rotation, the second element represents the material distortion after two passes and before three passes, assuming there is little to no effect on the material distortion shape at the left and right sides of the billet from the second pass, 3p arrow represents a 3rd pass of ECAE, and the third element represents the material distortion after the third pass.
**Figure 3.8:** OIM map showing the deformed microstructure of Nb after 4 passes of ECAE (ε ≈ 3.72) where (a) is the top section (b) is the middle section and (c) is the bottom section

**Five passes.** The micrographs of the fifth pass sample of ECAE are shown in Figure 3.9. The top section in Figure 3.9a showed elongated grains aligned with the shear direction. The crystal direction of the top section was primarily (212) with a random dispersal of all other directions throughout. There were some finer grains spread around and bordering larger feathered shear banded grains.

The middle section shown in Figure 3.9b showed heavy grain refinement with grains aligned with and perpendicular to the shear direction. The crystal orientations of the middle section were mainly (001), (112), and (212). The amount of shear banding showed a large decrease with more orientation splitting within the grains.

The bottom section showed in Figure 3.9c showed heavily deformed subgrain structure. The primary direction of the bottom was (101) with some (102) and (112) directions visible. There was no apparent grain shape orientation preference visible in the micrograph of the bottom section.
Six passes. The microstructure of the billet cross section is shown in Figure 3.10. The top section shown in Figure 3.10a showed more grain refinement with the grains elongated and aligned primarily with the shear direction. There was mainly (111) and (001) directions present with some (102) and (112) directions sparingly throughout. The grain structure started to become more homogeneous within the top section.

The middle section shown in Figure 3.10b showed a very homogenous and mostly equiaxed grain structure with a crystal direction of mainly (111) and (001) crystal orientations with a few (102) and (212) directions evenly spread throughout. The grains shapes showed little to no alignment preference with any of the primary directions of the billet or shear plane.

The bottom section shown in Figure 3.10c showed less refinement that the top and bottom sections. The crystal orientation of the grain in the bottom section showed an even spread of (112), (111), (212), and (101) directions evenly spread all over the bottom area. The grains have started to elongate and show a slight alignment with the ND of the billet after six passes of ECAE.

**Figure 3.9:** OIM map showing the deformed microstructure of Nb after 5 passes of ECAE ($\varepsilon \approx 4.65$) where (a) is the top section (b) is the middle section and (c) is the bottom section.
Eight passes. The micrographs for the eighth pass of ECAE are shown in Figure 3.11. The top section shown in Figure 3.11a showed a rotation of the crystal orientation to primarily the (101) direction with a few (001), (212) and (102) directions spread throughout. The grains were elongated and aligned with the shear direction. The concentration of subgrains within the HAGB decreased substantially.

The middle section shown in Figure 3.11b showed extensive grain refinement along with the rotation of the crystal orientation of the grains. The crystal orientation of the grains showed a rotation to mainly (101) directions with a homogeneous dispersion of low amounts of (102), (212), (111), and (001) directions throughout the section. The grains formed in this section after eight passes of ECAE were equiaxed and homogeneous.

The bottom section shown in Figure 3.11c showed fine elongated grains aligned about 15 degrees off of the ED axis. The crystal orientation of the grains were primarily (101) and (111) with lesser amounts of (112) and (212) directions evenly spread throughout.
Effect of ECAE on grain thickness

To determine the extent of grain refinement the thickness of the grains minor diameter was measured. This was done taking the point to point misorientation of the micrograph perpendicular to the major direction for all sections and determining the distance between the 15 degree misorientations. The result of this analysis is shown in Figure 3.12.

As can be seen the grain thickness of the niobium after 1p of ECAE the top and middle sections decreased from the AR size of 32.6 ± 0.7 μm to 11.4 ± 0.7 μm for the top and 10.1 ± 0.7 μm for the middle. The bottom section increased in grain size to 36.8 ± 0.7 μm. The second pass of ECAE had little influence on the decrease in the grain thickness of the first pass. After the third pass of ECAE however there was a large decrease across the entire sample. The top and middle sections were close again at a thickness of 2.85 ± 0.35 μm. The bottom section showed a decrease to 8.83 ± 0.35 μm after 3 passes. The fourth pass of ECAE continued to show refinement of the middle section down to the sub micron level of 0.85 ± 0.15 μm. The top and bottom sections of the 4p sample had a thickness of 1.35 ± 0.15 μm. The 5p sample showed little refinement in the middle section maintaining a thickness of 0.84 ± 0.15 μm while the top section decreased to 0.93 ± 0.15 μm. The bottom section of the 5p sample showed an increase in the grain thickness to 1.79 ± 0.15 μm. After six passes of ECAE the top and middle sections of the billet were 0.99 ± 0.15 μm and 0.84 ± 0.15 μm respectively. The bottom section after six passes again showed an increase to 2.42 ± 0.15 μm. After eight passes of ECAE all sections were refined to the submicron level. The middle section showed refinement to a thickness of

Figure 3.11: OIM map showing the deformed microstructure of Nb after 8 passes of ECAE (ε ≈ 7.44) where (a) is the top section (b) is the middle section and (c) is the bottom section
0.60 ± 0.15 μm while the top and bottom sections decreased in thickness to a value of 0.78 ± 0.15 μm.

**Figure 3.12**: Plot of the average grain thickness for the top middle and bottom sections with respect to the pass number

### Effect of ECAE on Grain Boundaries

**Middle Section.** Shown in Figure 3.13 is the GBCD of the middle section. As can be seen the CSL boundaries changes very little throughout the deformation process. The two to five degree boundaries show a marked increase from 35% for the AR sample up to 64% after the first pass then there was a steady decrease after each successive pass all the way to the 8p sample where the value is down to 26% two to five degree boundaries. The five to fifteen degree grain boundaries start at 13% for the AR sample increases for each pass up until the 3p sample at a value of 29%. After the forth pass the five to fifteen degree boundaries decreases to 20% where it stays for the 5p and 6p then drops again after the eighth pass to 15%. The HAGB (15 to 180
deg) starts at 46% for the AR sample and then drops after the first pass down to 13% then steadily rises for each successive pass until the eighth pass finishing at 52%.

**Figure 3.13:** Plot of the GBCD of the middle section with respect to pass number

**Top Section.** Shown in Figure 3.14 is the GBCD of the top section of the samples versus the sample number. Again the CSL boundaries do not change much throughout the eight passes of the ECAE process. The two to five degree boundaries start at 35% for the AR sample and increase after the first and second passes to 77% then there is a sudden decrease in the two to five degree boundaries after the third pass to 52%. The two to five degree boundaries then slowly decrease for the rest of the passes where it finally ends up at 43% at the end of eight passes. The five to fifteen degree low angle boundaries do not change very much throughout the deformation process going from 13% for the AR sample up to 22% for the 8p sample. The HAGB start at 46% and decrease suddenly down to 8% after the third pass where they start to increase steadily through to the eighth pass to 31%.
Shown in Figure 3.15 is the GBCD for the bottom section of the billet plotted versus the sample number. Again the CSL value does not change much throughout the ECAE process for the bottom section. The two to five degree boundaries again start at 35% and increases to 85% after one pass then to 89% after the second pass. The two to five degree boundaries fall after the third and fourth passes down to 49% where it stays through the sixth pass. After the eighth pass the two to five degree boundaries drops to 33%. The five to fifteen degree boundaries drops from the 13% for the AR sample down to 7.5% after the second ECAE pass then it increases to 21% after the third pass where it stays relatively constant for each successive pass. The HAGB start at 46% then drops to 3% after the second pass. The HAGB number fraction increase after the third and fourth pass to around 24% where it stays through the sixth pass, after the eighth pass the HAGB show an increase to 43%.

Figure 3.14: Plot of the GBCD of the top section with respect to pass number
Grain boundary maps. Microstructure OIM maps, grain width analysis, and GBCD analysis were not enough to determine if the grains developed via ECAE were UFG or UFSG. Further investigation was needed. The middle section of the billet showed significant grain refinement, as previously seen in Figure 3.11b and 3.12. It was also seen that the GBCD changed substantially throughout the ECAE process from the first pass through to the eighth pass (Figure 3.13). From these figures it can be said that the grains are refining, yet there is no evidence if they are UFG or UFSG.

Grain boundary maps of the middle section were used to determine UFG or UFSG production. Figure 3.16 shows the grain boundary maps of the middle section with the 5-15º sub-grain boundaries represented in green and the HAGBs represented in red.
Figure 3.16: Grain boundary maps for ECAE Nb (a) AR, (b) 1P, (c) 2P, (d) 3P, (e) 4P, (f) 5P, (g) 6P, and (h) 8P with green representing the 5-15° boundaries and red representing the HAGB
As can be seen in Figure 3.16 the grain boundaries of the middle sections changed dramatically throughout the ECAE process. For the AR sample (Figure 3.16a) the grains were composed of mostly HAGB with a few 5-15º boundaries completing the grain boundary. After the first pass (Figure 3.16b) there was an introduction of low angle grain boundary (LAGB) sub-grains within the HAGB grain microstructure. This trend continued for the second pass (Figure 3.16c) where there were more LAGB sub-grains introduced with fewer HAGB grains developed. After the third pass (Figure 3.16d) the high angle and sub-grain boundaries both increase in density, with there being noticeably more sub-grains than high angle boundary grains. Upon completion of the fourth pass (Figure 3.16e) of ECAE there was a transition to more HAGB grains with fewer LAGB sub-grains. This trend continued through onto the eighth pass (Figure 3.16h) where there were mostly HAGB complete grains with few LAGB sub-grains in the microstructure.

**Effect of ECAE on texture**

Texture development in bcc materials such as niobium subjected to simple shear have been shown to produce two primary partial fibers, \(\{h k l\}<1 1 1>\) (or \(<1 1 1>\)-fiber for short) and \(\{1 1 0\}<u v w>\) (or \(\{1 1 0\}\)-fiber) as shown as the solid black lines connecting the components in Figure 3.17, the components are described in detail in Table 3.1.

![Figure 3.17](image)

**Figure 3.17:** (110) pole figure showing the locations of the main ideal orientations and fiber textures of bcc materials subjected to 45º ECAE deformation. [3]
**Macro-texture.** Shown in Figure 3.18 are the changes in the macrotexture of the center area throughout the ECAE process. As can be seen after the first pass (Fig. 3.18a) a strong <111> and {110} fiber texture was developed. The second pass (Fig. 3.18b) showed the same <111> and {110} fiber textures evolving in roughly the same orientation. After the third pass (Fig. 3.18c) the strength of the fiber texture changed substantially and the materials texture was shown to be more of the J and $\overline{J}$ shear components. The fourth pass (Fig. 3.18d) showed an increase in the <111> and {110} partial fiber texture again. And after the fifth pass (Fig. 3.18e) the partial fiber texture decreased showing mainly the $D_1$ and $D_2$ components. After the sixth pass (Fig. 3.18f) of ECAE the material showed an increased strength of the <111> and {110} partial fibers while there was also a slight rotation, of about 15º CCW, of the partial fiber texture developed. Upon completion of the eighth pass of ECAE (Fig. 3.18g) the <111> and {110}...
partial fibers were once again strong and rotated back to the standard orientation of the components shown in Figure 3.17.

![Figure 3.18](image)

**Figure 3.18:** Experimental (110) pole figures from XRD macrotexture for Nb processed by ECAE (a) 1p, (b) 2p, (c) 3p, (d) 4p, (e) 5p, (f) 6p, and (g) 8p

**Micro-texture.** Shown in Figure 3.19 is the (110) pole figure micro-texture development of the center section of Nb subjected to ECAE as taken from OIM analysis. As can be seen in Figure 3.19a texture development after one pass showed a weak and incomplete <111> and {110} partial fiber. After two passes (Fig. 3.19b) the texture strength increases and the texture moves from being distributed along the partial fibers condense into finite shear components such as both D and both J shear components there was also about a 15º CCW rotation of the orientations in the pole figure. The third pass (Fig 3.19c) showed a complete breakdown in the <111> and {110} fiber textures with a few of the shear components shown at the orientations as seen in Figure 3.17. After the fourth pass (Fig. 3.19d) a weak <111> and {110} partial fiber texture reappeared and was once again rotated about 12º CCW from the orientations shown in Figure 3.17. The fifth pass (Fig. 3.19e) of ECAE showed a weaker <111> and {110} partial fiber when compared to the fourth pass and the texture components were rotated back about the center of the pole figure to the normal ECAE components as shown in Figure 3.17 and also rotated about the horizontal axis up about 10º from the standard texture. The sixth pass (Fig 3.19f) the texture strength remained the same while showing almost the full <111> and {110} partial fibers in the same orientation as the second pass (Fig 3.19b). Upon completion of the eight pass (Fig 3.19g) the texture strength increases slightly and showing a rotated <111> and {110} partial fiber in roughly the same orientation as the fifth pass (Fig 3.19e).
The micro-texture of the top section of the Nb subjected to ECAE processing is shown in Figure 3.20. After the first pass (Fig. 3.20a) the <111> and {110} partial fiber texture was developed rotated by about 8° CW about the center of the pf. The second pass (Fig. 3.20b) showed similar <111> and {110} partial fiber texture at about the same orientation. After the third pass (Fig. 3.20c) the partial fiber texture was developed once again however it was rotated about 14° CW about the center of the pf. The fourth pass (Fig. 3.20d) showed the <111> and {110} partial fiber texture developed but rotated back to the location of the standard ECAE texture orientation. The fifth pass (Fig 3.20e) there was a rotation of the developed <111> and {110} partial fiber texture again to return to the same orientation as the third pass. The sixth pass of ECAE shown in Figure 3.20f has the developed <111> and {110} partial fiber texture rotated about 5° CW. After the eighth pass (Fig. 3.20g) there was a full but extremely weak <111> and {110} partial fiber apparent rotated about 5° CCW from the orientations shown in Figure 3.17.
The micro-texture of the bottom section of niobium subjected to ECAE is shown in Figure 3.21. As can be seen there was no development of the partial fibers in the bottom section during any pass of ECAE. The texture development was chaotic and random while the texture strength varies greatly throughout the process for the bottom section.

![Figure 3.21](image)

**Figure 3.21**: Experimental (110) pole figures from OIM microtexture of bottom of sample of Nb processed by ECAE (a) 1p, (b) 2p, (c) 3p, (d) 4p, (e) 5p, (f) 6p, and (g) 8p

The texture strength was quantified for all texture files using equation 11. From this formula the strength of the textures could be compared for each pass of ECAE. The results from the middle section of both the macro-texture and micro-texture are represented in Figure 3.22.

\[
T_n = \int [f_n(g)]^2 \, dg
\]  

(11)

As can be seen the strength of the texture developed during the ECAE process was similar for both the macro and micro-texture of the center section. The strength started at 38 for the XRD macro-texture and 29 for the OIM micro-texture. The macro-texture then decreases to 17 after the first pass ending at 9 after the third pass. After the fourth pass the XRD texture strength increased slightly to 13 where it started to decrease again to 8 for the sixth pass. The texture strength after the eighth pass increased again to 12. The OIM micro-texture coincided with the XRD macro-texture except for the second and third pass of ECAE. For these passes the micro-texture strength deviated greatly from the macro-texture strength due to the limited area of the scans for the micro-texture compared to the large area of the scans or the macro-texture.
The micro-texture strength was also taken for the top, middle and bottom sections as shown in Figure 3.23. As can be seen the first pass of ECAE decreased the texture strength from the as received sample. After the second pass there was an increase in the texture strength for all sections of the sample with the bottom increasing greatly from the first pass by a factor of about 4 while the top and middle sections showed only slight increases from 20 to 28. After the third pass the texture strength of the bottom section decreased to 49 while the middle section increased to 65 the top sections texture strength decreased to 17. After the fourth pass the strength of the micro-texture for all samples was between 10 and 16. For the remainder of the scans the top and middle sections remained between 10 and 16 while the bottom section fluctuated between 13 and 30.

Figure 3.22: Experimental texture strength for middle section from XRD and OIM
Modeling of the ECAE process

The ECAE process used in this study was modeled using a combination of ABAQUS and LApp in an attempt to predict the strain and texture development during the ECAE process. A three dimensional computer model made using ABAQUS FEA program, in the same dimensions as the actual die used in the experimental process, was performed and the strain results of one pass are shown in Figure 3.24. As can be seen from Figure 3.24a the billet undergoes a non-uniform plastic deformation similar to what was shown in Figure 1.7c where the strain encountered in the billet varies from the top to the bottom. At the top the billet experiences a pure plastic equivalent strain (PEEQ) of about 1.5 mm/mm while the middle section showed a PEEQ value of about 1.0 to 1.1 mm/mm and for the bottom section the billet experiences a PEEQ value of about 0.7 to 0.8 mm/mm. The strain from side to side however is relatively symmetrical and uniform when compares to the top to bottom strain.

Figure 3.23: Experimental texture strength for top, middle, and bottom sections from OIM
Taking the components of strain from sets of elements shown in the appendix in Figure A.41 at the top, middle, and bottom sections and applying formulae 12 through 16 it was possible to determine the equivalent von Mises strain from the simulation to compare the differences between the three regions of interest.

\[
\varepsilon_{eq} = \frac{2}{3} \sqrt{\frac{3}{2} (\varepsilon_{11}^2 + \varepsilon_{12}^2 + \varepsilon_{33}^2) + \frac{3}{4} (\gamma_{12}^2 + \gamma_{23}^2 + \gamma_{31}^2)}
\]

(12)

\[
\varepsilon_{11} = \frac{2}{3} \varepsilon_{11} - \frac{1}{3} \varepsilon_{22} - \frac{1}{3} \varepsilon_{33}
\]

(13)

\[
\varepsilon_{22} = -\frac{1}{3} \varepsilon_{11} + \frac{2}{3} \varepsilon_{22} - \frac{1}{3} \varepsilon_{33}
\]

(14)

\[
\varepsilon_{33} = -\frac{1}{3} \varepsilon_{11} - \frac{1}{3} \varepsilon_{22} + \frac{2}{3} \varepsilon_{33}
\]

(15)

\[
\gamma_{ij} = 2\varepsilon_{ij}
\]

(16)

Figure 3.25 showed the comparison of the equivalent von Mises strains of three regions of the billet plotted against the extrusion distance when deformed to one pass using the simulation. As expected the top section is the first to experience the strain imparted onto the billet and it also experiences the highest strain of about 1.47. The middle section is the next section to experience the deformation from the ECAE process and the magnitude of the strain is
about 1.33. Finally the element chosen for the bottom section is the last to experience the deformation and also has the lowest eq. von Mises strain value of about 1.13.

![Simulated Equivalent von Mises strain vs. Extrusion Distance](image)

**Figure 3.25**: Equivalent von Mises strain from simulated strain components

A comparison of the results of the modeled texture to the experimental texture for the middle sections is shown in Figure 3.26. The experimental texture is in Figure 3.26a while the simulated texture development is shown in Figure 3.26b. As can be seen the simulated texture development using ABAQUS and LApp agrees very closely with the experimental micro-texture. The simulated micro-texture failed to show any changes in the orientations of the components of the shear such as the rotations about the center of the pf however they do represent the $<111>$ and $\{110\}$ partial fibers that are developed. The strength of the simulated texture of the middle sample showed an apparent strengthening during the process.
Shown in Figure 3.27 is a comparison of the micro-texture (Fig. 3.27a) and simulated texture (Fig. 3.27b) of the top section of the billet subjected to ECAE. The simulated texture again showed an accurate prediction of the texture components of the top section. Also the simulated texture also showed an apparent decrease in the texture strength for each successive pass of ECAE past the first.

Figure 3.26: Comparison of experimental texture (a) versus simulated texture (b) for middle section

Figure 3.27: Comparison of experimental texture (a) versus simulated texture (b) for top section
In Figure 3.28 is a comparison of the experimental micro-texture (Fig. 3.28a) and the simulated texture (Fig. 3.29b) of the bottom section. Due to the symmetrical properties of the simulated texture process the bottom section simulation is very similar to the top section simulation. Due to this the simulated texture does not replicate the experimental texture development of the bottom section accurately. The bottom section simulated texture also showed decrease in the texture strength.

Using Equation 11 from above the texture strength of the simulated ECAE process was quantified and is shown in Figure 3.29. The texture strength of the middle section showed an initial decrease from the AR sample to the second pass of ECAE where it then showed a constant increase for each successive pass until the eighth pass. For the top section the texture strength after the first pass showed a slight increase then a steady decrease for the second, third, fourth, and fifth passes where it appeared to level off to a constant value for the rest of the simulation. The bottom section showed an increase in the texture strength for the first and second pass and then the texture strength showed a marked decrease for the third pass only to reach a steady value for the fourth through eighth pass of the simulation.
Discussion of Results

The macro- and microtexture produced by the route \( B_C \) ECAE process was shown to be of the common \{110\} and \(<111>\) partial fiber texture. This texture was developed after the first pass and continued through to the eighth pass for the top and middle sections as shown in Figure 3.18 for the macrotexture, Figure 3.19, and Figure 3.20 for the microtexture of the middle and top sections respectively. The microtexture developed in the bottom section showed little development of any of the components of the shear fiber texture as can be seen in Figure 3.21.

The microstructure of the billets three zones also showed that the material does not uniformly deform from the top to the bottom of the billet. There was a noticeable difference in the microstructure of the top, middle, and bottom sections that was evident after the first pass of ECAE and carried through to the final pass of ECAE. There was noticeably less shearing of the bottom sections compared to that of the middle and top sections. The combination of the texture results and the microstructure micrographs supports the slip line solution and deformation gradient shown in Figure 1.7 developed by Segal et al for round corner dies which he stated that

![Simulated Texture Strength](image)

**Figure 3.29:** Simulated texture strength for the top, middle, and bottom sections
the outer portion of material did not deform primarily by shear but was mostly a rigid rotation of the material.

The effect of the ECAE process on the Nb, specifically the grain size and microhardness, was best understood by an application of the Hall-Petch relationship. The Hall-Petch relationship is a general relationship between the yield stress and grain size of a material. The main concept that the Hall-Petch equation was based on was that grain boundaries acted as barriers to dislocation motion. The standard Hall-Petch equation [19] is:

\[ \sigma = \sigma_i + kD^{-1/2} \]

Where the \( \sigma_0 \) is the yield stress, \( \sigma_i \) is the “friction stress,” which represents the overall resistance of the crystal lattice to dislocation movement, \( k \) is the “locking parameter,” which measures the relative hardening contribution of the grain boundaries, and \( D \) is the grain diameter. In order to determine the value for \( k \) and \( \sigma_i \) the yield stress of the material, \( \sigma_0 \), was plotted versus \( D^{-1/2} \) from this plot the slope of a line through the points was the \( k \) value and y intercept of the trendline was the \( \sigma_i \) value [19].

The values for \( \sigma_0 \) are found by using the relationship,

\[ \sigma_i = \frac{H_V}{3} (0.1)^{n'-2} \]

Where \( H_V \) represents the Vickers hardness number and \( n' \) is the exponent in Meyer’s law. However since Meyer’s law states that for fully strain hardened materials, as is the case for ECAE materials, the exponent \( n' \) is equal to 2 reducing the above equation to \( \sigma_0 = H_V/3 \). In this study the grain thickness was measured instead of the grain diameter and this value was used in the Hall-Petch relation. The plot for the Hall-Petch relationship of the middle section can be seen in Figure 3.30. The value for the constant \( k \) was determined to be 0.148 and the value for \( \sigma_i \) was 43 from the plot. As can be seen the Hall-Petch relationship holds true for the 1P through the 6P samples within ±5%. However for the 8P sample the Hall-Petch relation no longer holds true because the actual hardness was more that 12% higher than what the Hall-Petch relation predicted.
For the top section the Hall-Petch relation was shown to hold true within ±6% again as can be seen in Figure 3.31. The first pass of ECAE fully strain hardens the material and the Hall-Petch relation fully predicted the hardness for each pass thereafter. The reason the 8P sample did not deviate from the Hall-Petch line as was seen in the Figure 3.30 was because the strain at the top region trails the strain at the middle section due to the route B_{c} processing.

**Figure 3.30**: Hall-Petch correlation of the middle section of Nb subjected to ECAE SPD.

**Figure 3.31**: Hall-Petch correlation of the top section of Nb subjected to ECAE SPD.
The Hall-Petch relation was not as accurate for the bottom section as shown in Figure 3.32. The 1P and 2P samples at the bottom section were below the line predicted by the Hall-Petch relation because the material was not fully strain hardened after the first two passes as can be seen in Figure 3.4b and Figure 3.5b microstructure micrographs. For the 3P through the 8P samples the Hall-Petch relation accurately predicted the hardness of the material in the bottom region to within ± 6%.

![Comparison of Exp. Data to HP Relationship for the Bottom Section](image)

**Figure 3.31:** Hall-Petch correlation of the top section of Nb subjected to ECAE SPD.

Upon examining the microstructure, the GBCD, and the grain boundary plots of the middle section it can be said that the material produced was an UFG material with equiaxed submicron grains with a rotated simple shear partial fiber texture. These characteristics were prevalent within the middle two thirds of the bulk material. This material may exhibit superplastic forming behavior at elevated temperatures due to the fact that the equiaxed grains were less than 10 μm in diameter and the grain boundaries consisted of primarily HAGB. This could be useful in producing cylindrical specimens used for hydroformed RF cavities by back extruding the bulk material at elevated temperatures.
CHAPTER 4

CONCLUSION

The purpose of this study was to characterize and analyze the ECAE process of commercially pure Nb. Many different techniques were used to qualitatively and quantitatively analyze this process such as X-ray diffraction, OIM/EBSD analysis, Vickers microhardness testing, and FEM computer modeling. The conclusions drawn from the data collected are as follows:

i. The ECAE process developed the standard \{110\} and \langle 111 \rangle partial fiber duplex texture components for the top and middle sections of the billet after each pass. The bottom section showed no development of the \{110\} and \langle 111 \rangle partial fiber duplex texture for any of the eight passes. These results prove that the slip line solution developed by Segal holds true for Nb deformed by ECAE with a round corner die in that the material in the outer section does not deform by shear as the rest of the sample does.

ii. The Hall-Petch relation held true for the top and bottom sections once the material became fully work hardened all the way through the eighth pass. For the top section the Nb was fully work hardened after the first pass and the hardness could be accurately predicted by the Hall-Petch relation to ±6%. The bottom section did not appear to fully work harden until after the third pass of ECAE, from this pass through to eight passes the hardness could be accurately predicted to ±6%. The work hardening developed in the middle section was in agreement with the Hall-Petch relationship for the first through sixth pass of ECAE to ±5%. The hardness for the eighth pass sample was not accurately predicted by the Hall-Petch relationship indicating that there was another hardening mechanism affecting the material besides grain refinement over a true strain of about six.

iii. Very few subgrains remained after the eighth pass of ECAE via route B\textsubscript{C}. Eight passes of ECAE on Nb led to UFG production within the middle section. The UFG structure with HAGB misorientation might possibly lead to superplasticity upon deformation at elevated temperatures. This could possibly make this type of deformation process suitable producing cylinders via back forging for with properties suitable for use in RF cavity hydroforming.

iv. The combination of an ABAQUS FEA model of the ECAE process with the LApp texture modeling software was shown to accurately predict the texture developed in the middle section of BCC materials. The top and bottom sections were not as accurately predicted by this model.
RECOMMENDATIONS FOR FUTURE WORK

For a full understanding of if this processing technique will be able to produce Nb with superplastic forming properties future studies need to be performed. The deformed material should undergo heat treatment and be tested for superplastic forming capabilities. The deformation should also be performed with a die with a sharper outer bend corner to maximize the amount of material refined by the shear mechanism and minimize the rigid zone of material apparent with this die design.

The simulation should be tuned to generate the proper texture rotations developed during the route $B_C$ processing, and also to possibly predict the textures produced at the top and bottom of the material during processing.

The secondary hardening mechanism that causes the deviation from the Hall-Petch relation should be identified and a new Hall-Petch relationship with this second mechanism should be developed.

The process should also be scaled up in size to process large enough samples to be able to produce large seamless cylinders used for RF cavity production.
APPENDIX

Figure A.1: Extrusion force versus extrusion distance for 1P Nb ECAE.

Figure A.2: AR Nb Vickers microhardness top to bottom traverse.
**Figure A.3**: 1P Nb Vickers microhardness top to bottom traverse.

**Figure A.4**: 2P Nb Vickers microhardness top to bottom traverse.
Figure A.5: 3P Nb Vickers microhardness top to bottom traverse.

Figure A.6: 4P Nb Vickers microhardness top to bottom traverse.
**Figure A.7**: 5P Nb Vickers microhardness top to bottom traverse.

**Figure A.8**: 6P Nb Vickers microhardness top to bottom traverse.
Figure A.9: 8P Nb Vickers microhardness top to bottom traverse.

Figure A.10: X-ray diffraction 2 theta peak location scan for AR in dark blue, 1P in yellow, 2P in purple, 4P in green, and 8P in light blue.
Figure A.11: AR Nb macrotexture complete pole figure from popLA.

Figure A.12: 1P Nb macrotexture complete pole figure from popLA.
Figure A.13: 2P Nb macrotexture complete pole figure from popLA.

Figure A.14: 3P Nb macrotexture complete pole figure from popLA.
Figure A.15: 4P Nb macrotexture complete pole figure from popLA.

Figure A.16: 5P Nb macrotexture complete pole figure from popLA.
Figure A.17: 6P Nb macrotexture complete pole figure from popLA.

Figure A.18: 8P Nb macrotexture complete pole figure from popLA.
Figure A.19: AR Nb microtexture complete pole figure from TSL/EBSD.

Figure A.20: 1P Nb microtexture complete pole figure of top section from TSL/EBSD.

Figure A.21: 1P Nb microtexture complete pole figure of middle section from TSL/EBSD.
Figure A.22: 1P Nb microtexture complete pole figure of bottom section from TSL/EBSD.

Figure A.23: 2P Nb microtexture complete pole figure of top section from TSL/EBSD.

Figure A.24: 2P Nb microtexture complete pole figure of middle section from TSL/EBSD.
**Figure A.25**: 2P Nb microtexture complete pole figure of bottom section from TSL/EBSD.

**Figure A.26**: 3P Nb microtexture complete pole figure of top section from TSL/EBSD.

**Figure A.27**: 3P Nb microtexture complete pole figure of middle section from TSL/EBSD.
Figure A.28: 3P Nb microtexture complete pole figure of bottom section from TSL/EBSD.

Figure A.29: 4P Nb microtexture complete pole figure of top section from TSL/EBSD.

Figure A.30: 4P Nb microtexture complete pole figure of middle section from TSL/EBSD.
Figure A.31: 4P Nb microtexture complete pole figure of bottom section from TSL/EBSD.

Figure A.32: 5P Nb microtexture complete pole figure of top section from TSL/EBSD.

Figure A.33: 5P Nb microtexture complete pole figure of middle section from TSL/EBSD.
**Figure A.34**: 5P Nb microtexture complete pole figure of bottom section from TSL/EBSD.

**Figure A.35**: 6P Nb microtexture complete pole figure of top section from TSL/EBSD.

**Figure A.36**: 6P Nb microtexture complete pole figure of middle section from TSL/EBSD.
Figure A.37: 6P Nb microtexture complete pole figure of bottom section from TSL/EBSD.

Figure A.38: 8P Nb microtexture complete pole figure of top section from TSL/EBSD.

Figure A.39: 8P Nb microtexture complete pole figure of middle section from TSL/EBSD.
Figure A.40: 8P Nb microtexture complete pole figure of bottom section from TSL/EBSD.

Figure A.41: Element locations from ABAQUS for use in the equivalent von Mises strain calculations.
Figure A.42: 1P BCC simulated microtexture complete pole figure of top section from LApp and popLA.

Figure A.43: 1P BCC simulated microtexture complete pole figure of middle section from LApp and popLA.
Figure A.44: 1P BCC simulated microtexture complete pole figure of bottom section from LApp and popLA.

Figure A.45: 2P BCC simulated microtexture complete pole figure of top section from LApp and popLA.
Figure A.46: 2P BCC simulated microtexture complete pole figure of middle section from LAApp and popLA.

Figure A.47: 2P BCC simulated microtexture complete pole figure of bottom section from LAApp and popLA.
Figure A.48: 3P BCC simulated microtexture complete pole figure of top section from LApp and popLA.

Figure A.49: 3P BCC simulated microtexture complete pole figure of middle section from LApp and popLA.
Figure A.50: 3P BCC simulated microtexture complete pole figure of bottom section from LApp and popLA.

Figure A.51: 4P BCC simulated microtexture complete pole figure of top section from LApp and popLA.
Figure A.52: 4P BCC simulated microtexture complete pole figure of middle section from LApp and popLA.

Figure A.53: 4P BCC simulated microtexture complete pole figure of bottom section from LApp and popLA.
**Figure A.54**: 5P BCC simulated microtexture complete pole figure of top section from LApp and popLA.

**Figure A.55**: 5P BCC simulated microtexture complete pole figure of middle section from LApp and popLA.
Figure A.56: 5P BCC simulated microtexture complete pole figure of bottom section from LApp and popLA.

Figure A.57: 6P BCC simulated microtexture complete pole figure of top section from LApp and popLA.
Figure A.58: 6P BCC simulated microtexture complete pole figure of middle section from LAApp and popLA.

Figure A.59: 6P BCC simulated microtexture complete pole figure of bottom section from LAApp and popLA.
Figure A.60: 7P BCC simulated microtexture complete pole figure of top section from LAApp and popLA.

Figure A.61: 7P BCC simulated microtexture complete pole figure of middle section from LAApp and popLA.
Figure A.62: 7P BCC simulated microtexture complete pole figure of bottom section from LAApp and popLA.

Figure A.63: 8P BCC simulated microtexture complete pole figure of top section from LAApp and popLA.
Figure A.64: 8P BCC simulated microtexture complete pole figure of middle section from LAApp and popLA.

Figure A.65: 8P BCC simulated microtexture complete pole figure of bottom section from LAApp and popLA.


BIOGRAPHICAL SKETCH

Trever H. Carnes was born in Palatka, Florida on December 5th, 1979. He graduated from Palatka High School in 1998. He acquired his AA degree from St. Johns River Community College in the spring of 2003. He moved to Tallahassee, Florida in the fall of 2003 to pursue his Bachelor’s degree in Mechanical Engineering which he received in the spring of 2006. He received his Master’s of Science degree in Mechanical Engineering in the spring on 2009 under the advisement of Dr. Peter Kalu.