Effect of tube processing methods on the texture and grain boundary characteristics of 14YWT nanostructured ferritic alloys

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Abstract:

Texture and microstructure of tubes and plates fabricated from a nanostructured ferritic alloy (14YWT), produced either by spray forming followed by hydrostatic extrusion (Process I) or hot extrusion and cross-rolling a plate followed by hydrostatic tube extrusion (Process II) have been characterized in terms of their effects on texture and grain boundary character. Hydrostatic extrusion results in a combination of plane strain and shear deformations which generate low intensity α - and γ -fiber components of $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ together with a weak ζ -fiber component of $\{011\}\langle 211 \rangle$ and $\{011\}\langle 011 \rangle$. In contrast, multi-step plane strain deformation by hot extrusion and cross-rolling of the plate leads to a strong texture component of $\{001\}\langle 110 \rangle$ together with a weaker $\{111\}\langle 112 \rangle$ component. Although the total strains are similar, shear dominated deformation leads to much lower texture indexes compared to plane strain deformations. Further, the texture intensity decreases after hydrostatic extrusion of the alloy plate formed by plane strain deformation, due to a lower number of activated slip systems during shear dominated deformation. Notably, hot extruded and cross-rolled plane strain plate deformation to ~50% engineering strain creates only a modest population of low angle grain boundaries (LABs), compared to the much larger population observed following the combination of plane strain and shear deformation of ~44% engineering strain resulting from subsequent hydrostatic extrusion.

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1. Introduction:

Nanostructured ferritic alloys (NFAs) are leading candidates for structural components in nuclear reactors due to their exceptional irradiation tolerance, high strength and resistance to oxidation/corrosion under extreme conditions of temperature and stress [1-6]. There has been substantial progress on optimizing the production of larger heats of NFAs consolidated by hot extrusion (HE), and in some cases, hot isostatic pressing (HIP) [7]. Recently, an NFA with an approximately uniformly distributed population, $\sim 7 \times 10^{23}/\text{m}^3$, of ~ 2.5 nm Y-Ti-O rich nano-oxides (NOs) has been developed by Los Alamos National Laboratory (LANL), Oak Ridge National Laboratory (ORNL) and University of California Santa Barbara (UCSB) in collaboration [8]. The presence of coarsening resistant NOs, along with predominantly fine grain size ($< 1 \mu\text{m}$) and high dislocation densities imbue NFAs with higher strength and remarkable thermal stability compared to conventional ODS alloys. Moreover, the NOs help to recombine displacement damage and trap neutron irradiation transmutation product, He, in extremely small bubbles, thereby suppressing void swelling and grain boundary embrittlement [2,4,9,10].

Although significant progress has been made, some major issues associated with HE NFAs include: severe texturing (the distribution of grain orientations) as well as anisotropic microstructures and properties; and, bimodal grain size distributions [11-19]. The primary detrimental consequences of these characteristics include low fracture toughness and low creep strength in directions parallel to, and transverse to the HE axis, respectively [3,7,12,19-22]. Additional deformation processing steps to improve properties and/or fabricate shapes are also severely impacted by these characteristics of the HE condition. For example, HE cross-rolled plates develop microcracks [23] and deformation processed tubes experience radial through-wall cracks [24]. Extensive research in Japan and France have identified semi-optimized processing paths for tubing that differ in detail, but in both cases involve a series of cold-pilgering steps to reduce the wall thickness, with intermediate and final heat treatment softening schedules [5,25,26]. This general processing path has been most successful with lower 9 Cr transformable ($\gamma \rightarrow \alpha/\text{martensite}$) steels [7], but 14YWT ferritic stainless NO strengthened alloys remain difficult to process into thin-walled tubing.

Recent studies have suggested that material with low ductility can effectively be processed to a near net shape using hydrostatic extrusion. It can also produce the final product with enhanced

and more isotropic mechanical properties, as well as high powder consolidation densities [27-29]. For example, a recent review [30], and related research on hydrostatically extruded NiAl [31], revealed improved properties and beneficial changes in texture [32] due to the shear dominated stress state generated in hydrostatic extrusion. One hydrostatic extrusion path uses a high-pressure fluid in a die chamber to impose compressive stresses that lower ram pressures and temperatures, resulting in better formability and surface finishes [30-33].

In this study, the effects of different processing routes on texture and grain boundary characteristics of as processed 14YWT NFA tubes have been investigated using neutron and electron diffraction techniques.

2. Experimental Procedures:

The composition of the NFA tested in this study is 12.8wt%Cr-2.95W-0.38Ti-0.22Y-Fe for both tubes. In order to understand the effects of different processing methods on the resulting texture and grain boundary characteristics, 14YWT tubes were produced by two different initial routes followed by a novel hydrostatic extrusion process to produce tubes that were processed to full density. Process I utilizes the initial processing method of thermal spraying while Process II uses a conventional mechanical alloying method followed by hot extrusion. Figure 1 shows that Process I is the combination of gas atomization reaction synthesis of Fe-based NFA precursor powders, deposited by high velocity oxy-fuel (HVOF) thermal spray methods on tubular pre-forms, followed by hydrostatic extrusion to process the tube to final dimensions. A “mother tube” was manufactured through HVOF deposition on an Al mandrel with Fe-based NFA precursor powders (20-53 μ m dia.). The fugitive Al mandrel was removed from the deposit section with a phosphoric acid etching treatment and initial machining was performed to remove the interior ($\sim$ 250 μ m) layer of intermetallic phase (FeAl or Fe₃Al) that appeared to form during the deposition process. The samples were then heat treated at 1000°C for 5hr under vacuum following thermal spray deposition to promote the oxygen exchange reaction and Y-Ti-O dispersoid formation within the coating. Finally, hydrostatic extrusion was applied two times with a 4:1 area reduction ($\sim$ 44% engineering strain) at 815 °C by using a solid support mandrel plus external can to create a beneficial stress state for extrusion.

The schematic for Process II is shown in Figure 2. Powders were first produced by gas atomization method by ATI Powder Metals Laboratory (Pittsburgh, PA). After atomization, the Y phase separates and requires ball milling to create a more homogenous chemistry. So, the nominal 14wt%Cr-3W-0.35Ti-0.25Y iron alloy powder was ball milled by Zoz, GmbH (Wenden, Germany) with the addition of FeO powder for 40 h in a CM100b attritor mill. The powders were sealed in cans backfilled with Ar to remove any atmosphere contamination and degassed at 400 °C. The canned powders were then hot extruded at 850 °C to create a solid billet. After extrusion, the alloy was annealed for 1 h at 1000 °C and then hot cross-rolled at 1000 °C to a ~50% of thickness reduction resulting (50 % engineering strain) in a final thickness of ~10 mm. The solid billet was then electro-discharge machined (EDM) in the direction perpendicular to hot cross-rolling direction to produce a tube. This tube was then hydrostatically extruded at 815 °C using the same process described for the thermally sprayed tube. Figure 3 shows the tubes with a final wall thickness of 0.5 mm and outer diameters of ~8 mm and ~3.5 mm for Process I and Process II, respectively.

Grain boundary characteristics were investigated by orientation imaging microscopy (OIM) while texture analysis was implemented using both OIM and neutron diffraction methods. OIM by electron backscatter diffraction (EBSD) using a scanning electron microscope (SEM) was used to examine the grain boundary characteristics and texture of the plate and tubes. It should be noted that EBSD is limited to the grain size studies down to 60 nm [34,35]. Here, EBSD was carried out in an FEI Inspect FEG SEM equipped with TSL EBSD detector. All the experiments were performed by tilting the sample at 70° from the horizontal, at an acceleration voltage of 20 kV and aperture size of 50 µm. The sizes of the scanned regions was either 60µmx90µm or 240µmx320µm, where the step size for the data collection was chosen based on the grain and substructure dimensions. Samples for the EBSD scans were obtained from the tube and plate faces that were prepared to achieve very smooth surfaces by standard metallographic techniques followed by jet electropolishing using a solution of perchloric acid (5%) and methanol at -40°C with an applied voltage of 20 V.

Bulk texture measurements probing the grain orientation distribution function over the complete volume of each sample were collected on the HIPPO instrument at the pulsed neutron spallation source at LANSCE [36]. The samples were located ~9m from the neutron moderator and

117 diffracted neutrons were detected by 1240 ^3He tubes arranged on panels distributed over five
118 conical rings with scattering angles ranging from $2\theta = 40^\circ$ to 150° . Samples were glued on
119 standard HIPPO holders, with their tube axis parallel to the holder axis, and then loaded into the
120 robotic sample-changer. The entire ~ 10 mm long tubes were centered in a 10 mm diameter beam,
121 thus capturing the entire sample volume. Diffraction patterns were collected over ~ 20 minutes
122 for samples rotated around the vertical axis at 0, 67.5 and 90 degrees. The diffraction data was
123 analyzed by simultaneous Rietveld analysis of 135 diffraction patterns using the E-WIMV
124 texture algorithm with a 10 degree resolution of the orientation distribution function.

125 Orientation distribution function (ODF) analysis and pole figure plotting for both electron and
126 neutron texture measurements was performed using the MTEX package [37]. The EBSD data
127 was analyzed using the TSL OIM Analysis 7 software.

128 **3. Results:**

129 ***3.1. Tube Processing by Process I***

130 The only deformation that occurs in consolidation by Process I is by hydrostatic extrusion and
131 the corresponding microstructures isolate the unique effects of this processing path. Figure 4.a
132 shows the orientation distribution of the grains obtained by the OIM analysis of the 14YWT
133 sample produced by Process I. Even though the physical extrusion direction is shown in a
134 diagonal direction in the map, the coordinates of the map have been rotated in such a way that
135 the extrusion direction is into the page in order to determine the grain orientations in the
136 extrusion direction. Consequently, predominantly existing green color infers that most of the
137 grains are oriented with a (101) plane normal that is parallel to the extrusion direction. The band
138 contrast map in Figure 4.b together with Figure 4.c show that Process I results in wide grain area
139 fraction distribution as a function of size. However, there are many more small grains that yield a
140 corresponding average diameter of $\sim 2\mu\text{m}$. Figure 4.d shows that hydrostatic extrusion results in
141 a large fraction ($\sim 53\%$) of low angle boundaries (LABs) with misorientation angles between
142 $2^\circ < \theta < 15^\circ$.

143 Figure 5 shows the pole figures of the sample produced by Process I as determined by both
144 neutron diffraction (Figure 5.a) and EBSD (Figure 5.b). Even though data processing functions
145 and probed sample volumes are greatly different for neutron diffraction and EBSD, both produce

similar pole figures with only a slight difference in the texture intensity. From the pole figures it is obvious that the hydrostatic extrusion to 44% strain produces a typical α -fiber texture $\langle 110 \rangle \parallel \text{ED}$, generally common to deformation processed ferritic alloys [38,39].

The ideal orientations of texture components in bcc materials are shown schematically for $\varphi_2 = 0^\circ$ and 45° ODF sections in Figure 6 and Table 1 [40]. As noted previously, for bcc steels the important fiber textures are the α -fiber ($\langle 110 \rangle \parallel \text{RD}$), ε -fiber ($\langle 011 \rangle \parallel \text{TD}$), γ -fiber ($\langle 111 \rangle \parallel \text{ND}$), η -fiber ($\langle 100 \rangle \parallel \text{RD}$), θ -fiber ($\langle 001 \rangle \parallel \text{ND}$), and ζ -fiber ($\langle 011 \rangle \parallel \text{ND}$) [41,42] where RD, TD and ND stand for rolling direction, transverse direction and normal direction, respectively. It should be noted that 'RD' will be replaced by 'ED' throughout the paper.

Although EBSD might not give fully representative results because of its limited probed volume, Figure 5 confirms that the probed area on the tube surface using OIM analysis represents the bulk texture obtained by neutron diffraction well. However, there might be a texture gradient through the thickness of the tube wall. In order to further examine and represent the bulk texture, detailed analyses of ODF plots and inverse pole figures were obtained using the neutron diffraction method. It is worthwhile to point out that while neutron diffraction probes a much larger volume than OIM, all spatial information is lost. Hence, while the detailed texture analyses were employed using neutron diffraction, only OIM can provide grain misorientation angles and orientation maps.

Figure 7 shows the neutron diffraction resulted ODF maps for the 14YWT sample produced by Process I at constant $\varphi_2=0^\circ$ and $\varphi_2=45^\circ$. Comparing these maps with Figure 6 shows that the dominant texture is α -fiber at $\langle 110 \rangle$ having the strongest intensity on $\{001\}\langle 110 \rangle$. Furthermore, weaker γ -fiber component of $\{111\}\langle 110 \rangle$ and ζ -fiber on $\{011\}\langle 211 \rangle$ and $\{011\}\langle 011 \rangle$ can be observed. The overall texture index was calculated as 2.6 using ODF neutron diffraction data indicating a moderate texture strength relative to a texture index of 1 for a perfectly random texture and infinity for a single crystal.

3.2. Tube processing by Process II:

Figure 8 shows EBSD measurements on the 14YWT intermediate tube, EDM-cut from the hot extruded and cross-rolled plate, with information analogous to that shown in Figure 4 for the tube, only hydrostatically extruded in Process I. Note the tube is microstructurally identical to the

cross-rolled plate, differing only in its geometric reference orientation. Since this alloy condition was characterized before the final hydrostatic extrusion step, as illustrated in Figure 2, it is designated as being “intermediate”. Figure 8.a is an orientation distribution map. The arrows indicate the initial hot extrusion (ED) and the transverse cross-rolling directions (RD). The predominantly red color infers that most of the grains are oriented with (100) plane normal parallel to the tube wall thickness. Figure 8.b, 8.c and 8.d show that the intermediate tube has a tri-modal grain size distribution and fewer LABs (~32%) compared to the tube produced by Process I (53%).

Since the intermediate tube was cut from the cross-rolled plate, coordinates of the plate were transformed into the tube coordinates as shown in Figure 9. There is a common extrusion direction (ED) for the plate and tube. Radial wall thickness directions of the tube are designated ThD and directions corresponding to the normal and rolling directions are designated as ThND and ThRD, respectively. However, the tube surface ED is only parallel to that for the plate surface at 0° and 180° around the circumference, while it is parallel to the plate sides at 90° and 270°.

Figure 10 shows the pole figures of the intermediate tube determined by both neutron diffraction (Figure 10.a) and EBSD (Figure 10.b) at 0° (or 180°). The pole figures calculated by both methods are again similar, with slight intensity differences is observed. Based on the coordinate system defined above, pole figures indicate that the intermediate tube has texture of both $\langle 110 \rangle \parallel \text{ThRD}$ and $\langle 200 \rangle \parallel \text{ThND}$. Moreover, as it must, the intermediate tube RD shares the $\langle 110 \rangle$ α -fiber texture with the cross-rolled plate. As discussed elsewhere [24], (100)-type planes lie parallel to the plate faces as well as to the tube surface at 0° and 180°, while they are normal to the tube surface at 90° and 270° as illustrated in Figure 9. The $\{001\} \langle 110 \rangle$ system is oriented for brittle cleavage fracture leading to microcracking in the plate [24] and radial tubing cracks, especially at 90° and 270°. The intermediate tube has a strong neutron diffraction texture index of 10.1.

Figure 11 shows the sections of the neutron diffraction resulted ODF maps in Euler space for the intermediate 14YWT tube at constant $\phi_2=0^\circ$ and $\phi_2=45^\circ$. When compared with Figure 6, it can be seen that intermediate tube has strong θ - and ε -fiber on $\{001\} \langle 110 \rangle$ together with a weak γ -fiber on $\{111\} \langle 112 \rangle$. It should be noted that the main deformation axis is considered as the axis for

all tubes, which is the extrusion direction. However, as shown in Figure 10, intermediate tube has a stronger texture in rolling direction through the wall thickness (at 90° and 270°), which is transverse to the extrusion direction. Since the rolling direction was taken as the transverse direction during ODF calculations, the corresponding plots of the intermediate tube by reference frame definition exhibit θ - and ε -fiber texture ($\langle 110 \rangle \parallel \text{TD}$) rather than α fiber ($\langle 110 \rangle \parallel \text{RD}$) which is a typical texture for ferritic steels, as found in the plate. Again, the actual microstructures are identical in both cases.

Figure 12.a shows the orientation map of the grains in the extracted tube after final hydrostatic extrusion as described in Figure 2. The coordinates of the map have been rotated in such a way that the radial direction of the tube (normal direction-ND) points through the page. Consequently, the predominantly existing red color infers that most of the grains are oriented with (100) plane normal parallel to the sample normal direction. The band contrast map in Figure 12.b and grain size plot in Figure 12.c show that very small and very large grains exist, suggesting a bimodal grain size distribution with an average grain size of $\sim 0.5 \mu\text{m}$. Figure 12.d shows that fraction of LABs increases from 32% to 46% after hydrostatic extrusion.

Figure 13 shows the pole figures of the tube after hydrostatic extrusion derived by both neutron diffraction (Figure 13.a) and EBSD (Figure 13.b), yielding different results in this case. While the neutron diffraction indicates a broken fiber texture of $\langle 110 \rangle \parallel \text{ED}$, both the EBSD orientation map and pole figures show a maximum texture index in $\langle 200 \rangle \parallel \text{ND}$. However, the difference between the bulk texture obtained by neutron diffraction and surface texture determined by EBSD is the result of the position of the EBSD sample on the SEM stage. Since the data shows the maximum texture on $\langle 200 \rangle$ rather than $\langle 110 \rangle$, the EBSD reflects a region that is aligned 45° to the extrusion direction. Since the samples are tubes, their orientation reference frame determines the nominal texture that must be considered in reaching any conclusions or in making comparisons.

The neutron diffraction data shows that the initial processing steps of hot extrusion, annealing and hot cross-rolling, leads to discontinuity in the fiber texture as shown in Figure 13.a. The overall texture index was calculated as 8.6 using neutron diffraction. The texture index decreased from 10.1 to 8.6 after hydrostatic extrusion with 25% strain in the intermediate tube, since the principal deformation direction is in the opposite to that for the hot cross-rolling. These results in

combination with those observed for Process I, indicate that hydrostatic extrusion produces relatively weak textures.

Figure 14 shows the neutron diffraction resulted ODF maps of the 14YWT final tube produced by Process II at constant $\varphi_2=0^\circ$ and $\varphi_2=45^\circ$. If they are compared with Figure 6, it can be seen that α -fiber has the maximum intensity on $\{111\}<110>$ along with a weak discontinuous γ -fiber on $\{001\}<110>$. For $\varphi_2=0^\circ$ there is a very weak ζ -fiber texture on $\{011\}<211>$ and $\{011\}<011>$.

4. Discussion:

Large-scale deformation behavior of polycrystalline Fe is quite complex since it activates a number of different slip planes like $\{110\}$, $\{112\}$ and $\{123\}$, and unlike fcc materials, is not confined to only closed packed planes. Depending upon various factors like mode of deformation, processing history (strain path), composition, initial microstructure and orientation of the grains, deformation involves different combinations of these slip systems. These multiple factors also affect the dislocation substructure and local misorientations between sub-grains. The result is the production of a wide range of possible textures in the final microstructure [43]. In the present study two different large deformation processing methods were used to fabricate tubes. Both Process I and II create microstructures with a strong $<110>\parallel$ RD texture with various grain sizes.

Rolling and extrusion are usually regarded as plane strain deformations. However, a gradient in the stress state exists in the through thickness of a rolled or extruded plate. Deformation in the near surface region, in contact with the die, is dominated by a shear component. The center of the plate experiences compressive stresses and deformation occurs under plane strain conditions [44]. In contrast, during hydrostatic extrusion of a thin-walled tube the outer layer is dominated by a state of compressive stress, while the central layer is deformed by a dominant shear stress component [45].

It is observed that both hydrostatic extrusion and conventional hot extrusion processes produce a strong texture on $\{001\}<110>$; however, there are remarkable differences in the texture intensities. The tube produced through hydrostatic extrusion in Process I shows maximum texture index of 2.6, whereas the tube produced at the intermediate step in Process II which experienced a similar amount of deformation shows a much stronger texture index of 10.1. Pal et

al. [24] has investigated the inverse pole figures of the process II produced intermediate plate before EDM cutting tubes from it as well as after hydrostatically extruding the intermediate tube. The texturing found here is generally consistent with their study; while the textures in the plate and the hydrostatically extruded tube are similar in the extrusion direction, the maximum texture intensity changes from $\langle 100 \rangle$ to $\langle 111 \rangle$ in the tube thickness normal direction as a result of the hydrostatic extrusion. This rationalizes the observation that while the intermediate plate exhibits an extensive amount of microcracking, the hydrostatically extruded final tube with a texture on $\langle 111 \rangle$ ND resists such cracking in Process II [24]. Similarly, comparing the inverse pole figures of the hydrostatically extruded final and intermediate tubes shown in Figure 15 reveals that while hydrostatic extrusion develops a texture of a mixture of $\langle 111 \rangle$ and $\langle 100 \rangle$ along the ND, $\langle 100 \rangle$ is more profound along the ND of the intermediate tube. Hot working of the bcc steels results in cold rolling textures if the material does not undergo any kind of ferrite to austenite transformation or complete recrystallization [44,46]. Since 14YWT steels are fully ferritic and do not recrystallize during the present hot-working conditions, the observed texture differences can be explained in terms of cold rolling textures. Raphanel et al. [47] predicated the cold rolling texture of bcc steel based on a relaxed constant modification of Taylor theory. Simulations by reducing ε_{13} and ε_{23} shear strain components increases the intensity of $\{001\}\langle 110 \rangle$ texture and produces an elongated pan-cake shaped grains, as was observed in the cross-rolled plate and intermediate tube. Consequently, formation of high volume fraction of $\{001\}\langle 110 \rangle$ texture components in the intermediate tube orients the $\{001\}$ plane normal along the tube thickness normal direction (shown in Figure 15.b).

Our results show that after hydrostatic extrusion of the intermediate tube in Process II, the texture index is weakened from 10.1 to 8.6. Raabe et al. [48] also reported that the shear stress weakens the texture relative to the pre-deformed condition. This is simply attributed to a lower number of activated slip systems during pure shear deformation. The major texture component originating under simple shear deformation of a bcc steel is the ‘Goss’ texture component $\{110\}\langle 001 \rangle$. However, the ‘Goss’ texture component is not very stable, and can easily transform to the stable components of $\{001\}\langle 110 \rangle$ of α -fiber or $\{111\}\langle 110 \rangle$ of γ -fiber depending upon the relative strength of each fiber [48]. This is responsible for the origin of weak shear texture components of $\{111\}\langle 110 \rangle$ and $\{001\}\langle 110 \rangle$ producing a partial $\langle 111 \rangle$ ND texture.

The compressive part of the stress in hydrostatically extruded tubes introduces the common α -fiber. The nature of the deformation process is further corroborated using the number fraction vs. misorientation angle plots in Figure 16. The hydrostatically extruded tubes contain large fraction of LABs indicating the association of shear deformation and high amount of local reorientation [43], which in turn produces a continuous spread of orientation along α -fibers rather than developing a sharp texture. The effect of hydrostatic extrusion is more drastic when the starting material possesses a pre-existing texture (due to hot cross-rolling and extrusion), completely changing the texture. In this case, the maximum texture intensity is observed on the $\{111\}\langle 110 \rangle$ component of γ fiber due to the restriction of activated slip system and activation of high Taylor factor shear planes. Development of strong γ fiber has been reported to be good for the deep drawing ability of high chrome-ferritic steels [48]. It also supports the finding that the hydrostatically extruded final tube in Process II can be produced without cracking as reported by Pal et al. [24].

5. Conclusions:

In this study, the effect of different processing routes on texture and grain boundary characteristics for 14YWT NFA tubes have been investigated. Hydrostatic extrusion which introduces a combination of plane strain and shear deformations results in α - and γ -fibers on $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ as well as weak textures of ζ -fiber on $\{011\}\langle 211 \rangle$ and $\{011\}\langle 011 \rangle$. On the other hand, hot extrusion and cross-rolling processes result in plane strain deformations in the case of intermediate plate and tube in Process II, leading to a strong texture on $\{001\}\langle 110 \rangle$ together with weak texture on $\{111\}\langle 112 \rangle$. While both conventional hot extrusion and innovative hydrostatic extrusion produce a pronounced texture on $\{001\}\langle 110 \rangle$, shear deformation introduced during hydrostatic deformation produces $\langle 111 \rangle$ ND texture component. Furthermore, hydrostatic extrusion results in weaker textures compared to the hot extrusion and rolling textures. Even though the final tube produced by Process I and the intermediate tube in Process II are exposed to similar amounts of deformation, the former has a texture index of 2.6 while the latter has a texture index of 10.1. Similarly, when the intermediate tube is hydrostatically extruded, its texture index decreases to 8.6. Together with a decrease in the texture index, α -fiber on $\{001\}\langle 110 \rangle$ weakens and γ -fiber on higher Taylor factor planes, $\{111\}\langle 110 \rangle$, strengthens as a result of a reduced number of activated slip systems during shear

deformation. Moreover, the effect of hydrostatic extrusion on the grain boundary characteristics is quite strong. Local reorientation during shear deformation results in high fractions of LABs compared to the intermediate tube produced by plane strain deformation.

Acknowledgements:

This research was partially supported by DOE-NE Fuel Cycle Research and Development Program under the contract number DE-AC52-06NA25396.

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Figure 1: Processing steps of Process I

Figure 2: Processing steps of Process II (RD: rolling direction, TD: transverse direction, ED: extrusion direction)

Figure 3: Photos showing the final dimensions of the tubes produced by: a) Process I, and b) Process II.

Figure 4: An OIM showing a) the crystallographic orientation of the grains; b) the corresponding band contrast gray scale map of the grain structure; c) the grain area distribution as a function of the grain size; and, d) the grain boundary misorientation angle distribution in 14YWT samples produced by Process I.

Figure 5: Pole figures of 14YWT samples produced by Process I and obtained by a) Neutron diffraction and b) EBSD techniques. (ED: extrusion direction)

Figure 6: Schematic illustration of the important texture components in bcc materials (Reproduced from Ref [40]).

Figure 7: The neutron diffraction produced ODF, represented as sections in Euler space, for the 14YWT tube produced by process I at ϕ_2 values of 0° and 45° .

Figure 8: EBSD characterization of the intermediate tube alloy condition showing a) an orientation map of the crystallographic distribution of the grains; b) a band contrast map of the grain structure in gray scale; c) the grain area distribution as a function of size; and, d) the grain boundary misorientation angle distribution.

Figure 9: Coordinate system of the intermediate tube transformed from the plate form.

Figure 10: Pole figures of intermediate 14YWT tube obtained by: a) Neutron diffraction, and b) EBSD techniques.

Figure 11: ODF, represented as sections through Euler space, of the intermediate 14YWT tube at constant φ_2 values of 0° and 45° .

Figure 12: a) Orientation map showing the crystallographic distribution of the grains; b) band contrast map showing the microstructure in gray scale; c) grain size distribution plot; and, d) grain boundary misorientation angle distribution in 14YWT final tube produced by Process II.

Figure 13: Pole figures of 14YWT final tube after hydrostatic extrusion obtained by: a) Neutron diffraction, and b) EBSD techniques.

Figure 14: ODF, represented as sections through Euler space, of the 14YWT final tube after hydrostatic extrusion at constant φ_2 values of 0° and 45° .

Figure 15: Neutron diffraction produced inverse pole figures of a) hydrostatically extruded final tube in Process I, b) intermediate tube in Process II and c) hydrostatically extruded final tube in Process II.

Figure 16: Grain boundary misorientation angle distribution of three different tubes produced by Process I and II.

Table 1: Important fiber textures and orientations for bcc materials [40].

Fiber name	Fiber axis	Components
α	$\langle 110 \rangle \parallel \text{RD}$	$\{001\} \langle 110 \rangle, \{112\} \langle 110 \rangle, \{111\} \langle 110 \rangle$
ε	$\langle 011 \rangle \parallel \text{TD}$	$\{001\} \langle 110 \rangle, \{112\} \langle 111 \rangle, \{111\} \langle 112 \rangle, \{011\} \langle 100 \rangle$
γ	$\langle 111 \rangle \parallel \text{ND}$	$\{111\} \langle 110 \rangle, \{111\} \langle 112 \rangle$
η	$\langle 100 \rangle \parallel \text{RD}$	$\{001\} \langle 100 \rangle, \{011\} \langle 100 \rangle$
θ	$\langle 001 \rangle \parallel \text{ND}$	$\{001\} \langle 100 \rangle, \{001\} \langle 110 \rangle$
ζ	$\langle 011 \rangle \parallel \text{ND}$	$\{011\} \langle 100 \rangle, \{011\} \langle 211 \rangle, \{011\} \langle 111 \rangle, \{011\} \langle 011 \rangle$