Bulk Texture Evolution of Nanolamellar Zr-Nb Composites Processed Via Accumulative Roll Bonding

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Abstract

It was recently demonstrated that bulk two-phase 50/50 Zr-Nb nanolayered composites with 90 nm individual layers can be fabricated from an initial coarse-layered composite with 1 mm layers via the severe plastic deformation process of accumulative roll bonding. During the deformation, the Zr phase retained its hcp crystal structure and the Zr-Nb interface remained sharp. Here we use a combination of neutron diffraction and dislocation-based polycrystal plasticity constitutive modeling to assess the evolution of texture and deformation mechanisms over a four order-of-magnitude range in layer thicknesses. The phase textures in the nanocomposite strongly deviate from that of Zr or Nb rolled in monolithic form, becoming highly peaked and intense. The model suggests that texture development in the Nb phase is associated with multiple slip and contributions from both $\{112\}\langle 110 \rangle$ slip and $\{110\}\langle 110 \rangle$ slip. In the Zr phase the model suggests that the texture develops due to a predominance of prismatic $\langle a \rangle$ and basal $\langle a \rangle$ slip.

Keywords: texture, severe plastic deformation, accumulative roll bonding, neutron diffraction, composites, nanostructures

Introduction:

When the layer thickness (h) is reduced to the nanoscale, multilayered bimetallic composites have been shown to exhibit improved strength [1-3], thermal stability [4, 5], resistance to shock damage [6], and resistance to radiation damage [7, 8] compared to the constituent materials in bulk form. The majority of research in this area has been conducted on bimetallic systems fabricated using a bottom-up approach such as magnetron sputtering or other deposition methods [1-8]. The low deposition rate of these synthesis techniques makes them unsuitable for producing large volumes of material, precluding structural application of these otherwise exceptional materials.

Accumulative roll bonding (ARB) is a severe plastic deformation (SPD) technique, developed by Saito et al. [9], that has been used in recent years as a route for creating bulk multilayered composites of two dissimilar metals. As the ARB process uses standard metalworking equipment such as rolling mills, this technique is seen as both an economically viable and industrially scalable processing route for creating ultra-fine grained and nanograined metallic composites [10]. This technique has been applied successfully to create bimetallic composites with a variety of crystal structure combinations such as fcc/fcc [11-13], fcc/bcc [14], fcc/hcp [10, 15-20] and bcc/hcp [21]. Large, single-pass reductions are often necessary to achieve bonding during ARB between vicinal neighboring layers. These large reductions coupled with different shear strengths and work hardening rates can promote intense shear banding and break-up of the layered structure prior to reaching the nanoscale regime [16]. The nanoscale regime, where $h < 100$ nm, is where unique property combinations were achieved during bottom-up fabrication methodologies such as physical vapor deposition. In the past, ARB has had success in reducing $h < 100$ nm with combinations of cubic crystal structures like with Cu-Nb [14] and Ag-Fe [11].

Due to the reduced number of slip systems available, hcp materials have not been as amenable to nanostructuring through SPD methods as cubic materials. Without considering composites, many

efforts have been made to make nano-grained Zr, Ti, Be, Mg, and Zn via SPD techniques, such as ball milling, equal channel angular extrusion (ECAE), ARB, and high pressure torsion (HPT) [15, 22-31]. Some success was achieved in creating small quantities of nanoscale hcp material using ball milling and HPT. The coarse-grained structure of commercial purity (CP) Ti was successfully refined to 80 nm by HPT in [32, 33] and by ARB at room temperature [34] as well as under warm conditions [35]. On the other hand, SPD techniques have only achieved 0.5 μm grain size in Mg alloys [22, 36-38]. Prior attempts to refine the grain structure in Zr via SPD either led to submicron grains with grain diameters > 100 nm [23, 24, 39, 40] or a fraction of nanograins of Zr with an alternate crystal structure (ω -Zr, β -Zr or fcc Zr) [23-25, 41]. With single-phase commercial purity Zr, the final grain size reached with the ARB technique was 400 nm [42]. Bimetallic composites with hcp constituents such as Mg-Al [18], Ti-Al [20], Cu-Zn [16], Cu-Zr [17], Zr-Nb [40], and Al-Zn [15] have been synthesized using ARB. A controlled, continuous layered structure with $h < 100$ nm layers, however, has not been achieved.

Nanolamellar Zr-Nb composites has recently been synthesized using the ARB process [21]. The key to achieving nanoscale layers appeared to be intermediate annealing treatments of 575°C for one hour between roll bonding passes. These annealing steps effectively suppressed shear banding and allowed for continued roll bonding to ~ 90 nm individual layer thicknesses. This novel material can potentially lead to a number of interesting findings. In this work, we focus on texture evolution and deformation mechanisms during processing to the nanoscale.

Much is known about the large-strain deformation behavior of the individual constituents, Nb and Zr, in monolithic, polycrystalline, and coarse-grained form (> 1 μm and larger). For instance, their rolling textures after straining with or without subsequent annealing have been reported [43-45] and will be discussed in the results section of this paper. When combined with polycrystalline modeling for texture and stress-strain behavior, the plastic deformation mechanisms activated in rolling and other modes of deformation have been deduced. Based on these studies, these metals are generally expected

to deform via multiple slip modes and not predominantly by twinning at the room temperature and quasi-static strain rates of the ARB process. Specifically, the Nb composition studied here has been found to deform primarily by $\{110\}\langle 111 \rangle$ slip and secondly by $\{112\}\langle 111 \rangle$ slip with no deformation twinning during large-strain rolling or uniaxial deformation [40, 46] over a wide range of temperatures and strain rates [47]. The high-purity Zr composition used here has been shown in [48] to deform primarily by prismatic slip, secondly by pyramidal $\langle c+a \rangle$ slip, and after large strains also by basal slip. The latter phenomenon is interesting because generally, after small to moderate strains in single crystal and polycrystalline Zr, basal slip is expected to be negligible. However, in Yapici et al [39] who studied the large-strain behavior of high-purity Zr using ECAE, texture analysis and modeling suggested that basal slip in addition to prismatic and pyramidal slip was induced during the ECAE pass (~100% simple shear per pass). Likewise, a study of deformation mechanisms in the Zr phase within a non-nanoscale Zr-Nb composite fabricated via ARB (a total accumulated strain of 4) indicated that combinations of prismatic, pyramidal and non-negligible amounts of basal slip were responsible for its texture development [48]. The texture of the polycrystalline Zr phase in the Zr-Nb composite was very similar to that of monolithic Zr, suggesting that the substantial basal slip was an outcome of the SPD and not the coupling with Nb [40]. Since prior electron backscatter diffraction (EBSD) and neutron diffraction (NeD) revealed only a modest amount of twinning in this Zr composition at room temperature [49, 50], deformation twinning was not considered in this calculation.

Although much is known about the texture and deformation mechanisms in Zr and Nb, the extreme strains characteristic of SPD techniques can substantially alter the texture and microstructure of single-phase and two-phase composites [51, 52]. Moreover, it has been shown that combinations of extreme strains and nanoscale grain dimensions can result in significant changes in deformation mechanisms from those expected of the constituents when deformed alone [53-57].

In this work, we take advantage of the unique opportunity to study the plastic behavior of ultra-fine and nanograined Zr and Nb deformed to extreme levels of strain..

We will focus on these three specific areas:

- 1) The texture evolution of Nb during ARB processing of a Nb/fcc composite has been well-characterized for several processing conditions [14, 53]. The Zr-Nb material synthesized for the present work allows for a comparative study of the texture evolution of Nb during processing of Nb-hcp and Nb-fcc composites with similar processing history, length scales, and microstructures.
- 2) The texture evolution of hcp Zr as grain size is reduced in the presence of a bcc metal will also be examined.
- 3) Texture is an indicator of the deformation mechanisms that accommodate plastic strain during roll bonding. The texture evolutions studied in 1 and 2 will be combined with polycrystalline modeling to ascertain the deformation mechanisms that are active at the nanoscale in these materials.

Experimental Methods:

Initial materials for the accumulative roll bonding (ARB) process consisted of 2 mm thick sheets of high-purity Zr and reactor-grade, high-purity Nb (99.97% pure, ATI-Wah Chang). The nominal composition of the Zr expressed in weight parts per million (Wt. ppm) is Hf 35, Fe <50, Al <20, V <50, O <50, N <20, C <22, Zr balance. The composition for the Nb in (Wt. ppm) is C<20, Fe <25, H <3, Hf <30, Mo <30, N <20, Ni <20, O<40, P<30, Si<25, Ta=110, Ti<30, W<30, Zr<30 and Nb balance.

Zr plate processing [58] consisted of upset forging, clock rolling, and annealing in order to provide a plate with a strong fiber texture aligned with the c-axis in the normal direction. Clock rolling is similar to traditional rolling except that the rolling direction is rotated around the plate normal for every

subsequent pass such that the rolling direction is continuously changing. After this initial processing, the plate underwent further rolling and heat treatments [58]. The Zr plate utilized during the ARB process had equiaxed, twin-free grains of approximately 40 μm in diameter and strong basal texture. The Nb plate was annealed such that it exhibited a recrystallized microstructure with a (111) fiber texture parallel with the normal direction (ND) of the plate. Nominal grain size in Nb after heat treatment was larger than that of Zr with an approximate grain diameter of 300 μm . Figure 1 in [40] provides EBSD-based inverse pole figure grain maps coupled with pole figures for the starting Nb and Zr material used in this study.

The ARB process involves stacking plates of material and performing a single-pass room temperature rolling reduction ($\sim 50\%$) to bond the initially separate sheets. The Zr and Nb plates, after initial processing, were cleaned in an ultrasonic acetone bath and then wire brushed with a 76 mm diameter wheel of fine stainless steel bristles rotating at 1200 revolutions per minute. This surface preparation, repeated prior to all ARB steps, served to minimize retained oxide within the microstructure as well as to promote bonding between the layers by removing the native oxide and enhancing the surface roughness. The Zr and Nb sheets were stacked and then roll bonded via a single high-strain rolling pass on a 2-high rolling mill (Waterbury-Farrel) with 457.2 mm diameter rolls and a strain rate of $\sim 50 \text{ mm/mm} \cdot \text{sec}$. Mill settings during roll bonding were chosen in order to achieve a target of 50% reduction in overall sample thickness. Due to differing amount of work hardening present in each sample, however, actual reductions ranged from 40-60%. After successful roll bonding, the plate was cut in half and stacked such that the process could be repeated. In this way, material was fabricated that had various nominal layer thicknesses and various amounts of accumulated strain. In addition, the Zr and Nb sheets were initially stacked in a Zr-Nb-Zr sandwich with a Nb sheet of 2 mm and Zr sheets of 1 mm. The Zr-Nb interface bond was created only during the first pass and all subsequent

bonding steps were Zr on Zr. This means that any retained oxide along the initial Zr-Nb interface was then spread over kilometers of interfacial length during the roll bonding process.

Intermediate annealing treatments were conducted in order to minimize the occurrence of shear bands. Static anneals at 575°C for 60 minutes under vacuum followed by furnace cooling were performed after every second roll bonding pass or at strain intervals of ~1.4. The time and temperature combination was chosen in order to minimize intermixing of the Zr and Nb which may be expected to occur above temperatures of around ~500°C [59].

Characterization via diffraction was performed after both roll bonding steps and annealing treatments. Although roll bonding/restacking steps and annealing treatments were performed between $h = 676$ nm and $h = 92$ nm, material was not removed for characterization in order to conserve material for further roll bonding steps. Table 1 provides the layer thicknesses and total strain associated with each sample that was investigated in this study. Due to the initial Zr-Nb-Zr stacking sequence, the average layer thickness, h , reported in Table 1 represents the nominal layer thickness for all layers except the two surface layers of Zr, which are approximately $h/2$. Total strain was calculated via equation (1):

$$\varepsilon = \ln \frac{h_0}{h} \quad (1)$$

Table 1. Number of layers, layer thickness, total strain and latest processing step for the material characterized through NeD. A (^) indicates processing steps where material was not retained for characterization.

Number of Layers	Average Layer Thickness, h , um	Total Strain, ε	Latest Processing Step
2	845	0.9	Roll Bonding
4	466	1.5	Roll Bonding
4	466	1.5	Annealing

8	198	2.3	Roll Bonding
16	106	2.9	Roll Bonding
16	106	2.9	Annealing
32	59	3.5	Roll Bonding
64	26	4.3	Roll Bonding
64	26	4.3	Annealing
128	12	5.1	Roll Bonding
256	6	5.8	Roll Bonding
256	6	5.8	Annealing
512	3	6.5	Roll Bonding
1024	1.5	7.2	Roll Bonding
1024	1.5	7.2	Annealing
2048	0.676	8.0	Roll Bonding
4096[^]	0.372	8.6	Roll Bonding
4096[^]	0.372	8.6	Annealing
8192[^]	0.155	9.5	Roll Bonding
16384	0.092	10.0	Roll Bonding

NeD was performed on both the initial materials and processed samples using the High Pressure Preferred Orientation (HIPPO) neutron time-of-flight diffractometer at the Los Alamos Neutron Science Center at Los Alamos National Laboratory (LANL) [60, 61]. Unlike X-ray diffraction or EBSD, NeD provides a bulk measurement of specimen texture. The techniques used for both the collection of NeD data and the accompanying analysis to capture phase-specific orientation distribution functions (ODFs) was consistent with those described in [14]. A small change within the HIPPO diffractometer set-up is

the addition of two new detector panels at $2\theta = 60^\circ$ and 120° . The samples for which bulk textures for Nb and Zr within the composite were measured are listed in Table 1.

Transmission electron microscopy (TEM) was used in order to investigate the planarity of the interfaces and confirm the nominal thickness of the layers when $h = 92$ nm. Chemical sharpness of the interfaces at this length scale was explored in a previous publication [43] and indicated that the interfaces were chemically sharp, despite the mechanical straining and intermediate annealing treatments. Bright Field TEM was performed using a Tecnai F30 (FEI, Hillsboro, OR, USA) electron microscope operating at 300 kV on cross-sectional samples viewed along the transverse direction (TD). The samples were prepared using a Helios Nanolab DualBeam (FEI, Hillsboro, OR, USA) Focused Ion Beam (FIB). A traditional approach of tripod polishing, dimpling, and ion milling lead to damage in the Zr layers that prevented successful characterization. In this case, FIB lift-outs were extracted from a plan view section of the composite. Prior to extraction a chemical polish of 25 ml lactic acid, 15 ml of HNO_3 , and 5 ml HF was applied for an extended period of time in order to remove surface layers damaged during previous preparation steps.

Experimental Results and Discussion:

Microstructure:

Figure 1 shows electron microscopy results of the $h = 92$ nm Zr-Nb sample. Selected area diffraction patterns such as the one shown in Figure 1b indicate that the hcp crystal structure of Zr was maintained during fabrication. Figure 1c presents a scanning transmission electron microscopy (STEM) bright field image with phase boundaries overlaid in red based on the corresponding high angle annular dark field image (not shown). Figure 1c clearly shows that the lamellar structure is maintained, though signs of non-homogenous deformation are present such as variations in layer thickness. It is also observed in Fig. 1c that, despite the fine nanoscale dimensions, grain boundaries are present within both the Zr and Nb layers. The relatively thin layers tend to have a single grain through the thickness

whereas the relatively thicker layers tend to have multiple grains through the layer thickness. In the bright field TEM diffraction contrast image in Fig. 1a, significant dislocation-based contrast is evident within both Zr and Nb. The frequency of grain boundaries and stored dislocation content within the grains is much higher than in nanostructured ARB Cu-Nb multilayers strained to approximately the same h [53].

As stated earlier, due to the intermittent annealing treatments, shear bands did not form in this material and this is confirmed by optical microcopy [40]. Although shear banding is suppressed, interface roughening is observed. Interface roughening is not limited to the Zr-Nb material system and has been observed in the Cu-Nb, Cu-Zn, and Cu/Zn/Al systems [14, 16, 62] and is predicted in two-phase crystal plasticity finite element calculations [47, 63, 64].

Texture evolution:

Nb phase in the composite: An α -fiber plot for the Nb phase within the composite is shown in Figure 2 for varying layer thicknesses and processing steps. The α -fiber is a characteristic fiber for rolled body centered cubic (BCC) materials that is comprised of orientations that share the $\langle 110 \rangle$ crystal axis parallel to the rolling direction (RD).

Generally we find that the most significant layer size effect is the Nb texture is weak for all $h \geq 1.5 \mu\text{m}$ but noticeably stronger for $h = 92 \text{ nm}$. For $h \geq 1.5 \mu\text{m}$, very small peaks in intensity are found for the $\{001\}$, $\{113\}$, and $\{334\}$ orientations. The $h = 92 \text{ nm}$ as-rolled sample, however, exhibits a more intense α -fiber containing a relatively strong peak at the $\{001\}\langle 1\bar{1}0 \rangle$ orientation and two minor peaks at $\{115\}\langle 1\bar{1}0 \rangle$ and near $\{334\}\langle 1\bar{1}0 \rangle$.

Figure 2 shows the corresponding γ -fiber plot for the Nb phase within the composite. The γ -fiber plot provides ODF intensity data for orientations with the $\{111\}$ parallel to the normal direction (ND) of the sample. Like the α -fiber, the γ -fiber is weak for $h \geq 1.5 \mu\text{m}$ but is more intense for $h = 92 \text{ nm}$.

Comparing the α and γ -fibers in the as-rolled (AR) and annealed (AN) samples reveals that annealing had very little effect on the texture. This may be expected, as the melting temperature of Nb is high relative to the temperature of the intermediate static annealing steps. However, in prior works, a strengthening of the γ -fiber during similar annealing treatments of Nb has been reported [65]. As Fig. 2 shows, such a strengthening of the γ -fiber is not seen in this study.

Comparison with textures in the literature: Figure 3 compares the α and γ fibers of the resulting texture in the Nb phase at $h = 92$ nm with those of other rolled or roll-bonded Nb textures reported in the literature [14, 40, 45]. Of these reported textures, one is the rolled single-phase Nb in [45] and another is the Nb phase texture within the roll-bonded Zr-Nb with $h = 4$ μ m, which was made without intermediate annealing steps [40]. These two Nb textures are similar to each other but different than the Nb texture within the coarse- and fine-layered Zr-Nb composites made here. To compare with another nanolayered Nb phase, we also provide the Nb phase texture within the roll-bonded Cu-Nb at $h = 86$ nm that also had undergone intermittent annealing treatments [14]. Unlike the Nb phase texture in the ARB Zr-Nb, the Nb phase texture in the ARB Cu-Nb contains a peak near the $\{112\}\langle 1\bar{1}0 \rangle$ orientation.

The Nb phase textures within the present Zr-Nb samples at all h are significantly different than these three other Nb textures. For instance, in all the other cases, the $\{001\}\langle 1\bar{1}0 \rangle$ component diminished and the $\{112\}\langle 1\bar{1}0 \rangle$ or a component near it strengthened with increasing strain. In contrast, in the Nb phase texture in the present Zr-Nb composite the most intense component in the α fiber was the $\{001\}\langle 1\bar{1}0 \rangle$ component and the $\{112\}\langle 1\bar{1}0 \rangle$ component did not strengthen as the strain increased. It may be that the deformation mechanisms operating during rolling in the Nb phase within the present composites deviate from those expected in these other Nb based systems. Later, we investigate using crystal plasticity the slip activity associated with the development of the Nb texture.

Zr phase in the composite: Figure 4 shows the measured $\langle 11\bar{2}0 \rangle$ and $\langle 10\bar{1}0 \rangle$ fibers for the Zr phase within composites of different layer thickness. As h was reduced from 106 μm to 92 nm, the texture strengthened, becoming peaked at two components. In Fig. 4a for the $\langle 11\bar{2}0 \rangle$ fiber, a peak centering on the $\{3\bar{3}017\}\langle 11\bar{2}0 \rangle$ component increases with strain. In Fig. 4b for the $\langle 10\bar{1}0 \rangle$ fiber, the peak develops at the $\{1\bar{2}112\}\langle 10\bar{1}0 \rangle$ component with increasing strain. The texture in the nanolayered composite is remarkably sharp.

The fiber plots for the $h = 26\text{ }\mu\text{m}$ and $h = 1.5\text{ }\mu\text{m}$ after static annealing are also shown in order to assess the effect of annealing on texture. For the thicker $h = 26\text{ }\mu\text{m}$ sample, we observe that annealing affects the texture, shifting the peak from the $\{3\bar{3}017\}\langle 11\bar{2}0 \rangle$ component to the $\{7\bar{7}024\}\langle 11\bar{2}0 \rangle$ component along the $\langle 11\bar{2}0 \rangle$ fiber. This result is consistent with the observations of Jiang et al. [44] on single-phase rolled Zr. There it was shown that annealing at over 600° C leads to a similar shift in the $\langle 11\bar{2}0 \rangle$ fiber. In addition, we also observe a noticeable change in the $\langle 10\bar{1}0 \rangle$ fiber, namely a peak shift from the $\{1\bar{2}112\}\langle 10\bar{1}0 \rangle$ component to the $\{4\bar{8}425\}\langle 10\bar{1}0 \rangle$ component.

Most interestingly, we find that the peak shift caused by annealing in the coarser layer 26 μm is not apparent in the finer layer 1.5 μm . From the two fiber plots, we see that annealing this finer layer sample weakened the overall α fiber texture. A similar length-scale dependent hindrance of recrystallization at similar layer thicknesses between 1-2 μm was reported in the Cu phase of a Cu-Nb layered composite produced by ARB [66]. There it was linked to either the noticeable reduction in dislocation density seen in Cu at 1-2 μm layers or interface constraints. Given the significant dislocation content observed in the Zr phase in Figure 1a, it could be that suppression of certain recrystallization effects on texture at this 1-2 μm length scale is due to layer constraint.

Comparison with textures in the literature: Figure 5 compares the texture for Zr in this study at $h = 92\text{ nm}$ with a previously reported texture for Zr in a roll bonded Zr-Nb composite with 4 μm layers fabricated without intermediate annealing treatments [40]. Quantitative texture comparisons in this

paper are limited to prior Zr-Nb results due to the inadequacy of pole figures and inverse pole figures presented in other studies to delineate between intensity associated with the $\{3\bar{3}017\}[11\bar{2}0]$ and $\{1\bar{2}112\}[10\bar{1}0]$ peaks. The Zr phase textures of this study, both the coarse-layered and the nanolayered (92 nm), develop very different textures. First, from Fig. 5, we clearly see that the Zr phase texture from [40] shows a broad peak of intensity centered on the $\{8\bar{1}6827\}[10\bar{1}0]$ component in the $\langle 10\bar{1}0 \rangle$ fiber and little to no intensity along the $\langle 11\bar{2}0 \rangle$ fiber. Second, the texture developed in the 92 nm composite is distinctively much more peaked than previously reported Zr textures. Thus, we would expect that the deformation mechanisms operating in the Zr-Nb composites made here (with intermediate annealing plus roll bonding) are different than those in conventionally rolled polycrystalline Zr, particularly as the layer thickness reduces to the nanoscale. We explore this hypothesis in the modeling section.

Interfacial Analysis:

Figure 6 provides inverse pole figures (IPFs) for both Zr and Nb measured via NeD at $h = 92$ nm in the as-rolled condition. As the interfaces are relatively planar and perpendicular with the sample ND, the ND IPF can be utilized as an estimate of the interface plane normal distribution.

Consistent with the rolled monolithic Zr in [44] and the TEM micrograph of the Zr phase in the present composites in Figure 1, the Zr IPF for the RD shows intensity near the $\{1\bar{1}00\}$ planes. However, unlike the texture of the monolithic Zr, the IPF for the ND shows a band of intensity at approximately 15° from the $\{0001\}$ planes and not 25° away as seen in [44]. This significant difference is sufficient to indicate a possible change in deformation mechanisms.

In Nb, two peaks dominate the ND IPF intensity – $\{100\}$ and $\{111\}$. These features point to interfaces largely being either $\{110\}_{Nb}^{RD} || \{1\bar{1}00\}_{Zr}^{RD}$ and either $\{111\}_{Nb}^{ND} || \{1\bar{2}110\}_{Zr}^{ND}$ or $\{100\}_{Nb}^{ND} || \{1\bar{2}110\}_{Zr}^{ND}$. Therefore, it appears that, in contrast to roll bonded Cu-Nb [53], a single, stable interface has not developed in Zr-Nb at $h = 92$ nm. Nonetheless, considering the strong textures

in both Zr and Nb, the distribution of interface characters could be narrow and thus Zr-Nb could be useful as another material system to apply theories of stable interfaces under extreme straining [67].

Polycrystal Modeling:

The neutron diffraction indicates two interesting results: 1) the texture of the Nb phase in the present Zr-Nb nanoscale composites differs noticeably from the textures of Nb in other material systems deformed in similar ways and 2) a significant transition in the texture of the Zr phase as the material is refined from micron-layer thicknesses to nanolayer thicknesses. We suspect that these changes could be signatures of deviations in slip activities from those expected or reported previously. To explore this possibility, the viscoplastic self-consistent (VPSC) crystal plasticity polycrystal model is used to relate dislocation slip activity to the texture development observed in the Zr and Nb polycrystalline phases within the present composite [40]. This model does not account for the effects of the Zr-Nb interface and therefore cannot reveal the role the interface plays in texture evolution. It also does not account for intermediate annealing steps. Despite these limitations, the model can be useful, at least as a first step, in determining the active slip systems and their relative activity needed to attain the observed textures.

Modeling methodology:

In brief, VPSC is a mean-field polycrystal formulation [50, 68]. It represents a polycrystal as a group of grain orientations with a distinct orientation and volume fraction. In calculation, each grain orientation is modeled as an ellipsoidal inclusion embedded in a homogeneous effective medium (HEM) with average properties of the polycrystal. Like most polycrystal models, it accounts for orientation and hardening evolution on each slip system within each grain. Unlike simpler upper and lower bound mean-field polycrystal models, such as Taylor, Relaxed constraints, and Sachs [69]; however, VPSC accounts for the evolution of grain shape.

Important assumptions when utilizing the model are selecting which slip systems are made available for crystallographic slip and how dislocation hardening evolves in these slip systems. It has

been shown that in conventional coarse-grained Zr and Zr alloys, slip occurs most often on the prismatic $\langle a \rangle$ and pyramidal $\langle c+a \rangle$ slip systems [50, 70, 71], and at large strains basal $\langle a \rangle$ slip systems can be additionally activated [39, 48]. Here, these three modes are made available to the Zr crystals. In Nb, slip occurs most often on the $\{110\}\langle 111 \rangle$ and $\{112\}\langle 111 \rangle$ slip systems [72, 73] and these were made available to Nb. We will refer to these systems, respectively, in short as $\{110\}$ and $\{112\}$ slip.

In this work, the hardening laws for each slip system in each phase are based on thermally activated dislocation motion and vary as a function of temperature and strain rate [50]. In this hardening model, denoted the DD model, the evolution of dislocation density (DD) is different for each slip mode and is governed by material parameters such as Peierls stress, activation energy for depinning locked dislocations, and dislocation trapping rates.

Table 2 Material parameters for evolution of slip resistance in the coarse-grained Zr phase [50].

α – slip mode	prismatic slip $\alpha=1$	basal slip $\alpha=2$	pyramidal slip $\alpha=3$
$\tau_{o,f}^{\alpha}$ [MPa]	20	700	160
k_i^{α} [m^{-1}]	1.25×10^8	1.25×10^{10}	2.25×10^8
g^{α}	3.75×10^{-3}	3.675×10^{-2}	6.5×10^{-2}
D^{α} [MPa]	330	330	100
$\dot{\epsilon}_o^{\alpha}$ (s^{-1})	10^7	10^7	10^7
q^{α} [MPa]	88	0	0
H^{α} [MPa]	100	100	170

For the Zr phase the DD model material parameters for the three slip modes, α : prismatic ($\alpha=1$), basal ($\alpha=2$), and pyramidal slip ($\alpha=3$), were established earlier in [50] and are given in Table 2. These achieved good agreement with the textures seen in rolled monolithic Zr and the coarse-layered (4 μm) Zr phase in a straight-roll-bonded Zr-Nb composite. In the table, $\tau_{o,f}^s$ is the initial friction stress, H^{α} is a Hall-Petch-like constant, $\dot{\epsilon}_o$ is a reference strain rate, g^{α} is an effective activation enthalpy, D^{α} is a

drag stress, and q^α is a dislocation recovery rate coefficient defining the fraction of α -type dislocations that do not annihilate but become substructure.

In bcc metals, many recent studies have demonstrated the importance of non-Schmid effects on the motion of dislocations belonging to the $\{110\}$ and $\{112\}$ slip systems [74-76]. For the Nb phase, we make use of a modified hardening law that introduces non-Schmid effects into the DD model [77]. While the material parameters we use have been characterized for the particular Nb composition studied here [40], the extra parameters associated with the non-Schmid effects were not altered from those used previously for a Ta-W alloy. This simplification was done primarily due to a lack of data and evidence suggesting that these parameters would not depend sensitively on material details. The DD material parameters used for both $\{110\}$ and $\{112\}$ slip systems in the coarse-grained Nb phase are given in Table 3. The assumption of equal resistances to $\{110\}$ and $\{112\}$ slip will be addressed in the results.

Table 3 - Constitutive parameters for evolution of slip resistance in the Nb phase [40].

α – slip mode	$\{110\}$ & $\{112\}$ slip
$\tau_{o,f}^\alpha$ [MPa]	135
k_1^α [m^{-1}]	4.4×10^8
g^α	3.5×10^{-2}
D^α [MPa]	210
$\dot{\epsilon}_o^\alpha$ (s^{-1})	10^7
q^α [MPa]	14
H^α [MPa]	50

Deformation twinning in either phase takes place at colder temperatures than the room temperature used here [78]. As it is not observed in significant fraction ($> 1\%$) in the Zr and Nb phases in post-deformation TEM and EBSD analyses, its contribution is neglected in the model calculations.

Finally, in all VPSC calculations, we use boundary conditions of plane strain compression, an idealization of rolling that best applies to the center of the material, and apply low strain rates at room temperature to match laboratory conditions [47]. In the model, the texture is initialized using the measured starting texture (Fig. 1 in [40]). Reduction of Zr-Nb layer thickness for modeling of the nano-

grained material was from 1.5 μm to 92 nm. In all calculations, the simulations were carried out to 0.6 strain.

Modeling Results and Discussion:

Comparison with Simulated VPSC-Based Nb Textures: Figure 7 compares the α and γ -fibers for the Nb phase in the present Zr-Nb nanocomposite sample with $h = 92$ nm with the VPSC simulation. The model predicts both the γ -fiber and α -fiber reasonably well. It captures the higher intensity of the $\{001\}\langle 1\bar{1}0 \rangle$ component and the less intense peak near the $\{334\}\langle 1\bar{1}0 \rangle$ component. To compare the textures, Fig. 8 shows the $\{001\}$, $\{011\}$, and $\{111\}$ pole figures from the measurement and the VPSC calculation. Good agreement is again achieved. From these calculations, it is possible to extract the average slip activity, which is related to the number and distribution of slip shear on the active systems for each grain, averaged over all the grains in the polycrystal. As shown, the relative activities of $\{110\}$ and $\{112\}$ slip are similar with $\{110\}$ slip being slightly more active (Fig. 8c).

Two aspects are worth noting. First, we find that predicted texture evolution corresponds to multi-slip conditions inside the grains. Thus, multi-slip still persists in nano-sized crystals. This result is consistent with the dense dislocation structures seen in the TEM analysis (Fig. 1). Second, good agreement is also achieved when the initial slip resistance for $\{110\}$ slip increases to 145 MPa. This change only makes $\{112\}$ slip a little less active than $\{110\}$ slip. Thus, it appears that the Nb phase texture is associated with more or less similar contributions from both $\{110\}$ and $\{112\}$ slip.

Comparison with Simulated VPSC-Based Zr Textures: Figure 5 compares the measured $\langle 11\bar{2}0 \rangle$ and $\langle 10\bar{1}0 \rangle$ fibers at $h = 92$ nm with two VPSC simulations. Figures 9 and 10 make the same comparison for the entire textures using $\{0001\}$, $\{10\bar{1}0\}$, $\{11\bar{2}0\}$ pole figures and for the $\phi_2 = 0$ and 30° ODF sections. In one simulation (labeled Sim. All Systems Active), we use the parameters in Table 2, which provides a texture characteristic of rolled Zr and roll-bonded Zr in a coarse-layered Zr-Nb composite [40]. The measurement of the latter sample is also shown in Fig. 5 and it can be seen that this VPSC simulation

captures the weak $\langle 11\bar{2}0 \rangle$ fiber, albeit with much less intensity, and the strong $\langle 10\bar{1}0 \rangle$ fiber with a peak at the $\{8\bar{1}6\ 8\ 27\}\langle 10\bar{1}0 \rangle$ component. The corresponding slip activity is given in Figure 8e. Prismatic slip is the most active, followed by pyramidal slip. As straining proceeds, prismatic and pyramidal slip harden, which is seen to promote basal slip. The amount of basal slip predicted (reaching ~25%), although uncommonly large for Zr, is still consistent with other studies of Zr deformed to large strains [39, 79]. However, as noted before, the Zr in the present 92 nm nanocomposite develops a strikingly different texture than straight-rolled Zr and straight roll-bonded Zr. This difference suggests that the slip activity in the 92 nm composite is likely different than that shown in Figure 8e.

Figure 5 compares a second VPSC simulation (labeled Sim. SPIB) where the initial slip resistances for pyramidal slip and basal slip were altered (to 1400 and 400 MPa, respectively), such that the pyramidal slip was nearly suppressed and the basal slip significantly enhanced. Figures 9a,b and 10a,b show the same comparison respectively in the form of the $\phi_2 = 0$ and 30° ODF sections and pole figures. From Fig. 5, we see that with this new calculation the $\langle 10\bar{1}0 \rangle$ fiber attains the strong peak at the $\{1\bar{2}\ 1\ 12\}\langle 10\bar{1}0 \rangle$ component, as seen experimentally. It is important and can be concluded that stabilization of the $\{1\bar{2}\ 1\ 12\}\langle 10\bar{1}0 \rangle$ component in plane-strain compression requires an unusual predominance of prismatic and basal slip with pyramidal suppressed.

The VPSC simulations do not explain the occurrence of the large peak at the $\{3\bar{3}\ 0\ 17\}\langle 11\bar{2}0 \rangle$ component in the $\langle 11\bar{2}0 \rangle$ fiber, particularly prominent in the nanoscale composite. In the calculation, the $\langle 11\bar{2}0 \rangle$ fiber was not as strong and stable. Since our VPSC simulations do not include the effects of intermediate annealing treatments, it may be inferred that the observed stability of this component could be a result of annealing. Prior work on ARB of monolithic Zr [44] found that annealing is responsible for a rotation of peak intensity to this component in the $\langle 11\bar{2}0 \rangle$ fiber. To gain insight on whether annealing was involved, we carried out VPSC simulations starting with an annealed texture at 1.5 μm with the predominant $\{3\bar{3}\ 0\ 17\}\langle 11\bar{2}0 \rangle$ component. However the calculations find that the

slip induced under plane-strain compression destabilizes this orientation. Therefore, it is doubtful that the persistence of this orientation should be attributed to intermediate annealing. It is more likely a consequence of the interface, which would have a growing influence as layer thickness decreases.

The foregoing analysis provides some hints that the interface affects texture development. At the fine length scales and high interface densities associated with nanolayer thicknesses, it is reasonable to think the influence of the interface is enhanced. It has been proposed, for instance, in fcc-bcc composites that the bimetal interface can influence slip and twinning activity in several ways, such as via nucleation and transmission of certain slip systems [80, 81] and twinning systems [78], geometric constraints [80], kinematic constraints associated with co-deformation [46] and its formation energy [67]. Many of these studies are carried out using atomic-scale simulations [78, 82-84], involving small numbers of dislocations and short time scales, and thus it is not straightforward to translate these results to texture evolution. Some attempts have been made to incorporate such dislocation-interface interactions into larger scale polycrystalline plasticity models, like VPSC or 3D full-field spatially resolved techniques, such as crystal plasticity finite element [46, 47, 85-87]. Only very recently has CPFE been extended to examine the effect of slip transmission on texture evolution [88]. Clearly more effort in this area of interface-driven slip activity and texture evolution is needed. At present, we can only speculate that the stabilization of predominant $\{11\bar{2}10\}\langle11\bar{2}0\rangle$ component with strain in the nanoscale composite could be due to kinematic constraints at the hcp/bcc interface that become more prominent as the interface density increases and/or local shear components.

Conclusions:

In this study, we investigated the texture evolution of a Zr-Nb nanolayered composite processed via accumulative roll bonding. Average individual nanoscale layer thicknesses of 92 nm were achieved via severe plastic deformation (over strains of 10) and intermediate static annealing steps between roll-

bonding steps. The lamellar structure was maintained, the Zr phase remained hcp, and the interfaces were relatively sharp. The main conclusions of this work are:

1. Using neutron diffraction we show that the as rolled textures of the Zr and Nb phases within the composite deviated from those observed in Zr [58] and Nb [55] when rolled separately, in Nb in accumulative roll bonded Cu-Nb [14], and in the Zr and Nb phases within accumulative roll bonded Zr-Nb without an intermittent annealing treatment [64].
2. We observed a marked increase in the intensity of the Nb phase texture of the $h = 92$ nm composite that was not seen in the coarser micron-layered composites. The $h = 92$ nm as-rolled sample exhibited an intense α -fiber containing a relatively strong peak at $\{001\}\langle 1\bar{1}0 \rangle$ orientation and two minor peaks at $\{115\}\langle 1\bar{1}0 \rangle$ and near $\{334\}\langle 1\bar{1}0 \rangle$.
3. The Zr phase texture of the $h = 92$ nm composite developed a noticeably sharp texture with two main peaks, one centering on the $\{3\ \bar{3}\ 0\ 17\}\langle 1\ 1\ \bar{2}\ 0 \rangle$ component of the $\langle 11\bar{2}0 \rangle$ fiber and another at the $\{1\ \bar{2}\ 1\ 12\}\langle 1\ 0\ \bar{1}\ 0 \rangle$ component in the $\langle 1\ 0\ \bar{1}\ 0 \rangle$ fiber.
4. A dislocation density based hardening model with non-Schmid effects was implemented into the polycrystal visco-plastic self-consistent scheme (DD-VPSC) to associate the active deformation slip modes with texture development. The model suggested that the Nb phase texture in the 92 nm composite is associated with multiple slip and similar contributions from $\{110\}\langle 111 \rangle$ and $\{112\}\langle 111 \rangle$ slip.
5. The DD+VPSC model was also applied to the Zr phase. The model suggested that the predominant $\{1\ \bar{2}\ 1\ 12\}\langle 1\ 0\ \bar{1}\ 0 \rangle$ component can be attributed to an unusual slip activity of prismatic and basal slip with little to no pyramidal slip in plane-strain compression.
6. Analysis of the inverse pole figures for the Zr-Nb nanocomposite with $h = 92$ nm suggested that a few interface characters may predominate: $\{110\}_{Nb}^{RD} || \{1\ \bar{1}00\}_{Zr}^{RD}$ and either $\{111\}_{Nb}^{ND} || \{1\ \bar{2}\ 1\ 10\}_{Zr}^{ND}$ or $\{100\}_{Nb}^{ND} || \{1\ \bar{2}\ 1\ 10\}_{Zr}^{ND}$.

Acknowledgements:

This work was supported in part by the *Center for Materials at Irradiation and Mechanical Extremes*, an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under Award Number 2008LANL1026. This work was also supported in part by the Los Alamos National Laboratory Directed Research and Development (LDRD) project 20140348ER. This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Los Alamos National Laboratory, an affirmative action equal opportunity employer, is operated by Los Alamos National Security, LLC, for the National Nuclear Security Administration of the U.S. Department of Energy under contract DE-AC52-06NA25396. Neutron diffraction results were collected on the High Pressure Preferred Orientation (HIPPO) beam line at the Los Alamos Neutron Science Center. Electron microscopy was performed at the Los Alamos Electron Microscopy Laboratory.

Figure Captions:

Figure 1: (a) TEM-Based diffraction contrast image of Zr-Nb multilayer when $h = 92$ nm. (b) Selected area diffraction shows that the Zr phase is highly textured and consistent with the NeD texture. (c) BF STEM image of Zr-Nb layers with contrast showing grain boundaries within layers. Red lines indicate phase boundaries which were determined by complimentary HAADF STEM.

Figure 2: Fiber plots for α and γ fibers for Nb for varying layer thicknesses and processing history. For coarser layers ($h \geq 1.5$ μm) little to no change in texture is observed between roll bonding or annealing steps. The observed texture is inconsistent with that of rolled Nb [45]. At $h = 92$ nm, a sharp peak is present at $\{001\}\langle 110 \rangle$ with other peaks at $\{115\}\langle 110 \rangle$ and $\{334\}\langle 110 \rangle$. For comparison, the ODF intensity associated with the $\{112\}\langle 110 \rangle$ Nb peak for ARB Cu-Nb at similar length scales was 15 [14].

Figure 3: α and γ fiber plot for Nb from the Zr-Nb material at $h = 92$ nm in this study compared with literature. Nb in non-annealed Zr-Nb, annealed Cu-Nb and rolled single phase Nb all show a peak in intensity near the $\{112\}\langle 1\bar{1}0 \rangle$ orientation. Nb in annealed Zr-Nb, shows a more complex array of peaks.

Figure 4: Fiber plots for Zr for varying layer thicknesses and processing steps. (a) $\langle 1\ 1\ \bar{2}\ 0 \rangle$ fiber plot. At coarser layers (106 and 26 μm) annealing leads to a reduction in $\{3\ -3\ 0\ 17\}\langle 1\ 1\ -2\ 0 \rangle$ component and an increase in $\{7\ -7\ 0\ 24\}\langle 1\ 1\ -2\ 0 \rangle$ component. This effect is less apparent for the sample with $h = 1.5\ \mu\text{m}$ annealed. Roll bonding steps exaggerate the $\{3\ -3\ 0\ 17\}\langle 1\ 1\ -2\ 0 \rangle$ component at the expense of both $\{0001\}\langle 11\ -20 \rangle$ and $\{7\ -7\ 0\ 24\}\langle 1\ 1\ -2\ 0 \rangle$ components for all length scales from 26 μm to 92 nm. (b) $\langle 1\ 0\ \bar{1}\ 0 \rangle$ fiber plot. At coarser layers (106 and 26 μm) annealing leads to a reduction in $\{1\ -2\ 1\ 12\}\langle 1\ 0\ -1\ 0 \rangle$ component and an increase in $\{4\ -8\ 4\ 25\}\langle 1\ 0\ -1\ 0 \rangle$ component. This effect is less apparent for the sample with $h = 1.5\ \mu\text{m}$ annealed. Roll bonding steps exaggerate the $\{1\ -2\ 1\ 12\}\langle 1\ 0\ -1\ 0 \rangle$ component at the expense of both $\{0001\}\langle 1\ 0\ -1\ 0 \rangle$ and $\{4\ -8\ 4\ 25\}\langle 1\ 0\ -1\ 0 \rangle$ components for all length scales from 26 μm to 92 nm.

Figure 5: Fiber plots for Zr compared with literature and simulations. (a) $\langle 1\ 1\ \bar{2}\ 0 \rangle$ fiber plot. For Zr-Nb without annealing and VPSC-based simulations (which do not include annealing), $\{3\ -3\ 0\ 17\}\langle 1\ 1\ -2\ 0 \rangle$ texture is not observed to the level seen in this study. (b) $\langle 1\ 0\ \bar{1}\ 0 \rangle$ fiber plot. Zr-Nb without annealing has a large broad peak that is centered on the $\{8\ -16\ 8\ 27\}\langle 1\ 0\ -1\ 0 \rangle$ component. Note that suppressing pyramidal slip coupled with increased basal slip (SPIB) improves the VPSC fit along the $\langle 1\ 0\ -1\ 0 \rangle$ fiber for the Zr in this study. When all slip systems are active, VPSC performs well in matching the Zr texture along the $\langle 1\ 0\ -1\ 0 \rangle$ fiber for Zr-Nb without annealing.

Figure 6: IPFs for Zr (top row) and Nb (bottom row) for $h = 92$ nm. RD, TD, and ND refer to the sample direction with ND providing an estimate of the interface plane normal distribution.

Figure 7: α and γ fiber plot for Nb from the Zr-Nb material at $h = 92$ nm compared with VPSC simulation results.

Figure 8: Pole figure representations of (a) NeD results for Nb in Zr-Nb at $h = 92$ nm, (b) VPSC simulation results for slip activity as shown in (c).

Figure 9: ODF slices for Zr based on (a) NeD results for Zr in Zr-Nb at $h = 92$ nm, (b) VPSC-based simulation results with pyramidal slip activity suppressed and increased basal activity as seen in (d), (c) simulation results for slip activity as shown in (e), (d) slip activity as a function of strain for the VPSC simulation with suppressed pyramidal activity and increased basal slip activity (SPIB). (e) slip activity as a function of strain for the VPSC with all systems active.

Figure 10: Pole figure representations of (a) NeD results for Zr in Zr-Nb at $h = 92$ nm, (b) VPSC-based simulation results with pyramidal slip activity suppressed and basal slip increased as shown in Figure 9(d), (c) Simulation results for slip activity as shown in Figure 9(e).

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Figure 1

Figure 2

Figure 3

Figure 4

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Figure 6

Figure 7

Figure 8

Figure 9

Figure 10