

## LA-UR-15-29645

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Title:	Towards intelligent microstructural design of Nanocomposite Materials: Lightweight, high strength structural/armor materials for service in extreme environments
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Intended for:	Response to: Request for Information for DoD Long Range Research and Development Program Plan
Issued:	2015-12-21

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**Towards intelligent microstructural design of Nanocomposite Materials: Lightweight, high strength structural/armor materials for service in extreme environments**

Technical Areas of Relevance:

1. Space Technology
2. Undersea Technology
3. Air Dominance and Strike Technology
4. Air and Missile Defense Technology
5. Other Technology-Driven Concepts: Lightweighting of Air/Sea Drones for Defense Applications

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**Response to: Request for Information for DoD Long Range Research and Development  
Program Plan**

## Executive Summary:

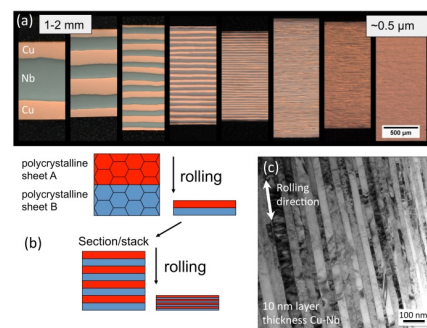
Materials under extreme environments have received significant attention recently in the context of next-generation of energy, defense and transportation technologies. These applications require materials to perform at “extremes” of stress, temperature, irradiation dose, and corrosive environments. **In land or air vehicles, lightweight, high strength structural materials are needed to increase fuel efficiency, maneuverability and threat/crash resistance.** Other applications, such as the next-generation of nuclear power reactors, require structural materials capable of withstanding elevated temperatures and radiation fluxes in highly corrosive environments for long periods of time without failure. The increased demands of future technologies cannot be met by incremental improvements in conventional materials. New concepts in materials design are needed to manufacture materials that resist damage at mechanical and irradiation extremes which are designed and manufactured based upon the control of the mechanisms responsible for material damage. It has long been known that surfaces, grain boundaries and interphase boundaries are sinks for deformation and radiation-induced defects.

Recent work on the design and synthesis of bulk structural nanocomposites with enhanced strength, fracture toughness, and formability. As layer thicknesses decrease through the microscale to the nanoscale ( $t=100$  nm or less,  $\sim 100$  times smaller than the thickness of a human hair!), prior work has shown orders-of-magnitude improvements in material performance behavior such as high strength and energy absorption, thermal stability, and ultrahigh resistance to radiation damage. Such outstanding properties are tied to both the high density of interfacial content and to specific interfacial structures in nanolaminar composites. That is, with the “right” characteristics, bi-material interfaces can possess significantly enhanced abilities to absorb and eliminate defects. Through their unparalleled ability to mitigate damage accumulation induced under severe loading and/or severe environments, they will provide their parent composite with a highly effective healing mechanism and consequently an unrivaled robustness not possible in existing advanced structural materials. Specific nanoscale layered composites can potentially be designed to resist specific threats and weight reductions.

Programs at Los Alamos National Laboratory and elsewhere have investigated this class of materials in thin film form. Thin film processing typically provides material thicknesses on the order of a micron, limiting structural engineering applications to high hardness coatings. Other than LANL, no current program addresses the manufacturability of nanocomposites with controlled interfacial structures in bulk form, which is a problem that intrinsically lies at the mesoscale. Therefore, the intent of such a research effort is to prove the hypothesis that: ***Through the employment of controlled processing parameters which are based upon integrated advanced material characterization and multi-physics material modeling, bulk nanolayered composites can be designed to contain high densities of preferred interfaces that can serve as supersinks for the defects responsible for premature damage and failure.***

## Highlights:

- Bulk metallic nanocomposites possess order-of-magnitude improvements in strength, shock damage resistance, high temperature resistance, and radiation damage tolerance.
- This class of materials offers the unique opportunity to reduce weight of personnel carriers, armored vehicles, aircraft, spacecraft, sea vessels, and drones by up to 75%.
- Industrially scalable severe plastic deformation techniques can be used to engineer stable interfaces that facilitate self-healing behaviors under extreme environments.
- A unique combination of pilot manufacturing capability, novel material characterization techniques, and multi-physics material modeling enables design of highly effective armor at substantial weight savings.



**Figure: (a)** Optical micrographs of ARB Cu-Nb processed at LANL from starting thicknesses of  $\sim 2$  mm down to  $0.5 \mu\text{m}$ .

**(b)** Schematic of ARB process of repeated stacking and rolling. **(c)** TEM micrograph of 10 nm layer thickness Cu-Nb material.

## Operational Opportunity:

Materials under extreme environments have received significant attention recently in the context of next-generation of energy, defense and transportation technologies. These applications require materials to perform at “extremes” of stress, temperature, irradiation dose, and corrosive environments<sup>3</sup>. **In land or air vehicles, lightweight, high strength structural materials are needed to increase fuel efficiency, maneuverability, threat/crash resistance, and reduce exhaust gas emissions.** Other applications, such as the next-generation of nuclear power reactors, require structural materials capable of withstanding elevated temperatures and radiation fluxes in highly corrosive environments for long periods of time without failure<sup>3-5</sup>.

The increased demands of future technologies cannot be met by incremental improvements in conventional materials. New concepts in materials design are needed to manufacture materials that resist damage at mechanical and irradiation extremes<sup>5</sup> which are designed and manufactured based upon the control of the mechanisms responsible for material damage. It has long been known that surfaces, grain boundaries and interphase boundaries are sinks for deformation and radiation-induced defects<sup>6-9</sup>. **With their high interphase boundary content, nanocomposite materials have shown orders-of-magnitude improvements over their constituent materials with respect to strength and performance under thermal, shock, and irradiation extremes.** If a new, ideal armor material or armor system based upon a specific threat were to be designed, it would possess precisely these properties. Current designs are limited by the properties/performance of off-the-shelf alloys that are often developed for other applications in mind, and are not tailored to a particular defense application – damage and failure avoidance and energy absorption. **Here, we seek to develop superior structural and armor materials with the ability to self-heal under extreme conditions (shock, burst, temperatures, irradiation) where traditional alloys are inherently unstable or are of high density and weight.**

However, the detailed mechanisms at the level of the atomic structure of an interface that enable a nanocomposite to be stable under high strains or high irradiation flux are only just beginning to be elucidated through studies on model systems where ion irradiation or implantation experiments are closely integrated with atomistic modeling<sup>10,11</sup>. Likewise, methods to process bulk nanocomposites, where the key is not just to refine the microstructure but also to produce interfaces that are stable at extreme conditions, are still under development<sup>12-15</sup>.

### *Current State-of-the-Art in Self-Healing Bulk Nanocomposites:*

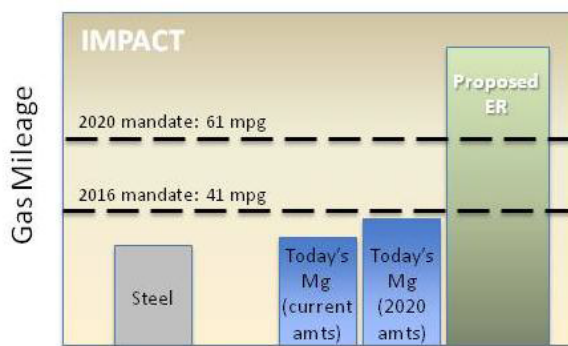
Recent work focuses on the design and synthesis of bulk structural nanocomposites with enhanced strength, fracture toughness, and formability. As layer thicknesses decrease through the microscale to the nanoscale ( $t=100$  nm or less,  $\sim 100$  times smaller than a human hair!), prior work has shown orders-of-magnitude improvements in material performance behavior such as high strength and energy absorption, thermal stability, and ultrahigh resistance to radiation damage. Such outstanding properties are tied to both the high density of interfacial content and to specific interfacial structures in nanolaminar composites<sup>2,13-16</sup>. That is, with the “right” characteristics, the bi-material interfaces can possess significantly enhanced abilities to absorb and eliminate defects. Through their unparalleled ability to mitigate damage accumulation

induced under severe loading and/or severe environments, they will provide their parent composite with a highly effective healing mechanism and consequently an unrivaled robustness not possible in existing advanced structural materials. Specific nanoscale layered composites can potentially be designed to resist specific threats and weight reductions.

Despite the attractive properties of this class of materials and blossoming ability to predict and control the defect sink strength of given bi-material interface structures, **the top-down manufacturing of nanolayered composites in bulk form remains a significant mesoscale hurdle** to their use in engineering applications. The authors have had substantial success with the manufacture of bulk nanolamellar composites<sup>17-21</sup>, in the form of Copper-Niobium (Cu-Nb), Copper-Silver (Cu-Ag), and Zirconium-Niobium (Zr-Nb) systems, providing the key discovery that specific dominant interface structures form under conditions such as rolling to extreme strains (1000's of percent strain). However, it is not yet clear why or how these structures form during synthesis. Both Cu-Nb and Cu-Ag have application for conductors in high-field magnets, and Zr-Nb the potential application for structural materials in irradiation extremes. The specific interface structures that evolve during processing are responsible for the amazing properties of this class of material, and so the lack of understanding of the mechanisms and energetics that drive preferred interfaces to form during processing represents a fundamental mesoscale knowledge gap. **If the connection between processing and interfacial structure could be established, it would enable the intelligent design of bulk materials with self-healing interfacial structures.** Pilot programs to expand this instrumental new set of discoveries into material systems relevant to the defense and transportation industries such as Steel and Magnesium based systems are underway at LANL.

*Example: Magnesium-based nanocomposites:*

With pressure increasing to reduce gas consumption, Magnesium alloys ('Mg' for short) have risen to the top of the list as the next structural materials to replace steel<sup>22-25</sup>. Mg has a high strength-to-weight ratio and low density, being 35% lighter than Al and 78% lighter than steel. Thus, if all the steel in your vehicle were replaced with Mg, then the weight reduction would boost fuel efficiency by 54.6% (e.g., from 30 to 46 mpg)<sup>1</sup>. While impressive, this enhancement will just meet CAFE (Corporate Average Fuel Economy) standards for 2016 (41 mpg)<sup>26</sup>. Yet there is plenty of room for vast improvement that remains untapped to date. With a tensile yield strength of  $\sim 250 \text{ MPa}$ <sup>27</sup>, Mg is not strong enough on its own. Imagine, if the strength of bulk Mg were to increase ten-fold, only a tenth of the Mg would be required, leading to a boost in gas mileage (from 30 to 76 mpg!), and a radical transformation in vehicle manufacturing (Figure 1).



**Figure 1:** Projected impact of nano-Mg-X on gas mileage. Calculations based on<sup>1</sup>. Currently 14-26 kg of Mg are used per car and the projected amounts for 2020 are 160 kg.

For many other structural metals (e.g., Al, Cu, Ni, steel), ten-fold increases in strength have been realized by severe plastic deformation (SPD) techniques<sup>28,29</sup> leading to specialized material microstructures. These bulk processing techniques transform traditional coarse-grained metals (> 100  $\mu\text{m}$ ) into nano-grained materials (<100 nm), leading to increases in strength up to a factor of 10. The original dimensions of the sample are retained, so if it starts as a bulk material, it stays a bulk material (>  $\text{cm}^3$ ). Unfortunately, all attempts, thus far, to make nano-Mg in bulk sizes have failed<sup>30-37</sup>. Ongoing work in this area has proven that nano-Mg-Nb composites are deformable<sup>38</sup>, with the next step being scale-up of production to bulk quantities of material, much as what has been done for Cu-Nb, Cu-Ag, and Zr-Nb. In the case of Mg, **if material with the strength of steel, but 75% less weight were employed in manned or unmanned vehicles, or used to enhance current armor technologies, this would represent a new paradigm in materials research, and would revolutionize current and future military vehicle designs.**

### Enabling Technologies:

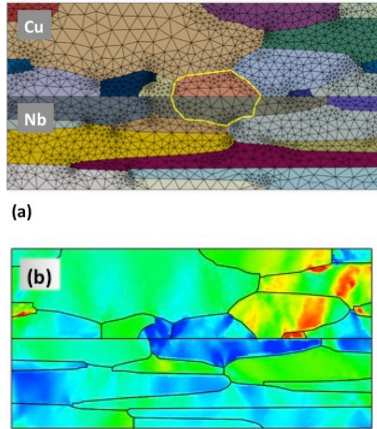
Programs at LANL and elsewhere (sponsored by DOE-Office of Science) have investigated the correlations between structure and interfacial content in thin film form. Thin film processing typically provides material of thicknesses on the order of a micron, limiting its engineering application to high hardness coatings. Other than LANL internal funding for manufacture of bulk Mg-Nb composites, no current program addresses the manufacturability of nanocomposites with controlled interfacial structures in bulk form, which is a problem that intrinsically lies at the mesoscale. Therefore, the intent of such a research effort is to prove the hypothesis that: *Through the employment of controlled processing parameters, bulk nanolayered composites can be designed to contain high densities of preferred interfaces that can serve as supersinks for the defects responsible for premature damage and failure.*

Over the past 5 years, we have been able to successfully process bulk Cu-Nb<sup>18,19,39</sup>, Zr-Nb<sup>20,21,40</sup>, and Cu-Ag<sup>17</sup> nanocomposite material in kilogram-quantities using industrially scalable SPD thermomechanical processing (see subsequent Technical Approach section for details). These techniques use scalable forming technologies commonly found in metallurgical processing, such as rolling, extruding, and swaging. Yet parameter space associated with the effects of all possible combinations of constituent materials, processing routes, processing temperatures, etc. is vast. Guidance of an experimental program using advanced modeling techniques is of paramount importance to the success of the overall effort. Our past successes have coupled theory/simulation with experiment to predict not only which interfaces evolve in a bulk nanocomposite under a given processing route, but the material stability under subsequent perturbation in other extremes such as strain<sup>15,41</sup>, high temperature<sup>14,15,42,43</sup>, irradiation<sup>2</sup> or shock<sup>44</sup>.

We lack an experimentally validated, atomistically informed mesoscale model that can predict the family of stable interfaces that will arise during composite SPD processing, and can then be extended to guide production of technologically relevant alloys for engineering applications. Specifically needed is a better understanding of the collaborative roles of the nanostructure, dislocation slip, deformation twinning, and the bimetal interface on the dynamics of deformation and performance of the formed composite. Meso-scale modeling techniques that integrate atomic-level physics associated with interface character have tremendous potential to address



these issues. They treat the time and length scales corresponding to laboratory conditions, typically used in bulk metal working processes. The meso-scales are also relevant to understanding the effects of interfaces on texture and interface crystallography development. Our recent crystal plasticity finite element models (See Figure 2), using 2D and 3D microstructures



**Figure 2:** 3D crystal plasticity finite element simulations of a two-phase Cu-Nb composite under deformation.

(a) experimentally determined mesh of the initial grain structure and (b) deformation map. The colors correspond to the amount of lattice reorientation. This analysis enables us to determine which bi-phase interfaces are orientationally stable under deformation.

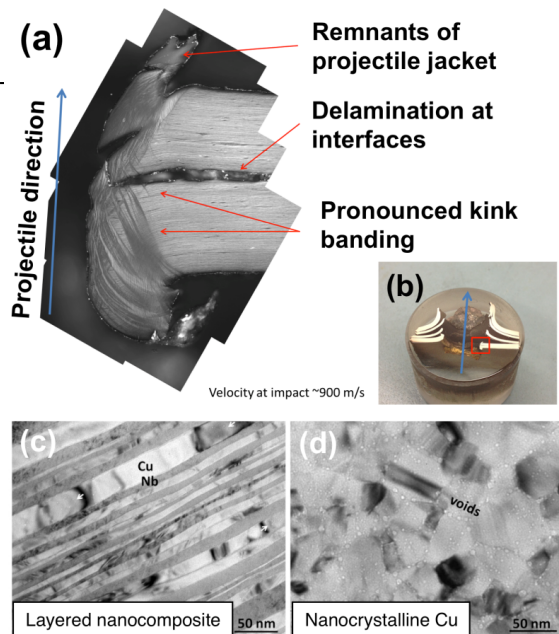
taken from experimental characterization, have been developed to account for co-deformation and/or slip transmission across a bimetal interface<sup>45,46</sup>. They have demonstrated that these effects have profound influence on the stability of interface character and texture during plastic deformation<sup>47</sup>. Currently, the effort to integrate the atomic-level physics of defect-interface

interactions into larger scale models, which are able to account for dynamic performance to be used for structural design, is still in its infancy. If this effort to develop experimentally validated meso-scale were expanded, it will provide a critical tool for predicting the interfaces that will arise under a given thermomechanical processing schedule, and in turn, the enhanced properties that result from the designed composite structure.

#### *Enhanced properties of bulk nanocomposites under extreme conditions:*

As mentioned before, this class of materials exhibits strengths and resistance to shock at levels that represent over a 10x improvement over conventional coarse-grained materials. Examples of such behavior have been published extensively in the literature<sup>13,18</sup>, but two specific cases are seen in Figure 3. In Figure 3, a 3mm thick plate of Cu-Nb bulk nanocomposite with individual layer thickness of 60 nm has been

**Figure 3:** (a and b) optical micrographs of a 60 nm individual layer thickness Cu-Nb bulk nanocomposite after 30-caliber round impact. (a) enlarged view of the red boxed region in (b). Extensive kink banding, an energy-absorbing process, occurs at high stresses over 1 GPa in this material. (c) Irradiated ARB NL Cu-Nb composites with 20 nm individual layer thickness are nearly void free. Arrows indicate small, isolated voids in the thicker Cu layers. (d) Irradiation damage (voids) in nanocrystalline Cu under identical conditions<sup>2</sup>.



subjected to impact by a 30-caliber projectile traveling at 900 meters/second. Figure 2(a) is a close-up cross-sectional optical micrograph of the red boxed region shown in (b). Not only does this material possess strength approaching ~2 GPa, but also exhibits pronounced kink banding

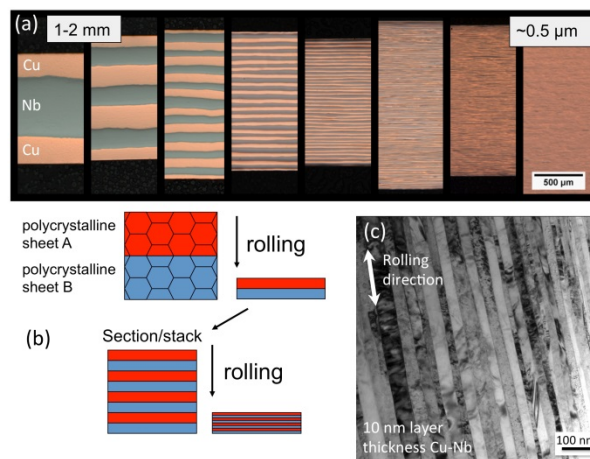


near the impact site. Kink banding is found in material systems with limited deformation pathways, which in this case is believed to be due to the fine layered structure, and interfacial effects<sup>16</sup>. In this preliminary result, this mechanism is expected to dissipate large amounts of impact energy attributable to the high stresses needed to produce the kink bands.

Nanolayered composites have also proven to be radiation damage tolerant when exposed to light ion irradiation. In Figure 3(c), Cu-Nb material with 20 nm layer thickness exhibits little to no void formation after exposure to He ion irradiation<sup>2</sup>. In contrast, pure Cu, even in nanocrystalline form (Figure 3(d)) shows extensive void formation at grain boundaries. This points to the fact that high boundary density alone is a necessary, but insufficient condition for damage tolerance—only the specific interfaces found in the Cu-Nb nanolaminate possess both the thermal stability and sink strength for irradiation-induced defects to provide radiation damage resistance.

### Technical Approach to design of bulk nanocomposite materials for enhanced performance under extreme conditions:

*Synthesis:* Identification of the optimized biomaterial combination is critical to success of the current investigation. Bulk ( $> \text{cm}^3$ ) laminar composites with controllable layer thicknesses down to the submicron or nano-scale range can be fabricated via accumulative roll bonding (ARB), a severe plastic deformation (SPD) processing technique. The ARB process is in itself an extreme condition. Imposing over thousands of percent strain, ARB refines the microstructure of ordinary coarse-grained composite metals down to submicron and nanoscales, and is a current capability at LANL. (See Figure 4) *ARB is ideal for this investigation* for three reasons: 1) it produces a 2-D layered microstructure, 2) it imposes monotonic deformation in a familiar manner (rolling), and 3) it allows for controllable accumulated strain and layer thickness (from 1 mm to 10 nm). This methodology is similar to the manner in which ancient cultures may have used repeated forging and folding to create high-strength blade materials since ~300 B.C.E.<sup>48</sup> However, the ancients did not have the modern characterization tools to understand exactly why their craft produced superior material or the ability to move beyond trial-and-error exploration. More recent work has focused on fcc/fcc (Ag-Cu) and fcc/bcc (Cu-Nb) nanocomposites (fcc=face-centered cubic, bcc=body centered cubic), chosen for their immiscibility over a wide range of temperature, and lack of formation of intermetallic phases to give a well-defined interface. Similar criteria apply to the Zr-Nb and Mg-Nb, although in this case, the crystal structures are hcp/bcc (hcp=hexagonal close packed). It is expected that the differences in



**Figure 4:** (a) Optical micrographs of ARB Cu-Nb processed at LANL from starting plate thicknesses of ~1-2 mm down to 0.5  $\mu\text{m}$ . (b) Schematic of ARB process of repeated stacking and rolling. (c) TEM micrograph of 10 nm layer thickness ARB Cu-Nb material.

propensity of dislocation slip relative to deformation twinning in hcp metals will drive unique texture development in at least one phase during processing, which when combined with dislocation transmissibility across bimetal boundaries of differing crystal orientations, can result in the formation of new preferred interfacial structures that can be compared with existing data on fcc/fcc and fcc/bcc composites. These represent ideal candidates for model systems (the Zr-Nb system has already been successfully ARB processed at LANL), and have broad impact in other areas, such as nuclear engineering (Zr-based) and strong, lightweight alloys (Mg-based). Since ARB processing is based on rolling, a commonly used manufacturing technique, the fundamental knowledge of interfacial evolution during large rolling strains gained from this study could be eventually applied to enhance Fe-based or Al-based alloys.

#### *Microstructural Characterization and Mechanical Property Measurement:*

Measurements are carried out at intervals throughout processing from microns down to nanometers to investigate interfacial evolution as a function of strain and layer thickness.

There are several key parameters that must be determined in such an investigation to link properties and performance to the material microstructure:

- 1.) Grain orientation distribution (texture)
- 2.) Grain morphology (shape)
- 3.) Interface plane and local grain orientation (interface geometry)
- 4.) local atomic structure (faceting, interface morphology, local chemistry)

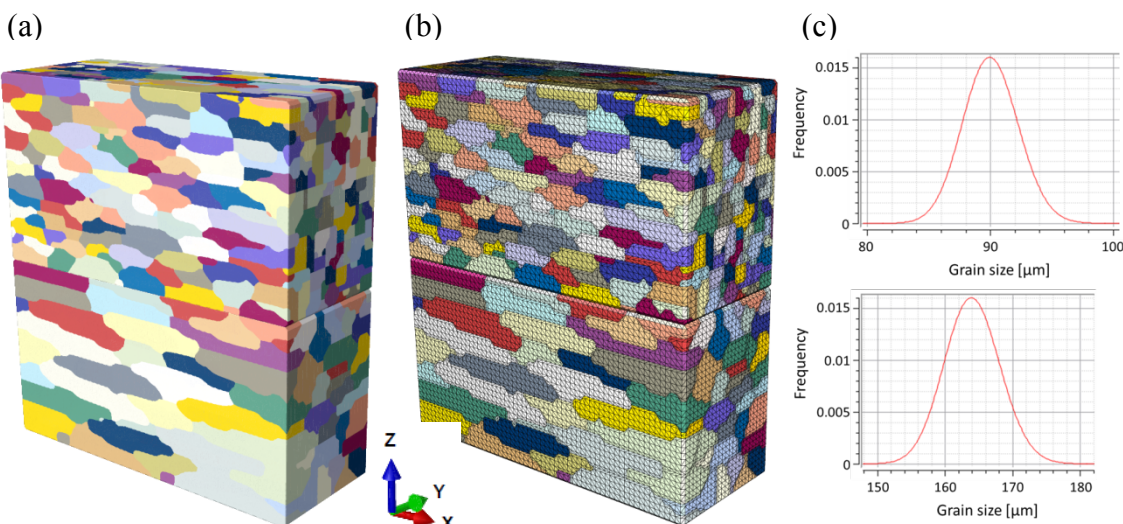
The ARB technique yields bulk specimens (approx. 2-4 mm thick by 5 cm wide by 30 cm long), enabling bulk texture analysis via either neutron or x-ray diffraction. Local texture, grain morphology, and 5-dimensional analysis to determine interface crystallography and interface plane normal is conducted using a combination of Electron Backscatter Diffraction (EBSD) in the Scanning Electron Microscope (SEM) and Transmission Electron Microscopy (TEM) as is length scale appropriate. Analysis of the interfacial structures on the atomic scale will be carried out via aberration-corrected High Resolution TEM. Mechanical behavior will be measured by a combination of nanomechanical testing (nanohardness, micropillar compression, tension) and bulk mechanical testing (tension/compression) along different loading directions to investigate anisotropy effects. Through these methods, the influence of synthesis pathway on specific crystallographic textures, bimetal interfaces, and interface defect structures will be determined. Their influence on subsequent mechanical response and fracture will then be investigated.

#### *Predictive Microstructural Modeling for Designed Interfaces:*

Simulations will be carried out using crystal plasticity finite element (CPFE) method for these material systems. We will develop a material model of the two-phase multi-layered laminates that contains 3D grain structures (Figure 5). The morphology and relative sizes of these grains will be based on experimental characterization (Figure 5c). Creation of such 3D microstructures will make use of DREAM.3D (Figure 5a) and a novel meshing tool for the grains and grain boundaries and interfaces (Figure 5b). The CPFE model will account for differences in elastic anisotropy, temperature and rate-dependencies of slip, and crystallographic slip systems between the two phases. Under deformation, these differences can potentially cause heterogeneity in

stress, strain, and lattice reorientation, particularly near the interface (Figure 2), and notably, this model can capture such effects. For experimental comparison and model validation, we will output texture and grain morphology distributions. From the same deformed microstructure, we can model subsequent mechanical testing to see the effect of post-processing microstructure on mechanical performance. The processed internal stress state can also affect performance and is another potential design variable to enhance penetration and damage resistance of the armor system.

For the proposed program, such two-phase CPFE simulations can be designed to determine how interface crystallographic character evolves as a function of processing path. In this respect, the modeling and experimental efforts will work together to identify candidate processing pathways for specific target interfaces. For the finer nanolayered composites ( $< 10$  nm layer thicknesses), we will plan to advance the models to treat interface-dislocation interactions and the process of defect annihilation. For instance, we have uniquely extended this CPFE model to treat slip transmission across the interfaces and its effect on texture evolution<sup>45,46</sup>. Based on prior MD simulations, we are knowledgeable about other factors that will play an important role in the deformation of nanolayered composites, such as interface formation energy and dislocation annihilation at the interface<sup>12</sup>. As part of this program, we plan to implement these factors in order to make the model more predictive as the layer thicknesses reduced below a few tens of nanometers.



**Figure 5:** (a) Zr-Nb synthetic polycrystalline aggregate consisting of 3D grain structure generated in DREAM.3D, and (b) corresponding mesh of 3D grains (c) Experimentally measured grain size distribution used in DREAM.3D to generate the 3D grain structure in (a).

### Proposed Budget Summary/Justification:

The main body of work in the literature on bulk nanomaterial synthesis and evolution during processing was carried out in the past 5 years as a combination of efforts from the Center for Materials at Irradiation and Mechanical Extremes, a DOE Energy Frontier Research Center, and focused on Cu-Nb and Cu-Ag systems (\$19.8m/year over 5 years). This center was led by LANL and included academic collaborators from numerous institutions, including MIT,

University of Illinois, Carnegie Mellon University, and UC Santa Barbara, among others. Additional funding from LANL internal sources for specific, targeted studies, such as mesoscale modeling of microstructure evolution during ARB processing, amounted to ~\$6m over a similar time span. A similar ~\$25m investment over 5 years would provide significant inroads into industrially-relevant magnesium, aluminum, and steel-based systems, paving the way for engineering application in the 10-15 year time frame. Smaller pilot programs, on the order of ~\$1m over 3 years could be employed to solve focused problems, but the integration of such pilots into a comprehensive effort would incur additional project management costs.

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